STUDIES ON KINETICS OF SETTLING AND CONCENTRATION OF CASSAVA STARCH SUSPENSION

By

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CERTIFICATE

This is to certify that the thesis entitled **"STUDIES ON KINETICS OF SETTLING AND CONCENTRATION OF CASSAVA STARCH SUSPENSION"** in part fulfillment of the requirements for the degree of **DOCTOR OF PHILOSOPHY** in **AGRICULTURAL PROCESS ENGINEERING** to the Tamil Nadu Agricultural University, Coimbatore is a record of bona fide research work carried out by **Mr. M. S. SAJEEV** under my guidance and that no part of this thesis has been submitted for the award of any other degree, diploma, fellowship or other similar titles or prizes and that the work has not been published in part or full in any scientific or popular magazine.

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ABSTRACT

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Cassava or tapioca (*Manihot esculenta* Crantz.), in addition to being an important food crop, is an industrial crop for its high starch content (about 25-30%). Cassava is mainly processed into starch and sago and there are about 800 small to medium scale industries in Tamil Nadu alone for processing cassava. The process of extraction of starch involves washing, peeling, crushing, screening, gravitational settling, pulverisation and drying and it requires about 4.5-7.3 m^3 of water per tonne of roots. The settling process takes about 10-12 hours and this longer detention time causes fermentation; producing alcohol and organic acids, which give foul smell near the factory area. Removal of water from the starch milk immediately after screening reduces the volume of effluent and helps to reuse the same water for rasping operation.

Settling process – a prime unit operation in cassava starch extraction process was studied in detail to understand the kinetics of starch settling, settling characteristics in the presence of chemicals, electrolysis, inclined columns and centrifugal settling. The thermal and

rheological properties of settled starch in presence of chemicals and electric field were evaluated to find their effect on starch quality. A hydrocyclone for cassava starch milk concentration was designed according to the starch and water properties and tested at different feed concentrations and operating pressures. Experiments on a battery of hydrocylcone and recycling of the overflow stream were also done to obtain minimum starch content in the overflow stream, so that it could be reused for rasping operation.

Cassava starch in suspension was found to follow discrete or flocculant settling at low concentration upto 6 % and hindered or compression settling was predominant above 10 % concentration.

Free falling velocity as measured according to Stoke's law was 0.169 mm/s and the velocity of particle in suspension was found to be lower than this value. Settling velocity was found to be reduced significantly with concentration and height of suspension, but diameter of settling column did not produce any effect. Settling time for fresh starch was found to be more than the reconstituted dry starch due to hydration repulsion effect.

Mathematical models were developed to describe the settling velocity, final water content of the settled starch cake, and effect of inclined column on settling.

Acids and sodium metabisulphite aided settling in more effective way, however, more compactness of settled starch was obtained with sodium hypochlorite followed by sulphuric acid, sodium metabisulphite, hydrochloric acid and alum.

Gelatinization temperature was enhanced by chemical treatment where as rheological properties were lowered or not affected. Starch properties were severely affected by sulphuric acid and alum, whereas hypochlorite and sodium metabisulphite did not produce much effect

Electroflocculation produced faster settling of the starch, without affecting the gelatinization temperature, but reduced rheological parameters.

Settling velocity of starch particle in a centrifugal field can be increased to 373 times than the gravitational settling velocity by keeping the peripheral speed of the modified rotor as 23.16 m/s.

Based on the properties of starch and water, a 30 mm hydrocyclone was designed and tested. Underflow concentration, total efficiency and reduced efficiency was found to be increased with increasing pressures and decreasing concentrations. When operated at 3 kg/cm^2 feed pressure; 62.68% and 41.87% total and reduced efficiency, respectively were achieved. Underflow volume split was not effected by the operating parameters.

Experiments with recycling of overflow stream showed that at 1.8 kg/cm^2, feed concentration of 3.5% reduced to 0.69% in overflow after 9^{th} pass. About 43% of the feed stream with this amount of starch could be reused for rasping operations.

Battery of five hydrocyclones of 30 mm diameter gave about 2.28% starch in over flow when operated at 3 kg/cm^2 with 3.5% feed concentration. But modified battery of five units with equal inlet and overflow diameter gave about 1.56% overflow concentration at the above condition.

When the overflow from the modified battery of hydrocyclone was recycled, it gave about 0.40% starch in the overflow stream after 3 passes and 0.46% starch in under flow after 4 passes with a net saving of 47% water which could be reused for rasping operation.

Operational cost of the modified battery was calculated as Rs.22/- per cubic metre of starch suspension to get an overflow stream of 1.56 % concentration, compared to 3.5% feed concentration

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mg	-	milligram
mha	-	million hectares
min	-	minimum
min	-	minute
ml	-	millilitre
mm	-	millimetre
mM	-	millimolar
mt	-	million tonnes
Pa.s	-	Pascal second
pp	-	page to page
rpm	-	rotations per minute
Rs.	-	rupees
s	-	second
SI	-	settling index
SVI	-	sludge volume index
t	-	tonne
T_e	-	end gelatinization temperature
T_o	-	onset gelatinization temperature
T_p	-	peak minimum gelatinization temperature
U_{vs}	-	underflow volume split
V	-	Volt
v/v	-	volume by volume
viz.,	-	namely
w/v	-	weight by volume
"	-	inch
%	-	per cent
≤	-	less than or equal to
≥	-	greater than or equal to
ε	-	fluid volumetric concentration
v_c	-	settling velocity in suspension
ΔH	-	gelatinization enthalpy
ΔT	-	gelatinization temperature range
°	-	degree
°C	-	degree Celcius
μm	-	micrometre

INTRODUCTION

CHAPTER I

INTRODUCTION

Cassava or tapioca (*Manihot esculenta* Crantz.) is one of the richest sources of starch (25-30%) among the tropical tuber crops with a recorded global production of 164.75 mt from an area of 16.37 mha (FAO, 1997). It has the potential to produce more food per unit area by withstanding adverse biotic and abiotic stresses and is well adopted to various agroclimatic conditions. In India, cassava is grown in Tamil Nadu, Kerala, Andhra Pradesh, Karnataka and few North Eastern States, spreading over an area of 0.24 mha yielding about 5.98 mt. In Tamil Nadu, the productivity is about 22 t/ha which is the highest in the world (Edison, 1999).

Cassava is mainly processed for starch and sago and in Tamil Nadu, there are about 800 small to medium starch and sago factories besides some cottage level industries producing value added products. Tamil Nadu stands atop in the processing of cassava into starch and sago and 80 per cent of the national demand in food and non food area is met by this state (Shegaonkar, 1996). Cassava starch, in addition to being used for the production of sago finds extensive industrial application in textile, paper, alcohol, pharmaceuticals, confectionery, laundry, adhesives, explosives etc. The emerging uses of starch include low calorie fat substitute, biodegradable plastics, edible packaging films and thermoplastic materials with improved thermal and mechanical properties.

In 1996, about 31,000 t of cassava products including raw tubers, flour meal, starch and chips were exported from India to European Union, Gulf Cooperation Council and some Asian countries and earned about 14.13 crores of foreign exchange (Edison, 1999). The export of cassava starch alone was 65.4 t valuing about 6.74 lakh rupees. Globally about 75% of the commercial requirement of starch comes from maize followed by cassava (Anon., 1999). Cassava starch is often preferred to maize starch in

view of its low cost, easy extractability, better qualities like high viscosity, bland taste, neutral flavour and easy degradation (Balagopalan *et al.*, 1996). Hence in India, cassava starch is well utilized along with maize starch.

Extraction of starch from raw tubers involves various unit operations *viz.*, washing, peeling, washing peeled tubers, rasping, screening, settling and purification, pulverisation and drying. Major technological problems associated with the cassava starch industry are high water consumption, more space requirement, longer detention time of starch in the settling tanks and weather dependent drying which adversely affect the end quality of the starch.

About 90% of the total water consumption in the starch factory is utilized for primary and secondary starch settling (Nandy *et al.*, 1996[a]; Nandy *et al.*, 1996[b] and Thangavel *et al.*, 1998[a]). Though settling tanks are relatively inexpensive, the process takes about 10-12 hours. The prolonged detention time can cause fermentation producing alcohols and organic acids which can produce foul smell, polluting the entire atmosphere around the plant. Also trampling by human labour commonly practiced to make starch granules free from dust particles and settling is unhygienic. Hence, an easy and quick separation of starch becomes a pre-requisite for good quality starch and a pollution free environment.

Cassava starch milk can be considered as a two phase coarse dispersed system (solid-liquid system). The separation of starch particles from its suspension in water by settling takes place under the action of gravitational force. During settling, colloidal interaction due to inter particle forces and hydrodynamic interaction will occur. Hence, the classical Stoke's sedimentation theory can not be applied as such for describing the settling behaviour of cassava starch. The settling characteristics of starch particles in its suspension in water at different concentrations helps to determine settling

velocity distribution and total solid settles as a function of time. The design curves so obtained from the laboratory settling column test can be used in designing settling basins or tables employed in starch extraction process.

During starch settling, interparticle interaction causes them to coalesce or flocculate and hence increase in mass of the aggregates will lead to faster settling. Alternate means for producing such agglomeration can hasten the settling process. When starch suspension is subjected to electrolysis, dissociation of water takes place releasing hydrogen and oxygen bubbles. During the movement of these gas bubbles, starch granules will be disturbed causing more frequent collision between the particles. This interaction can cause agglomeration and faster settling. Thus, the principle of electroflocculation may effectively be applied for faster separation and settling of starch.

The addition of small amount of chemicals may also improve the settling rate and compactness of settled starch. Any detrimental effect on the rheological and physico-chemical properties of the starch by the chemicals will lead to the lowering of starch value and marketability. So, the chemicals used should not affect the desirable properties of the extracted starch and should be within the permissible limit.

The rate of settling of suspended particles can be greatly increased if centrifugal forces are employed as the particle experiences a centrifugal force which is several hundred times greater than gravitational force. Hydrocyclone is a solid-liquid separator working on this principle. They have been used in large scale starch manufacturing since long to separate starch from impurities and to concentrate starch milk obtained from different crops (Van Esch, 1991 and Trim and Marder, 1995). Use of hydrocyclone not only reduces the time of contact between starch granules and fruit water, but also the total water consumption and thereby the quantity of effluent too. Trim and Marder (1995) and Thangavel *et al.* (1998[a] and 1998[b]) tested standard single hydrocyclone for cassava

starch milk concentration by varying different operational and equipment parameters. But to obtain maximum separation efficiency for a given starch milk concentration, they are to be designed according to the properties of starch and fluid. So far no attempt has been made in this direction.

If the overflow stream from the hydrocyclone is made to have minimum starch content, it can very well be diverted back for the various unit operations in extraction process especially for rasping operation. Hence the effect of recycling and performance of battery of hydrocyclones arranged in series are to be studied to minimize the water consumption. Settling and concentration – the prime unit operations in cassava processing are therefore to be examined to understand the kinetics of settling under gravity, centrifugal force, electric field and in the presence of different permissible chemicals and hydrocyclone system.

Under this background, the present study is undertaken with the following objectives.

1. To study the kinetics of starch settling in gravity field
2. To study the various processes to aid settling of cassava starch
   a. in the presence of chemicals
   b. in electric field
   c. in inclined columns , and
   d. centrifugal settling
3. To study the rheological and gelatinization properties of starch settled in the presence of chemicals and electrolysis
4. To design, develop and test a single and battery of hydrocyclone for cassava starch concentration.
5. To find out the techno-economic feasibility of the developed system.

REVIEW OF LITERATURE

CHAPTER II

REVIEW OF LITERATURE

This chapter is broadly divided into four sections *viz.*, cassava starch and its industrial applications, gravitational settling or sedimentation, processes to aid settling and properties of modified starch and hydrocyclones. The research work done on cassava starch production process, properties of cassava starch and the industrial application of starch in food and non-food areas are discussed in Section 2.1. Basic aspects of phase separation process, solid-liquid separation processes, types of sedimentation and settling characteristics of suspended solids are dealt in Section 2.2. In Section 2.3, various processes to aid settling like chemical addition, electroflocculation and settling in inclined column, and the properties of modified starch so obtained by these processes are presented. Basic aspects of hydrocyclone, its design, factors affecting its performance and application in biological processing are described in Section 2.4.

2.1 Cassava Starch and its Industrial Applications

Cassava is the most widely cultivated tuber crop, having wide applications in food, feed and industrial sector owing to its high starch content (25-30%). It cannot be kept fresh for more than a day or two due to primary and secondary deterioration making them unfit for human consumption or value addition and hence to be processed quickly after harvesting (Balagopalan *et al.*, 1987 and Kordylas, 1991).

2.1.1 Manufacture of cassava starch

The starch granules are usually locked up in cells together with other constituents and have to be separated from all other constituents to get the pure form of starch. Manufacture of cassava starch is carried out in three types of establishments *viz.*, cottage industries (50-60 kg/day/man), small scale industries (40-50 t/day) and large scale

industries (100 t/day and above) (Balagopalan *et al.*, 1987). Processing of tubers by wet milling is chiefly employed for the extraction of starch in all types of cassava industries irrespective of their production capacity.

2.1.2 Wet milling process for cassava starch production

The various unit operations involved in the wet milling process is given in Fig. 2.1.

a. Washing

It is done to remove and separate all adhering soil as well as protective epidermis to get colourless (white) pure starch. Roots are washed manually in tanks or by using mechanical washers worked on the principle of mechanical scrubbing as adopted in larger size factories.

b. Peeling

It is carried out with the help of special knives designed for peeling to minimise the loss of edible fleshy part of the tubers. To get a good quality product, washing is done before and after peeling of tubers.

c. Rasping

The tubers are turned into pulp or mash using a rasper which destroys the cellular structure, rupture the cell walls and release the starch as discrete, undamaged granules from other insoluble matters. Rasper consists of a solid wooden roller around which a purched metal sheet with its protrusions facing outside is nailed. The drum rotates inside a housing with a hopper at the top for feeding the tubers and with a perforated metallic plate underneath, through which the rasped pulp passes into the sump below. Water is continuously added during rasping. In this method,70-90% rasping effect is obtained during first rasping operation itself.

d. Sieving or Screening

It is done by rinsing the pulp mass on screens by addition/sprinkling of water, continuously to it. The pulp is pumped into a series of diminishing mesh sizes. The sieving is completed when the water running out of the screen is partially clear. The starch milk obtained after screening is collected in tanks and from where it is channelled for sedimentation. Residual pulp remaining on the screen after second pass is taken for drying in the sun and is used as an ingredient in cattle feed.

e. Settling or sedimentation

It includes a series of operations performed to separate the pure starch from other contaminants. Settling process should be completed as quickly as possible to prevent chemical, enzymatic and microbial reactions. Settling tanks or tables are used for this purpose. Starch milk is allowed to settle for a period of about 8-12 hours in the settling tanks whose capacity varies with the processing capacity of the factory. Starch settles at the bottom of the tank and the supernatant fruit water is let off through the outlets provided at different depths of the tank. The upper layer of settled starch contains many impurities and is scrapped off and rejected.

f. Tabling

It is a semi continuous settling process followed to reduce the time of contact between the starch and fruit water. The settling table consists of successive sets of slightly inclined channels or troughs. The starch milk is allowed to flow along the trough and when sufficient starch settles at the base of the channel, the flow of starch milk is temporarily stopped and the starch is removed manually.

g. Drying

Starch cake so settled contains 35-40% moisture. It is digged out and broken into small lumps and spread in thin layer on a large clean open area for sun drying to reduce the moisture content to 15-20% .

h. Bolting

Dried cassava starch consisting of hard agglomerates is pulverised or milled into powder form, screened to remove the foreign particles and ensure lump free uniform product. The starch powder so obtained through the bolting process is stored in a dry place and packed in polyethylene or gunny bags for marketing/storage.

2.1.3 Water consumption in cassava processing

Cassava processing for the production of starch requires large volume of water and produces equivalent volume of effluent. The unit operations where water consumption is necessary are washings, crushing and screening. About 90-95% of the water used was going as effluent (Nandy *et al.*, 1996[a] ; Nandy *et al.*, 1996[b] and Thangavel *et al.*,1998[a]).

Studies revealed that for processing of one tonne of cassava tubers, about 5.8-7.3 m^3 (Marder *et al.*, 1994) or 4.5 m^3 (Thangavel *et al.*, 1998[a]) of water was necessary. Water requirement for producing one tonne of sago was reported to be 31 m^3 (Trim *et al.*, 1993) and 16- 25 m^3 (Nandy *et al.*, 1996[a] and 1996[b]).

2.1.4 Properties of cassava starch

Starch and starch derivatives are the main products of cassava based agro- industries. In most of the applications, cassava starch is used primarily based on its special physico-chemical and functional properties.

Cassava starch is pure white in colour due to low contents of protein and phenolic compounds (Moorthy, 1994). It composed of two structurally distinct components *viz.*, amylose (17-18%) and amylopectin (82-83%) (Raja, 1995 and Kumar, 1995). Amylose is essentially a linear polymer of glucose with molecular weight varying from 1,50,000 to 10,00,000 depending on the biological origin. Amylopectin is a highly branched polymer and its molecular weight was reported to be 10×10^6 to 500×10^6. Shape of cassava starch

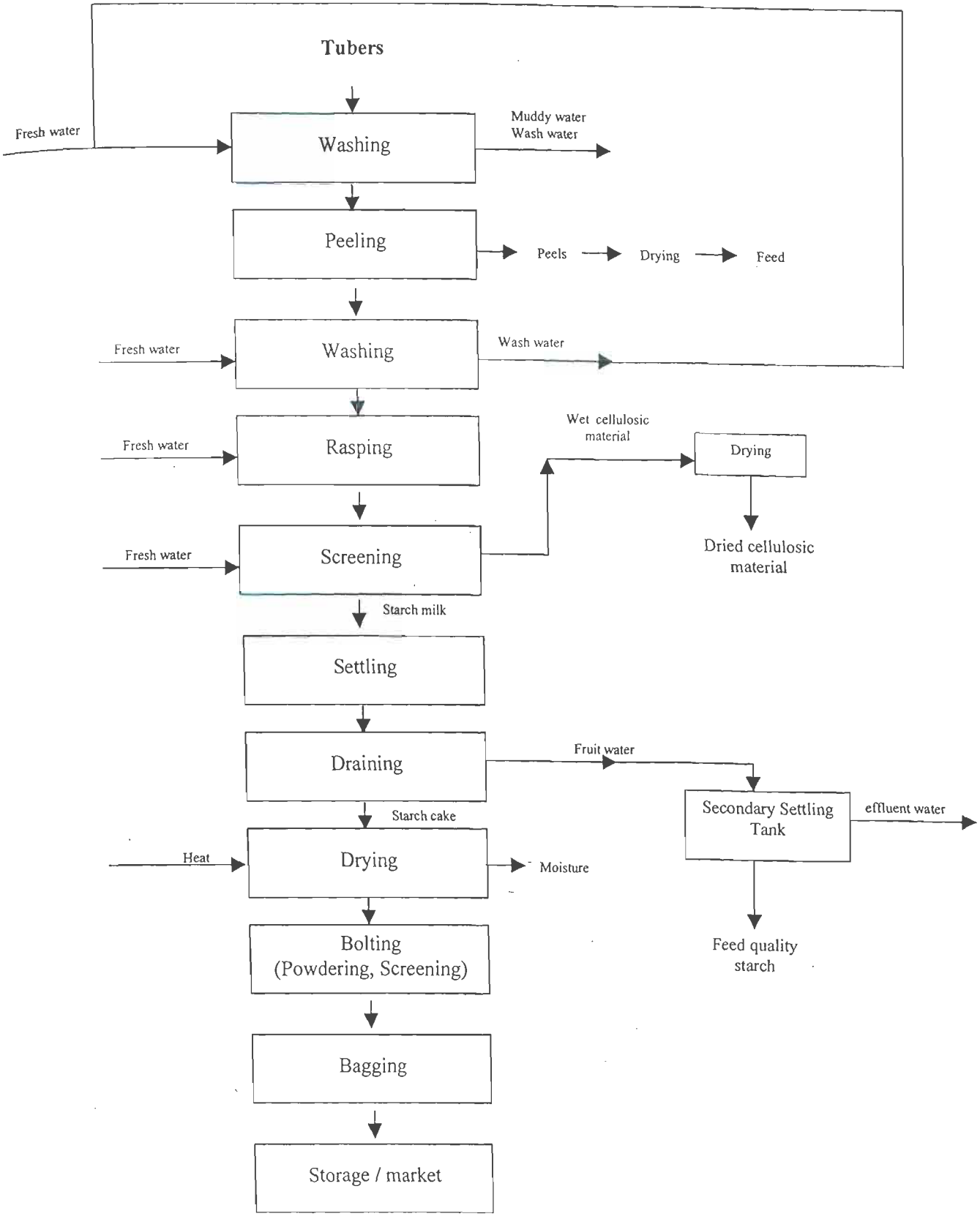


Fig. 2.1 Extraction of cassava starch by wet milling process

granules are round, oval or truncated having about 500 x 10^6 starch granules per gram of starch. Average number of starch molecules in one granule was about 200 x 10^{12} and surface area was about 200 m^2/kg (Kumar, 1995).

The granule size of cassava starch varied with the stage of maturity and origin of the tubers and the size ranged from 5-45 μm (Moorthy, 1985; Moorthy and Ramanujam, 1986 and Wurzburg, 1989).

Cassava starch granule size as determined microscopically gave a value of 5-40 μm wherein the majority of them were in the range of 25–35 μm and others in the range of 5- 15 μm (Balagopalan *et al.*, 1987).

A smaller range of granule size of starch was reported by Rickard *et al.* (1991) as 5-20 μm and Manikavasagan (1997) as 10-15 μm. Kumar (1995) and Raja (1995) reported the range of granule size as 4-35 μm with an average of about 15 μm.

2.1.5 Industrial applications of starch

In addition to being a major constituent of human diet, starch also functions as an excellent raw material for modifying the texture and consistency of foods owing to its ability to form visco-elastic gels when heated in water. Starch derived products have considerable applications in non food areas and are detailed in Table 2.1 (Ellis *et al.*, 1998). Koch *et al.* (1993) have remarked that it is possible to produce new generation detergents in which surfactant builders, cobuilders and bleaching activators could all be derived from starch. Starch could be an alternate material to meet the high demand for petroleum based chemicals in the non food industries like polymer synthesis. Polyamides are one of the most important synthetic polymers and methods had already been developed to synthesis carbohydrate or starch derived polymers (Thiem and Bachmann, 1994). Emerging uses of starch included as low fat calorie substitute, biodegradable plastic

(Wool, 1989; Arvanitoyannis *et al.*, 1994; Griffin, 1994 and Arvanitoyannis *et al.*, 1997) and edible films (Goheen and Wool, 1991; Arvanitoyannis *et al.*, 1996 and Psomiadou *et al.*, 1996).

Table 2.1. Industrial applications of starch

Industry	Applications
Adhesives	Adhesive products
Agrochemical	Mulches, pesticide delivery, seed coating
Cosmetics	Face and talcum powders
Detergents	Surfactants, bleaching agents and bleaching activators
Food	Viscosity modifier and glazing agents
Medical	Plasma extender/replacer, transplant organ preservation, absorbant sanitary products
Oil drilling	Viscosity modifier
Paper and Board	Binding, sizing, coating
Pharmaceuticals	Diluent, binder, drug deliver
Plastics	Biodegradable filler
Purification	Flocculant
Textile	Sizing, finishing, printing and fire resistance

2.2. Gravitational Settling of Cassava Starch

The starch milk obtained after crushing and sieving during wet milling of tubers is a suspension of starch in water. Hence, the separation of starch from its suspension becomes a prime objective in cassava processing. Starch milk can be considered as a two phase coarsely dispersed system in which the dispersed starch granules have a closed interface with the surrounding dispersing medium (water). Knowledge on the basics of disperse system and phase separation processes is imperative to study the settling kinetics of cassava starch milk.

2.2.1 Disperse system

A disperse system is that in which one phase is dispersed into small particles while the other phase is continuous and all the dispersed particles have a closed interface with the surrounding continuous phase (Rietma, 1982). According to the state of aggregation, structure, interphase interaction and phase distinguishability, Fridrikhsberg (1986) gave a detailed classification of disperse system. It is mainly divided into coarsely dispersed system with a particle size exceeding 1 μm and highly dispersed system or colloidal system with particle size below 1 μm. Coarsely dispersed system is one in which the particles of dispersed phase settle or rise to the surface in a gravitational field and do not pass through filter papers and are visible under an ordinary microscope whereas particles of highly dispersed system pass through ordinary filters, but are retained by ultra filters and do not settle and are not visible under an optical microscope. The fundamental behaviour of a two phase system is primarily determined by the hydrodynamic interaction between the two phases and further by the interaction between the separate particles of the dispersed phase itself (Rietma, 1982). Starch milk is a coarsely and freely dispersed system where the starch granules are not bound one another and can freely move in their suspension and settle due to gravitational force.

2.2.2 Phase separation

Separation processes are the most important unit processes to effectively separate the desired solid, liquid and gas from a variety of mixture. Phase separation is achieved by creating, increasing or controlling the slip velocity between the two phases which can be achieved by applying one or more of the principles as in Table 2.2 (Rietma, 1982).

Principle A

On the basis of controlling or increasing the slip velocity in a conservative force field to which a potential field can be ascribed. Gravitational, centrifugal or electrostatic field is mostly applied.

Principle B

On the basis of forced nullifying the dispersed phase velocity while the continuous phase can be pass through.

Principle C

On the basis of impingement, scrubbing or also adsorption in which case the velocity of the dispersed particle will be matched to the velocity of other generally coarsely dispersed particles which are more easy to separate.

Table 2.2 Phase separation principles

Phase system	Separation principles		
	Principle A	Principle B	Principle C
Gas-Liquid	Cyclones Electrostatic-precipitators	-	Scrubbing Wire mate
Gas-Solid	Cyclone Cottrell	Dust filter Sieving	Scrubbing, Adsorption in fluidised bed
Liquid-Gas	Cyclone	-	-
Liquid-Liquid	Settling Cyclones Centrifuges	-	Coalescers
Liquid-Solid	Sedimentation Cyclones Centrifuges	Cake filtration	Deep bed film adsorption in fluidised bed

2.2.3 Solid-Liquid separation

Solid-liquid separation is probably the most common phase separation operation. The most suitable techniques to use will dependent on size and nature of solid particles, solid concentration and feed rate of suspension and density and viscosity of fluids. Sinnot (1996) gave an elaborate description of various solid-liquid separation processes which are illustrated in Fig. 2.2.

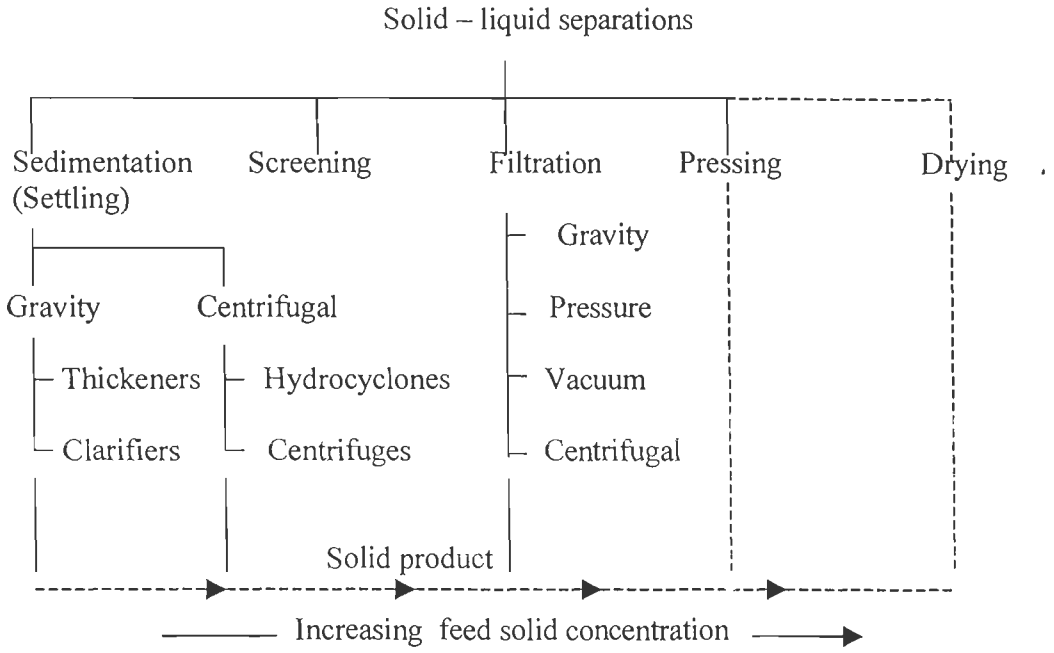


Fig. 2.2. Solid - liquid separation techniques

2.2.4 Settling or sedimentation

Settling or sedimentation is a process for removing suspended particles that are heavier than liquid by gravitational settling. Among the various solid-liquid separation processes, it is the most common method. It is a very old unit operation practiced from ancient times. As an important factor in the natural purification of stream, lakes and tidal water, it was observed centuries ago and used in ancient water works in Egypt and Roman empire (Ignjatovic, 1991). It appears to be an attractive method for removing fine solids from slurry owing to low cost of the equipment, simple process and its secondary functions in providing temporary storage (Martinez *et al.*, 1995).

2.2.4.1 Types of settling

On the basis of concentration of the suspension and the tendency of particle to particle interaction, settling process has been divided into four types (Shukla and Srivastava, 1992; Metcalf and Eddy, 1993 and Droste, 1997).

a. Discrete particle settling (Type I)

It is the sedimentation of particles in a suspension of low solid concentration in which particles settle as individual entities and there is no significant interaction with neighbouring particles.

b. Flocculant settling (Type II)

It refers to a rather dilute suspensions of particles that coalesce or flocculate during the sedimentation process. By coalescing, the mass of particle increases and settles at a faster rate.

c. Hindered Settling (Type III)

It refers to a suspension of intermediate concentration in which inter particle forces are sufficient to hinder the settling of neighbouring particles. The particle tends to remain in fixed position with respect to each other and the mass of particle settles as a unit. A solid-liquid interface develops at the top of the settling mass.

d. Compression settling (Type IV)

It refers to settling in which the particles are of such concentration that a structure is formed and further settling can occur only by compression of the structure. Compression takes place due to the selfweight of particles which are constantly being added to the structure by sedimentation from the supernatant liquid.

During a sedimentation operation, it is common to have more than one type of settling to occur at a given time and it is also possible to have all four types to occur simultaneously (Metcalf and Eddy, 1993).

2.2.5 Starch settling

Settling is one of the most important unit operations involved in cassava starch extraction process to separate the starch granule from its suspension in water. The oldest, but still practiced method for settling of starch is by using settling tanks where the starch milk is

allowed to stand for a period of 8-12 hours. When the starch settles down at the bottom of the tank, supernatant liquor is drained through the outlets located at various depths in the tank. It is a batch operation and analogous to settling basins employed for waste water treatment where settling occurs in the same manner as in quiescent settling containers of same depth.

2.2.5.1 Settling tables

It is desirable to complete the settling process as quick as possible to prevent the formation of alcohols and organic acids especially butyric acid. Tabling is a semi continuous settling process used to reduce the time of contact between starch and fruit water (Radley, 1976 and Balagopalan *et al.*, 1987).

Tabling process were used for the separation of maize starch (Berkhout, 1976), potato starch (DeWilligen, 1976) and rice and sweet potato starch (Radley, 1976)

Radley (1976) described the process of separation of cassava starch from its milk on more steeply inclined tables as "silting". In tabling process, the suspension generally contains starch about 25-30 g/l, but in silting, higher concentration upto 250 g/l was allowed.

Balagopalan *et al.* (1987) described the tabling process used in cassava starch settling. It consisted of successive sets of channels or troughs about 40-50 cm wide, 20-30 cm deep and 30-100 m long. The starch suspension is allowed to flow very slowly along a slightly inclined shallow trough. When sufficient starch settles at the base of the channel, the flow of starch milk is temporarily stopped and starch is removed manually.

2.2.6 Settling characteristics of suspended solids

Rate of settling of a particle in quiescent condition was first analysed by G.G. Stoke, an English Physicist in 1851 (Gee and Bander, 1990). He gave a relationship between the radius of particle and its rate of fall from a height in a fluid medium by considering the gravity force and buoyant force acting on the particle.

The relationship was

$$v_s = \frac{g\, d_s^{\,2}(\rho_s - \rho_f)}{18\,\mu_f} \qquad \text{.....2.1}$$

where,

v_s = Settling velocity, m/s

d_s = diameter of particle, m

ρ_s = density of particle, kg/m^3

ρ_f = density of fluid, kg/m^3

g = acceleration due to gravity, m/s^2

μ_f = viscosity of fluid, kg/ms

Basic assumptions made for deriving the Stoke's law are:

a) Particle must be sufficiently large so that Brownian movement will not influence the rate of fall.
b) Particle must fall independently.
c) There must be no slipping between the particles and liquid
d) Particles are smooth and spherical.
e) Velocity of fall must not exceed a certain critical value (laminar flow) so that the viscosity of the liquid remains the only resistance to the fall of particles.

Stoke's law for sedimentation is valid only for dilute suspension where, the effect of neighbouring particles on the movement of particle under consideration can be neglected *i.e.*, it is ideally suitable for discrete particle settling. But at high solid concentration, the interfering force of adjacent particles cannot be ignored (Iordache and Corbu, 1986). Starch in water will not obey the phenomena of discrete particle settling as it is composed of very large number of fraction of different size granules and will coalesce or interact with other particle and obey the principles of flocculant or hindered settling. So, the settling of cassava starch in water is entirely a different phenomena from Stoke's law and to be investigated separately.

The behaviour of suspended fine particles during sedimentation is profoundly affected by the solid concentration. In very dilute concentration, the particle settles at velocities which are very close to the free falling velocities. But in high concentration, when a particle settles down, due to their movement, an equal volumetric flow rate of displaced fluid takes place and particle must move with a higher velocity relative to the displaced fluid velocity (Mirza and Richardson, 1979). This velocity distribution generated in the fluid surrounding each moving particle causes an interaction between particles and also between fluid and solid particles.

There have been many investigations on the effect of concentrations on sedimentation rate of both uniform particles in suspensions and of the settling of naturally occurred solids, many of which becomes highly flocculated when in aqueous suspension.

Robinson (1926) modified Stoke's equation by substituting the density and viscosity of suspensions in place of that of fluid. Steinour (1944) studied the sedimentation of small uniform particles and obtained an expression for the velocity of particle relative to fluid as:

$$u_p = \frac{d^2(\rho_s - \rho_c)g}{18\,\mu_f} f(e) \qquad .....2.2$$

where,

u_p = Velocity of the particle relative to fluid, m/s

ρ_c = Density of suspension, kg/m^3

f(e)= a function based on the voidage of suspension

He found the value of f(e) for the sedimentation of tapioca in oil as $10^{-1.82(1-e)}$ where, e = voidage in decimal.

Richardson and Zaki (1954) conducted sedimentation experiments using a suspension having particle diameter ranged from 1-100 μm and suggested a relationship between sedimentation velocity and volumetric concentration as:

$$\frac{v_c}{v_o} = e^n = (1-c)^n \qquad \text{.....2.3}$$

where,

v_c = sedimentation velocity, cm/s

v_o = free falling velocity, cm/s

e = voidage of suspension, decimal

C = volumetric concentration, decimal

They found the exponent `n' as a function of the ratio of particle diameter to tube diameter (d/D) and of the particle Reynold's number (Re').

Maude and Whitemore (1958) established value of 'n' in Eq (2.3) as 4.65 for hindered settling for Re' < 0.2 and Richardson and Meikle (1961) found it as 4.8 for fine particles (<100 μm) and 10.5 for alumina of very fine particle size(4-7 μm).

Richardson and Shabi (1960) have shown that Eq (2.3) can also be applied to calculate the sedimentation rate of any sized particles in a poly dispersion suspension provided that the correction factor for the terminal falling velocity of the particle involves the total concentration of particles of all sizes that are present.

Gramme *et al.* (1971) analysed the effect of concentration of dairy bull manure on settling and observed that relatively little solid settled in 5.5 per cent total solid manure slurries compared to 1-3 per cent total solid slurries.

Lockett and A1 Habbooby (1973) suggested an equation for hindered settling velocity as:

$$v_s - v_f = \frac{g\, d_s^{\,2}(\rho_s - \rho_f)}{18\,\mu_f}\alpha_f\, F(\alpha_f) \qquad \text{.....2.4}$$

where,

v_s = particle velocity, m/s

v_f = fluid velocity, m/s

g = acceleration due to gravity, m/s^2

d_s = diameter of particle, m

ρ_s = density of particle, kg/m^3

ρ_f = density of fluid, kg/m^3

μ_f = viscosity of fluid, kg/ms

α_f = fluid volumetric concentration or the suspension voidage

$F(\alpha_f)$ = function accounts for particle concentration

This equation is a modified form of the classical Stoke's equation *i.e.*, when $\alpha_f = 1$, $F(\alpha_f) = 1$ and the equation becomes Stoke's equation.

The most acceptable form of the function $F(\alpha_f)$ as described by Richardson and Zaki (1954) is

$$F(\alpha_f) = \alpha_f^{2.7} \qquad .....2.5$$

According to Barmea and Mirzachi (1973)

$$F(\alpha_f) = \left[1 + (1-\alpha_f)^{1/3} \exp\frac{5(1-\alpha_f)}{3\alpha_f}\right]^{-1} \qquad .....2.6$$

Moore *et al.* (1975) found very little difference in the settling characteristic of 0.01, 0.10 and 1 % total solid concentration of dairy manure slurries and about 60% of the total solid was settled within 10 min in all the cases.

Dixon *et al.* (1976) studied the inertial effect on sedimentation using a simple model of an incompressible slurry. The behaviour of the model has also been calculated from the same equation but the inertia term neglected and different results were obtained, stressing that the inertial effect cannot be ignored in the studies of sedimentation process.

Smiles (1976) evaluated sedimentation in terms of non linear Focker- Plank equation and demonstrated that the rate of accumulation of liquid above the settling solid was depending on the liquid content at the top of the solids, also on the total amount of solid in the column.

Vesilend (1979) developed a general equation for hindered settling velocity at any concentration with two adjustable parameters 'a' and 'b', of the form

$$V_h = a \exp^{-bC} \quad .....2.8$$

where,

V_h = hindered settling velocity, m/s

C = concentration of suspension, decimal.

Mirza and Richardson (1979) developed a simple model for sedimentation of multi component mixtures of particles of different sizes and tested experimentally for binary system. Diagrammatic representation of the formation of zones during sedimentation of poly dispersed suspension was also given. They applied a correction factor $e^{0.4}$ to Eq (2.3) to allow for the interaction between the particles settling at different velocities.

Hills and Kemmerle (1981) carried out lab scale investigations to find out the dewatering capabilities of vibrating screens, sedimentation tanks and sand drying beds for dairy manure digester effluent having 6.5 per cent solid concentration. During sedimentation, very little separation was achieved *i.e.,* it was concentrated only to about 7.3 per cent after 48 h of settling.

Wilson and Lee (1982) used the equation $v_h = a\ C^b$ where, v_h = settling velocity, C = concentration, decimal ; a and b = constants describing the initial settling velocity of suspensions at any concentration.

Grootscholtan and Dejong (1985) measured particle velocities in a solid-liquid dispersion of poly sized particles at a solid concentration from 1.6 - 3.6 per cent (v/v). For particles smaller than 300 μm, the slip velocity unexpectedly increased with increasing solid concentration and became higher than that of free terminal falling velocity. The results indicated that particle to particle interaction played an important role in determining the velocities of separate particles in the suspension.

Law *et al.* (1987) studied the gravity separation of bidisperse suspension containing particle species lighter and heavier than the suspending fluid, both theoretically and experimentally using a vertical sedimenting tube. For a dilute suspension (<16 per cent), the natural segregation of light and heavy particles into clusters and formation of fingering flow structure do not occur, but the settling velocities of both particle species are retarded.

Keinath (1990) correlated the sludge volume index (SVI) and concentration (C) with hindered settling velocity (Vh) as

$$v_h = (25.3\text{-}0.061\ SVI)\ exp\ (0.426 + 0.00384\ SVI\text{-}0.000055^2)]\ C \qquad .....2.9$$

Hultman *et al.* (1991) evaluated different sludge indices to predict activated sludge sedimentation. The various indices studied were sludge volume index, diluted sludge volume index, sludge quality index and stirred specific volume index. A method was given to predict the maximum solid handling capacity in sedimentation basin. Initial sedimentation velocity (ISV) was found to vary with solid concentration (SS) as

$$Log\ (ISV) = 0.92 - 0.18\ (SS) \qquad .....2.10$$

Tokunaga (1991) conducted settling and filtration tests on 25 types of lake and river sediments. A linear relationship of initial settling rate and compression volume was obtained, indicating that the sediments of low initial settling rate do not easily loose water by compression. Data on particle size also showed a linear relation to the square of initial settling rate and compression volume.

Friedrich *et al.* (1993) studied the dependence of the particle size distribution and surface charge on the behaviour of sludge in dewatering. Based on lazer diffraction measurement to determine particle size distribution, it was found that specific proportion between fine and large sludge particles was necessary to produce good separation efficiency and to get the required shear resistant flocs.

Gregory (1993) explained the role of colloid interaction in solid-liquid separation. He discussed the most important types of colloid interactions *viz.,* vander Waals forces, electrical interaction, hydration forces and hydrophobic interaction. These short range interactions have little influence on the transport of particle, but have a major effect on collision efficiencies and on the adhesion between particles. Flocculation, coagulation, filtration depth, cake filtration and dissolved air flotation are the main solid-liquid separation processes where collision interaction are very important.

Lawler (1993) explored two methods *viz.,* turbulent flocculation and hetero disperse (curvilinear) flocculation to describe the collision and interaction between the particles during sedimentation. In turbulent flocculation theory, the velocity gradient required for collision frequency is a decreasing function of particle size. The curvilinear collision approach accounts for hydrodynamic interaction and vander Waals interaction, showed a dramatic reduction in the predicted collision frequency between smaller and larger particles.

Rajagopalan (1993) presented a modern thermodynamic theory, capable of predicting stability diagram for dense as well as dilute dispersion. He termed coagulation as a phase separation process, similar to gas/liquid, liquid/liquid and liquid/solid phase transition in atomic molecular system. This type of separation plays a significant role in water treatment, biochemical separation process, ceramic processing and food industry.

Tiller and Hsyung (1993) described that the concentration of slurry during settling was related to movement of particles as individual entities or as a mass in zone form. In dilute slurry containing particles of different sizes and densities, the particle settled individually with different velocities. As the concentration increased, the motion of each particle was affected by its neighbours, and ultimately a concentration was reached where zone settling prevailed and all the particles moved with same bulk velocity. Zone movement was divided into freely segmenting suspension and cake with structures capable of supporting stresses.

Vesilind and Jones (1993) explained the chanelling effect occurred during batch thickening of concentrated slurries which allowed water to escape from the settling slurries at high rate, thus increasing the interface settling velocity. This also showed that the upper layer of the settling column was progressively diluted and that the channel zone occurred at the interface between the diluted slurries and the slurry in compression. As the solids in the lower section of the columns compressed and expelled water, this water escaped by means of the channels.

Martinez *et al.* (1995) compared different separation process for the treatment of pig slurry. Sedimentation techniques were shown to have the potential to concentrate the insoluble matter of treated slurry into a sludge phase and a separation of 30-50 per cent was achieved.

Akhtar (1997) carried out a study in a specially designed settling columns to investigate the settling characteristics of effluent from Karachi tanneries. Design curves in terms of per cent removal of suspended solid versus loading rate and detention time were constructed using the data obtained from settling column tests and these curves could be applied in the design of settling tank in the waste treatment plant.

Settling or sedimentation is the most commonly and widely practiced method for separating starch from its suspension in water. The wide range and rather smaller granule size cause them to behave in a different manner than that of discrete particles during settling. The literature cited in this section are all concerned with settling characteristics of suspended solids especially in waste water treatments. The starch milk is a two phase coarsely and freely dispersed system and hence the fundamental principle governing the settling of disperse system can also be extended to starch granule settling in water. But reports on the settling characteristics of starch milk is scanty. Besides to obtain the settling characteristics, the design curves obtained from the batch settling test may help in the design of settling tanks or tables used in starch industry.

2.3 Processes to Aid Settling and the Properties of the Modified Starch

Efficiency of settling is determined by the speed of settling and also by the compactness of the settled starch. When starch settles into a firm cake, the starch loss occurred during the draining of fruit water will be less. Various chemicals such as acids and bleaching agents are added during the settling process by the starch industries without having clear cut knowledge about for what purpose and how much to be added. Most of the factories add chemicals for improving whiteness of starch. But knowledge on the effect of these chemicals on the rheological properties of starch, a prime criteria in deciding the quality of starch for various purposes is lacking. Chemicals are well known for modifying the functional properties of starch. Modification of the properties of native starches are mainly directed to improve their functions and expand the usefulness in industrial application. Hence, knowledge on the effect of various chemicals on settling, thermal and rheological properties of cassava starch is a prerequisite for the judicial addition of various chemicals during starch settling process.

2.3.1 Effect of chemicals on settling and properties of starch

Morgan (1940) reported that granules of oxidized corn starch gelatinized or became paste at progressively lower temperature as the level of oxidation by hypochlorite increased. Pasting temperature of oxidised starches were lower than those of modified starches.

Kerr (1944) reported that in settling, the rate of deposition was markedly affected by the nature and amount of electrolyte present and other factors being equal, the anion is more effective, the higher its valency. Phosphate is effective, but a small addition of sulphuric acid is sometimes favoured.

The rheological properties of acid modified corn starch was determined and found that the acid addition lowers the hot paste viscosity at a more rapid rate (Bechtel, 1950 and Lloyd and Kirst, 1963).

Kerr (1952) examined the amylose and amylopectin fraction of acid modified corn starch and showed that in the early stages of acid modification, the amount of amylose in the starch increased indicating that the acid preferentially hydrolyzed more accessible amylopectin.

Sulphuric acid is often added to aid sedimentation and for producing whiter starch. Addition of 0.001 ml acid/l starch suspension gave rapid sedimentation, but a rather soft sediment. It was reported that higher concentration above 0.001 ml per litre starch suspension decreased the starch paste viscosity (Holleman and Aten, 1956). He also reported that alum had a favourable effect on sedimentation and by adding 0.1 g/l starch milk, 50% increase in viscosity was observed.

Cowie and Greenwood (1957) investigated the acid hydrolysis of potato starch and showed that no granule swelling occurred and no loss of birefringence, suggesting that acid was preferentially attacking the amorphous rather than the crystalline region.

Leach and Schoch (1962) observed an increase in gelatinization temperature by 7°C than the unmodified corn starch by the acid treatment.

Schmorak *et al.* (1962) noted that oxidation by hypochlorite increased the size of wheat starch granules by approximately 16% but no change in size was noted with waxy corn starch.

Ganz (1965) reported that a 2.5 per cent sodium chloride solution increased the peak viscosity of corn starch and wheat starch in the Brabender Amylograph. The onset temperature for viscosity development was not affected by this treatment.

Effects of sulphur dioxide on starch settling were reported by Radley (1976) and Kordylas (1991). It helped to separate the starch from the other substances to which it was more or less firmly bound in the cell. It also checked bacterial and enzymatic action during the process and acted as a bleaching agent to get white starch. But viscosity was found to be reduced for the modified starches.

Radley (1976) reported that a compact sediment was obtained when one milligram alum was added to one litre starch solution and viscosity of the starch paste was found to be increased at this concentration. However, higher concentration (50 mg/l) resulted in a very soft and discoloured sediment and a product of very low viscosity.

Biliaderis *et al.* (1980) conducted DSC analysis to study the gelatinization phenomena of acid modified corn and pea starches and obtained a higher gelatinization temperature for the modified starches.

Rutenberg and Solarek (1984) reported that modification by oxidising agents like hypochlorite, permanganate or hydrogen peroxide improved whiteness and lowered the microbiological population in the starch. These oxidised starch found use where intermediate viscosity and soft gels were desired. They also had the unique property of improving adhesion of starch batters to fish and meat and were widely used in breaded foods.

DSC analysis for *Dioscorea ballophylla* and *Amorphophallus campanulatus* were carried out by Soni *et al.* (1985); sweet potato by Garcia and Walter (1998) and african yams by Farhat *et al.* (1999)

2.3.1.1 Differential scanning calorimetry

Starch granules are insoluble in cold water, but when an aqueous starch suspension is heated over a critical temperature, the hydrogen bond responsible for the structural integrity of the granules weakens, allowing penetration of water and consequently

hydration of linear segments of amylopectin takes place. As a result, the molecules start to form helices of coils creating a tangential pressure causing the granules to imbibe water and swell many fold to their original volume with a specific range of temperature, the hitherto opaque aqueous solution transforms to a translucent gel. The above process of transition that taking place in the starch granule is termed as gelatinization. The gelatinization results in loss of crystalline and birefringence properties, swelling of granules and leaching of amylose bringing about considerable changes in the viscoelastic properties.

Differential scanning calorimetry (DSC) is an important technique to study the thermodynamics of starch gelatinization, revealing several endothermic phenomena during heating of moistened starch. During gelatinization, DSC measures the extent of disruption, primarily of the hydrogen bonds that stabilizes the double helices within the starch granules and quantifies the heat energy *i.e.*, the enthalpy involved in transition of starch from a series of crystalline granule to an amorphous gel (Tester, 1997). This thermal analysis technique identified both first and second order thermal transition (melting and glass transition, respectively) and process of either exothermic and endothermic character can be measured during heating or cooling of the sample (Boerio Goates and Callaman, 1992). The onset (T_o) and final (T_e) gelatinization temperatures and enthalpy of gelatinization (ΔH) could be measured by DSC.

2.3.1.2 Rheological properties of starch

Starch constitutes an excellent raw material for modifying the texture and consistency of food because of its ability to form visco- elastic pastes or gels when heated in excess water. Visco-elastic materials have both elastic and viscous properties due to which they store or dissipate energy upon deformation. The study of every thing from elastic solid behaviour to viscous liquid behaviour, is the subject matter of "Rheology". The rheological methods give information about the sample structure and consistency,

quality control of raw materials and products, control of process conditions, rheological behaviour in relation to mouth feel of a product and interaction between the component in a composite material (Larsson, 1997).

Visco-elastic properties of starch samples are measured either by static or dynamic methods. In dynamic methods of measurements, the potential energy and the energy which is dissipated as heat can be separated into the storage/elastic modulus (G') and loss/viscous modulus (G"), respectively. The measurement of G' and G" gives an indication about the elastic and viscous properties of a material over a spectrum of time i.e., when G'>G" elastic properties and G" >G' viscous properties dominates.

2.3.2 Electroflocculation of cassava starch suspension

Starch particles when suspended in water acquires a motion of constant velocity due to gravitational force. They are not uniform in shape or size, and the size ranges between 5-45μm. This wide range of size causes random motion of starch granules during settling and larger particles attain high terminal velocity compared to smaller ones. The random motion and velocity gradient of particles leads to both perikinetic and/or ortho kinetic flocculation, respectively.

Schroeder (1977) gave details about the kinetics of flocculation. Perikinetic flocculation is due to the random motion of particles (Brownian movement) resulting from collision with fluid molecules and is significant for very small particles, whereas orthokinetic flocculation is induced by the presence of velocity gradients in the fluid. Because of the velocity gradients, particles travel at different velocities and collision results. Particle that collide may aggregate and the increased mass of the particles, enhanced the settling rate.

If the collision frequency of the starch granules are increased by some external aids, very faster settling can occur. Gas bubbles can be generated electrolytically in starch milk by passing direct current through electrodes immersed in the milk. These gas bubble of

smaller sizes, during their upward movement came in contact with the starch granules and caused frequent collision between the particles. This results flocculation to occur at faster rate and thereby enhances the settling rate. This is a new concept as against the electroflotation in waste water treatment where the finer suspended solids in water is carried along with the gas bubbles to the surface, and we could term it as **"Electroflocculation"**. So the principle of electrolysis and electrokinetic phenomena should be known for the study of electroflocculation.

2.3.2.1 Electrolysis

The principle of modern electrochemistry was built up during 1950's. When metal electrode is dipped in aqueous solution, charge transfer occurs at the metal-solution interface as current is passing through the system resulting in a desired chemical change. Electro chemistry is the science that deals within and around phenomena arising from electron transfer at interface.

Andrews and Kokes (1965) explained in detail about the overall reaction during water electrolysis as $2H_2O \rightarrow 2\ H_2 + O_2$, at one electrode, hydrogen gas is produced through the effect of electric current and oxygen gas is given off at the other electrode.

The gas bubbles so generated during electrolysis forms the basis for electroflotation for the treatment of dilute suspension. The bubbles attach to the suspended particles which then rose to the surface. The high surface volume ratio of small bubbles make flotation process very effective in suspension of fine particles (Coulson and Richardson, 1980). It allowed the dilute suspension to be separated into a concentrated slurry and clear liquid. Unlike waste water having very fine suspended particles at very dilute conditions, starch milk contained comparatively bigger particles at high concentrations and hence the chance of carrying these particles to the surface is less, instead, they may collide with each other and form aggregates and settle at the bottom, we can term this phenomena as '**Electroflocculation**'.

2.3.2.2 Electrokinetic phenomena

Electrokinetic phenomena deals with either the mechanical motion occurring between different phases of heterogenous system having at least one liquid phase under the action of an externally applied electric potential or setting up of potential difference due to the relative motion between boundaries (Michael, 1926).

Samex (1932) described the process of cataphoresis in which the starch constituents were transported to positive poles in the electric field. The speed of migration was highest for the amylo phosphate and much slower for electrolyte free carbohydrates so that a partial separation occurred during cataphoresis.

If solid particles are suspended in water, they are free to move under electric stress and Holmes (1934) termed this as electrophoresis as some particles moved to cathode or anode.

Farely and Hixon (1942) conducted electrolysis of starch and found that in the initial phase of oxidation of starch by electrolysis in alkaline sodium chloride solution, the viscosity increased and beyond 0.2 equivalent chlorine per glucose unit, the viscosity reduced continuously.

Electrophoresis (movement of sol particles with respect to the liquid); Electro osmosis (movement of liquid with respect to particles in a porous diaphragm); sedimentation potential (reverse of electrophoresis) and streaming potential (reverse of electroosmosis) are the electrokinetic phenomena generally encountered in colloid chemistry (Weiser, 1958).

Bokris and Nagy (1974) explained in detail about the various electrochemical treatment processes *viz.*, electro dialysis, electoflotation, electroflocculation, electro filtration and electrostatic precipitation for waste water/air treatment.

Deyl *et al.* (1979) described about the electrode reactions and transport phenomena. Electrode reactions are characterised by an immediate exchange of electrones and the solution and as a result of this reaction, concentration gradient originate near the electrodes causing diffusion and migration flow. Vacik (1979) explained the theory of electromigration process where separation was based on the difference in the mobility of electrically charged particles in an electric field.

Coulson and Richardson (1980) gave an illustration of an electroflotation plant for waste water treatment in which a potential difference of 5-10 V was applied to electrodes kept at a distance of 5-40 mm to give a current density of 100 A/m^2. The floated sludge containing about 95 per cent of the solids form blanket on the surface which can be continuously removed by means of slowly moving scraper mounted across the top of the tank.

Electroflotation has been first applied to the treatment of domestic sewage in 1911 in the US and Fukui and Yuu (1984) narrated the advantages of electroflotation process as: the apparatus for the process was small and compact, the amount of generated gas was suitably adjusted by controlling electric current, the diameter of electrolysed gas bubbles was as small as 20 μm and small flocs were collected and floated. Since the reaction of oxidation by electrolysis occured, besides the generation of gas bubbles, it could reduce BOD and COD values. They developed a new apparatus for electro flotation by using impeller of stirred tank as an electrode. The principle was that gas bubbles generated by electrolysis float the flocs while flocculation is promoted by the rotation of electrodes.

Vik *et al.* (1984) studied the electro coagulation of potable water and they compared this process with conventional flocculation-coagulation treatment process and came up with the following advantages for the electro coagulation process: the amount of chemical used was 1/10 of the amount required in conventional treatment, lesser amount of sludge was formed, time needed was reduced and operation and maintenance of the system was simple.

Kondoh and Hiraoka (1990) studied the application of electroosmosis to conventional filter press dehydrator to reduce water content in the sludge generated from the waste water treatment process and found about 50-60 per cent reduction of water content in the sludge.

Ju (1991) investigated the electroosmotic dewatering of bentonite clay suspension using DC power supply (4-8V) with 5 cm diameter column. He found that electroosmosis removed 20-60 per cent of the water with energy expenditure well below the energy required to vapourise the water and amount of water removed was directly proportional to the voltage and current applied.

Pouet *et al.* (1992) studied the electro-coagulation flotation tangential flow micro filtration processes for waste water treatment. This combined process was found to be helpful to reduce the fouling of membrane and was compact and effective.

Rahimi and Mirzai (1997) exploited the principle of electroosmosis for the transmission of moisture in soil. They tried to remove water in the soil porous media using electroosmosis and supply enough moisture in the root zone so that the plant could use the moisture for growth without any surface irrigation.

2.3.3 Effect of inclined column on settling

From an economic point of view, the depth of settling basin should be minimum so as to minimise the overall size of the basin. Also, it was reported that solid removal in an ideal basin is dependent only on overflow rate and not much on fluid depth. (Droste, 1997).

A possible geometry for achieving shallow depth is bundles of tubes arranged either parallel or slightly inclined at the bottom surface of the basin. Camp (1945) was the first Environmental Engineer who attempted to exploit this concept by inserting number of sub floors into a horizontal sedimentation basin to increase the surface area. But he faced with the problem of sludge removal.

Pearce (1962) studied the effect of downward facing surface by considering an inclined tube and observed a considerable effect on sedimentation rate and he opined that it was possible to obtain an accelerated rate of settling in inclined plates.

Yao (1970) analysed the performance of the tube settlers and suggested an angle greater than 40° for better results. Culp and Culp (1974) however, found that angle greater than 45° were needed to permit the sludge to slide back down the table.

Ignjatovic (1991) described the design principles and theoretical results for a new type of settling tank, where within the settling zones of the tank, sedimentation took place in a tube as in quiescent container of equal depth. He reported that the new model was suitable where large sludge volume was to be handled or more putrefaction of sludge in contact with the flowing water was to be avoided.

Martinez *et al.* (1995) used a continuous sedimentation tank 180 x 80 x 30 cm dimensions with its base sloped at 10° to the horizontal for the sedimentation analysis of pig slurry. The results on the performance of the tank was comparable with settling column test.

2.4 Hydrocyclones

Cassava starch extraction by wet process require large quantity of water during various unit operations and separation of starch from the water used is a great problem in all the starch industries. One of the earliest, but still widely practiced method to separate starch from its suspension in water is the gravity settling in tanks or tables. Settling tanks held large volume and the process took several hours. The larger detention time of starch with fruit water causes fermentation, producing alcohols and organic acids (particularly butyric acid) which give foul smell, polluting the entire atmosphere, besides resulting undesirable changes in the starch properties. Sedimentation in settling tanks uses gravitational force to separate the starch granule from its suspension. But faster and effective separation is achieved if a combination of forces are applied on the particles.

The velocity of settling can be increased by applying centrifugal force. The most common application of this principle is the hydrocyclones. It is a solid-liquid separator working on the basis of centrifugal separation where an accelerated form of sedimentation takes place by centrifugal force which is several hundred times greater than the gravitational force.

2.4.1 Principle of hydrocyclone

The working principles of hydrocyclone have been described in detail by Svarovsky (1977); Bloor and Ingham (1983); Gomez (1992[a]) and Khatavkar and Pansare (1992).

Hydrocyclone-a simple and robust separating device- is used for solid-liquid and liquid-liquid separation, and also for solid classification. It has been suggested as a practical alternative in solid-liquid separation involving biological materials and suspensions (Ortega and Medina, 1996). It is often the least cost alternative for separation of particles having 4-500 μm sizes (Sinnot, 1996).

Hydrocyclone consisted of a conical vessel, usually of small included angle surmounted by a cylindrical section. Top of the cylindrical section is covered by a lid, centrally through which a tube known as vortex finder is connected to allow the overflow to pass through it. Inlet pipe is attached to the upper part of the cylindrical body which allows the feed to enter tangentially into the cylindrical part. At the apex of the cone, there is an orifice through which some of the fluid entered the cyclone is allowed to leave as a concentrated underflow stream (Fig. 2.3).

Fluid containing particles of different density or even two fluid is injected tangentially at high velocity into the upper part of the cylindrical section. The whirling motion so generated produce centrifugal acceleration which causes the particle to move relative to the fluid and therefore offer the possibility of separation or classification, subject to some means of collecting the distinct phase (Bloor and Ingham, 1983).

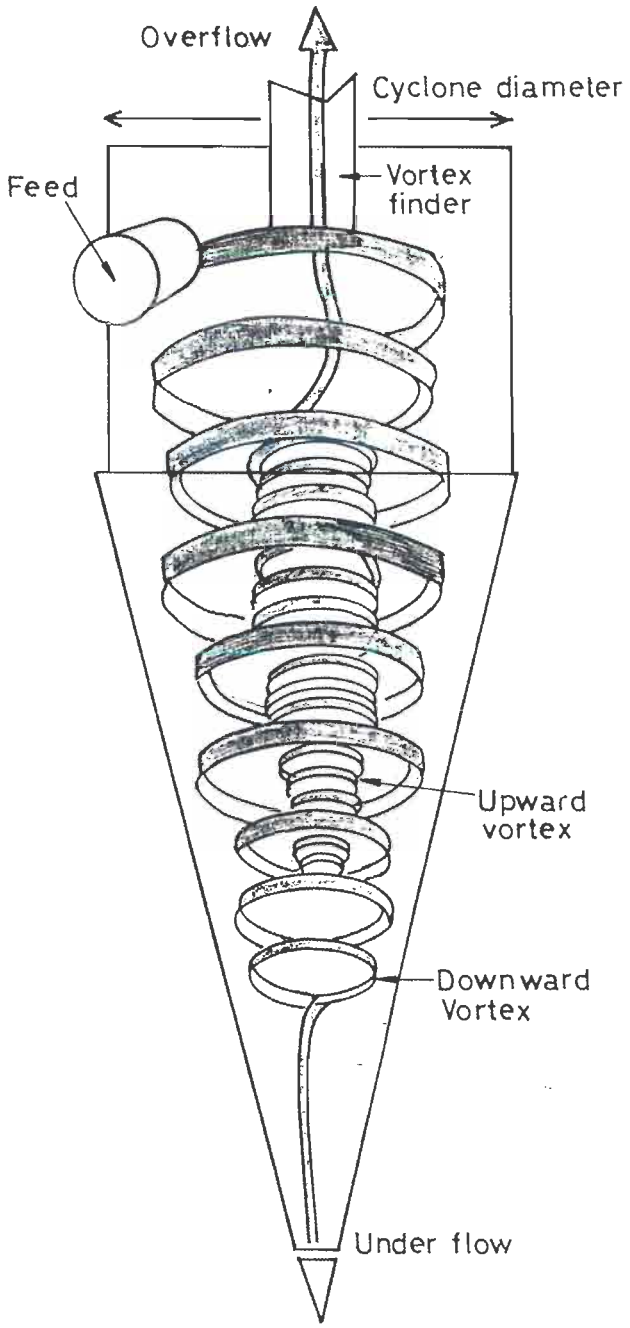


Fig. 2.3 Working principles of hydrocyclone
(after Gomez, J.V. 1992)

The very high radial acceleration produced to allow for a rotational rapid migration of particles, thus allowing a large rate of volume flows through the equipment. The stream being discharged out at the top of the hydrocyclone through the vortex finder consisted of the bulk of feed fluid together with very fine solids and is commonly known as overflow. The stream containing the remaining liquid and coarse solid is discharged through the bottom orifice as underflow.

2.4.2 Advantages of hydrocyclone

Hydrocyclones are easy to install, operate and require very limited space. Because of their low purchase price and operating cost, they are one of the first choices to accomplish solid-liquid separation. Since it contains no moving parts; installation, operation and maintenance are easy. It can be operated in a continuous manner at very low cost than most of the solid-liquid separation equipments. It can be constructed with a wide range of materials including plastics which is a corrosion resistant material. However, they generally are smaller in size causing low throughput capacity and efficiency.

2.4.3 Design of hydrocyclones

Hydrocyclones have been used in the process industries for more than 50 years. But the selection was usually according to manufactures specifications. Though the process is simple, it still remains as a complicated equipments perhaps due to the involvement of many variables that influence its performance.

Bradley (1960) suggested an empirical equation for finding out d_{50} particle size as a function of diameter, feed flow rate and liquid and solid properties.

Design consideration of hydrocyclone geometric dimensions were given by Rietma (1961) as a function of its diameter. Using these standard, it is possible to predict the separation and capacity in solid-liquid separation.

Zanker (1977) outlined a nomograph method to preliminary estimate the size of hydrocyclone based on Bradley (1960).

Schwallbach (1988) gave a graphical method to obtain design parameters of hydrocyclone *viz.,* cyclone chamber diameter, feed pressure and feed flow rate for sand-water suspension having solid content <250g/l and cyclone diameter 25-1000 mm.

Gomez (1992^a, 1992^b) presented an algorithm that make use of analytical expression for finding the diameter of hydrocyclone based on the data points taken from the graphical method given by Schwallbach (1988) for sand-water suspension. This method gave an equation for the specific flow capacity and feed pressure to be applied to the hydrocyclone. He also suggested a correction equation to apply the same equations for solids and liquids other than sand and water.

2.4.4 Hydrocyclone performance

Molyneaux (1962) conducted a series of experiments using mixture of screened particles of sand with salt and potassium permanganate in two hydrocyclones having 15 and 30 mm diameter. The extraction efficiency was found to be increasing with decreasing solid flow rate in both units.

Bradley (1965) gave a limiting size of hydrocyclone of about 10 mm below which, he reported, the separation of fine particle did not take place.

Rao and Rao (1975) described the hydrocyclone performance by an actual efficiency curve which was a plot of the particle size versus the percentage of the feed of that size entering the under flow discharge. An equation for finding out the rate of over flow stream as a function of feed rate and spigot diameter was developed.

Purchas (1981) reported that the pressure drop was the only operating cost for a hydrocyclone and for the typical range of 2-3 bar, it corresponded to a power consumption of 0.5 to 5 kw/m^3 feed/min.

Van Duijn and Rietma (1983[a]) investigated the hydrodynamical behaviour of large-cone-angle hydrocyclone for a suspension of sand (particle size - 280 μm and density 2650 kg/m^3) in water. They reported that at the bottom, a bed of suspended solid rotated which was in a fluidised condition. The pressure drop at high solid content was found to be hardly influenced by the volume fraction of solids in the feed, indicating that the change of inlet velocity head was almost balanced by the change of friction.

Van Duijin and Rietma (1983[b]) determined the separation properties of large cone-angle hydrocyclone of two different size at different cyclone configurations and input variables using a mixture of sand and magnetite. They showed that if a high separation efficiency was wanted at high throughput of solids and a low energy consumption, the cyclone should operate with a high vortex finder bottom clearance.

Rovinsky (1991) presented a model for the dynamics of the rotating flow of a fluid with suspended particles. The separation capabilities of hydrocyclone decreased dramatically with a decrease of particle size and small difference between particle and liquid densities. He suggested a new design to separate highly dispersed particle or in case where the density difference between particle and liquid was too small.

Van Esch (1991) studied the effect of size of hydrocyclone on the separation efficiency and found that the cut size of the particle to be separated decreased with cyclone diameter to the power 1.5.

Banerjee and Dey (1993) explained that increasing the diameter of hydroyclone with other conditions constant, reduced efficiency of separation. Effect of particle shape was also studied using flattened type particle and found that increasing overall size of particle lead first to a nominal increase in efficiency, by then to a sudden tempering off and then fairly rapid decrease in efficiency.

2.4.5 Hydrocyclones for biological materials

A hydrocyclone called "germ cyclone" was used for maize starch processing (Berkhout, 1976). The maize slurry from the fooz mill was forced through the tangential inlet and entered in a swirling motion. The light fractions containing germ and fibres discharged through the overflow and the main parts of the slurry containing the other solids discharged through the underflow opening.

Besso (1976) reported the use of hydrocyclone in cereal starch extraction process in place of germ separator. The size of hydrocyclone was about 100 mm installed singly or in batteries to separate lighter germ from heavy husk and efficient germ recovery was achieved with two pass system.

Fecske (1983) described two different arrangements of individual cyclones in parallel operations called ALCL and MOCL system. ALCL system consisted of a hydrocyclone deck having 20 cyclones of 10 mm diameter arranged radially with their areas horizontally like spokes on a wheel lying flat. In MOCL, the hydrocyclones of either 10 or 15 mm are held radially in a frame consisting of two concentric stainless steel tubes. The pressure drop was found to be varied with flow rate to the power 2.5 for both 10 and 15 mm units at free discharge conditions. This new hydrocyclone system combined with screens and centrifugal separators reduced the water consumption of 300-350 l/t of potatoes for starch extraction and refining.

Van Esch (1991) studied the suitability of hydrocyclone for different starches and concluded that they were effective for washing potato and maize starches, but not for rice and wheat starches. He also presented different methods to express the efficiency of hydrocyclones. He concluded that to wash starch economically using hydrocyclone, the reduced efficiency must be above 0.5.

Singh and Eckhoff (1991) developed a hydrocyclone system for starch-protein separation in lab corn-wet milling process and got a higher starch yield of 3-4 per cent for dent corn and 2-3 per cent for waxy corn. The hydrocyclone method reduced the time required for starch-protein separation by 75 per cent apart from eliminating the large floor area for starch tables.

Bendixen and Rickwood (1994) examined the comparative effect of passing mammalian and nucleated red blood cells through solid-liquid hydrocyclone and observed the cell disruption as a result of high operating pressure, but yeast cells were not disrupted by the highest operating pressure.

Satyamoorthy (1995) suggested the use of hydrocyclone in tapioca starch processing and reusing the water reduced the volume of effluent by 60 per cent.

Trim and Marder (1995) gave a basic performance data with selected hydrocyclone configuration for cassava starch milk concentration and reported a high and efficient levels of starch separation with considerable potential for reduction in water consumption and effluent released from starch factories. Standard hydrocyclones (25 and 50 mm) were used and experiments were conducted by changing the vortex finder and spigot diameter using different feed concentrations and operating pressures. They showed that there was no appreciable differences in the performance of the hydrocyclone unit with suspensions made up from dried starch and fresh starch.

A small hydrocyclone (25.4 mm) was used for primary sludge thickening of domestic waste water from a treatment plant by Ortega and Medina (1996). The thickening capability of hydrocyclone measured as the ratio between underflow and feed concentration was directly affected by the pressure drop and underflow diameter.

Yuan *et al.* (1996) investigated the possible use of hydrocyclone for the removal of yeast from beer and found that separation efficiency was affected by the concentration of yeast suspension and temperature with lower temperature resulted in poorer separation.

Marder (1998) explained various aspects of hydrocyclone technology for use in cassava starch/sago factories. He pointed out the various benefits from using this technology as:

i. Water consumption could be reduced by upto 60 per cent and hence the same amount of effluent too.
ii. Volume of settling tank required was reduced.
iii. Quicker settling of starch and reduction in quality loss.
iv. The quantity of dirt collected on starch in settling tank was reduced.
v. Less starch would be lost in the overflow from the settling tanks.

He stressed that the only operating cost required for running the pump would be offsetted by the savings made from the reduction of fresh water that would be pumped from the open or bore wells.

Thangavel *et al.* (1998[a]) studied the water consumption and product out put pattern in sago factories and a hydrocyclone of 10cm diameter was tested with varying feed pressures, feed inlet diameters, overflow diameters and underflow diameters to study the effect of these parameters on the underflow concentration. At 5 per cent feed concentration, a maximum increase of 206 per cent in underflow concentration was obtained for the inlet diameter of 19 mm, overflow diameter of 19 mm and underflow diameter of 5 mm at 49 Kpa. The operational cost of the developed single hydrocyclone to concentrate one m^3 of starch milk was Rs.1.50/-.

Thangavel *et al.* (1998 [b]) developed a single hydrocyclone of 10 cm diameter and tested for concentration of cassava starch milk. They found that the underflow concentration was increased with increase in feed pressure and decrease in underflow diameter and the effect of underflow diameter was more pronounced at lower feed concentration at all pressure levels.

Though hydrocyclones have been used in large scale starch manufacturing units for many years to separate the extracted starch from fibre and other impurities, their selection was mainly according to the manufactures specification without giving any consideration to the properties of disperse system used in the process. Selection of hydrocyclone diameter and feed pressure has a significant role in deciding its performance. Earlier works on design of hydrocyclone were reported for mineral processing. Very little work is reported on the design aspects of hydrocyclone for starch processing. Though they are widely used, the low throughput capacity of single hydrocyclone makes them unpopular in starch industries. Hence, batteries of hydrocyclones with individual inlets were used, but the cost of such system is too high to afford by the small or medium industries. Attempt should be made to operate batteries of hydrocyclones in series with one inlet for the first unit and the overflow from this unit serves as an input to the second unit and so on. It is also desirable that the overflow from the hydrocyclone system should contain minimum starch content so that it can be directed back to the starch extraction process (rasping), for this, effect of recycling of overflow stream on its final concentration is to be studied.

MATERIALS AND METHODS

CHAPTER III

MATERIALS AND METHODS

This chapter deals with the various experimental set up and techniques used for studying the settling characteristics of cassava starch in suspension. It also describes the experimental procedures for different processes to aid settling like electroflocculation and chemical addition and centrifugal settling. Design of hydrocyclone for cassava starch milk concentration and performance evaluation of a single hydrocyclone and a battery of five units in series are also dealt in detail in this chapter.

3.1 Settling Characteristics of Cassava Starch Suspension

Settling or sedimentation is the physical separation of suspended materials from water by the action of gravity. The mode of settling of particles in dilute suspension was found to be different from that of concentrated ones and hence a wide range of concentrations of the starch suspensions were taken to study the settling characteristics. Also, the settling characteristics of both reconstituted dried starch and freshly extracted starch were studied.

3.1.1 Preparation of starch suspension

3.1.1.1 Reconstituted dried starch suspension

Dried starch obtained from Sago Serve, Salem was used for all the experiments. Starch suspensions of varying volumetric concentration were prepared by adding water to a beaker containing weighed quantity of starch. The content was stirred well to make a homogeneous suspension and addition of water was stopped when the volume of suspension reached the required level.

3.1.1.2 Freshly extracted starch suspension

Fresh starch suspension was prepared from the cassava tubers, purchased from the local market. The tubers were washed, peeled and again washed thoroughly with water to remove all the dirt, dust and other impurities attached to the tubers. They were cut into small pieces (2-3 cm) and crushed using a pulper after adding enough quantity of water. Crushing was continued till a mash was obtained and it was screened through a 30 mesh sieve by adding water to remove fibrous and other smaller uncrushed tuber pieces. This suspension was again passed through 8 mesh sieve to get almost pure starch.

3.1.1.3 Measurement of concentration of fresh starch suspension

Hydrometer method was employed for measuring the concentration of fresh starch suspensions. Starch suspension was taken in one litre measuring cylinder and was agitated thoroughly to prevent settling of starch particles, immediately the hydrometer (1.0-2.0 range with each division marked as .01) was freely immersed in the suspension and the reading was noted. The procedure was repeated for the suspensions of different concentrations and the actual concentration or solid content in suspension was determined by the method given by Trim and Marder (1995). Samples (100 ml) from each experiment were taken in containers and kept over night to get a compact settled starch at the bottom of the container. The clear supernatant liquor was decanted out and the starch cake was then dried to a constant weight in a mechanical oven at 50°C. Volumetric concentration of starch was calculated as gram per litre suspension.

A calibration equation was developed between hydrometer reading and concentration of the fresh starch suspension. Suspensions with required concentration were roughly prepared by noting the hydrometer reading obtained from the equation. But to get the actual concentration of suspension, the above procedure given by Trim and Marder (1995) was employed.

3.1.2 Free falling velocity of starch particles

Free falling or settling velocity of discrete starch particles in water in quiescent condition was measured by employing Stoke's law

$$v_s = \frac{g\, d_s^{\,2}(\rho_s - \rho_f)}{18\,\mu_f} \qquad \ldots. 3.1$$

where,

v_s = Free falling velocity, m/s

g = acceleration due to gravity, m/s^2

d_s = particle size, m

ρ_s = density of particles, kg/m^3

ρ_f = density of water, kg/m^3

μ_f = viscosity of water, kg/ms

3.1.3 Particle density of cassava starch

Specific gravity bottle method as described by Mohsenin (1977) was employed for measuring the density of starch particle.

3.1.4 Particle size

Particle size was measured microscopically using an optical microscope (LEICA DMLB Research Microscope) with a magnification factor of 400X.

3.1.5 Settling characteristics of starch particles in suspension

Settling characteristics of starch particles in suspension in water was analysed by Laboratory Column Settling Tests. It consisted of graduated cylinders of varying diameters as detailed below. A scale marked in millimeter was fixed to the cylinder along its length. Starch suspensions of different volumetric concentrations was transferred to the column for settling to take place in quiescent condition. The position of the interface between the settling starch particles and an almost clear fluid with respect to time was noted.

The observation was stopped when there was no change in the interface height for two or three consecutive readings *i.e.,* settling process was almost completed. The preliminary tests showed that for suspensions upto 6% concentration, the interface was not clearly visible for the initial periods and hence the height of starch settled at the bottom (sediment height) at different time intervals was noted. The experiments were conducted in two concentration ranges *viz.,* low concentration (≤6%) and high concentration (≥10%) as the settling mechanism at these ranges was found to be different.

3.1.5.1 Rate of fall of interface

The rate of fall/subsidence of interface at high concentration was calculated as:

$$H = \frac{h_2 - h_1}{t_2 - t_1} \qquad \text{.... 3.2}$$

where,

h_2 = height of interface at time t_2, mm

h_1 = height of interface at time t_1, mm

3.1.5.2 Starch settling index (SI)

The term starch settling index analogous to sludge volume index in waste water treatment was introduced and determined. Sludge volume index is a calculation which indicates the tendency of solid sludge to thicken or concentrate during the sedimentation process.

It was calculated as:

$$SI = \frac{SV}{SS} \qquad \text{.... 3.3}$$

where,

SI = Starch settling index, ml/g

SV = settled starch volume, ml/l

SS = volumetric starch concentration, g/l

The mechanism of starch settling at different concentrations was visually observed to represent schematically the settling behaviour of starch suspensions.

3.1.5.3 Modelling of settling characteristics

The hindered settling velocity of the suspension was measured as settling velocity of the interface during the initial straight line portion of the interface settling curve as given by Metcalf and Eddy (1993) and Droste (1997) by noting the slope of the initial straight line portion of the curve.

The variation of the hindered settling velocity with fluid volumetric concentration was plotted and the equations of best fit was determined using a Curvefit computer programme for different settling columns and height of suspension similar to the equation given by Richardson and Zaki (1954) for mono dispersed system, but with slight modification. A general equation representing the relation between settling velocity, fluid volumetric concentration, ratio of height of suspension to the diameter of settling column was developed.

3.1.5.4 Experimental layout

Experiments were carried out in the following way:

Diameter of settling column	: 37 mm (250 ml) and 49 mm (500 ml)
Height of suspension	: 100, 200, 300 mm
Concentration of starch	: Low concentration - 2, 4 and 6 % (w/v)
	High concentration - 10, 12, 14 and 16 % (w/v)

In addition, settling columns having 22 mm (50 ml) and 28 mm (100 ml) with height of suspensions 200 mm were also used to verify the settling mechanism at low concentrations, as with higher diameter column, measurement of sediment height was found to be difficult and hence not considered.

3.1.6 Effect of settling time and concentration on water content of the settled starch cake

During settling, separation is achieved by the downward movement of solids relative to the water allowing a more concentrated slurry to form at the bottom. As the concentration increases, the liquid tends to move up through the interstices of contacting particles. As settling continues, compressed layers of particles begin to form at the bottom of the cylinder. Final compression and compaction allows most of the liquid to escape through the small interstices between the particles and hence the final water content of settled starch cake will be reduced. The amount of water present in the cake will give an idea about the level compaction of starch cake and also the total time required for settling .

Experiments were conducted to find out the final moisture content of the settled starch with 5, 15 and 25% concentrations for different settling periods upto 8 h. After each hour of settling, the supernatant liquor was decanted out and cake was kept in shade for 30 min to remove the surface film of water. Keep the sample in an oven at 45-50°C for partial removal of moisture. Then it was transferred to the oven at 100°C for 3-4 hours to find out the final moisture content.

3.1.7 Batch settling tests

Batch settling tests were conducted to study the settling characteristics of a suspension of low concentration where coalescence or flocculation occurs. Column diameter can be of any size, but should be equal in height to the depth of settling tables / tanks (Metcalf and Eddy, 1993).

Settling column made of plastic cylinder having height equal to the depth of settling tables used in starch industries (40 cm) were used for finding out the velocity distribution of starch suspension. Sampling ports were provided along the height of the column at uniform intervals as shown in Plate 3.1. Sampling ports made up of surgical

Plate. 3·1 Settling column

flexible tubes of 5 mm diameter and 100 mm length was fixed to the cylinder wall. The distance between the ports were fixed in such a way that they occupied the 100 ml mark in the cylinder giving a spacing of about 6 cm each and the ports were clamped on while doing the experiments.

Starch suspension was added to the column by closing the sampling ports and allowed to stand for a predetermined time intervals, after which the samples were collected from all ports after opening the ports. The solid content or concentration of the sample was measured by the method explained in Section 3.1.1.3

The data obtained from the test consisted of the concentration of suspension at various depths of column at different time intervals and were anlaysed to get a plot of the settling trajectories for various fractions of the suspended starch by the method given by Droste (1997).

The per cent starch settled (removed) at each depth was calculated by

$$r_{td}\,(\%) = \frac{C_0 - C_{td}}{C_0} \times 100 \qquad \ldots. 3.4$$

where,

C_0 = Initial concentration of the sample, %

C_{td} = Concentration of sample at time 't' and depth 'd', %

A computer programme 'Surfer' was employed to trace the settling trajectories by plotting the per cent starch settled or removed as a function of depth and time. The data from the figure can be used to estimate the total starch settled at any time .

3.1.7.1 Total starch settled

Fractional settling at any time (r_i) was calculated by using the equation :

$$r_i = \frac{di}{D} \Delta Pi \qquad \text{.... 3.5}$$

where,

d_i = average depth reached by the i^{th} fraction in time t_d , cm

D = total effective settling depth *i.e.*, depth above the bottom of the port, cm

P_i = fraction difference, %

The total starch settled at any given time was obtained by adding the per cent particles settled where the isosettling lines met with time axis and the sum of all the fractional settling at that time.

3.2 Settling in the Presence of Different Chemicals

There are only isolated reports on the use of chemicals for settling of starch. In addition to improve settling, it is also necessary that the starch quality should be maintained. Some of the major requirement of starch are based on the rheological and thermal properties. Any detrimental effect on these properties can lead to the lowering of the starch value. Hence, it was decided to compare the effect of some easily and commonly available chemicals on the settling of starch and also the effect of these chemicals on the rheological and physico-chemical properties of starch. The chemicals used include acids (sulphuric acid and hydrochloric acid), coagulant (alum) and bleaching and oxidising agents (sodium metabisulphite and sodium hypochlorite). The concentration of the chemicals were used based on the earlier works in this area. Kordylas (1991) reported the concentration to be used for different chemicals as 0.001 ml for sulpuric acid, 0.1 g for alum, 0.4 g for sulphur dioxide and 1 mg for chlorine per litre starch suspension and the available data explained only the viscosity change due to the addition of chemicals.

3.2.1 Sample preparation

A suspension of the freshly prepared starch was made by adding water to give 4 per cent concentration indicated by the hydrometer reading. This suspension was used for all the experiments related to chemically aided settling processes and quality evaluation. Chemicals of varying concentrations were added to the starch suspensions and stirred well to get a homogeneous starch suspensions. Settling characteristics were found out by the method described in Section 3.1.5. For quality evaluation, they were allowed to stand overnight to settle the starch. The supernatant liquor was decanted out and settled starch was dried in the sun and the samples were analysed for their rheological properties.

3.2.1.1 Sulphuric acid

A solution was prepared by carefully mixing 98 g of sulphuric acid with one litre of water which served as stock solution (1 molar concentration). Five milllitre of the stock solution was added to one litre starch suspension to give 5 mM acid concentration. Similarly 10, 20, 30 and 40 ml stock solutions were added to give an acid concentrations of 10, 20, 30 and 40 mM.

3.2.1.2 Hydrochloric acid

Stock solution was prepared by mixing 109.5 g acid with one litre water which gave 3 Molar solution; 5, 10, 20, 30 and 40 ml stock solutions were added to get 15, 30, 60, 90 and 120 mM acid concentrations.

3.2.1.3 Sodium hypochlorite

Sodium hypochlorite containing approximately 4 % chlorine was used and hence for getting one gram chlorine, 18.5 g hypochlorite was used. The amount of chlorine used were from 0.0125, 0.025, 0.05, 0.075 and 0.1 g for which 0.17, 0.35, 0.70, 1.06 and 1.41 mM chlorine concentrations was obtained, respectively.

3.2.1.4 Sodium metabisulphite

One hundred and ninety grams of sodium metabisulphite can release 128 g sulphur dioxide and 29.6 g of bisulphite in one litre of water can give a concentration of about 312.5 mM SO_2 per litre. Five, 10, 15, 20 and 25 ml stock solutions were added to one litre starch suspension to give 1.56, 3.12, 4.68, 6.29 and 7.8 mM sulphur dioxide concentrations, respectively per litre of starch suspension.

3.2.1.5 Alum

Stock solution was prepared by dissolving 94.8 g of alum in one litre water to give 0.1 molar solution. 200, 400, 600, 800 and 1000 ml of stock solutions were added to one litre starch suspension to give 16.7, 28.5, 37.5, 44.4 and 50 mM salt concentrations, respectively.

3.2.2 Gelatinization and rheological properties of starch

During electrolysis or chemical addition, besides affecting the settling rate, the properties of starch will also be affected. So measurement of rheological properties and thermal analysis of starch were carried out using Bohlin Rheometer and DSC apparatus, respectively. These experiments were carried out at Department of Food Technology, University of Lund, Lund, Sweden.

3.2.2.1 Differential scanning calorimetry (DSC)

DSC of the sample was run by using a Seiko 6200 Differential Scanning Calorimeter equipped with a built in software. Weighed quantity of the starch using a micro balance (four digits) was transferred into previously weighed aluminium pan and weighed quantity of water was added to give a water starch ratio of 3:1. The pan was sealed hermetically and transferred to the heating pan of the DSC. An empty pan was used as reference. Indium was used as standard. The sample was heated from 10-140 °C at 10 °C/min and cool back to 20 °C at the same rate. Gelatinization temperatures *viz.*, T_{onset},

T_{max} and T_{end} were obtained from the graph by using the built in software. Gelatinization enthalpy (ΔH) *i.e.,* the heat change occurring during gelatinization was also obtained from the graph for each samples. Experiments were conducted in three replications and mean values were reported.

3.2.2.2 Bohlin rheometer

Rheological properties of the starch sample was carried out in a VOR Bohlin Rheometer. The starch sample of 2.5 g was taken in a stoppered test tube, 25 g of distilled water was added to it and shaken well to provide a homogeneous suspension. To avoid settling of the starch during the test in the rheometer, each sample was heated in a water bath to 75°C or until all the starch became a paste, before placing the sample in the rheometer. After heating in the water bath, the slurries were poured into the cup of a C25 measuring system of VOR Bohlin Rheometer operated in the oscillation mode.

The experimental conditions used were:

Torsion element	:	1.67 g cm
Amplitude	:	3%
Sensitivity	:	1%
Thermal equilibrium time	:	10 s
Heating rate	:	1.5 °C/min

The samples were heated from 75 to 95 °C at 1.5 °C/min and held at 95 °C for about 10 min and cooled at the same rate as heating, from 95 to 35 °C. It was allowed to remain at 35 °C for about 60 min. The built in Bohlin software package recorded the time, temperature, phase angle, viscosity and dynamic modulii (viscous modulus and elastic modulus). Elastic modulus (G') is associated with the periodic storage and complete release of energy in a sinusoidal deformation process and viscous modulus (G") reflects the non recoverable use of applied mechanical energy to cause flow in the specimen.

3.3 Electroflocculation of Cassava Starch

The experimental set up for conducting the electroflocculation studies is given in Plate 3.2. It consisted of a direct current continuous variable auto transformer, stainless steel electrode assembly, electrode holder and sample container. The variable auto transformer is equipped with an in built current and voltage indicator. The electrode assembly was made up of two stainless steel electrodes (3 x 10 cm) separated by a non conducting wooden block (3 x 3 x 3 cm) to get an electrode gap of 3 cm. The terminals from the direct current transformer was connected to the steel electrodes by means of bolt and nut fixed at the top middle portion of the electrode and the electrode assembly was hung from a stand and immersed in the starch suspension present in the container.

The starch suspension of 4% concentration was taken in the beaker and the electrode assembly was immersed into the suspension. The electrodes were kept 3-5 cm from the bottom of the container to give enough space for settling. Experiments were conducted by applying different electrode potential levels and corresponding current drawn was noted. Temperature rise, if any, was noted using a digital thermometer. Experiments were conducted using three types of water obtained from different sources *viz,* borewell water (ordinary salt water), dam water (potable water) and double distilled water, quality of these water was analysed as per standard methods. Depending upon the type of water, voltage required to initiate electrolysis varies and hence different ranges of voltage were selected for the study in each experiments (15, 20 and 30 V for borewell water; 40, 50 and 60 V for dam water and distilled water). Electrolysis was done for 20 min and the suspension was allowed to stand for 5 min so that movement of particles will be stopped. The supernatant starch suspension (50 ml) was syringed out in a container to measure the suspended solids. The starch settled at the bottom was dried in shade and rheological properties were analysed.

3.4 Effect of Inclined Column on Settling

A tilting top arrangement was made and used for analysing the effect of inclined bottom surface of the container on settling. It consisted of a frame (32.5×32.5× 5 cm) to which a tilting surface was attached. The surface was a thick hard board sheet (30×30×0.5 cm) hinged at full length to the front side of the frame so that it can be tilted to any position as shown in Plate 3.3.1. A slotted arc of 30 cm radius and 30 cm height was fixed at one side end of the frame enabling to measure upto 90° inclination. A flat iron piece of 7.5 cm length and 1.5 cm width was fixed to the left top corner of the surface to which 1.25 cm bolt was attached, which could be freely moved inside the slotted arc and the surface can be fixed at any angle using a nut. Four cylindrical pipe sections of 3.5 cm height and 10 cm diameter were fixed on the surface as sample holders to keep the beakers containing starch samples.

Starch suspensions (200 ml) prepared in beakers were placed in the sample holders. Before placing the samples, the surface was fixed at the required angle using bolt and nut arrangement. Sample was allowed to settle for about 10 minute and 50 ml of the supernatant suspension was carefully syringed out and kept in a container for over night. The concentration of the sample was determined as in Section 3.1.1.3.

Experiments were carried out at four concentrations (2, 4, 6 and 8%) and angle of inclination ranging from 0 - 40° at 5° intervals and each experiment was replicated thrice.

3.5 Centrifugal Settling of Cassava Starch

Experiments on centrifugal settling was carried out by using a lab model centrifuge (REMI Laboratory Centrifuge-R80) with its rotor replaced by a small gadget as shown in Plate 3.3.2. The gadget was made with 20 × 8 cm mild steel sheet having 2mm thickness. Each end was bent 5 cm vertically upward and 5 × 5 cm central portion of which was cut and removed to fix a sample holder of about 6.5 cm diameter cylindrical ring with 2 cm length. It was welded at an angle of about 55° from vertical to the bottom flat portion of the gadget and to avoid outward sagging of the holder during rotation, it was connected with

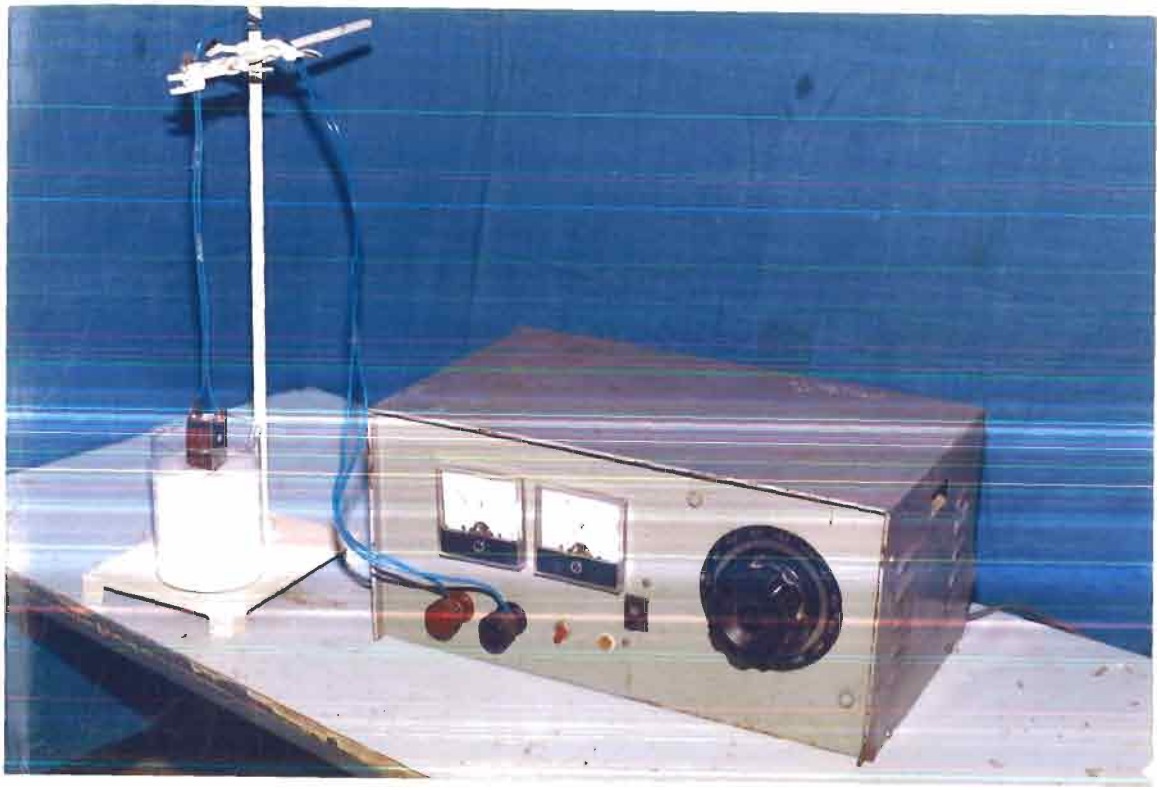


Plate. 3·2 Experimental set up for electroflocculation studies

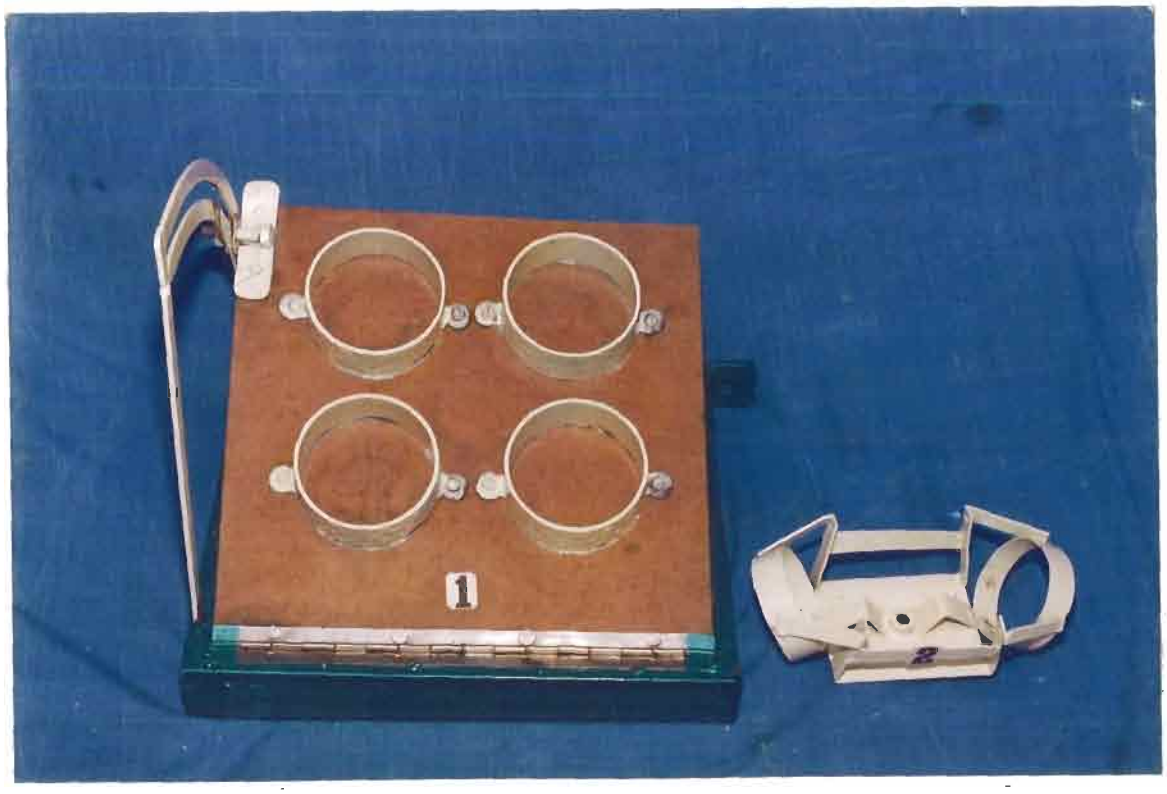


Plate. 3·3 1. Tilting top arrangement
2. Rotor for centrifugal settling

tie bars to the vertical arm of the rotor. The vertical arms are attached with a small beam of one centimeter width to further prevent sagging during high centrifugal force. The gadget was fixed to the centrifuge shaft through a slot provided in the centre of the plate. Suspension was taken in a plastic cylindrical container (6 cm diameter and 11 cm long) to which, at the top, a ring with nut and bolt is provided to hold it in the ring, which keep the cylinder in position even at high speed. The entire assembly was fixed to the centrifuge and operated at different speeds for different time intervals with starch suspension of different concentrations (Plate 3.4).

Experimental plan was as follows:

Speed	:	500, 1000 and 1500 rpm
Time of settling	:	15, 30, 45, 60, 90 and 180 seconds
Concentrations	:	2, 4, 6 and 8%

The experiments were also conducted with fresh starch to compare the effect of centrifugal force on the settling of it with dried starch.

3.6 Design of Hydrocyclone

Hydrocyclones are designed mainly based on the properties of the fluid and solid particles to be separated, *viz.*, density and size of the particles and density and viscosity of the fluid. Design procedure given by Gomez (1992[a] and 1992[b]) was applied for the calculation of the diameter of the experimental hydrocyclone. The other geometric proportions of the hydrocyclone were chosen according to the correlation given by Rietma (1961).

Based on Schwallbach(1988) graphical representation, Gomez (1992[a] and 1992[b]) developed a method for designing hydrocyclone. The design was based on d_{T50} particle size *i.e.*, one half of the particle will be found in the underflow and another half in the overflow or d_{T50} is defined as the particle diameter for which the hydrocyclone has 50 % efficiency of removal.

Particle with a diameter other than d_{T50} can be related to this size by means of either a correlation equation given by Bennet (1936) or from the monograph published by Zanker (1977).

Bennet (1936) gave the equation as

$$E = 100\left[1 - \exp\left(\frac{d}{d_{T50^*} - 0.115}\right)^3\right] \quad \text{.... 3.6}$$

where,

d = particle diameter, μm

d_{T50^*} = required separation mesh for suspension other than sand water suspension, μm.

E = the efficiency of the hydrocyclone in separating any particle of diameter d, %

Thus d_{T50^*} size for a given separation can be found out by the equation by knowing the size range of particles in the feed stream and by selecting the efficiency of cyclone. Schwallbach (1988) reported the performance data for hydrocyclone in terms of separation achieved for a suspension of sand in water. However, it can be applied to solid and liquid other than sand and water by means of the following correction equation (Gomez, 1992[a])

$$d_{T50}{}^* = d_{T50}{}^{\eta}\left[\frac{\rho_{sand} - \rho_w}{\rho_s - \rho_l}\right]^{0.5} \quad \text{.... 3.7.}$$

where,

η = Kinetic strength of solid, can be taken as unity

ρ_{sand} = density of sand particles (2.65 kg/l)

ρ_w = density of water (1 kg/l)

ρ_s = density of solid particles, kg/l

ρ_l = density of liquid containing the suspended solid, kg/l

After determining the d_{T50} particle size, the hydrocyclone can be sized using the following correlation as a function of d_{T50} (Gomez, 1992[a]).

$$D_{min} = \left[\frac{(3.111 + 0.2607 d_{T50})}{(1 + 0.003998 d_{T50} - 0.00001589 d_{T50}^{2})} \right]^{2} \quad \text{.... 3.8}$$

$$D_{avg} = \left[\frac{(2.327 + 0.4037 d_{T50})}{(1 + 0.008217 d_{T50} - 0.00004059 d_{T50}^{2})} \right]^{2} \quad \text{.... 3.9}$$

$$D_{max} = \left[\frac{(0.6924 + 0.7774 d_{T50})}{(1 + 0.02144 d_{T50} - 0.0001546 d_{T50}^{2})} \right]^{2} \quad \text{.... 3.10}$$

where,

D_{min}, D_{avg} and D_{max} are the minimum, average and maximum diameters of hydrocyclone, respectively.

3.6.1 Dimensions of hydrocyclones

Rietma (1961) correlations were used for fixing the other dimensions of the hydrocyclone. They are as follows :

$$\frac{L}{D} = 5,\ \frac{l}{D} = 0.4,\ \frac{D_i}{D} = 0.28,\ \frac{D_0}{D} = 0.34,\ D_u : D_0 = 1:4,\ L_1 : L_2 = 1:5$$

where,

L	=	Length of cyclone, cm
D	=	cyclone diameter, cm
l	=	length of vortex finder, cm
D_i	=	inlet diameter, cm
D_0	=	vortex finder (overflow) diameter, cm
D_u	=	Diameter of underflow orifice, cm
L_1,L_2	=	Length of cylinder and cone, respectively, cm

3.6.2 Feed pressure

Inlet feed pressure should be adequate to keep a centrifugal field inside the hydrocyclone and keep up for static pressure losses including friction losses and centrifugal head.

The allowable feed pressure related to the diameter of hydrocyclone was given as

$$P_{min} = 1.907 - 0.2126 \ln (D) \quad \text{.... 3.11}$$

$$P_{avg} = 3.068 - 0.3373 \ln (D) \quad \text{.... 3.12}$$

$$P_{max} = 5.330 + 0.0003104D - 0.6693 \ln(D) + \frac{20.88}{D} \quad \text{.... 3.13}$$

where P_{min}, P_{avg} and P_{max} are the minimum, average and maximum feed pressures, respectively.

3.6.3 Flow capacity of hydrocyclone

The minimum (V_{min}), average (V_{avg}) and maximum (V_{max}) flow capacities of the hydrocyclone as a function of the nominal diameter were given as follows :

$$V_{min} = 0.00330 \times D^{1.702} \quad \text{.... 3.14}$$

$$V_{avg} = 0.00516 \times D^{1.702} \quad \text{.... 3.15}$$

$$V_{max} = 0.00702 \times D^{1.702} \quad \text{.... 3.16}$$

Gomez (1992) method was intended for using within the diameter 25 - 1000 mm and solid concentration less than 250 g/l.

3.6.4 Calculation

Step I :

Required mesh separation for starch particles was calculated using Eq.(3.6)

By substituting E = 90% and d = 20 μm, we get d_{T50}^{*} = 15.26 μm

Step II

Related the required separation mesh for starch/water (d_{T50*}) with an equivalent separation mesh for sand /water (d_{T50}) using Eq (3.7). by substituting

ρ_{sand} = 2.65 kg/l, ρ_{water} = 0.993 kg/l, ρ_{solid} = 1.52 kg/l, ρ_{liquid} =1kg/l in equation ,

then d_{T50} = 8.567 μm

Step III : Calculation of diameter

Substituted d_{T50} = 8.567 μm in Eqs (3.8), (3.9) and (3.10) to give D_{min}, D_{avg} and D_{max} as 26.76, 29.38 and 39.33 mm, respectively.

The average diameter of hydrocyclone was found to be 29.40 mm ≈ 30 mm. The other geometric proportions were worked out according to Rietma (1961). Slightly bigger diameter (50 mm) was also chosen to see the effect of increase in diameter on hydrocyclone performance. The geometric proportions of hydrocyclone for 30 and 50mm diameter is given in Table 3.1 .

Table 3.1 Hydrocyclone dimensions based on Rietma's proportions

	Dimensions, mm	
Cyclone diameter	30	50
Cyclone length	150	250
Vortex finder length	12	20
Cylinder length	25	42
Cone length	125	208
Inlet diameter	8.4	14
Overflow diameter	10.2	17
Underflow diameter	2.6	4.3

Step IV : Calculation of feed pressure

The value of D was substituted as 30mm in Eqs. (3.11), (3.12) and (3.13) to give P_{min}=1.184 bar (1.20 kg/cm^2), P_{avg}=1.921 bar (1.95 kg/cm^2) and P_{max}=3.759 bar

Step V: Calculation of flow capacity

From Eqs.(3.14), (3.15) and (3.16), the V_{min}, V_{avg} and V_{max} were calculated as 1.078, 1.685 and 2.293 m^3/h, respectively.

3.7 Hydrocyclone

The experimental set up comprised of a feed pump (reciprocating type), hydrocyclone assembly, pressure gauges, hydrocyclone connecting pipe works, feed tank *etc.* and the same is illustrated in Fig.3.1 and Plate 3.5.

3.7.1 Feed pump

A reciprocating or positive displacement pump of 3 hp was used for pumping the feed suspension to the hydrocyclone. It was equipped with a by-pass valve by which the feed pressure at the hydrocyclone could be regulated to the desired level. The inlet and outlet of the reciprocating pump was connected with 12.5 mm rubber hoses. The inlet hose was fixed to a strainer at the bottom through which the suspension from the feed tank was drawn into the pump. The by-pass valve was adjusted to get required pressure reading in the gauge connected to the inlet pipe of the hydrocyclone and feed was introduced into the unit from the outlet of the pump.

3.7.2 Hydrocyclone connecting pipe assembly

The outlet hose of the pump was attached to a 12.5 mm GI pipe through a hose connector to which the inlet of the hydrocyclone was connected using suitable connections.

The diameter of the inlet of the hydrocyclone was fixed according to the size of the hydrocyclone (diameters), details of which are given in Table 3.1. A 12.5 mm gate valve was connected to the GI pipe to get a final adjustment of the pressure in the inlet line.

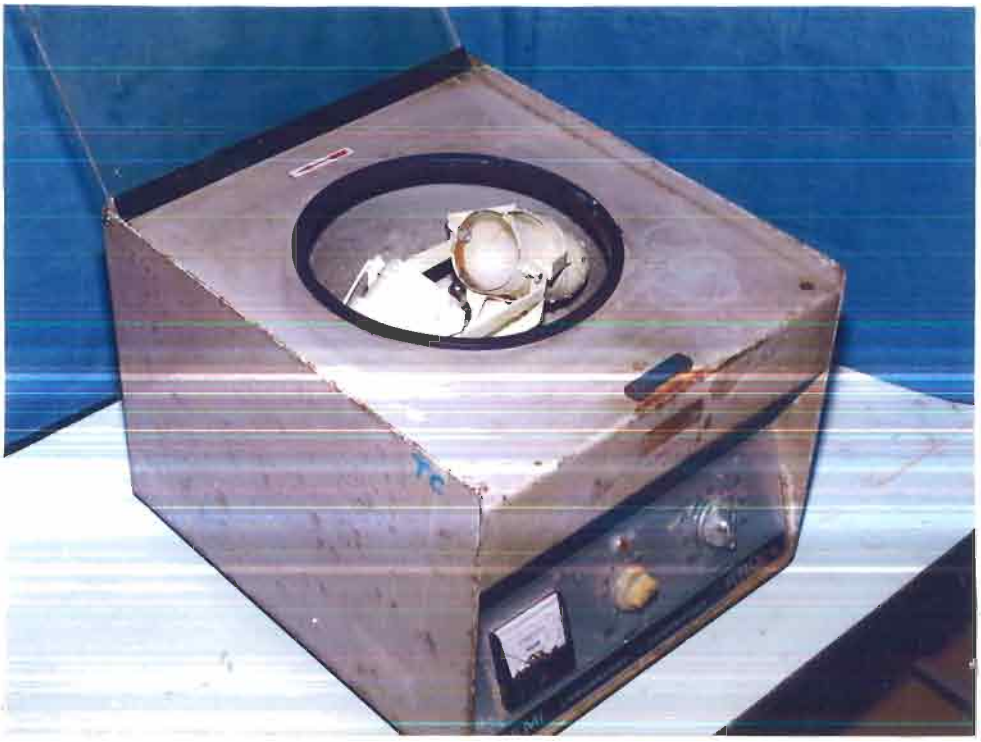


Plate. 3·4 Experimental set up for centrifugal settling

Plate. 3·5 Single hydro cyclone (30 mm dia meter)

The vortex finder was connected with a pipe line of same dimension using a bent to which a pressure gauge was fixed to record the pressure drop occurred during the course of separation..

3.7.3 Overflow and underflow product collection

Flexible hoses were attached to the vortex finder and underflow orifices to collect the samples for making measurements like flow rate and starch concentration. Volume and time method was used for measuring the flow rate from the underflow and overflow pipes. Samples were collected in buckets for every 60 seconds and quantity was measured in litre per second.

The starch concentration in the feed, overflow and underflow stream was measured by the oven method as described in section 3.1.1.3.

3.8 Testing of Hydrocyclone

Two hydrocyclones were tested with five concentration levels and by varying the inlet pressure at five levels. Different concentration of the starch suspensions were obtained by mixing dry starch in predetermined quantity of water in a tank. Trim and Marder (1995) reported that there was no appreciable difference in the performance of the hydrocyclone unit with suspensions made from dried and fresh cassava starch. Hence, dry starch reconstituted to different concentration levels were used in the present study. Hydrocyclone inlet feed pressure was regulated by adjusting the by-pass valve in the reciprocating pump and then the gate valve in pipe assembly. At each pressure and concentration, the hydrocyclone was allowed to operate continuously for about 15 minutes to attain steady flow conditions and then the samples were collected for analysis.

The details of the experimental design is as follows :

Diameter of hydrocyclones	:	30 and 50 mm
Concentration of starch suspension	:	2, 3.5, 5, 6.5 and 8% (w/v)
Feed inlet pressures	:	0.6, 1.2, 1.8, 2.4 and 3 kg/cm^2.

3.8.1 Effect of recycling of overflow stream

It is intended that a hydrocyclone would be installed to get an overflow stream with a starch concentration as minimum as possible so that it can be directed back to the starch extraction process for rasping operations where as the thickened or concentrated underflow stream is directed back to the settling tanks. Hence, tests were carried out to examine the effect of recycling the overflow stream on final starch concentration.

The overflow stream from the hydrocyclone was collected separately and recycled different times and in each recycling, the underflow and overflow concentration was noted.

3.8.2 Battery of hydrocyclones

It is expected from the principle of hydrocyclone that overflow stream contains finer particles than that in the underflow and smaller the particles, the smaller the hydrocyclones that must be used. If the overflow is used as the feed stream to the inlet of another hydrocyclone unit, size of the second unit should be minimum.

Test I

Tests were conducted by connecting a 50 and 30 mm hydrocyclones in series and underflow and overflow concentrations from each unit was measured along with the pressure drop.

Test II

A battery of hydrocyclones, 5 units of 30 mm diameter were connected in series as shown in Plate 3.6 such that the overflow from the preceeding unit will be the inflow (inlet) for the succeeding units. Pressure gauges were fixed on the inlet of each units to note the pressure drop occurred during operation. The concentration of the underflow from each unit and overflow of the last unit was noted. Also the overflow and underflow streams were collected separately and recycled different times and concentration of the resulting

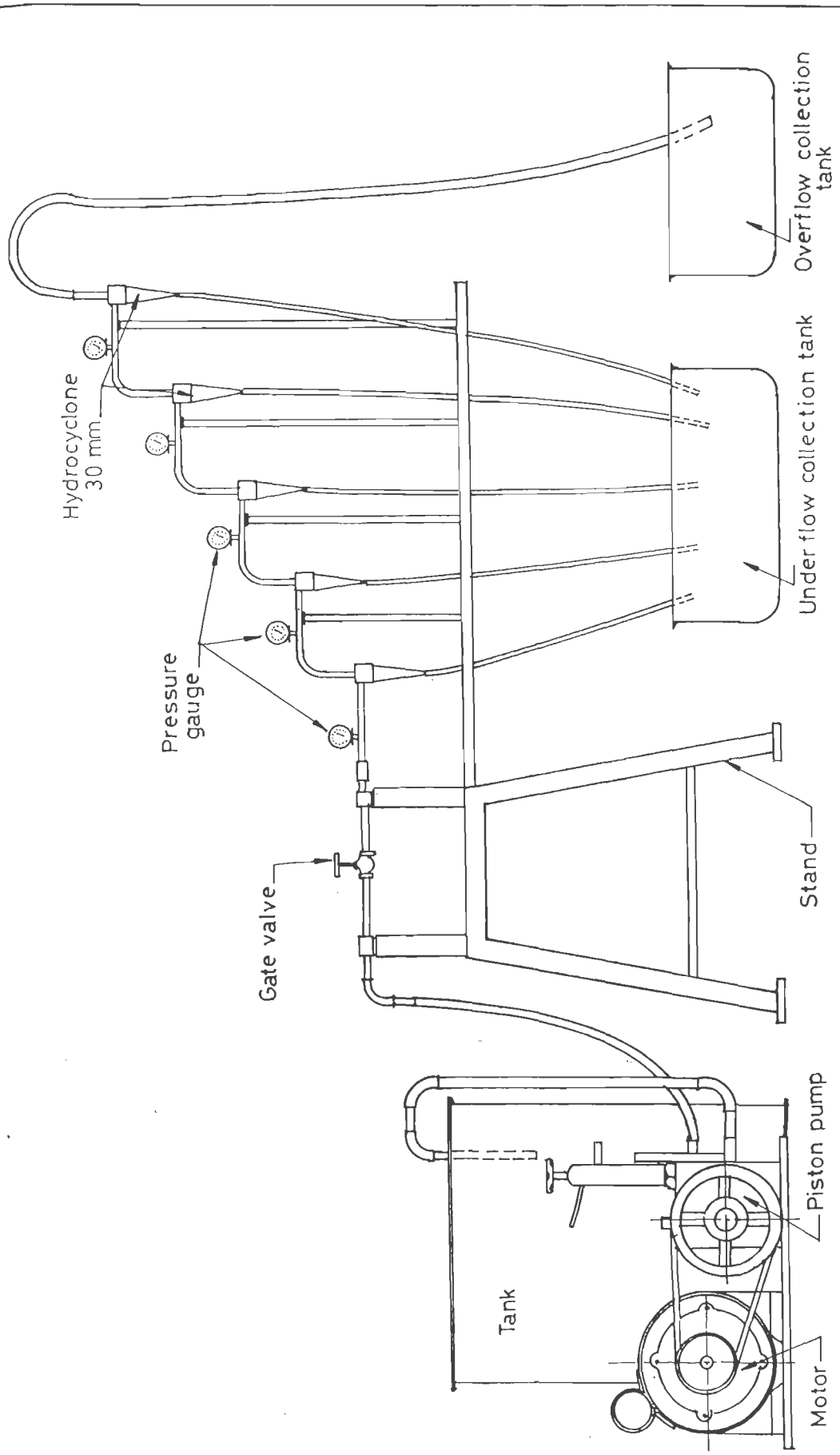


Fig. 3·1 Experimental set up of a battery of hydrocyclone

Plate. 3.6 Battery of hydro clones

Plate. 3.7 Modified battery of hydro cyclones

streams were noted to analyse the effect of recycling in battery system. Performance of the battery was not satisfactory due to the effect of back flow/back pressure and hence a modification was made which was against the design principle, *i.e.*, the inlet and overflow pipes were made equal (Plate3.7).

3.8.3 Performance evaluation of hydrocyclone

The performance of the hydrocyclone was evaluated by the method given by Van Esch (1991) and Trim and Marder (1995).

3.8.3.1 Total efficiency (E_T)

$$E_T = \frac{M_u}{M_s} \qquad \text{.... 3.17}$$

where,

M_u = Mass flow rate of solid material in underflow, kg/s

M_s = Mass flow rate of solid material in feed, kg/s

The above equation gives an efficiency equal to the underflow / supply ratio (R) even if no separation takes place in the hydrocyclone. Hence a better definition is one, where in case no separation takes place, an efficiency of zero is found, for this R should be subtracted from the efficiency and in order to bring the maximum efficiency back to 1, division by 1 - R is needed. Hence, a term Reduced Efficiency (E_R) is expressed as :

$$E_R = \frac{E_T - R}{1 - R} \qquad \text{.... 3.18}$$

where,

E_T = Total efficiency

R = Underflow / supply ratio

3.8.3.2 Underflow volume split (U_{VS})

It is the ratio by volume of the underflow stream to the feed stream.

$$U_{VS} = \frac{V_u}{V_f} \quad \text{.... 3.19}$$

where,

V_u = Volume of underflow, l/s

V_f = Feed rate, l/s

3.8.3.3 Underflow concentration

Increase in underflow concentration was calculated by the equation:

$$C = \frac{C_u - C_f}{C_f} \quad \text{.... 3.20}$$

where,

C_u = Underflow concentration, %

C_f = Feed starch concentration, %

3.8.3.4 Recycling efficiency (R_C)

Recycling experiments were performed to get an overflow stream with only very little quantity of starch which can be taken back for rasping operation and hence water consumption can be reduced. A term "Efficiency of recycling" was introduced here to see the effect of recycling on overflow concentration and was calculated as :

$$R_C = 1 - \frac{C_o}{C_f} \quad \text{.... 3.21}$$

where,

C_o = Overflow concentration, %

C_f = Feed concentration, %

RESULTS AND DISCUSSION

CHAPTER IV
RESULTS AND DISCUSSION

This chapter deals with the results of the different experiments carried out to study the kinetics of cassava starch settling in water. Effects of chemical addition, electroflocculation, inclined column and centrifugal force on settling and the thermal and rheological properties of modified starch are also presented and discussed. Performance evaluation of newly designed single and battery of hydrocyclones for concentration of starch suspension is discussed in detail in this section.

4.1 Kinetics of Starch Settling

Settling characteristics of cassava starch in water at various concentrations are discussed in this section.

4.1.1 Cassava starch settling process

The starch suspension was prepared with different solid concentrations and settling phenomena occurred during the process was visually observed. Different types of settling process are found to be encountered depending upon the concentration of the suspension. During a sedimentation operation, it is common to have more than one type of settling to occur at a given time, and it is possible to have all four types to occur simultaneously *viz.*, discrete, flocculant, hindered and compression settling (Metcalf and Eddy, 1993). The settling of starch suspension at low concentration (≤6%) was found to be entirely different from that of higher concentration (≥10%) as detailed below.

4.1.1.1 Starch suspension of low solid concentration (≤6%)

The starch suspension of low concentration was placed in a glass column and observed the settling phenomena with respect to time (Fig.4.1a). At the initial phase of settling, starch particles found to accumulate at the bottom. The concentration of the

suspension was found to be gradually increased with depth from the top of the column. At the start of the experiment, a line of demarkation between the settled starch and concentrated suspension was noticed, but there was no clearly defined interface. After five minutes, zones of varying concentrations but with different interfaces were visualized. The height of settled starch gradually increased and after 15 min, a well defined interface between the top layer of almost clear water and settled starch was formed, showing the initiation of zone or hindered settling. This observation clearly indicated that discrete or flocculent settling was more predominant at the initial phase of the settling process. The suspended particles in water had a natural tendency to agglomerate. Due to the coalescence, size of the agglomerates increased which in turn increased the settling velocity of the particle and reached to the bottom of the container. Concentration of the particles after a definite period, increased to such a level that more physical contact or interaction between the particles was more, giving rise to hindered or compression settling. Hence, clear interface was formed between the supernatant liquor and settled starch and it was continuously subsided until an equilibrium position was reached.

4.1.1.2 Starch suspension of high solid concentration (≥10%)

The mode of settling of starch particles in highly concentrated suspension was found to be entirely different from that of dilute suspensions (Fig.4.1b). The starch suspension was poured in to a cylindrical column and after a short time of 1-2 min, a zone of relatively clear water `A' was observed at the top of the column. Below this, the concentration was progressively increased and zone D of more concentrated starch began to accumulate at the bottom of the container. After five minutes, the layer B was found to be divided at C below which the concentration changed more towards D and above which more towards B. After 10 min, the zone B merged with the zone C and the interface subsided to C to show three different zones. As settling proceeds, the concentration continued to increase at the bottom and the interface between C and D raised and after

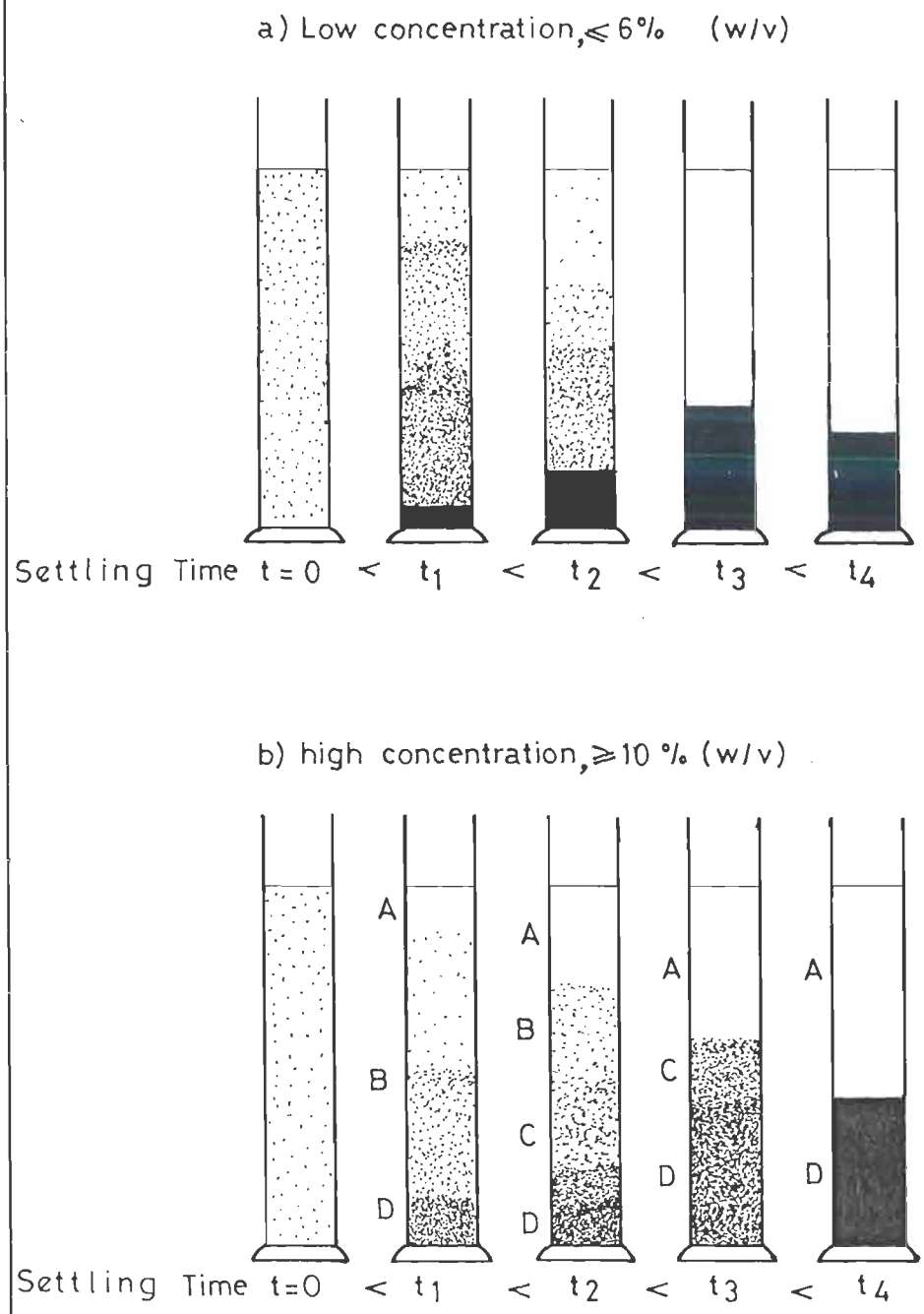


Fig. 4.1 Schematics of settling of cassava starch in water

20-25 min, coincided with the falling interface A to give only two zones A and D *i.e.*, clear water and settled starch. In the zone D, a layer of compressed particles was formed due to the weight of starch particles, settled just above it, causing the interface to decrease further till an equilibrium position attained, indicating the formation of more compacted (settled) starch.

In this case, discrete or flocculant settling was assumed to occur for a short duration at the early phase of settling, proved by the accumulation of starch at the bottom. But hindered and compression settling was more noticeable in the longer period of settling. At 8% concentration, neither sediment height nor interface could be noticed at the initial periods of settling and interface was found to be developed after 20-30 min only. Hence, settling at this concentration could not be grouped under any one of the above two groups.

4.1.2 Settling characteristics of cassava starch in water

4.1.2.1 Low starch concentration

Cassava starch suspension in water with different concentrations were poured in marked cylinders in quiescent conditions and the sediment (settled starch) heights were measured as a function of time and depicted in Fig.4.2 and data of the same are presented in Appendix A

From the Fig. 4.2a, it was observed that the sediment height was increased at the initial phase of settling and then decreased due to the compaction and attained almost constant values at latter stage of settling except at 2% concentration where it registered a progressive increase initially and remained almost constant afterwards. For 37 mm column diameter, the sediment height increased to 15 and 27 mm for 4 and 6% concentrations, respectively after 15 min, then it was decreased to 13.75 mm after 40 min for 4% concentration and 20.5 mm after 60 min for 6% concentration. This results showed that compaction had taken place only after 15 min of initial settling and as the concentration increased, time of settling also increased due to the decelerating effect of neighbouring particles on movement of the particles.

Starch suspension, being poly-disperse in nature contains particles of varying sizes, densities and shapes, which caused particle to travel with different settling velocities. As a result, larger and denser particles will move downward at higher velocities than smaller ones. The fast moving particles will collide with slow moving particles during the course of their settling and this collision resulted the coalescence to occur between the colliding particles and aggregates with increased size and settling velocity are formed. Also, this particle to particle interaction will result in transfer of momentum, thus affecting the velocity of the colliding particles (Grootscholtan and Dejong, 1985). The denser particles thus formed settled at slower rate than that of smaller ones due to loss of energy during the course of collision. So, the initial period of settling may be characterised by settling of more smaller ones than larger ones, with which, the weight may not be sufficient to form a compact mass at the bottom and hence height of settled starch increased at the initial stage. As more and more heavier particles settled, more consolidated starch sediment was formed. This was confirmed by noting the particle size distribution of the starch granules settled at the bottom of the settling column at different time intervals. The average particle size was found to be 11.45, 11.35, 11.15, 14.9 and 16.8 μm after 15, 30, 45, 60 and 90 min. of settling, respectively. During consolidation, water present at the interstices was displaced to the top and more closely packed sediment was formed.

On the final stage of settling, zone or compression settling may be predominant owing to the higher concentration of the suspension at deeper zones. Starch particle being cohesive in nature, at higher concentrations, they settle as a mass with distinct interface between settled starch and clear water. This greater interaction between the downward moving particles and upward raising displaced water caused a retarding effect on the settling velocity.

The experiments were repeated to verify the above pattern of settling using different column diameter (22 and 28 mm) and found to follow the same pattern confirming the above findings (Fig. 4.2b and 4.2c).

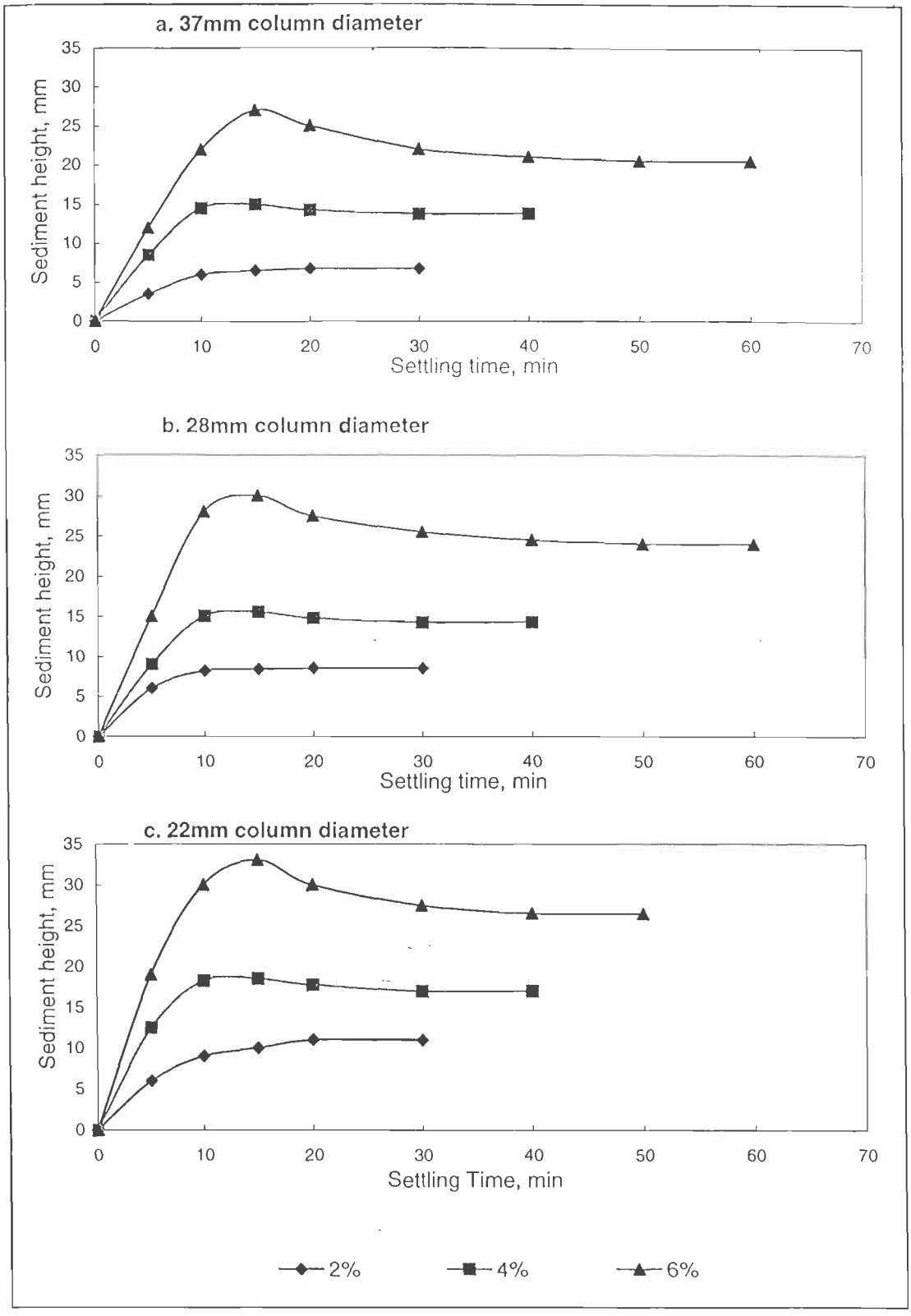


Fig.4.2 Settling characteristics of cassava starch in low concentrated suspensions

4.1.2.2 High starch concentration

The settling characteristics are studied by timing the position of the interface between clarified water and zone settling regions, as against the sediment height in low concentrated suspension and are represented in Figs. 4.3 to 4.5 and Appendix B and C for 37 and 49 mm diameter columns. In all the cases, it was found that during the initial phase of settling, interface subsided quite rapidly and thereafter, the rate of decrease was gradually reduced until it become almost parallel to time axis. For 37 mm diameter settling column, the interface between the settling particles and clear water rapidly subsided from 100 mm to 23.5, 33.0, 40.5 and 58.5 mm after 15 min; from 200 mm to 46.5, 56.0, 56.0 and 93 mm after 30 min; from 300 mm to 61.25, 70.5, 99.5 and 152 mm after 40 min, for 10, 12, 14 and 16% starch concentrations, respectively. Time needed for the completion of settling (indicated by the concordant readings) was found to be increased with height of column and concentration. Settling process was completed at about 60, 70, 80 and 90 min after accumulating a sediment height of 17.75, 22, 25, 28 mm for 10, 12, 14 and 16% concentrations, respectively in 37 mm column diameter with 100 mm height of suspension. For 200 mm height of suspension, the starch suspension took about 100, 100, 150 and 170 min with a sediment height of 33, 37.5, 44 and 49 mm for 10, 12, 14 and 16% starch concentrations, respectively. Similarly, for 300 mm column height, the final settled starch occupied a height of 46.5, 51, 61 and 68.5 mm after 120, 140, 160 and 180 min for 10,12, 14 and 16% concentrations, respectively. Similar trends were also observed with 49 mm diameter column as shown in Fig.4.4b and 4.5a and b. Settling process was found to be completed in 60, 70, 80 and 100 min for 100 mm height of suspension; 100,120,140 and 160 min for 200 mm height and 110,140, 150 and 200 min for 300 mm height for 10, 12, 14 and 16% concentrations, respectively.

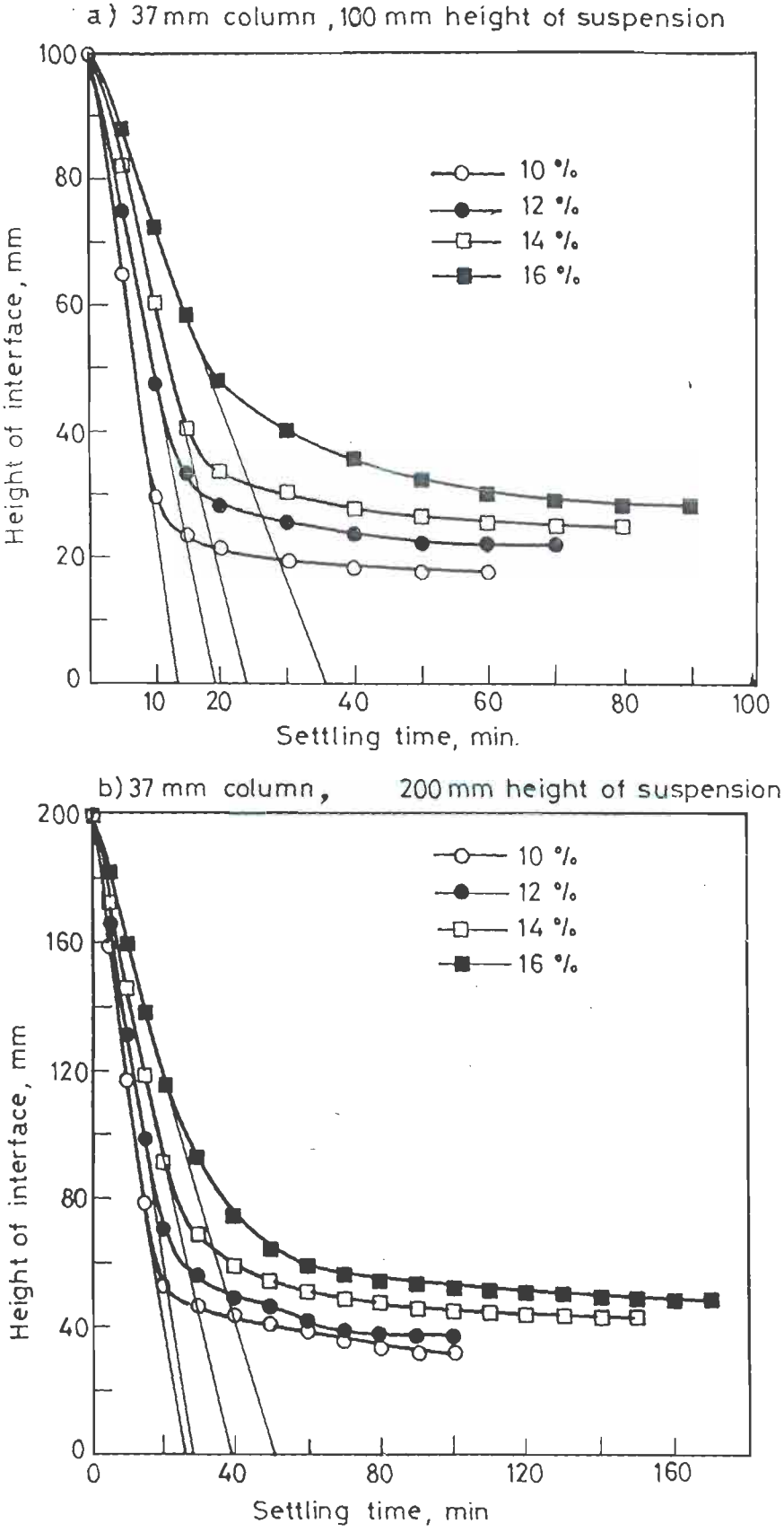


Fig. 4·3 Effect of high concentrated suspension on settling of cassava starch

Settling characteristics curves for zone settling suspension were studied by many authors (White, 1978; Coulson and Richardson, 1980; Metcalf and Eddy, 1993 and Droste, 1997). According to them, in the initial phase of settling, suspension settles at a velocity which is characteristic of the initial solid concentration. As the solids accumulates at the bottom of the container, a zone with higher solid concentration is developed. When this zone meets the sharply defined interface, the rate of settling of interface decreased. As more and more starch settle at the bottom, causing to increase the concentration, a compression zone is developed at the bottom. The particles in this region apparently form a structure in which the particle will be in physical contact with each other. The weight of the particle is partly supported by the lower layer of particle leading to a progressively greater compression with depth leading to compaction of settled starch. The interface will continue to subside in compression until an equilibrium position is reached.

The settling curve at the final phase declined very little, showing the final consolidation of the sediment. It is the slowest part of the process as the displaced fluid has to flow through the small pore space between the particles. As consolidation starts, the rate falls off because the resistance to the flow of water through the starch progressively increases. Porosity of the sediment is less at the bottom most layer because of the self weight of particles (Coulson and Richardson, 1980). Similar trend was observed in the present study also.

4.1.2.3 Rate of fall of interface

The data on the rate of fall of interface is presented in Tables 4.1 and 4.2. It was observed that for the same column height, as concentration of the suspension increased, the rate of fall decreased; and with height, an increase in rate of fall was observed. As settling progressed, the rate was decreased as expected. However, during the initial stage, an increasing trend was observed, which may be due to the stirring of suspension done to avoid

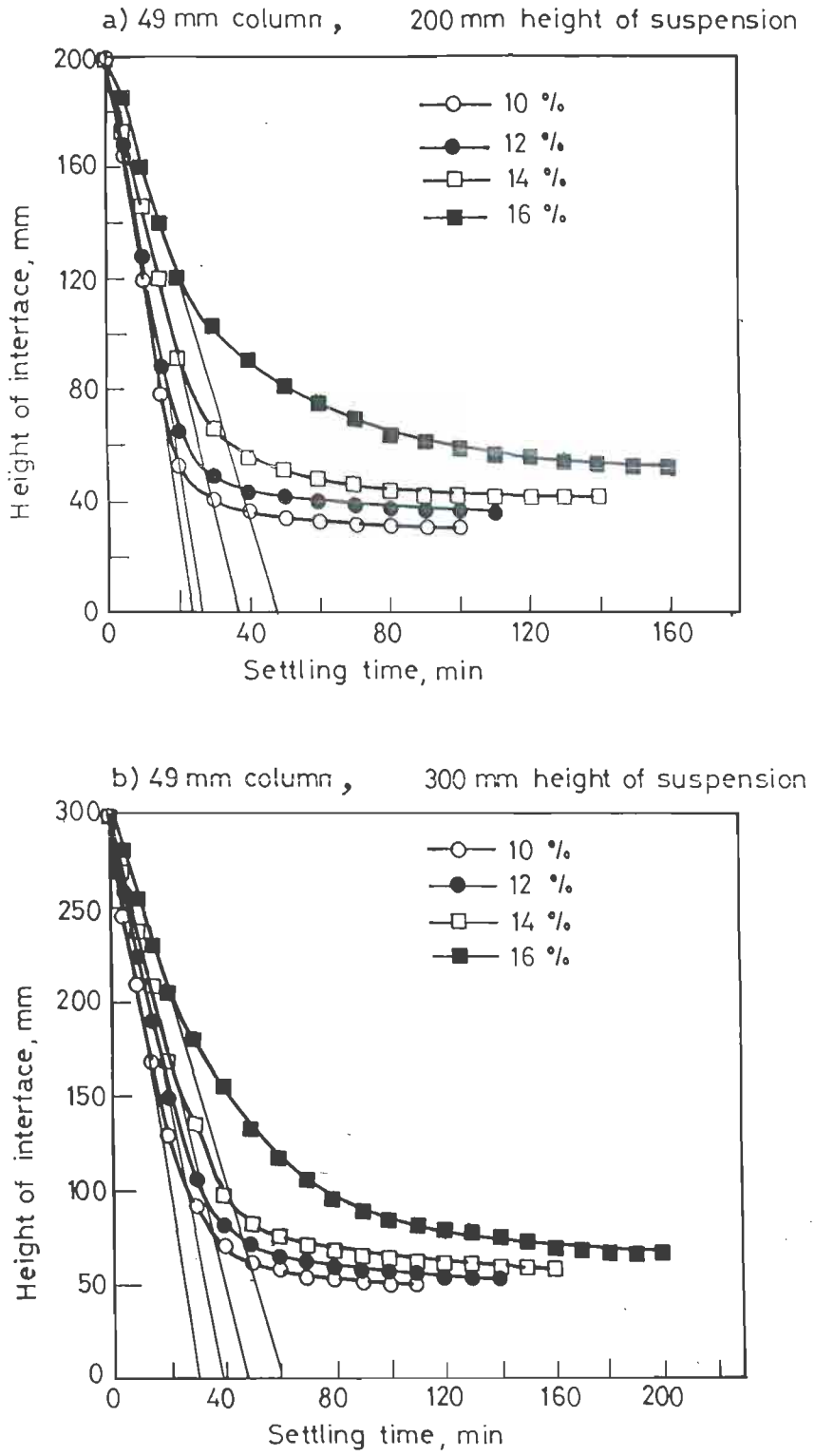


Fig. 4.4 Effect of high concentrated suspension on settling of cassava starch

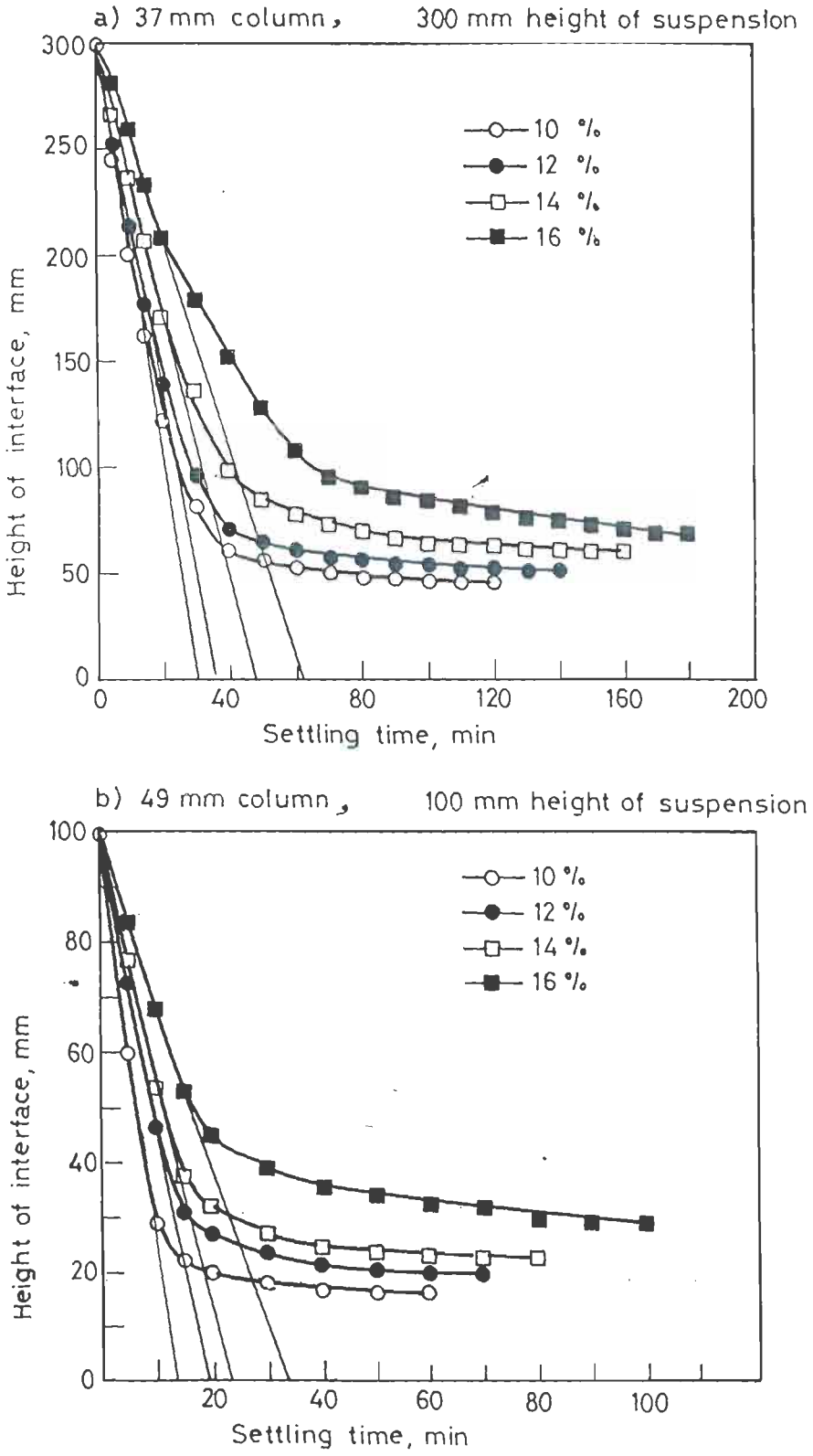


Fig. 4.5 Effect of high concentrated suspension on settling of cassava starch

Table 4.1 Rate of fall of interface during settling of cassava starch in 37 mm diameter column

Time, min	Rate of fall of interface, mm/min											
	Concentration, %											
	10			12			14			16		
	Height of suspension, mm			Height of suspension, mm			Height of suspension, mm			Height of suspension, mm		
	100	200	300	100	200	300	100	200	300	100	200	300
5	7.02	8.10	10.90	5.00	6.70	9.60	3.50	5.40	6.30	2.40	3.60	3.40
10	7.88	8.40	8.90	5.50	7.10	7.60	4.40	5.20	6.00	3.10	4.40	4.60
15	1.20	7.60	7.70	2.90	6.40	6.10	4.00	5.60	6.10	2.80	4.40	5.20
20	0.45	5.30	8.00	1.00	5.60	7.60	1.40	5.40	7.20	2.10	4.60	5.20
30	0.20	0.65	4.05	0.25	1.50	4.30	0.33	2.25	3.55	1.80	2.20	2.90
40	0.10	0.25	2.08	0.20	0.65	2.55	0.25	1.04	3.70	0.45	1.80	2.70
50	0.05	0.28	0.48	0.13	0.35	0.60	0.13	0.43	1.45	0.35	1.10	2.40
60	0.00	0.20	0.33	0.03	0.45	0.40	0.10	0.33	0.65	0.20	0.45	2.00
70		0.33	0.25	0.00	0.28	0.25	0.05	0.23	0.48	0.10	0.30	1.30
80		0.2	0.20		0.10	0.20	0.00	0.15	0.30	0.10	0.20	0.50
90		0.10	0.08		0.03	0.15		0.15	0.33	0.00	0.10	0.35
100		0.00	0.10		0.00	0.10		0.08	0.23		0.05	0.20
110			0.05			0.15		0.05	0.08		0.05	0.30
120			0.00			0.05		0.05	0.10		0.05	0.30
130						0.05		0.03	0.10		0.10	0.30
140						0.00		0.03	0.05		0.05	0.10
150								0.00	0.05		0.05	0.15
160											0.05	0.25
170											0.00	0.20
180												0.00

the wall effects and immediate settling which caused to drop the interface rapidly at the initial periods and this effect was continued only for a short period. The slow rate of fall at the latter stage of settling showed the compaction of settled starch nearer to equilibrium position. The decelerating effect of concentration on rate of fall of interface was also observed by many researchers who dealt with mono, bi or poly dispersed suspension of varying sizes and densities (Richardson and Shabi, 1960; Richardson and Jeronino, 1979; Coulson and Richardson, 1980; Grootscholtan and Dejong, 1985; Metcalf and Eddy, 1993; Tiller and Hsyung, 1993 and Droste, 1997). Higher concentration produced greater interaction between particles due to greater cohesive force between them and thereby restrict the motion of the particles relative to each other. Also, at higher concentrations, the upward velocity of displaced fluid was greater which hindered the downward movement of interface and this resulted in lower rate of fall of interface (Coulson and Richardson, 1980).

4.1.2.4 Settling of fresh starch

4.1.2.4.1 Determination of concentration of fresh starch suspensions

Fresh starch suspension was prepared as explained in Section 3.1.1.2. Though the required concentration was adjusted by noting the hydrometer reading, actual concentration was found to be slightly different. Hence hydrometer was used to get an approximate estimation and the actual concentration of the suspension used for all the experiments was determined by the oven method procedure and reported.

Starch suspensions of varying concentrations were prepared and hydrometer reading for each case was recorded and the data obtained were analysed to get the line of best fit and it yielded model $Y = 0.99 + 0.0004\ X$, where X = concentration, % and Y = hydrometer readings. The correlation coefficient was found to be 0.99. Only slight difference was noticed between observed and predicted values using this equation.

Table 4.2 Rate of fall of interface during settling of cassava starch in 49 mm diameter column

Time, min	Rate of fall of interface, mm/min											
	Concentration, %											
	10			12			14			16		
	Height of suspension, mm			Height of suspension, mm			Height of suspension, mm			Height of suspension, mm		
	100	200	300	100	200	300	100	200	300	100	200	300
5	8.00	7.10	10.80	5.40	6.40	8.20	4.50	5.30	6.10	3.20	3.00	3.6
10	6.25	8.80	7.10	5.30	8.00	7.00	4.70	5.20	6.0	3.20	5.00	5.2
15	1.30	8.40	8.50	3.05	7.90	6.80	3.25	5.35	5.9	3.0	4.00	5.20
20	0.45	5.10	7.60	0.80	4.70	8.20	1.15	5.80	8.40	1.60	4.00	5.0
30	0.23	1.20	3.70	0.38	1.60	4.40	0.48	2.55	3.30	0.60	1.80	2.50
40	0.10	0.45	2.15	0.23	0.60	2.45	0.25	1.03	3.70	0.35	1.10	2.50
50	0.06	0.20	0.90	0.10	0.15	1.00	0.10	0.50	1.58	0.15	1.00	2.25
60	0.00	0.18	0.30	0.06	0.20	0.70	0.80	0.33	0.68	0.15	0.60	1.50
70		0.08	0.38	0.00	0.13	0.15	0.02	0.20	0.43	0.10	0.60	1.25
80		0.08	0.23		0.13	0.40	0.00	0.18	0.33	0.20	0.55	1.00
90		0.03	0.18		0.08	0.15		0.03	0.28	0.05	0.25	0.65
100		0.00	0.13		0.03	0.10		0.10	0.18	0.00	0.25	0.40
110			0.00		0.00	0.15		0.10	0.08		0.25	0.40
120						0.05		0.05	0.10		0.05	0.20
130						0.05		0.03	0.10		0.10	0.20
140						0.00		0.00	0.15		0.10	0.20
150									0.00		0.00	0.25
160												0.30
170												0.10
180												0.10
190												0.05
200												0.00

4.1.2.4.2 Low starch concentration

Experiments with fresh starch suspension kept in 37 mm diameter column with 200 mm height of suspension also followed the same trend as dried starch, but more shifted at higher concentrations. At 4.04 and 6.45%, the sediment height was progressively increased initially and remained almost constant, but at higher concentration of 7.92 and 9.1%, it was increased initially and then decreased gradually to constant values (Fig. 4.6a). Also, it took longer time to get a compacted mass. At 4.04 and 6.45%, raw starch was settled in about 90 and 110 min whereas dried starch took only 40 and 60 min at 4 and 6% concentrations, respectively. At 7.92 and 9.10%, settling time for fresh starch was found to be 120 and 140 min, respectively.

4.1.2.4.3 High concentration

The subsidence of interface with respect to time was noted in the suspension kept in 37 mm diameter column with 200mm height of suspension and the observations are represented in Fig.4.6b and Appendix D. Like dried starch, the interface subsided quite rapidly at the initial phase, though the time for the termination of this rapid rate was different for each concentration. Interface fell rapidly from 200 to 57, 60, 65 and 84.5 mm for 13.43, 15.85, 16.97 and 20.01% concentrations, respectively at about 80 min and then followed a gradual decreasing trend. The time needed for the completion of settling was 180, 190, 210 and 230 min for 13.43, 15.85, 16.97 and 20.01%, respectively which were higher than that of dried starch and the height of the settled starch was found to be about 44.5, 47.5, 50 and 56 mm for the above concentrations, respectively. The above observations revealed that as concentration increased, the interface subside very slowly indicating the low rate of fall due to the interaction effect between the particles.

The higher settling time for fresh starch may be explained on the basis of hydration effect as described by Gregory (1993) for suspended solids in aqueous suspensions. All particles in aqueous suspension carry a surface charge due to ionization of surface

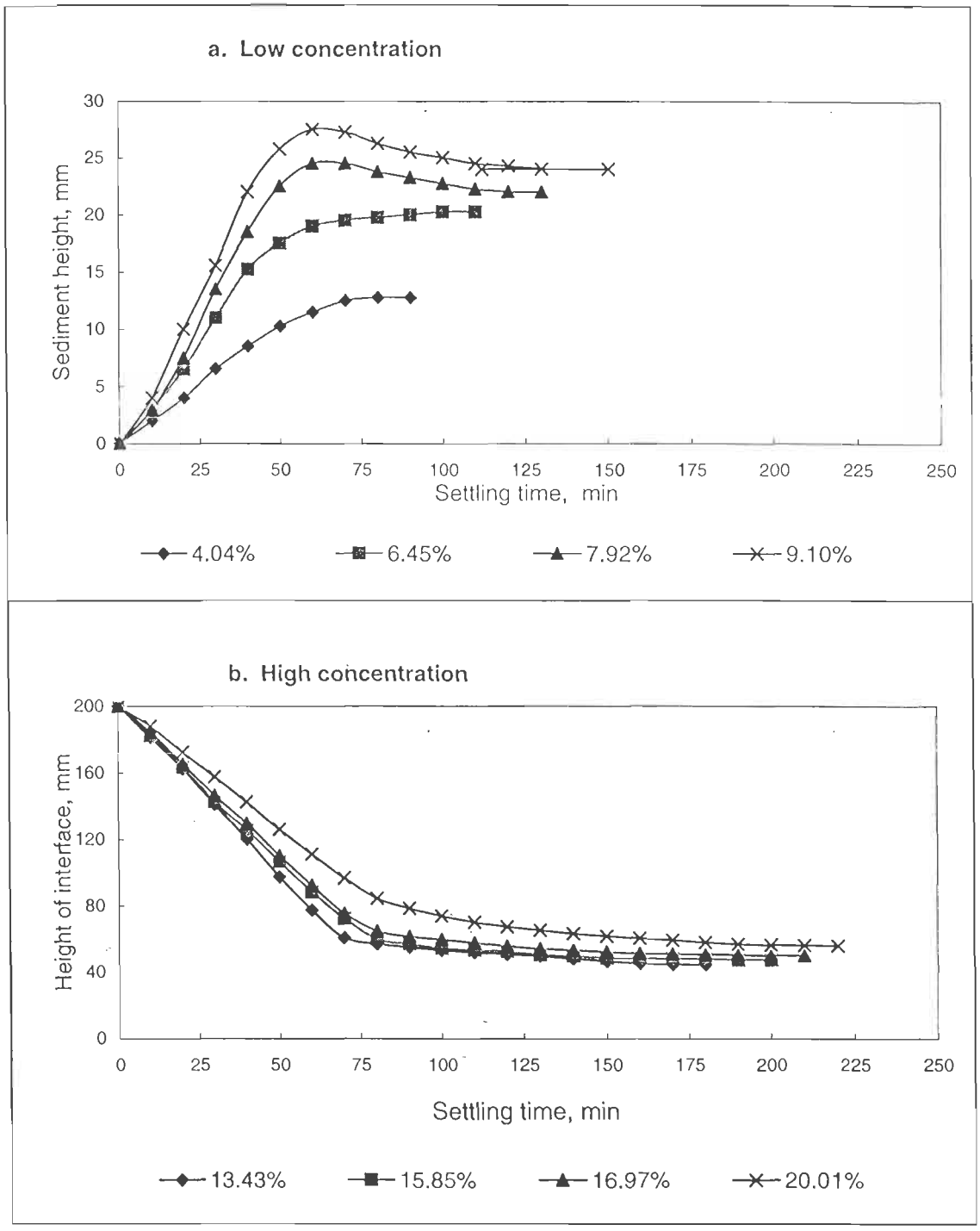


Fig.4.6 Settling charactreristics of fresh cassava starch in low and high concentrated suspensions

groups or specific adsorption of ions and some hydration of these groups would be expected, by analogy with ions in solution. Particles of biological origin have various types of hydrophilic material at their surface having quite large amount of bound water which play a part in the interaction of such particles. The approach of two particles with hydrated surfaces will generally be hindered by an extra repulsive interaction. This hydration repulsion arises essentially from fresh starch has to be dehydrated if true contact between them is to occur. According to this theory, dried starch though it is suspended in water, the hydration repulsion may not be to that extent of fresh starch. Fresh starch which is already be in hydrated stage (wet) before being mixed or suspended in water and hence experiences more hydration repulsion during settling than dried starch.

4.1.3 Settling velocity

Stoke's law was applied to calculate the free falling velocity (v_o) of the starch particle in water in isolation. The average particle size (d) and density of particle (ρ_s) were measured as 20 μm and 1520 kg/m^3, respectively. Properties of water in atmospheric temperature was adopted from Brown and Marcer (1958) as density (ρ_w)=993 kg/m^3 and viscosity (η_w) = 6.82 x 10^{-4} Pa.s. These values were substituted in Stoke's equation (Eq.3.1) and yielded a value for v_o as 0.169 mm/s.

Settling velocity of particles in suspension was different from that of particle in isolation. It was measured as the slope of the initial linear segment of the settling curve obtained by drawing height of interface versus time (Figs 4.3 to 4.5). The values of settling velocity are represented in Table.4.3. The concentration of starch particle (w/v) in suspension was converted into volume basis (v/v) so as to represent the settling velocity as a function of fluid (water) volumetric concentration or suspension voidage (ε).

From the table, it is clear that the settling velocity of particles in suspension (v_c) is less than that of the particle in isolation (v_o). v_o was calculated as 0.169 mm/s whereas

v_c was 0.122, 0.086, 0.069 and 0.047 for 10, 12, 14 and 16 % concentrations, respectively in 37 mm diameter column with 100 mm suspension height. v_c was found to be decreased to 0.066 and 0.081 mm/s for 200 and 300 mm height of suspension at 16% concentration.

Table 4.3 Settling velocity of starch particles in high concentrated suspensions

Solid concentration		Fluid volumetric concentration, %	Settling velocity, mm/s					
			Column diameter, 37 mm			Column diameter, 49 mm		
			Height of suspension, mm			Height of suspension, mm		
w/v, %	v/v, %		100	200	300	100	200	300
10	6.58	93.42	0.122	0.135	0.164	0.124	0.139	0.163
12	7.89	92.11	0.086	0.121	0.142	0.088	0.125	0.129
14	9.21	90.79	0.069	0.087	0.105	0.072	0.091	0.106
16	10.53	89.47	0.047	0.066	0.081	0.049	0.070	0.082

Similar retarding effect on sedimentation was also observed for 49 mm diameter column where v_c reduced to 0.049, 0.070, 0.082 mm/s when the concentration of the suspension was increased to 16% in 100, 200 and 300 mm column heights. When compared to v_o, the values of v_c were reduced by 27.81, 49.11, 59.17 and 72.19% for 10, 12, 14 and 16% in 37 mm column diameter with 100 mm height. Similarly for 49 mm diameter column with 100 mm height, 26.63, 47.93, 57.40 and 71.01% reduction was observed for v_c. For 200 mm column height, v_c was dropped by 60.95 and 58.58 % for 37 and 49 mm column diameter at 16 % concentration and these values were 52.07 and 51.48 % for 300 mm height of suspension for the above mentioned conditions. This clearly indicates that as concentration of the suspension was increased, the settling velocity of particle was reduced significantly when compared to free falling velocity. The results further revealed that column diameter had no effect on v_c whereas increase in column height increased the percent reduction in v_c.

This reduced settling velocity of starch particles in suspension is attributed by the greater interaction between the cohesive particles and the upward movement of the displaced fluid. During settling, the downward movement of the particles caused an equal volumetric flow rate of displaced water relative to which the particles must move. Also, for a given relative velocity, shear stresses will be greater in a concentrated suspension. As concentration of the suspension is increased, there is a greater interaction between the particles and the cohesive force between the particles are sufficient to restrict the movement of the particle relative to each other which caused to reduce the settling velocity (Mirza and Richardson, 1979; Richardson and Jeronino, 1979; Metcalf and Eddy, 1993 and Droste, 1997).

4.1.3.1 Effect of concentration

When the concentration of the suspension was increased, v_c registered a significant fall *i.e.*, for increase in concentration from 10 to 16% resulted 61.48, 51.11, 50.61% reduction in v_c for 100, 200 and 300 mm height, respectively in 37 mm column diameter. Similarly for 49 mm column, v_c was reduced by 60.48, 49.64 and 49.69% for 100, 200 and 300 mm height, respectively for the above said conditions. The significant reduction in v_c with respect to concentration is due to the interaction effect between the particles and upward flow of displaced fluid. These results are also in confirmation with the lower rate of interface subsidence at higher concentrations. The results revealed that the column diameter has no effect and hence v_c recorded more or less same values for 37 and 49 mm column diameters.

4.1.3.2 Effect of height

As the height of suspension increased, settling velocity was also found to be increased. For 37 mm column diameter, v_c was increased by 34.43, 65.12, 52.17 and 72.34% for 10, 12, 14 and 16% concentrations, respectively when the height was increased

from 100 to 300 mm. But for 49 mm column, the corresponding values were 31.45, 46.59, 47.22 and 67.35%. This trend is contradictory to the results of Coulson and Richardson (1980) who reported for 3% calcium carbonate suspension, height of suspension did not affect the settling velocity. This may be due to the differences in the concentration and origin of the particles. As starch particles are biological in origin and are of different sizes whereas calcium carbonate used was of same size (sieved material)

4.1.3.3 Effect of column diameter

Diameter of column did not have much influence on V_c compared to the effects of concentration and height of suspension. When the diameter was increased from 37 to 49 mm, V_c was changed by 1.64, 2.33, 4.35 and 4.26% for 100 mm height, 2.96, 3.31, 4.60 and 6.06% for 200 mm height and 0.61, 0.92, 0.95 and 1.23% for 300 mm height for 10, 12, 14 and 16% concentrations, respectively. This is in agreement with the general observation that, if the ratio of the diameter of the settling column to the diameter of particle is greater than 100, the wall of the container had no effect on sedimentation rate (Coulson and Richardson, 1980). In the present study, this ratio is of the order of several thousands and hence produced no wall effect and bridging of the suspension.

4.1.3.4 Statistical Analysis

Analysis of variance for settling velocity is presented in Table 4.4. It revealed that concentration and height of suspension have significant effect on settling velocity ($P \leq 0.01$) and column diameter had no significant effect. Concentration x height interaction was significant at 5 % level and other interactions were not significant.

Table 4.4 ANOVA Table for settling velocity of starch particles in water

SV	DF	SS	MS	F
Treatment	23	189.66	8.25	71.46**
Concentration (C)	3	143.12	47.71	413.41**
Height (H)	2	42.92	21.46	185.95**
Diameter (D)	1	0.12	0.12	1.02 ns
C x H	6	2.31	0.38	3.33*
C x D	3	0.15	0.05	<1
H x D	2	0.63	0.315	2.73 ns
C x H x D	6	0.42	0.07	< 1
Error	24	2.77	0.115	
Total	47	192.43		

** Significant at 1 % level, * significant at 5 % level, ns-not significant.

4.1.4 Modelling of cassava starch settling

Generally settling velocity equations were expressed by power regression equation relating free falling velocity as $V_c / V_o = \varepsilon^n$ (Richardson and Zaki,1954) where, the value of constant `n' depends on Reynold's number and ratio of particle diameter to column diameter. The value of `n' was calculated for mono, bi or poly dispersed suspension of non biological origin by taking particles of definite size and shape. But starch granules being bio origin, its suspension being poly dispersion in nature which prevented the use of above equations as such in the present study. Also statistical analysis revealed that apart from concentration, height of suspension also influenced the settling velocity. Hence, an attempt was made to model the settling velocity (V_c) in terms of free falling velocity (V_o), ratio of height of suspension to column diameter (H/D) and fluid volumetric concentration (ε).

The regression equation for different column diameters and height of suspensions followed the form $v_c = A.\ \varepsilon^{B/\varepsilon}$ where, A and B are constants and ε - fluid volumetric concentration or voidage, decimal (Fig. 4.7). The values for the constants in the equations for different column diameter and height of suspension are given in Table 4.5.

Table 4.5 Values of A and B in the settling velocity equations

Height of suspension, mm	Column diameter, 37 mm			Column diameter, 49 mm		
	A	B	r^2	A	B	r^2
100	0.45	17.95	0.99	0.43	17.39	0.99
200	0.41	14.46	0.97	0.40	13.87	0.97
300	0.47	14.11	0.99	0.42	13.15	0.99

Note: A and B are constants in the equation, r^2 = correlation coefficient

A. Equations for 37 mm column diameter

For 100 mm height:

$v_c = 0.45\varepsilon^{\frac{17.95}{\varepsilon}}$, by substituting Vo=0.169, H/D=2.70, the equation becomes

$$v_c = 2.66 v_o \varepsilon^{(6.65\frac{H}{D})/\varepsilon} \quad \text{....4.1}$$

For 200 mm height:

$v_c = 0.41\varepsilon^{14.46/\varepsilon}$, by substituting H/D=5.41

$$v_c = 2.43 v_o \varepsilon^{(2.67\frac{H}{D})/\varepsilon} \quad \text{....4.2}$$

For 300 mm height

$v_c = 0.47\varepsilon^{14.11/\varepsilon}$, by substituting H/D=8.11

$$= 2.78 v_o \varepsilon^{(1.74\frac{H}{D})/\varepsilon} \quad \text{....4.3}$$

B. Equations for 49 mm column diameter

100 mm height:

$v_c = 0.43\varepsilon^{17.39/\varepsilon}$, by substituting H/D=2.04

$$= 2.54 v_o \varepsilon^{(8.52\frac{H}{D})/\varepsilon} \quad \text{....4.4}$$

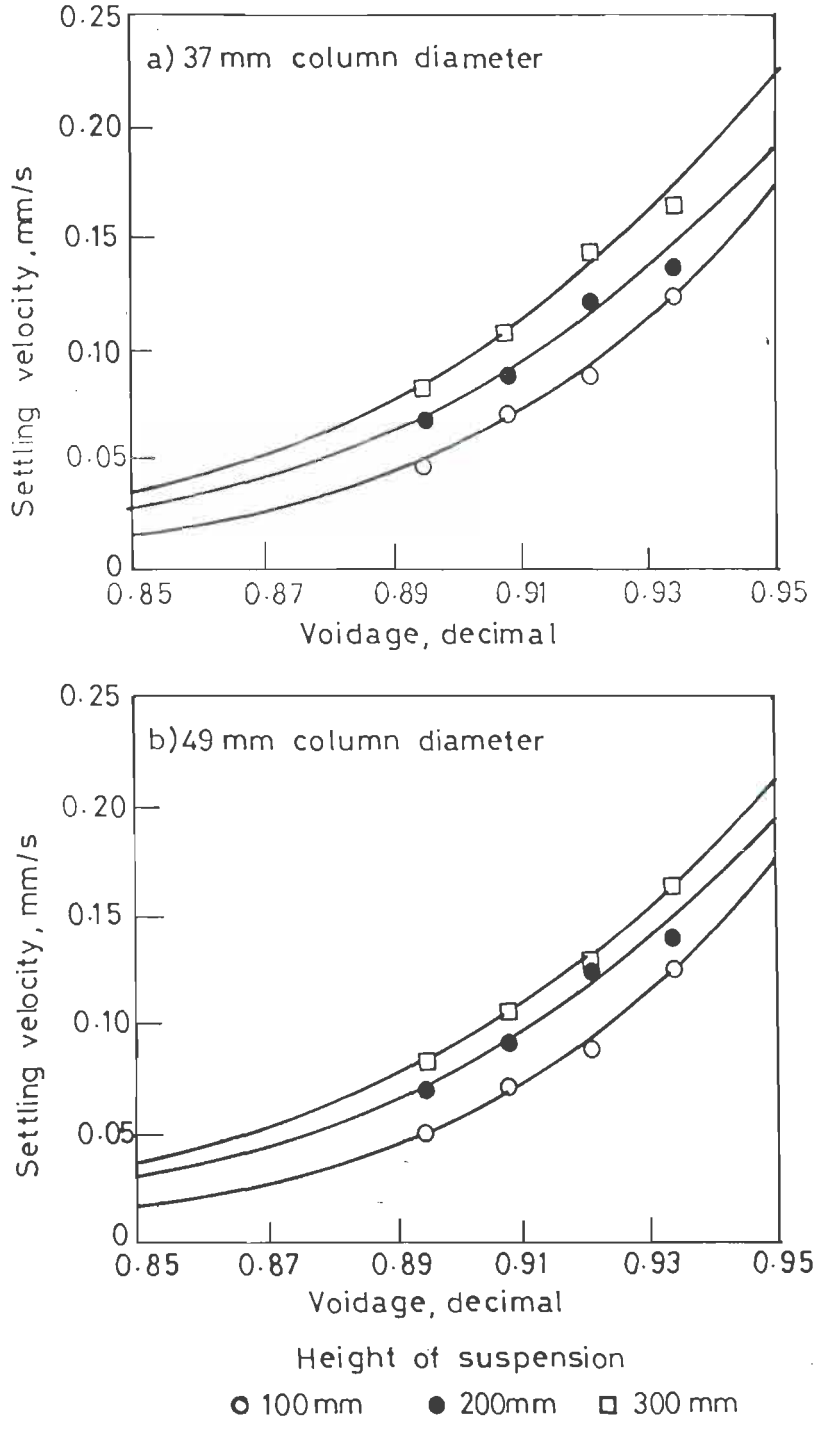


Fig. 4·7 Cassava starch settling velocity pattern in high fluid volumetric concentration (Voidage)

For 200 mm height

$$\nu_c = 0.40\varepsilon^{13.87/\varepsilon}, \text{ by substituting H/D=4.08}$$

$$= 2.37\nu_o\varepsilon^{(3.40\frac{H}{D})/\varepsilon} \quad \ldots.4.5$$

For 300 mm height

$$\nu_c = 0.42\varepsilon^{13.15/\varepsilon}, \text{ by substituting H/D=6.12}$$

$$= 2.49\nu_o\varepsilon^{(2.14\frac{H}{D})/\varepsilon} \quad \ldots.4.6$$

The values of A does not show any regular trend and on analysis, a polynomial equation of the form $A = 3.06 - 0.28\frac{H}{D} + 0.03\left(\frac{H}{D}\right)^2$ (Eq. 4.7) was found to be the best fit equation for the values of A.

The value of B had a decreasing trend with increasing H/D ratio and it was found to be varying with H/D according to the equation, $B = (-0.046 + 0.079\ H/D)^{-1}$ (Eq. 4.8).

Hence the final equations for settling velocity took the form as:

$$\frac{\nu_c}{\nu_o} = A\varepsilon^{(B.\frac{H}{D})/\varepsilon} \quad \ldots.4.9$$

where,

$$A = 3.06 - 0.28\frac{H}{D} + 0.03\left(\frac{H}{D}\right)^2$$

$$B = (-0.046 + 0.079\ H/D)^{-1}$$

The values of ν_c obtained from the experiments and predicted using the model is given in Table 4.6 and the equation describes the settling velocity within an error of 10% in all cases except four.

Table 4.6 Comparison of the experimental and predicted values of setting velocity using power equation

Fluid volumetric concentration, %	Settling velocity, mm/s											
	Column diameter ,37 mm						Column diameter ,49 mm					
	Height of suspension, mm											
	100		200		300		100		200		300	
	O	P	O	P	O	P	O	P	O	P	O	P
93.42	0.122	0.131	0.135	0.145	0.164	0.175	0.124	0.120	0.139	0.141	0.163	0.151
	(7.3)		(7.4)		(6.7)		(3.2)		(1.4)		(7.3)	
92.11	0.086	0.100	0.121	0.117	0.142	0.139	0.088	0.091	0.125	0.109	0.129	0.121
	(17.4)		(3.3)		(2.1)		(3.4)		(12.8)		(6.2)	
90.79	0.069	0.076	0.089	0.90	0.105	0111	0.072	0.068	0.091	0.085	0.106	0.094
	(10.1)		(3.4)		(5.7)		(5.5)		(6.5)		(11.3)	
89.47	0.047	0.058	0.060	0.070	0.081	0.086	0.049	0.050	0.070	0.666	0.082	0.024
	(22.3)		(26.6)		(6.1)		(2.0)		(5.7)		(9.7)	

*O - Observed value, P- Predicted value
The values in the parentheses are the % deviation of observed values from predicted values.

4.1.5 Batch settling test for low concentrated starch suspension

Data on the settling column test were analysed and the settling trajectories of various fraction of the suspended starch particles were obtained by plotting the lines of equal percent settled against time and depth by drawing data points on the contour map. This is represented in Figs. 4.8 and 4.9. for different concentrations for dried and fresh starch. Effective depth of column was taken as 24 cm as the region below the bottom port was taken as sedimentation or settling zone.

From the figures, initial phases of settling were characterised by closely spaced isosettling lines, indicating the faster settling rates. Afterwards, the gap between the settling lines were increased due to decreased settling rates. This result is in agreement with the observation made in Section. 4.1.2. As concentration increased, more time was required

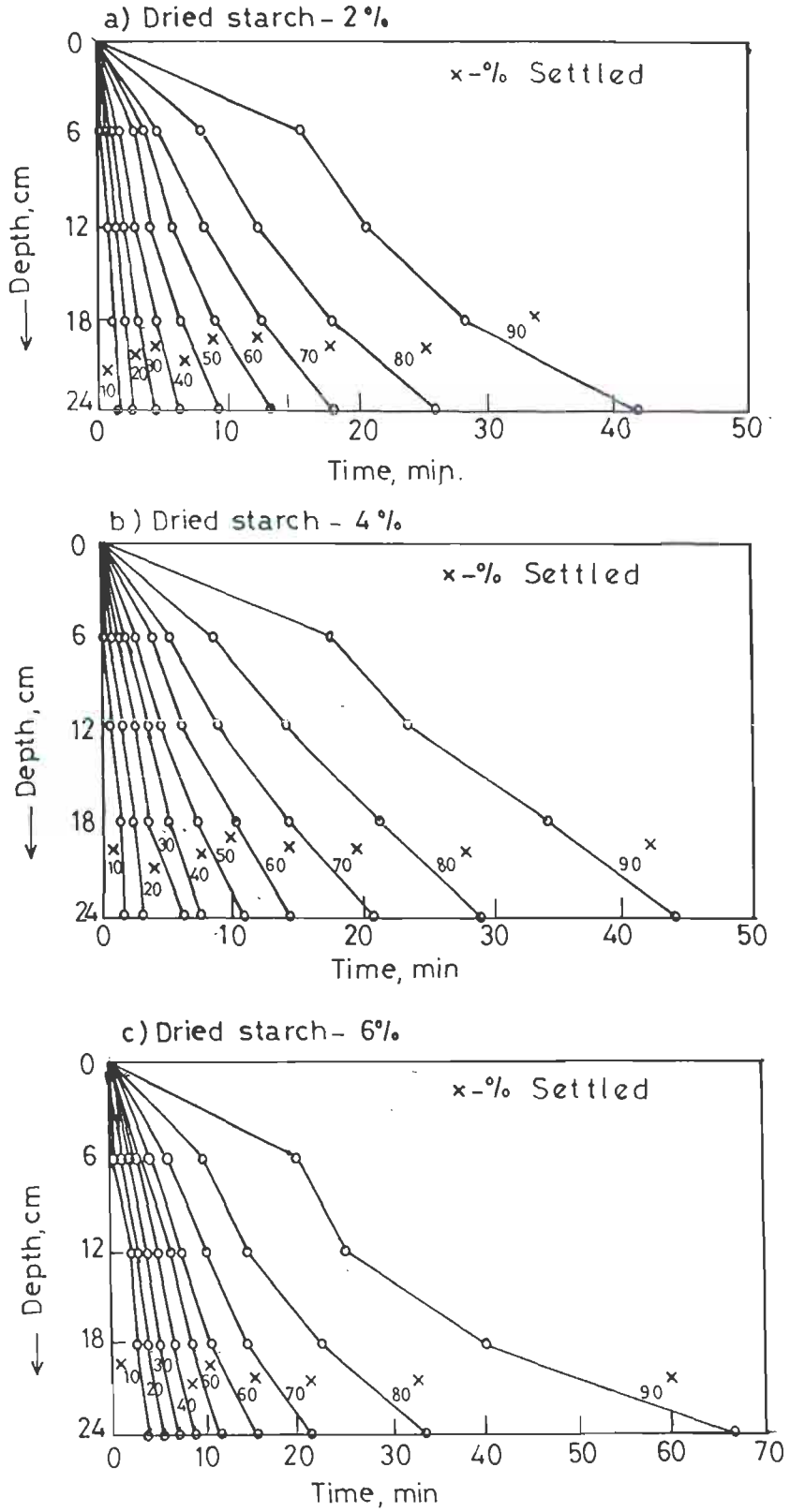


Fig. 4.8 Batch settling curves of cassava starch in low concentrated suspensions

for the starch to settle down. The total removal of 90% of the starch was obtained by noting the time where the isosettling lines meet the time axis. It was found that about 41, 45, 67 and 95 min were required for the complete settling of 90% of the starch particles in 2, 4, 6 and 8% concentrations, respectively for dry starch. In 2% concentration, the average settling velocity, calculated (as the ratio of the effective depth and time) was found to be 5.85 mm/min. It meant that 90% of the particles in the suspension had an average settling velocity greater than or equal to 5.85 mm/min in the first 41 min of settling time and were completely settled. Similarly for 4, 6 and 8% concentrations, 90% of the particles had settling velocities higher than or equal to 5.33, 3.58 and 2.53 mm/min, respectively *i.e.*, as concentration increased, the settling took more time and the same was confirmed by decreased settling velocity at higher concentrations.

Total amount of starch settled at various time intervals were obtained by adding the amount of starch completely settled or removed (*i.e.*, isosettling lines meet the X-axis) and the sum of the fractional starch removed for each fractions. The fractional removal for 2% concentrated dried starch suspension at 5 min of settling was obtained as 8.88, 6.67, 4.67, 3.15, 1.94, 1.67 and 0.33% for 30-40, 40-50, 50-60, 60-70, 70-80, 80-90, 90-100% fractions. These fractions are summed up to get 27.31% and is added with the 30% *i.e.*, completely settled in 5 min, giving about 57.31% of the starch particles which are totally removed in 5 min of settling (Fig. 4.8a). Similar calculations were done for other settling times and concentrations and a graph relating to the total removal or settling of starch versus time is depicted in Fig.4.10. From this data, after 40 min of settling, about 94.43, 93.80, 92.58, 89.12% of the starch particles were settled for 2, 4, 6 and 8% concentrations, respectively. Also for 2, 4, 6 and 8% concentrations, about 94% starch was settled after 40, 40, 60 and 90 min indicating slow settling of starch at higher concentrations. As the time of settling increased, progressive increase in the settling was observed in the initial stages,

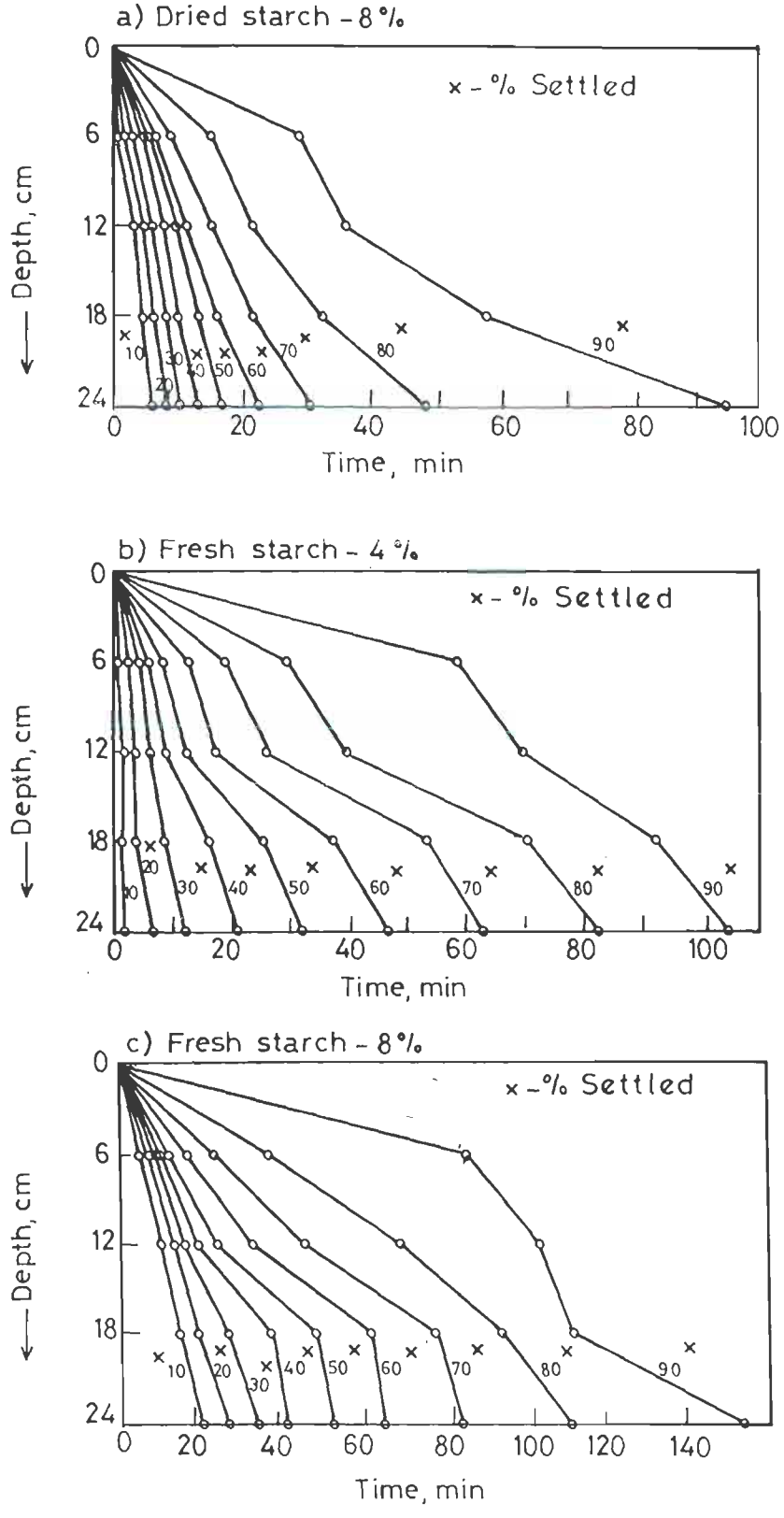


Fig. 4.9 Batch settling curves of cassava starch in low concentrated suspensions

then the increase was gradually reduced and finally it became nearly parallel to time axis. This time corresponding to the parallel segment of the curves gave the detention period for the particle to be in the settling tank.

4.1.5.1 Fresh starch

Settling analysis with fresh starch (Figs. 4.9b and 4.9c) showed that it took more time to completely settle a definite fraction of the particles *i.e.,* during first 40 min of settling, about 76.34 and 68.05% of the starch were settled at the bottom for 4 and 8% concentrations, respectively whereas for dried starch (Fig.4.9a), it was 94.43 and 93.80%, respectively. To achieve 94% settling in fresh starch with 4 and 8% concentrations, about 100 and 140 min were required. The enhanced time of settling of fresh starch was due to the hydration repulsion effect as explained in Section.4.1.2.4.3.

4.1.5.2 Settling velocity

Settling velocity distribution of suspension was drawn by noting the weight fraction of particle having velocities less than the stated velocities as shown in Fig.4.11. It gives an idea about the percent of starch particles having settling velocities less than a particular settling velocity with which the fraction of particles to be settled. At 40 min, settling velocity was found to be 6 mm/min and about 6, 6, 7, 11, 24 and 40% fraction of 2, 4, 6, 8% (dry) and 4 and 8% (fresh) starch concentrated suspensions were remained in the suspension without settling. These fractions are having velocities less than the above velocity of 6 mm/min and hence to achieve maximum settling, more time should be provided.

4.1.5.3 Settling tables

Data from the settling column tests could be used as a valuable tool for designing of settling tables *i.e.,* for the determination of detention period and loading rate. Settling tables used in the settling process could be considered as a continuous flow sedimentation tank.

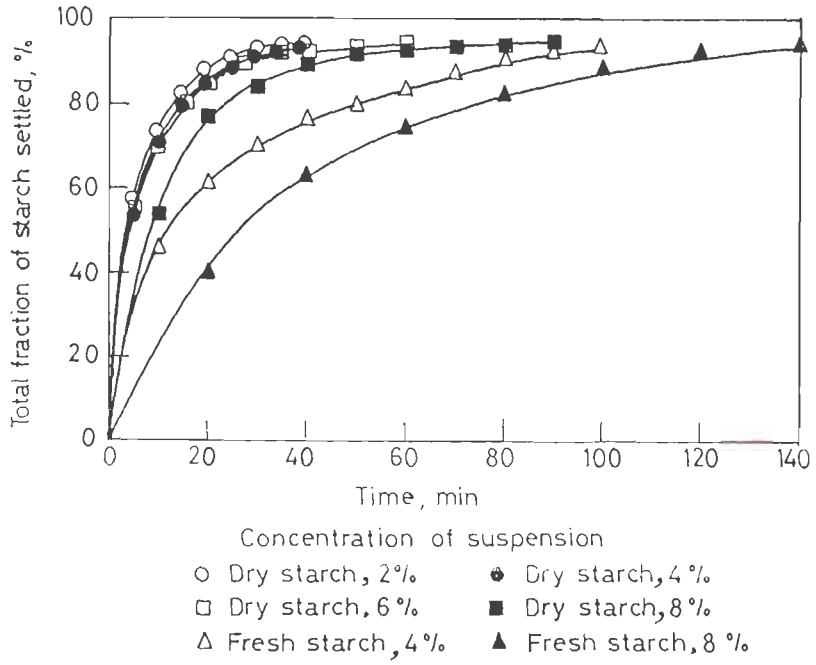


Fig. 4.10 Overall settling of cassava starch Vs detention time

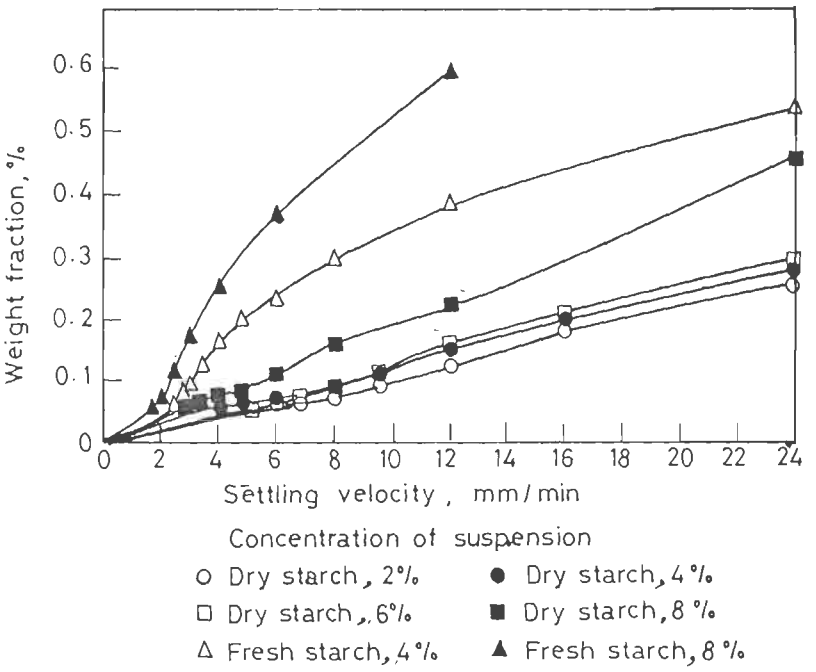


Fig. 4.11 Settling velocity pattern of low concentrated starch suspensions

The detention time of the starch particles in the tabling process should be selected in such a way that all the particles present in the suspensions should settle at the bottom of the tank, before the clarified water/supernatant is discharged out. Moreover, a compacted starch cake is desired after settling to take it out easily from the tank. The detention time calculated from the settling studies is enough only for settling the majority of the starch particles. For consolidation to occur for the starch mass, it should be allowed to stand for additional time. In the case of fresh starch at about 4% concentration within 100 min, about 94.2% of the starch particles were settled down. Hence, for consolidation to occur and or to meet with the actual field condition, the time should be taken as double. Droste (1997) reported a multiplication factor of 1.25-1.75 for the determination of detention period to meet with the actual field condition for waste water treatment. The present study also (as explained in latter Section 4.1.6) showed that after 3-4 h of settling, moisture content of the cake did not vary much i.e. cake was compacted to more or less same degree. Hence, maximum of 4 h settling is sufficient. This also confirmed the selection of 2 as multiplication factor in the present study.

Tabling process is carried out by flowing the starch suspension in an inclined trough/channel at very slow rate. Here, flow velocity is very critical criteria in designing the table. Having 200 min of detention period and 24 cm effective depth, settling velocity (Vs) will be 1.2 mm/min and hence the flow velocity should be less than this velocity. Otherwise, the particle will be discharged along with the overflow. Generally tables are 40-50 cm in width, 30-100 m long and 20-30 cm deep (Balagopalan, *et al.*, 1987). If a settling table of 50 cm width and 100 m long and 30 cm depth is considered; then area of the tank (As) will be 50 m^2. The loading rate (Q), settling velocity (Vs) and area (As) are related in an expression $Q = Vs.As$. Then the designed loading rate in the above example was calculated as 0.001 m^3/s

4.1.6 Water content in the settled starch cake

The cakes obtained after settling of starch suspension for different time intervals were analysed for their moisture content (Appendix E). It was observed that as the time of settling and concentration increased, the moisture content was found to be decreased (Fig.4.12). The water content was determined for the sample collected after 3h of settling so as to get a sediment which is compact enough to decant the supernatant water without disturbing the settled mass. The water content of the starch cake was found to be 86.54, 86.31 and 86.23% after 3 h of settling for 5, 15, and 25% concentrations, respectively. It was reduced to 81.42, 81.18 and 81.12% after 6 h of settling showing a progressive reduction in water content. During additional 2 h settling, the reduction was very less *i.e.*, it dropped to 80.20, 79.80 and 79.20% at the end of 8 h for 5,15 and 25%, respectively. When starch particle settled at the bottom, they squeezed out water present in the interstices of particles. During this thickening process, liquid flows upward through the aggregate and around individual particles and flocs in uniform seepage across the horizontal cross section of the thickening column (Vesilind and Jones, 1993). This behaviour is referred as channelling i.e. creation of flow path in a thickening suspension on a scale much larger than the size of the solid particle themselves (Dixon, 1982). As the time of settling increased, more compaction resulted in more displacement of water from the interstices of starch particles forming the cake and hence reduction in moisture content was obtained. The higher rate of reduction in moisture in the initial phase showed that more compaction has occurred due to self weight of more particles settled out. Almost constant values recorded at the end indicated the termination of compaction and further compaction was not possible as rearrangements of particle in the compression zone was almost completed and resulted in well packed mass. Though increase in concentration also reduced the moisture content of the cake, it was not that much pronounced as time of settling because the observations were taken after 3h of settling during which almost all starch particle might be in the compression zone and slight increase in compaction may be due to the self weight of the settled starch.

Table 4.7 represents the results of the analysis of variance showing the effect of concentrations and time of settling on the water content of the cake and it revealed that only time of settling have significant effect ($P \leq 0.01$) on water content and concentration and the interaction terms did not have any significant effect.

Table 4.7 ANOVA Table for water content of the settled starch cake

SV	DF	SS	MS	F
Treatment	17	220.94	13.00	13.69**
Time of settling (T)	5	217.26	43.45	45.77**
Concentration (C)	2	2.91	1.45	1.53 ns
T x C	10	0.70	0.08	<1
Error	18	17.09	0.95	
Total	35	238.03		

** Significant at 1 % level, ns -Not significant

4.1.6.1 Modelling of final moisture content

An equation correlating moisture content with time of settling was developed in the form of $M = A + B \ln(t)$ for each concentration and given below:

For 5%, $M = 94.45 - 7.55 \ln(t)$ ($r^2 = 0.97$) 4.10

For 15%, $M = 94.40 - 7.15 \ln(t)$ ($r^2 = 0.97$) 4.11

For 25 %, $M = 94.61 - 7.51 \ln(t)$ ($r^2 = 0.96$) 4.12

The values of A and B for different concentrations were changed very slightly, hence mean values were taken for the final equation. The mean value of 94.49±0.08 for A and 7.4 ±0.18 for B was obtained and hence the general equation took the form as:

$$M = 94.5 - 7.4 \ln(t) \quad \text{....4.13}$$

Water content values predicted with this equation are compared with the observed values and are given in Table 4.8.

The per cent deviation of the predicted value from the observed values ranged from ± 1.5 %. Hence the above equation M = 94.5 - 7.4 ln (t) can very well be used to describe the change of moisture content of the starch cake with time of settling.

4.1.7 Settling index

Settling index (SI) is a measure of the thickening ability or compactness of the starch in suspension during settling and was calculated as the ratio of the volume of settled starch to the concentration of starch in the suspension. It was measured in tune with sludge volume index (SVI) in waste water treatment for the settling of suspended solids, but with some modifications. SVI is calculated by allowing the sample for a certain prefixed period, whereas in the present study, the final volume of the settled starch was taken for which the time required will vary as our interest is to get an idea about the compactness of the settled starch. From the volume of the settled starch and total amount of starch present in the suspension of different initial heights, settling index (SI) was calculated and presented in Table 4.9

Though there was no significant difference in SI with concentrations, a slight reduction was observed with increase in concentration. But for height of suspension, the reduction was more predominant than that of concentration. For 37 mm column, when the concentration was increased from 10 to 16%, value of SI decreased from 1.82 to 1.75 ml/g for 100 mm,1.65 to 1.53 ml/g for 200 mm and 1.53 to 1.42 ml/g for 300 mm height of suspension. But with 49 mm diameter column, SI value varied from 1.62 to 1.51 ml/g, 1.55 to 1.44 ml/g, 1.50 to 1.36 ml/g for 100, 200 and 300 mm heights for an increase in concentration from 10 to 16%, respectively.

As the height of suspension increased from 100 to 300 mm, SI reduced from 1.82 to 1.53, 1.79 to 1.42, 1.77 to 1.42 and 1.76 to 1.42 ml/g for 10, 12, 14 and 16% concentrated suspensions, respectively in 37 mm column diameter. But for 49 mm column diameter,

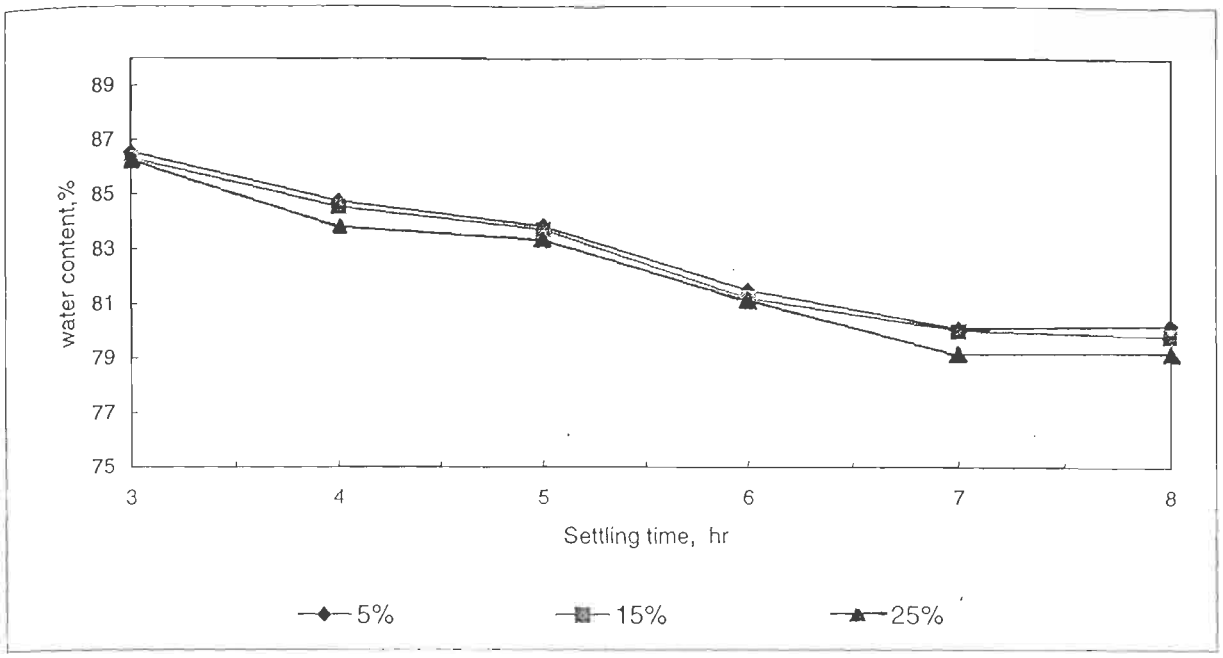


Fig.4.12 Effect of settling time on final water content of the starch cake for different concentrations

Table 4.7 Comparison of the predicted water content of the starch cake with actual values for different concentrations

Sl No	Conc. (%)	Descriptions	Water content, %(db)					
			Duration of settling, hr					
			3	4	5	6	7	8
1	5	Observed value	86.53	84.74	83.82	81.48	80.18	80.20
		Predicted value	86.37	84.24	82.59	81.24	80.10	79.11
		% deviation	0.20	0.59	1.46	0.29	0.10	1.36
2	15	Observed value	86.31	84.53	83.69	81.18	80.00	79.80
		Predicted value	86.37	84.24	82.59	81.24	80.10	79.11
		% deviation	-0.07	0.34	1.30	-0.07	-0.13	0.86
3	25	Observed value	86.23	83.82	83.32	81.12	79.15	79.20
		Predicted value	86.37	84.24	82.59	81.24	80.10	79.11
		% deviation	-0.16	-0.50	0.88	-0.15	1.20	-0.11

the reduction of SI value was from 1.62 to 1.50, 1.59 to 1.47, 1.53 to 1.38 and 1.51 to 1.36 ml/g for 10, 12, 14 and 16% concentrations, respectively. Lower the value of SI, higher will be the compactness of settled starch and from the data, it is clear that suspension with more initial height produced more compacted starch. This is due to self weight of starch particles and column of water standing over it.

Table 4.9 Settling index of cassava starch suspension

Height of suspension , mm	Concentration, % (w/v)	Volume of settled starch, ml	Amount of starch in suspension, g	Settling index, ml/g
37 mm column diameter				
100	10	19.56	10.75	1.82
	12	23.17	12.94	1.79
	14	26.44	15.05	1.767
	16	30.10	17.20	1.75
200	10	35.48	21.50	1.65
	12	40.32	25.80	1.56
	14	46.67	30.11	1.55
	16	52.69	34.41	1.53
300	10	49.50	32.26	1.53
	12	54.84	38.71	1.42
	14	64.13	45.16	1.42
	16	73.29	51.61	1.42
49 mm column diameter				
100	10	30.64	18.86	1.62
	12	35.98	22.63	1.59
	14	40.39	26.40	1.53
	16	45.56	30.17	1.51
200	10	58.46	37.72	1.55
	12	67.89	45.26	1.50
	14	77.79	52.80	1.47
	16	86.89	60.34	1.44
300	10	84.85	56.57	1.50
	12	99.94	67.89	1.47
	14	109.37	79.20	1.38
	16	123.52	90.52	1.36

Slight reduction in SI due to the increase in concentration is explained by the moisture present in the final cake as explained in Section 4.1.6. As concentration increased, a slight reduction in water content was observed indicating the expulsion of more water from the pore space between the settled mass which caused the volume to reduce, producing more compacted starch. The particles placed at higher elevation possessed higher energy. On collision, the impact of collision on the immediate particle was more which finally reached the settled starch particles and caused more squeezing effect on them, displacing more water present in the interstices of the settled mass, resulting more compaction. The dependence of SVI on suspended solid concentration was investigated by Stobbe (1969) and found that SVI is independent of the concentration if the sludge volume after 30 min of sedimentation is less than 250 ml/g. In the present study, the sludge volume is less than this quantity which attributed to the light dependence of concentration on SI

Settling index of fresh starch was calculated and found that the values could be compared well with that of dried starch. The values were 1.65, 1.50, 1.47 and 1.40 ml/g for 13.43, 15.85, 16.97 and 20.10% fresh starch concentrations, respectively and it was found to be decreased with increasing concentration, which is in tune with that of dried starch. For suspension of low concentration, SI was 1.68, 1.71 and 1.7 ml/g for 2, 4 and 6% concentrated dried starch suspension, whereas 1.58, 1.56, 1.39 and 1.32 ml/g for 4.04, 6.45, 7.92 and 9.10% fresh starch concentrations, respectively which recorded slightly lower values for fresh starch than dried starch, which may be due to the higher settling time or hydrated condition of fresh starch.

Analysis of variance indicated that only height of suspension significantly affected the settling index at 1% level whereas concentration and diameter are not having any significant effect on settling index, also the case for all the interactive terms.

4.2 Settling in the Presence of Different Chemicals

Settling characteristics of cassava starch settled in the presence of different chemicals and the quality attributes of the settled starch are explained in this section

4.2.1 Effect of chemicals on settling characteristic of cassava starch

The effect of chemicals used for settling of starch on the settling properties (settling time, settling index and hardness of the settled cake after drying) are presented in Table 4.10.

4.2.1.1 Sulphuric acid

Addition of sulphuric acid resulted in a slightly faster settling at higher concentrations of the acid. From Table 4.10, it is seen that settling time was reduced to a minimum of 70 min compared to control sample which took 100 min for settling. Settling index-a measure of the compactness of starch cake was found to be reduced from 1.19 to 1.02 ml/g indicating that addition of acid led to a more compact mass after settling. Hardness of the dried starch cake was found to be more than that of control at all the concentrations studied. For control sample, hardness was 0.88 kg, whereas it increased to 1.09 kg at 40 mM concentration. This increase in hardness confirmed that sulphuric acid helped to produce more compact starch cake.

4.2.1.2 Hydrochloric acid

Hydrochloric acid was found to aid settling process at all concentrations except at 15 mM, where the settling time was on par with that of control, i.e. 100 min. At all other concentrations, settling time was reduced by 30 min. Settling index for control sample was found to be 1.38 ml/g whereas for starch settled in acid showed a range of 1.24 to 1.15, lowest being for 120 mM. Hardness was also higher for all the treated samples compared to control except at 15 mM concentration which was equal to control. The value showed an increasing trend and the maximum value observed was 1.43 kg.

Reduction in settling time and settling index indicated that acid treatment aids in settling and getting more compact starch cake which is in agreement with the observation made by Holleman and Atten (1956).

4.2.1.3 Sodium metabisulphite

The study revealed that settling time was reduced (20-30 min) by the addition of sodium metabisulphite at all concentrations and the maximum reduction of 30 min was observed at 4.68 to 7.8 mM concentrations. Settling index values were nearly equal and all the values were lower than that of control. It varied from 1.19 to 1.14 ml/g when compared to control (1.38 ml/g). But the hardness was equal or slightly lower than that of control, ranging from 0.65 to 0.88 kg. The results indicated that sodium metabisulphite had different effects as compared to that of acids.

4.2.1.4 Sodium hypochlorite

Settling time was reduced by 20 min at higher concentrations *i.e.*, above 0.70 mM and the settling index was found to be lowered with increasing concentrations. The values were dropped to 1.02 ml/g compared to 1.38 ml/g for control. The pronounced effect at higher concentration is attributed to the action of chlorine gas acting as an oxidising agent. The reaction of chlorine gas with water produces hydrochloric acid and hence combined effect may be more pronounced at higher concentrations. Hardness of the starch cake was also found to be higher than that of control and a maximum of 1.53 kg was obtained at 1.41 mM concentration.

Though sodium metabisulphite and sodium hypochlorite are oxidising agents, hypochlorite was found to be more effective to give a more compacted mass, probably due to the combined effect as oxidising agent and acid.

Table 4.10 Effect of chemical addition on settling characteristics of cassava starch

Properties	Concentration of chemicals					Control
	Sulphuric acid					
	5 mM	10 mM	20 mM	30 mM	40 mM	
Settling time, min	90	90	80	70	70	100
Settling index, ml/g	1.02	1.02	1.02	1.02	1.02	1.19
Hardness, kg	0.93	0.93	0.98	1.07	1.09	0.88
	Hydrochloric acid					
	15 mM	30 mM	60 mM	90 mM	120 mM	
Settling time, min	100	70	70	70	70	100
Settling index, ml/g	1.24	1.19	1.16	1.15	1.15	1.38
Hardness, kg	0.89	1.2	1.22	1.39	1.43	0.88
	Sodium metabisulphite					
	1.56 mM	3.12 mM	4.68 mM	6.24 mM	7.8 mM	
Settling time, min	80	80	70	70	70	100
Settling index, ml/g	1.19	1.19	1.19	1.16	1.14	1.38
Hardness, kg	0.8	0.82	0.65	0.76	0.88	0.88
	Sodium hypochlorite					
	0.17 mM	0.35 mM	0.70 mM	1.06 mM	1.41 mM	
Settling time, min	90	90	80	80	80	100
Settling index, ml/g	1.24	1.19	1.16	1.02	1.02	1.38
Hardness, kg	1.02	1.04	1.12	1.17	1.53	0.88
	Alum					
	16.7 mM	28.50 mM	37.50 mM	44.40 mM	50.0 mM	
Settling time, min	100	90	100	90	90	100
Settling index, ml/g	1.39	1.36	1.35	1.36	1.36	1.41
Hardness, kg	0.53	0.4	0.47	0.48	0.78	0.88

4.2.1.5 Alum

Settling time of starch was reduced only by 10 min by the addition of alum at 50 mM concentration and it is the least effective among the chemicals tested in the present study. Settling index and hardness were found to be slightly lower than those of control at all concentrations studied.

Among the different chemicals used in the present study, acids and metabisulphite accelerated the settling process in a more effective way than hypochlorite and Alum. But more compactness was achieved by addition of hypochlorite followed by sulphuric acid, metabisuphite, hydrochloric acid and alum.

4.2.2 Gelatinization properties of cassava starch

The gelatinization properties of native starch and the starch settled by using water to which different chemicals have been added were studied by means of Differential scanning calorimetry (DSC) endotherms. They were characterised by the onset or start temperature (T_o), the temperature at peak minimum (T_p), offset or end temperature (T_e), gelatinization temperature range or melting interval (ΔT) and melting enthalpy (ΔH) and are presented in Table 4.11 and DSC thermograms are depicted in Fig.4.13.

4.2.2.1 Native starch

The DSC trace of the heating of cassava starch in excess water showed one sharp single endotherm, as expected. This reveals the co-operative melting behaviour of cassava starch where the granular structure responds uniformly to heating. The T_o, T_p and T_e values for cassava starch were 66.90, 70.07 and 85.07°C, respectively giving ΔT of approximately 18.17°C. These values are slightly higher than the values reported by Francisco *et al.* (1996), Perez *et al.* (1998[a]) and Farhat *et al.* (1999). DSC parameters for cassava starch were found to be higher than those for other root and tuber crop starches. The T_o, T_p and T_e values were 64.6, 73.9 and 84.6°C for sweet potato starch (Collado, 1999); 68.5, 68.5 and 85.0°C for arrow root starch (Perez *et al.* 1998[b]); 61.6, 64.8 and 74.5°C

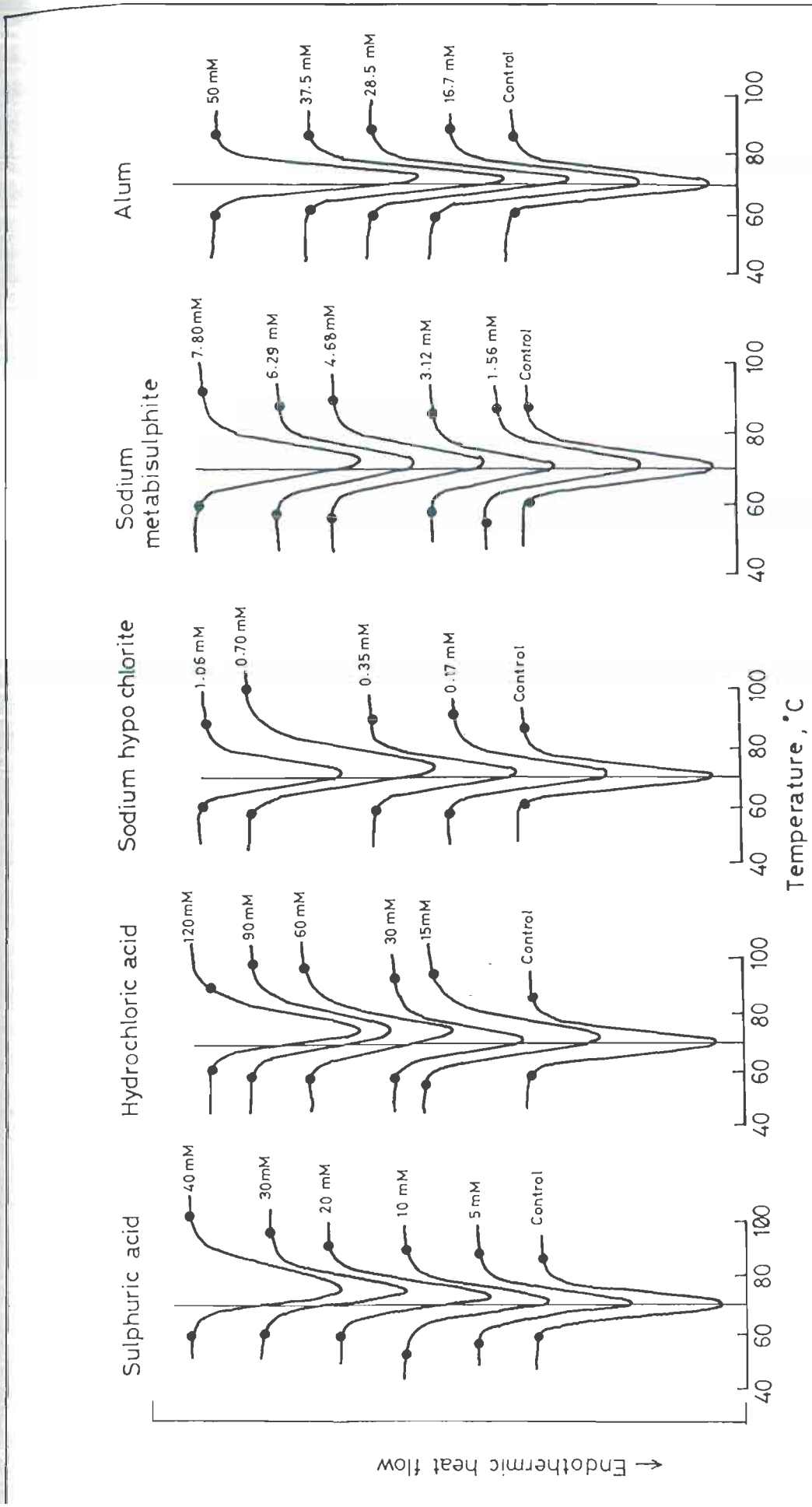


Fig. 4.13 Differential scanning calorimetric thermograms of cassava starch settled in the presence of different chemicals

for *Canna edulis* starch; 64.2, 68.2 and 74.8°C for *Dioscorea* starch (Gebre Mariam and Schmidt, 1998) and 66 , 69, and 80°C for potato starch (Perez, *et al.* 1998[a]). The present observation on ΔT values is in agreement with the general concept that gelatinization in excess of water always takes place over a temperature interval of 1-2°C for a single granule and for the whole population of granules, the interval may be greater than 10°C (French, 1984 and Eliasson and Gudmudsson, 1996).

ΔH is a measure of the thermal energy involved in the gelatinization process and at the molecular level, this may be expected to involve the cleavage of existing hydrogen-bonds between starch molecules and the formation of new bonds involving water to give a less ordered structure with increased entropy, so that the overall process is endothermic (Slevens and Elton, 1971). ΔH for cassava starch was found to be 15.55 J/g which was higher than that of sweet potato 12.9 J/g (Collado, 1999); arrow root 4.4 J/g; *Canna edulis* 1.8 J/g (Perez *et al.*, 1998[b]) and lesser than that of *Dioscorea* starches 19.2 J/g (Gebre Mariam and Schmidt, 1998) and potato starch 19.8 J/g (Gebre Mariam *et al.*, 1996). The ΔH value obtained for cassava starch in the present study was close to the value (15.6 J/g) obtained by Francisco *et al.* (1996). They also studied the DSC and light microscopic method to determine the gelatinization temperature and reported a melting range of 19°C for cassava starch. The slight difference in values may be attributed to both the innate difference in starch quality as influenced by the agro-climatic conditions, varietal characteristics as well as to the minor changes in the conditions adopted in the experiment (Asaoka *et al.*, 1991; Asaoka *et al.*,1992 and Moorthy *et al.*, 1996).

4.2.2.2 Cassava starch settled in presence of different chemicals

4.2.2.2.1 Sulphuric Acid

Treatment with sulphuric acid resulted in a noticeable shift in gelatinization temperature which increased with increase in concentration of acids (Table 4.11). The T_o values were enhanced from 66.90°C for the control to 70.53°C for the samples treated

with 30 mM sulphuric acid where as T_e values were shifted progressively from 85.07°C for the control to 98.0°C at 40 mM concentration. ΔT was also higher compared to control, which increased from 18.17°C for control to 27.87°C at 40 mM concentration. Leach and Schoch (1962) have observed an increase of 7°C in gelatinization temperature on acid treatment of corn starch. The authors attributed the higher gelatinzation temperature to increased miscellar organization in the granule. Though gelatinization temperature was increased, the damage brought about by the acid treatment in the granule rendered the viscosity quite low. However, the enthalpy of gelatinization did not show any definite trend for all the treatments, but the values were with in the range of 15.75±0.75 J/g when compared to 15.55 J/g for native cassava starch, indicating that most of the effect due to acid addition is on the amorphous region of the starch granule and the crystallinity is affected only to a very small extent. This is in agreement with the observations made by Wurzburg (1989). It is well known that during the acid modification of starch in the granular form, the linear portions containing a predominance of α-D (1→4) linkages are involved in the formation of crystallites are held together by hydrogen bonding; thus decreasing the availability of these segments to acid. The amorphous region containing branching points with α-D (1→6) linkage is more accessible to acid penetration and hydrolysis (Cowie and Greenwood, 1957 and Wurzburg, 1989).

4.2.2.2.2 Hydrochloric acid

T_o values for hydrochloric acid treated samples registered a progressive increase upto 90 mM concentration and then a slight decrease at 120 mM concentration. The T_o registered a rise from 66.90°C for the control to 70.65°C for samples treated with 90 mM concentration, but at 120 mM concentration, a minor reduction to 69.60°C was noticed. Similarly T_e values were increased from 85.07°C for the control to 90.60°C for samples at 90 mM concentration and then decreased to 89.30°C at 120 mM.

Biliaderis *et al.* (1980) also observed an increase in the T_o and T_e values for pea and corn starch when treated with hydrochloric acid. The ΔT of all the treated samples recorded higher values than the control. However, increase in acid concentration decrease the ΔT value except at 30 mM concentration which recorded the highest value.

Unlike sulphuric acid treatment, ΔH in hydrochloric acid treatment showed a steady decline except at 30 and 60 mM concentrations. The value dropped from 15.55 J/g for the control to 14.17 J/g at 120 mM concentration. ΔH values of hydrochloric acid modified corn and smooth pea starches were found to be lower compared to native starch (Biliaderis *et al.*, 1980) though the reduction was not predominant in the present study. Similarly, higher susceptibility of cassava starch to structural changes associated with acid modification by hydrochloric acid or nitric acid has been reported by Vasudevasingh *et al.* (1993).

4.2.2.2.3 Sodium metabisulphite

The variation in concentration of sodium metabisulphite did not affect much the T_o value which recorded higher values than the control (66.90°C). The T_e varied from 86.80 to 88.23 for the range of concentrations studied (1.56 to 7.80 mM) and showed a continuously increasing trend except at 6.29 mM where it recorded a lower value of 85.87°C. The same trend was also observed with ΔT. ΔH values were nearly same for all the treatments. This indicates that the sodium metabisulphite had no effect in the thermal energy required for gelatinization of starch.

4.2.2.2.4 Sodium hypochlorite

With sodium hypochlorite, T_o and T_e recorded higher values at all concentrations studied, than the control. T_o recorded a value of 68±0.5°C except at 0.17 mM and T_e recorded 88.75±0.25°C upto 0.70 mM concentration and then showed a wide variation in its value at higher concentration. Similar trend was also observed in ΔT also.

Table 4.11 Differential scanning calorimeter (DSC) gelatinization parameters of cassava starch settled in the presence of different chemicals

Treatments	Gelatinization temperatures, °C				Enthalpy ΔH, J/g
	T_o	T_p	T_e	ΔT	
Control (Native starch)	66.90	70.07	85.07	18.17	15.55
Sulphuric acid					
5 mM	67.30	70.50	86.73	19.43	15.98
10 mM	67.77	71.20	88.10	20.33	16.05
20 mM	69.33	72.20	89.15	19.82	15.00
30 mM	70.53	74.20	93.13	22.60	15.76
40 mM	70.13	75.07	98.0	27.87	16.59
Hydrochloric acid					
15 mM	67.40	71.10	87.46	20.06	15.28
30 mM	67.53	71.27	90.30	22.77	16.12
60 mM	68.93	72.57	88.93	20.00	15.40
90 mM	70.65	73.90	90.60	19.95	14.95
120 mM	69.60	74.30	89.3	19.70	14.17
Sodium metabisulphite					
1.56 mM	67.50	70.80	86.80	19.30	15.61
3.12 mM	67.57	70.87	86.97	19.40	15.27
4.68 mM	67.60	70.90	87.33	19.73	15.86
6.29 mM	67.53	70.80	85.87	18.34	15.32
7.80 mM	67.93	71.37	88.23	20.30	15.51
Sodium hypochlorite					
0.17 mM	67.50	70.97	88.97	21.47	17.17
0.35 mM	68.26	71.53	88.47	20.21	15.38
0.70 mM	68.16	71.34	88.68	20.37	15.25
1.06 mM	68.47	71.93	91.73	23.26	15.72
1.41 mM	68.07	71.37	85.53	17.46	16.25
Alum					
16.7 mM	67.73	71.00	86.40	18.67	15.01
28.5 mM	68.27	71.57	86.67	18.40	15.62
37.5 mM	68.60	71.57	86.30	17.70	15.28
44.4 mM	68.70	71.90	84.87	16.17	15.69
50.0 mM	68.53	71.65	84.57	16.04	15.20

T_o - Onset gelatinization temperature, T_p - Peak minimum temperature,
T_e - Final gelatinization temperature, ΔT - Gelatinization interval (T_e-T_o)

The higher values of gelatinization parameters as compared to control may be due to the oxidation as reported by Wootton and Bamunuarachchi (1979) and Eliasson *et al.* (1988). At low concentration (0.17 mM) and at high concentration (1.41 mM), gelatinization demanded higher thermal energy (ΔH is 17.17 and 16.25 J/g) than the control (15.55 J/g) whereas other concentrations recorded only 15.55 ± 0.30 J/g which is similar to the control values.

4.2.2.2.5 Alum

In the case of alum, except at low concentration (16.7 mM), all other concentrations recorded T_o values of 68.5±0.20°C which is higher than the control value, whereas at 16.7 mM concentration, T_o recorded 67.73°C which is also higher than control value but lower than that of other higher concentration treatment values. But the trend was some thing different for T_e *i.e.*, below 44.4 mM addition of alum to the suspension recorded higher values of T_e (86.5 ± 0.20°C) and further increase in addition decreased the value to 84.72± 0.15°C which is lower than the control. The ΔT showed an inverse relationship with concentration of alum. The addition alum from 16.7 to 50.0 mM, decreased ΔT values continuously from 18.67 to 16.04°C. This may be due to the salt effect of alum during the gelatinization process as explained by Eliasson and Gudmudsson (1996). The gelatinization enthalpy did not show much variation and it was 15.33±0.35 J/g which is more or less equal to the control value.

A comparative study on the gelatinization properties of native as well as treated samples revealed that the chemicals affect the granular thermal properties, the extent of which depends on the type and concentration of chemicals. The gelatinization temperatures T_o, T_p and T_e values were found to be enhanced by chemical treatments; the acid treatment having more pronounced effect. Shift in gelatinization temperature to a wider range was also observed at higher concentration of acids. Changes in ΔH values

were not significant except for hydrochloric acid showing that during heating, most of the changes are taking place on the amorphous region only. With hydrochloric acid, the crystalline region is also affected to a small extent.

4.2.3 Rheological properties of cassava starch

4.2.3.1 Native starch

The rheological properties of cassava starch *viz.*, viscosity, dynamic modulus [elastic modulus (G') and viscous modulus (G")] and phase angle were determined using Bohlin rheometer and the values are presented in Fig.4.14 and Table 4.12.

a. Viscosity

The data obtained from the rheogram indicated that viscosity undergone breakdown during holding at 95°C and again increased on cooling (Fig.4.14). Similar trend was also obtained in the Brabender viscographs for cassava starch (Moorthy, 1994). The viscosity dropped from 0.78 Pas at 75°C at the beginning of test and dropped to 0.57 Pas after holding at 95°C for 10 min. During cooling cycle, upto 75°C, viscosity was reduced to 0.50 Pas and then increased to 0.93 Pas after one hour holding at 35°C

The changes takes place during heating of starch in excess of water had been studied in detail by Whistler *et al.* (1984) and Wurzburg (1989). Native starch granules are insoluble in water, but when a starch suspension in water is heated above a critical temperature, the hydrogen bond responsible for the structural integrity of the granules weakens allowing the penetration of water and hydration of the linear segments of the amylopectin of the starch. During swelling, the amylose tends to leach out of the granule and along with the amylopectin, become highly hydrated. The suspension begins to clarify and the viscosity of the suspension rises to a peak where maximum swelling of granules takes place. In the experiments of present study, this critical initial rise in viscosity could not be observed as the starch sample was preheated at 75°C before

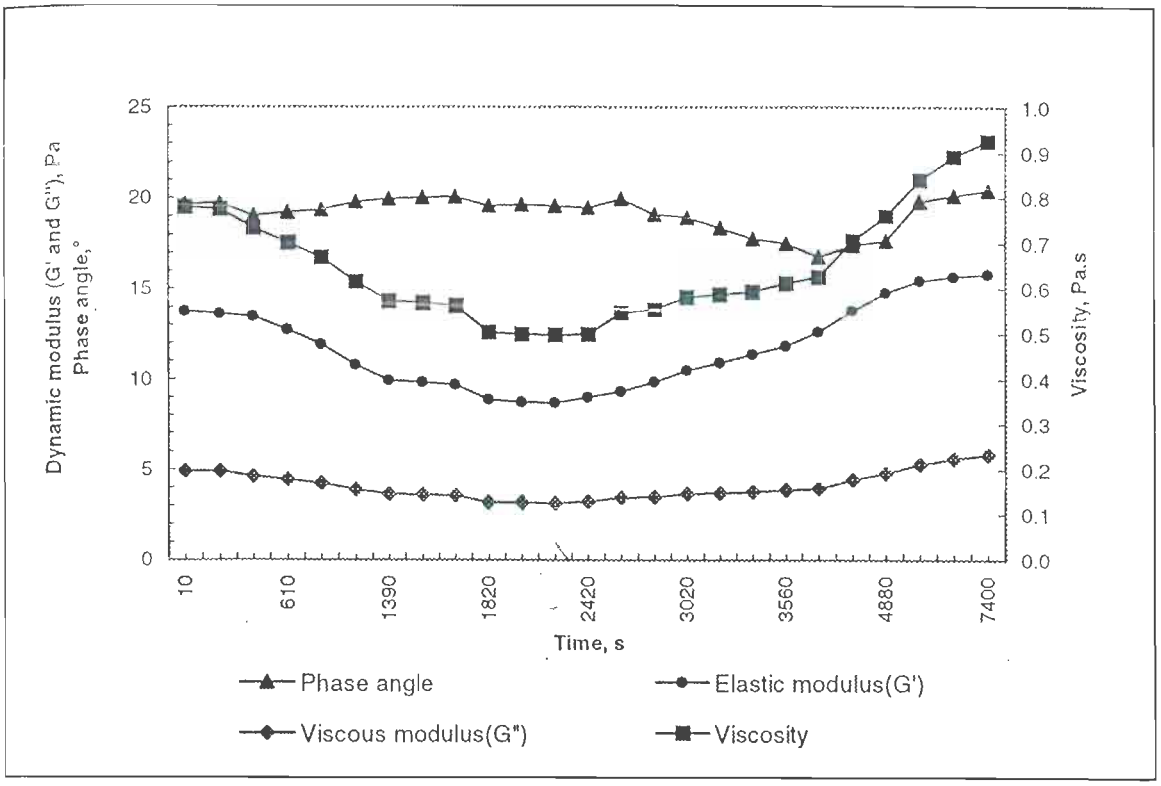


Fig.4.14 Rheological properties of native cassava starch

transferring to the rheometer and as heating continued, the granules tend to rupture and collapse and fragment, releasing the polymeric molecules and aggregates and hence a fall in viscosity was recorded. This trend was observed during heating at 95°C for 10 min and subsequent cooling upto 75°C. But after 75°C in the cooling cycle, the cohesive rubbery textured gel becomes thick and hence the viscosity increased due to the crystallisation or reorganisation in the starch granules.

b. Dynamic modulus

Elastic modulus (G') also followed the same trend as viscosity showing an initial decrease followed by a slow rise and then remaining nearly consistent. The G' decreased from 13.75 to 9.82 Pa on holding at 95°C for 10 min and during cooling cycle, after an initial fall increased to 15.45 Pa and remained constant during the holding period.

From the figure, it is clear that viscous modulus (G") also exhibited the same trend as G'. However, the decrease in the initial stage and increase in the latter stage were much less. G" values dropped from 4.9 to 4.2 Pa during the heating cycle and then registered a steady increase, finally reached to 5.8 Pa on holding at 35°C.

The elastic modulus was invariably higher than the viscous modulus at all the stages during heating and cooling of the sample, showing that starch paste has a tendency to behave more like solid under the experimental conditions. After the starch granules swell to a maximum extent, they become very soft, deformable and compressible causing a decrease in G' and G" (Evan and Haissman, 1979). From Fig.4.14, it is clear that the graph corresponding to G' and G" became parallel at the final stages of holding, indicating that the paste became a strong gel. This observation is similar to that obtained by Clark and Rossmurphy (1987) for guargum.

Table 4.12 Rhelogical properties of native cassava starch

Time, s	Temperature, °C	Phase angle, degree	Viscosity, Pas	Elastic modulus (G'), Pa	Viscous modulus (G"), Pa
10	75	19.65	0.78	13.75	4.90
190	80	19.70	0.77	13.60	4.87
370	85	19.00	0.73	13.45	4.61
610	90	19.20	0.70	12.70	4.41
790	95	19.35	0.67	11.90	4.20
1090	95	19.75	0.61	10.75	3.87
1390	95	19.95	0.57	9.93	3.60
1400	95	20.00	0.57	9.82	3.58
1580	90	20.05	0.56	9.65	3.54
1820	85	19.55	0.50	8.84	3.16
2000	80	19.60	0.50	8.71	3.14
2180	75	19.55	0.50	8.68	3.12
2420	70	19.45	0.50	9.00	3.23
2600	65	19.95	0.55	9.33	3.44
2780	60	19.10	0.55	9.84	3.48
3020	55	18.90	0.58	10.47	3.66
3200	50	18.35	0.59	10.91	3.70
3380	45	17.75	0.59	11.37	3.74
3560	40	17.50	0.61	11.83	3.85
3800	35	16.75	0.63	12.60	3.93
4280	35	17.40	0.71	13.80	4.44
4880	35	17.65	0.76	14.75	4.77
5660	35	19.80	0.84	15.45	5.27
6560	35	20.10	0.89	15.65	5.60
7400	35	20.40	0.93	15.80	5.80

c. Phase angle

The phase angle values which give information about the phase shift during heating/cooling cycle showed a very narrow range. During heating cycle from 75°C to 95°C and holding at 95°C, the values deviated only slightly from 20°. But during cooling cycle, value dropped marginally to 16.75° and then registered a progressive increase to 20.40° after one hour holding at 35°C. The low values of phase angle again confirmed the observation that starch paste has very low tendency to undergo deformation by placing the oscillatory strain and that system is predominantly elastic.

4.2.3.2 Cassava starch settled in the presence of different chemicals.

Rheological properties of starch settled in the presence of different chemicals are discussed in detail in this section and data for the same are presented in Appendix F to J and Figs. 4.15 to 4.19.

4.2.3.2.1 Sulphuric acid

a. Viscosity

Figure 4.15 clearly brings out the effect of concentration of acid on the viscosity of starch samples. On treatment with 5 mM concentration, the viscosity dropped rapidly to 0.11 from 0.78 Pas at 75 °C for the control. Similar trends were also observed at all other stages. Further rise in concentration led to the viscosity values of nearly zero indicating that starch has undergone considerable breakdown at these concentrations. Only at 5 mM concentration, starch was able to offer some resistance to acid. However, even at this level, 86% reduction was noticed. Wurzburg (1989) reported that the granules of acid modified starch resembled to those of unmodified starch at room temperature, but they behaved quite differently when heated in water. They do not swell many times their original size like unmodified starches, but developed radial fissures and disintegrate or fragment when they are heated. As a result, the hot paste of acid modified

starches are clear fluids showing more newtonian behaviour and act more like fluids causing the viscosity to come down to large extent. A decrease in viscosity was also reported by Balagopalan *et al.* (1987) for starch samples treated with sulphuric acid at 0.01 ml/l starch suspension. Acid being a hydrolysing agent, molecular weight of starch was decreased leading to the progressive reduction in viscosity due to treatment (Wurzburg, 1989).

b. Dynamic modulus

From Fig.4.15, it is clear that G' underwent noticeable reduction when compared to control. There was a slight increase in G' from 0.77 to 0.80 Pa during the heating cycle for 5 mM concentration. The value fell to 0.49 Pa during holding at 95°C for 10 min, rose and finally reached 1.0 Pa during cooling and remained steady. At higher concentrations, G' had reached insignificant values or nearing to zero due to the breakdown of starch and yielded a thin solution. Absence of increase in G' values even at 5 mM concentration showed that gelling tendency was also affected.

Viscous modulus (G") also followed the same trend as G', but the change was not so pronounced as G'. The trend was similar to control, but the values were much lower. G" values were lowered from 0.68 to 0.61 Pa during heating cycle and enhanced to 0.83 Pa at 35°C for 5 mM concentration. When compared to G' at 5mM acid concentrations, G" values were reduced to the maximum of 7 fold when compared to control. At higher concentrations, G" also had nil values similar to G'.

From the G' and G" values, it is clear that the native starch gel behaves elastic as G' values were 2-3 times greater that G" values. When treated at 5mM concentration, the G' underwent about 10 fold reductions while G" were reduced by 7 fold, showing it has a tendency to behave like a thin solution having more viscous properties.

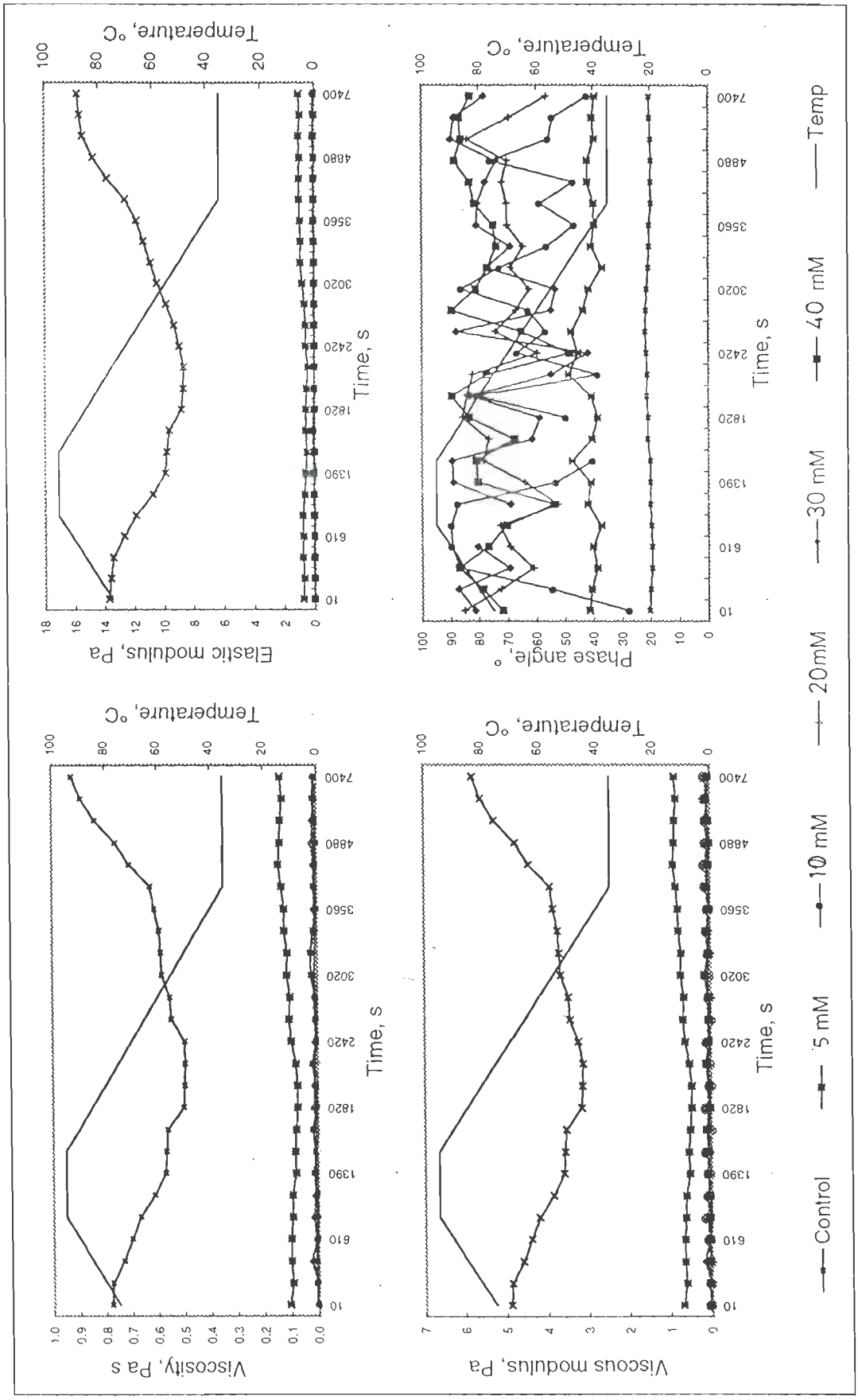


Fig. 4.15 Rheological properties of cassava starch settled in the presence of sulphuric acid

c. Phase angle

The phase angle values increased at all the concentrations. However, it did not show any regular trend except at low concentration where it showed a range of values between 37 and 48.6°. The other results were very irregular, again indicating the very flimsy nature of the paste. Phase angle values increased 4-5 folds at higher concentrations compared to control confirming that the paste thinned out at these concentrations.

The values of G' and G" approaching zero at higher concentrations of acids demonstrated that the paste behaves neither like elastic nor viscous fluid. In contrast, phase angle nearing 90° reflected the high tendency to form a very thin clear paste during heating-cooling cycle.

4.2.3.2.2 Hydrochloric acid

a. Viscosity

Rheological properties of the starch treated with hydrochloric acid resembled those of sulphuric acid treatment, though the severity of the effect was less pronounced in the former case. From Fig.4.16, it is clear that at lower concentration of 15 mM, viscosity values followed the same trend as that of control, but with lower values. Viscosity was found to be lowered from 0.68 Pas to 0.50 Pas at the end of holding period at 95°C. But during cooling cycle, the viscosity was higher compared to control. However, at the end of heating-cooling cycle, the values were nearly equal. The results showed that unlike sulphuric acid treatment, where the viscosity was affected during the whole cycle, reorganization was not very much influenced in case of hydrochloric acid treatment. There was a regular suppression of viscosity with increase in concentration so that at higher acid levels, the viscosity values were nearly zero, the reason being same as in the case of sulphuric acid treatment. Wurzburg (1989) quoted that cold aqueous hydrochloric acid

dissolves good portion of the granule content leaving a fragmented granular residue composed of short linear molecules, which cause reduction in viscosity. At lower concentrations, hydrochloric acid did not cause severe damage to the starch granules unlike sulphuric acid treatment, but at high concentration, the effect of both acids was similar.

b. Dynamic modulus

From Fig.4.16, it is clear that, just like viscosity pattern, there was an initial decrease in G' which recovered and overtook the values of the control during cooling cycle. As the concentration of acid increased, the G' was nearly zero. At 15 mM, the G' value decreased from 11.85 to 10.63 Pa during heating period and further reduced to 9.29 Pa during cooling to 90°C. The trend was similar to that of control sample. However, the G' values for the acid treated sample registered higher values (13.85 Pa) till the end of cooling cycle when compared to native starch (12.60 Pa) at 35°C. But at the end of cooling cycle, the G' values were almost constant (15.1 Pa), still lower than the control (15.8 pa). At other higher concentrations, G' values approached zero. The pattern of G' at 15 mM concentration confirmed that the starch quality was not affected to any noticeable extent at this level, Whereas from the viscosity graph, some detectable values obtained at 30 mM and 60 mM concentrations, these are not observed for G'.

G" values also followed the same trend as viscosity and elastic modulus. At the lowest concentration, there was a minor decrease during the initial stage and then a small increase at the latter stage. During heating cycle, G" values registered a fall from 4.29 to 3.14 Pa which is slightly lower than that of control. During cooling cycle, G" progressively increased and attained a value of 5.61 Pa compared to 3.93 Pa for the control and during holding at 35°C, there was not much variation for treated sample unlike control, for which a noticeable increase was observed. Unlike G', 30mM concentration exhibited a small G" values, but further increase in concentration resulted in nearly zero values.

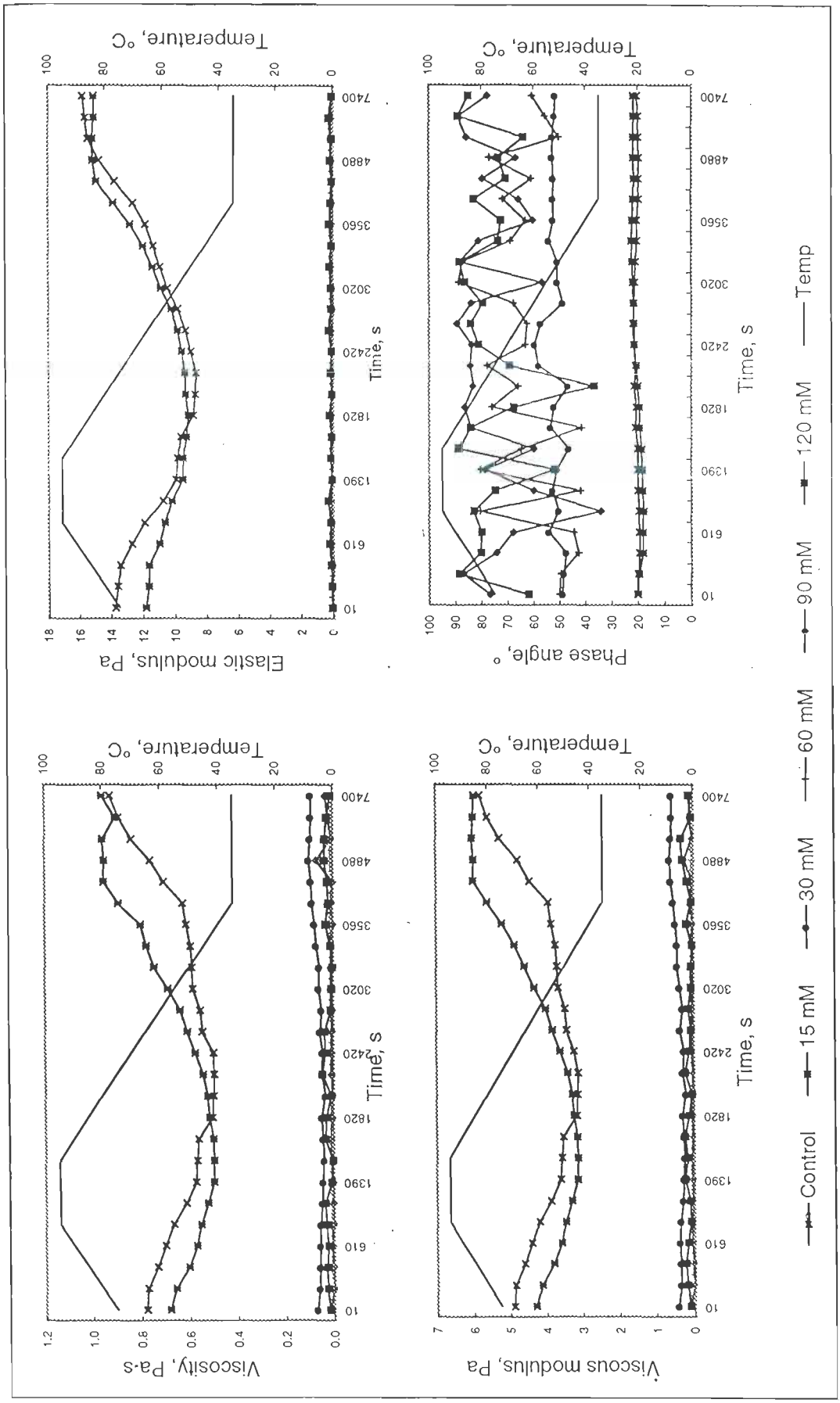


Fig. 4.16 Rheological properties of cassava starch settled in the presence of hydrochloric acid

c. Phase angle

Phase angle data for the hydrochloric acid treatment at 15 mM showed that very little change occurred and closely resembled the control values. The phase angle values changed from 19.4 to 21.80° for control and 17.95 to 22.60° for treated samples at 15 mM concentration. As expected, at higher concentrations, the values were very irregular similar to those obtained for sulphuric acid, though not to such an extent. The irregular nature of values indicated that the starch has undergone considerable breakdown leading to a very thin solution. The result again confirmed that only at 15mM concentrations starch remains unaffected.

4.2.3.2.3 Sodium hypochlorite

a. Viscosity

The data on viscosity of the starch settled using sodium hypochlorite solutions showed that there was a slight enhancement in the viscosity at the lowest concentration of 0.17 mM throughout the heating and cooling cycle, compared to control and similar trend was observed (Fig.4.17). The viscosity value steadily decreased from 0.83 Pas to 0.64 Pas at 80°C in the cooling phase and then progressively rose to 1.09 at 35°C, and then remained unaltered. For 0.35 mM concentration, though there was a small decrease during the heating cycle, it increased during cooling phase. For all other higher concentrations, viscosity was found to be lower than that of control. For higher concentrations of 0.7, 1.06 and 1.41 mM, the viscosity values were reduced by 24.4, 41.0 and 53.8% at the beginning of the heating cycle. At 35°C in the holding period, the viscosity values were 0.81, 0.56 and 0.44 Pas for the above concentrations showing a drop of 12.9, 39.8 and 52.7%, respectively compared to the control. These results clearly indicated that the oxidative action of chlorine was more noticeable at concentrations above 0.35 mM.

Drop in viscosity at higher level of hypochlorite has been well documented (Wurzburg, 1989). Farely and Hixon (1942) explained the changes taking place during oxidation of starch. During oxidation in the presence of chlorine gas, radial fissures are formed in the granule segment, the degree of which increases as the level of oxidation increases. When heated in water, instead of swelling like native starches, the granules tend to disintegrate along these fissures and fragment, causing lower viscosity at higher concentrations. But at the lower concentrations, the reagent may be acting as a salt rather than an oxidising agent, thereby elevating the viscosity levels. Many salts are known to increase the viscosity (Eliasson and Gudmudsson, 1996). The increased viscosity at lower level of hypochlorite and decreased viscosity at higher level have been attributed to the difference in pH (Wurzburg, 1989). Granule erosion which is known to enhance the viscosity predominates fragmentation at lower levels of oxidising agent, but gets reversed as the concentration increases. The hypochlorite treatment promotes solubilization of some of the impurities permitting them to be washed out of the starch. Hence, it can be assumed that at lower level, bleaching action is more pronounced and the leachate may have an influence on increasing viscosity. At higher concentrations, oxidation can lead to thinning of starch and hence reduction in viscosity.

b. Dynamic modulus

G' values followed the same trend as the viscosity, the pattern being similar to that of control except at the lowest concentration of 0.17 mM. G' values were observed to be higher at 0.17 mM and for all other concentrations, the values were lower than control. During heating to 95°C, the G' values were 12.50, 10.60 , 9.01, 6.09, 4.25 and 11.90 Pa for 0.17, 0.35, 0.70, 1.06, 1.41 mM concentrations and control, respectively. When holding the sample for one hour at 35°C the change was more pronounced for control and 0.17 mM concentrations, whereas for samples treated at high level of chlorine, the variation was negligible. The higher values of G' at 0.17 mM showed that

the paste is more elastic than viscous. But as the concentration increased, the value came down considerably showing a reduction in elastic properties of the gel. Also the higher values of G' at 0.17, 0.35 and 0.70 mM concentrations revealed that reassociation of structure takes place during cooling and subsequent holding at 35°C.

The data on G" also followed the trend of G', showing a higher value at 0.17 mM, where as for 0.35 mM concentrations, the values decreased during heating cycle, but increased in the later stage, while all other concentrations progressively recorded reduced G". At the end of heating cycle at 95°C, the G" values were increased by 12.9% and reduced by 6.2, 18.8, 38.8 and 56.2% at 0.17, 0.35, 0.7, 1.06 and 1.41 mM concentrations, respectively as compared to control. After cooling to 35°C, there was an increase of approximately 55.2, 43.0, 13.5% at 0.17, 0.35 and 0.70 mM concentrations and afterwards registered 23.2 and 41.5% reduction at the other concentrations. In these cases also, G' values were higher than G" showing the elastic tendency of the starch at all concentrations, inspite of some thinning effect at higher concentrations. Reassociation was also 3 evident at 0.17, 0.35 and 0.70 mM concentrations where higher values of G" were observed, whereas it was very low at 1.06 and 1.41 mM levels.

At the highest concentrations of 1.41 mM, during heating cycle, G' and G" values were decreased by 64 and 56% and but they were 72 and 53% lower than the control on holding at 35°C, showing more pronounced change in the elastic nature than the viscous nature.

c. Phase angle

The phase angle values for oxidised starch also followed the same trend as in acid treated and some clear trend was evident. At all concentrations, there was an increase in the phase angle, though the change was more predominant at 1.06 and 1.41 mM concentrations. There was a very little tendency for the values to decrease

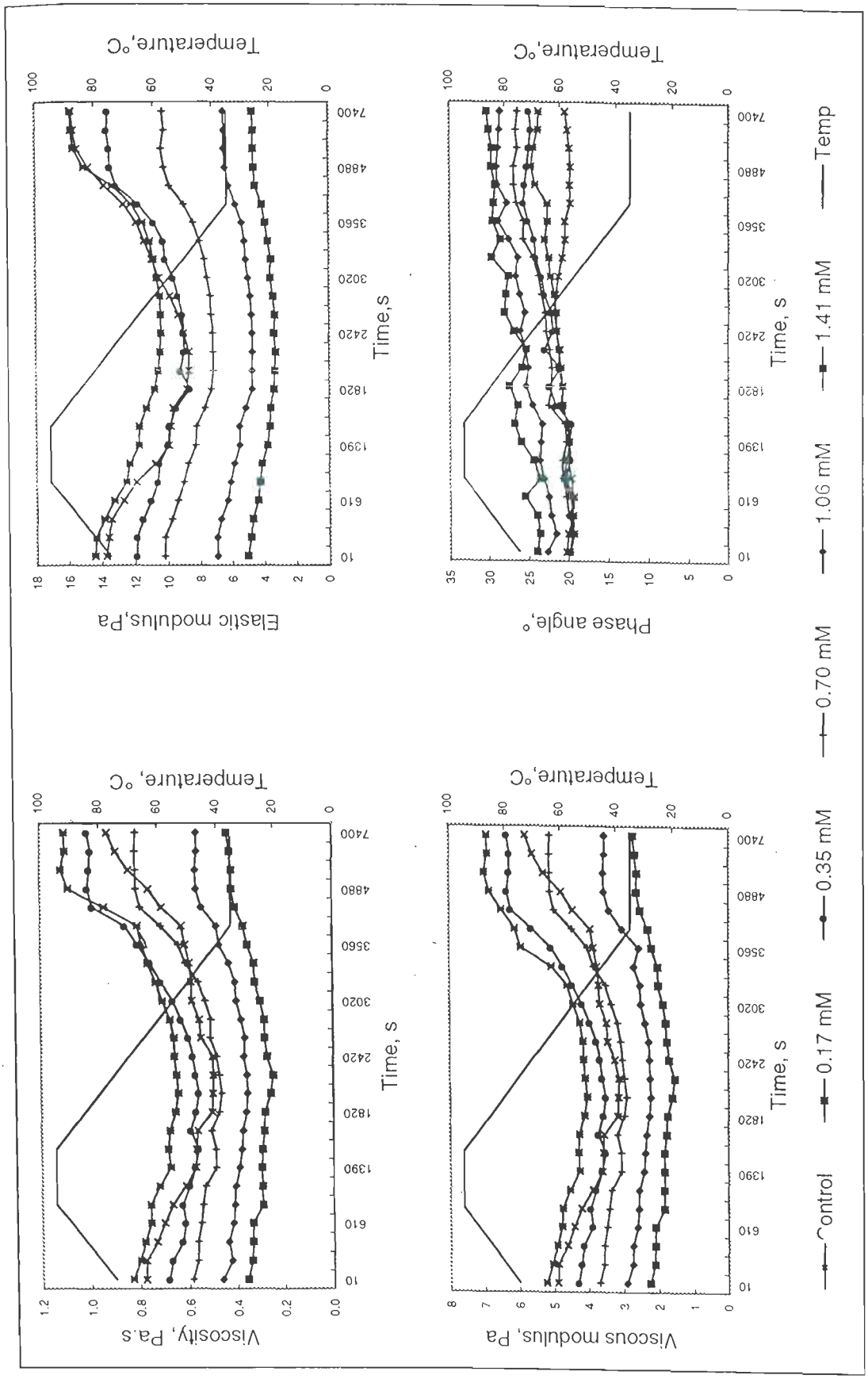


Fig. 4.17 Rheological properties of cassava starch settled in the presence of sodium hypochlorite

even during the later stages of experiments, showing the gelling tendency is affected drastically. The higher values of phase angle showed that some shift from elastic to viscous character was taking place, but the change is not much pronounced.

4.2.3.2.4 Sodium metabisulphite

The rheological properties of metabisulphite treated sample are represented in Fig.4.18 and Appendix I.

a. Viscosity

The treatment with 1.56, 3.12 and 4.88 mM brought about slight enhancement in viscosity at the cooling cycle, but 6.24 and 7.80 mM concentrations resulted in a decrease in viscosity compared to control, the values increased from 0.69 Pas for control to 0.92, 0.90 and 0.82 Pas for 1.56, 3.12 and 4.68 mM concentrations, then reduced to 0.62 and 0.40 at 6.24 and 7.80 mM concentrations, respectively. At the heating cycle, the viscosity values were decreased at all concentrations.

At lower concentrations, sodium metabisulphite may be acting as a simple salt causing an increase in viscosity, whereas at higher concentrations, it behaves more like a dilute acid affecting the viscosity properties. However, the influence was not very pronounced compared to mineral acids.

b. Dynamic modulus

G' was found to decrease with increase in concentration and the effect was more pronounced at 6.24 and 7.8 mM concentrations. At the end of heating cycle to 95°C, about 49.0 and 54.4% reduction in G' values were observed compared to control whereas at the end of holding period at 35°C, 50.3 and 61.7% reduction was observed for 6.24 and 7.80 mM concentrations showing that a noticeable thinning of starch had occurred.

As already mentioned, the acidic nature at higher concentrations may be the reason for this observation. At lower concentrations, though effect on viscosity was much less evident, effect on G' was prominent.

At higher concentrations of 6.24 and 7.80 mM, there was a considerable decrease in G" values. But at lower concentrations, in the initial stage, there was a decrease and thereafter increase over the control. At the end of heating cycle at 95°C, a reduction of 36 and 40.5% was observed with respect to control and at the end of the run, the reduction from control was only 27.2 and 40.3% for 6.24 and 7.8 mM concentrations. These values clearly indicated that elastic modulus is more affected compared to viscous modulus showing the thinning of starch.

c. Phase angle

Phase angle values were enhanced to a small extent by the treatment at all concentrations and the pattern resembled that of control sample. The phase angle for control sample ranged from 19.4 – 21.80° whereas it was 24.7 – 30.2° for the samples treated with 7.80 mM The observed increase of 10° in phase angle at the higher concentrations indicated a small decrease in strength or thinning of starch.

Unlike the acid treatment, which brought about considerable reduction in the values of rheological parameters, especially at higher concentrations, the effect of metabisulphite was much less pronounced. Metabisulphite is known to improve the whiteness of starch. Our results showed that, at the concentrations used in the study, rheological properties were not affected to any major extent proving that this reagent could be used without affecting starch quality.

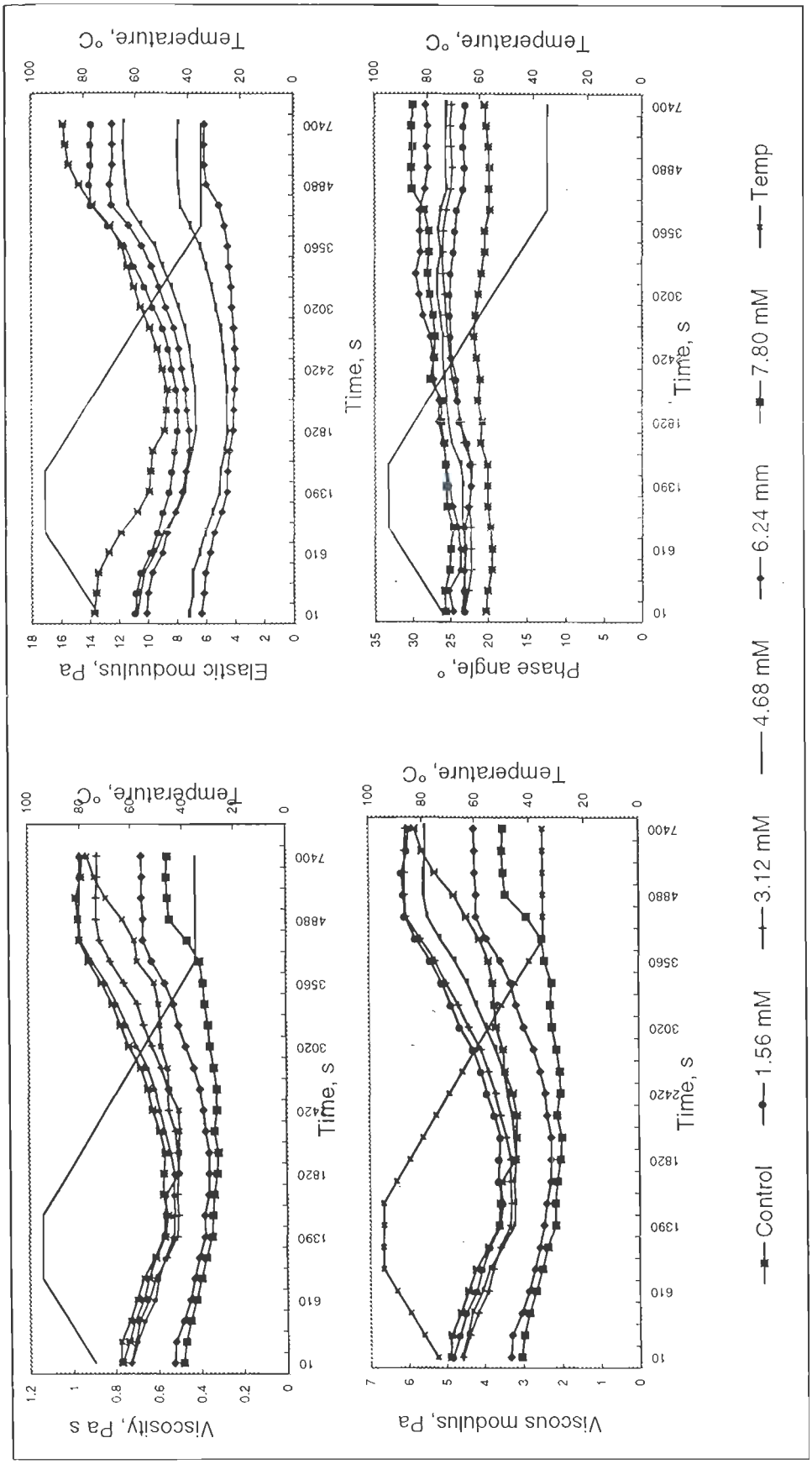


Fig. 4.18 Rheological properties of cassava starch settled in the presence of sodium metabisulphite

4.2.3.2.5 Alum

a. Viscosity

Viscosity of starch was drastically affected by alum at all concentrations (Fig.4.19 and Appendix J) The viscosity level came down about 80-85% compared to control. Breakdown as well as reassociation also did not take place as a result of the treatment. The viscosity value ranged from 0.02 to 0.12 Pas at all concentrations showing the effect is independent of the concentrations tried. This observation is contradictory with that of Radley (1976) who reported that an addition of 0.1 g/l to starch milk resulted in a 50% increase in viscosity. Alum is known to be a coagulating agent. The interaction between the swollen granules might have been reduced due to some surface phenomena occurring in the starch brought about by the alum. It is well known that the starch viscosity in mainly due to the interaction between the swollen starch granules. The poly electric nature of the starch granules (-ve) is being neutralised by the positively charged heavy atoms in alum, which may be responsible for the observed reduction in viscosity. It is also possible that alum may be affecting the water activity of the starch solution leading to reduced viscosity (Spies and Hoosner, 1982).

b. Dynamic modulus

Like viscosity, elastic modulus was also effected to similar extent by alum. At the end of heating cycle at 95°, the G' value was lesser than 1.0 when compared to 11.9 Pa for control showing a drastic reduction of over 95%. At the end of cooling cycle also the observed reduction in G' values was above 95% at all concentrations The substantial reduction in G' values brought about by the treatments again confirmed the effect of alum on the rheological properties of starch.

G" also followed the same trend as G' showing a reduction as the concentration increased. The G" value dropped from 4.20 for control to 0.36 Pa at 50 mM concentrations at the end of heating cycle at 95°C. When the samples were cooled to

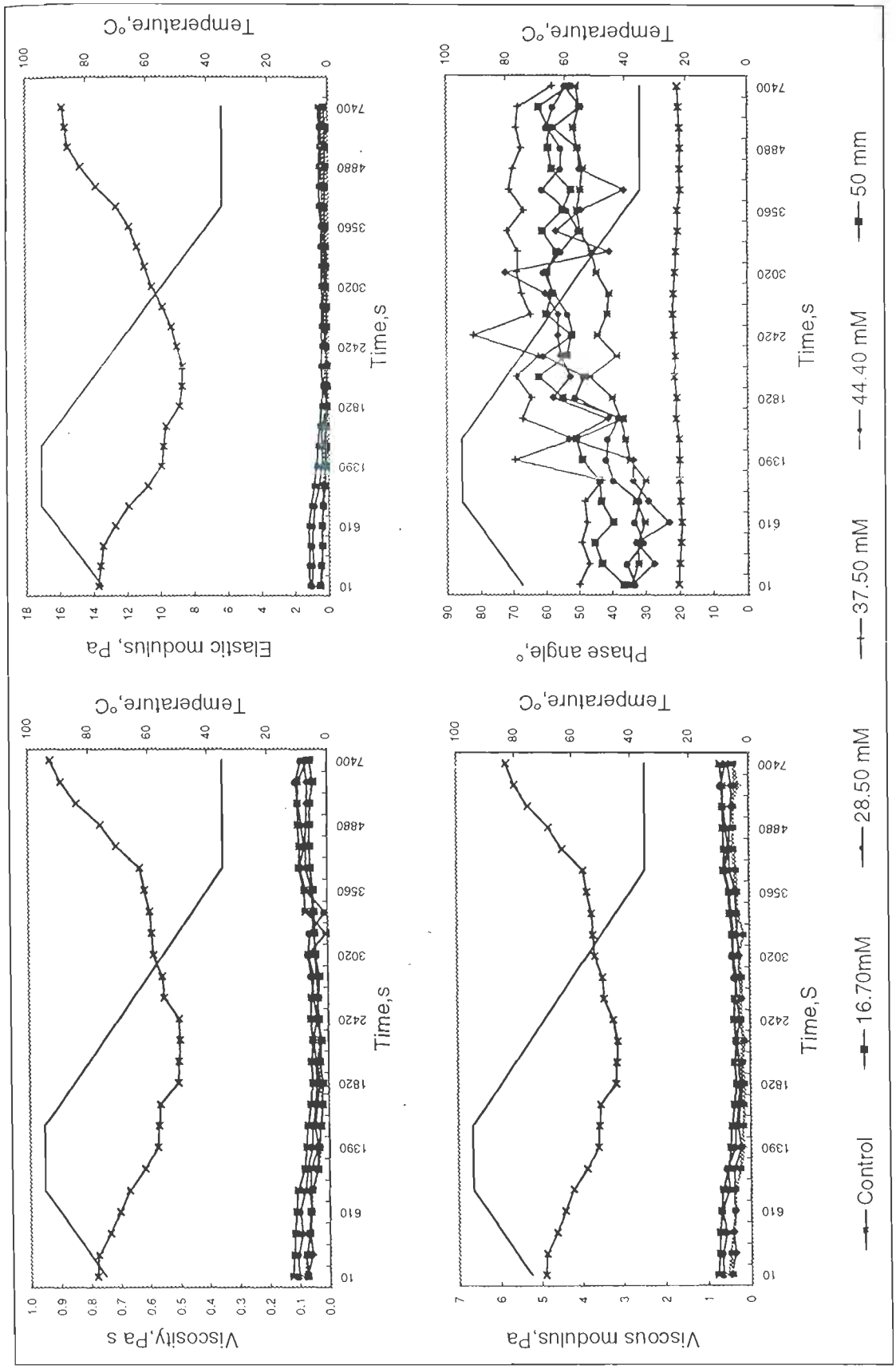


Fig. 4.19 Rheological properties of cassava starch settled in the presence of alum

35°C, the observed reduction in G" values were 84.9, 85.4, 91.1, 88.5 and 92.4% at 16.7, 28.5, 37.5, 44.4 and 50 mM concentrations, respectively. When compared to G' values, the extent of reduction was found to be almost same for G" and in effect there is preference for either viscous or elastic.

c. Phase angle

Figure 4.19 showed that the phase angle was increased by the treatment with alum. However, among the different concentrations studied, 16.70 mM concentration had less effect. The increase in phase angle was to the extent of 2-5 fold and treatment with 37.5 mM concentration seemed to reach to the maximum extent. However, there was no regular trend in all concentrations except at 16.70 mM. This also showed that whenever, G' and G" are every low, phase angle values show a lot of irregularities as observed in acid treatment. This result on treatment with alum stressed that it affects the rheological properties of starch considerably due to the high cationic nature of the salt.

4.3 Electroflocculation of Cassava Starch

Electrical conductivity of water varied with the amount of salts present in the water. Hence quality of water was assessed to find out the various salts present in the water obtained from different sources and are presented in Table 4.13.

Table 4.13 Quality of water from different sources

Type of water	pH	EC	CO_3^{-2}	HCO_3^{-1}	SO_4^{-2}	Cl^{-1}	Mg	Ca	Na	K
Bore well water	7.3	2.5	0.3	7.2	Excess	8.2	7.8	5.6	9.6	0.26
Dam water	7.5	0.2	nil	0.8	Trace	0.5	0.7	0.3	0.43	0.03

Note: EC- Electrical conductivity, mmhos/cm, CO_3^{-2}-Carbonate, $HCO3^{-1}$-Bicarbonate, SO_4^{-2}-sulphate, Cl^{-1}-Chloride, Mg –Magnesium, Ca–Calcium, Na-Sodium, K-Potassium and concentration of the salts are in milli equivalent per litre

From the table, it is clear that borewell water contains higher amount of various salts which increases the electrical conductivity of the water (2.5 mmhos/cm) when compared to dam water (0.2 mmhos/cm). This explains the delayed starting of electrolysis in dam water.

The data on the potential, current density, temperature rise and per cent starch in supernatant is given in Table 4.14.

Table 4.14 Electroflocculation of cassava starch at 4% concentration

Voltage, V	Potential, V/cm	Current, A	Current density, A/cm^2	Temperature, °C	Starch in supernatant, %
5	1.66	0.1	0.003	26.9	0.93
10	3.33	0.3	0.01	27.5	0.25
15	5.00	0.6	0.02	30.6	0.16
20	6.66	0.9	0.03	35.4	0.14
25	8.33	1.1	0.04	41.2	0.12
30	10.00	1.6	0.05	50.7	0.12
40	13.33	2.6	0.09	68.3	0.11

As voltage increased, current drawn was more as expected and it reached a maximum of 0.09 A/cm^2 at 13.33 V/cm The increase was gradual till 8.33 V/cm and afterwards registered a sharp increase. Similar trends were also observed for the temperature of the starch suspension during electrolysis. At 1.66 V/cm the temperature was same as that of ambient, but at 13.33, V/cm it was increased to 68.3°C. Amount of starch in the supernatant was about 2.4% for the control sample after 10 min of settling whereas it was reduced to a great extent by the process of electroflocculation of starch suspension. It was reduced to 0.93% at 1.66 V/cm and 0.12% at 8.33 V/cm and remained

constant thereafter showing the application of higher voltage has only slight effect on settling. However, at high voltage, the water was found to be brown coloured due to the disassociation of the electrode materials.

4.3.1 Gelatinization parameters of electroflocculated starch

The results on gelatinization temperature as observed from the DSC data indicated that the different source of water and applied voltage does not influence any of the thermal parameters to any noticeable extent (Table 4.15). The T_o values were around 61.94±0.8, 62±0.78 and 61±0.05°C for bore well, dam and distilled water treated samples, respectively, whereas control value was 61.05°C. Effect of salt is felt only to very small extent. Though the salts are known to influence the gelatinization parameters of the starch, the salt concentration in the present water samples studied are too low to induce any effect. It is also confirmed by the nearly equal values for T_e *i.e.*, 79.58 ± 0.72, 79.48 ± 1.30, 78.67±1.32°C for bore well, dam and distilled water treatment and 79.90°C for control. Similarly gelatinization enthalpy also exhibited very insignificant variation.

4.3.2 Rheological properties of electroflocculated starch

Cassava starch suspension at 4% concentration (w/v) was subjected to electrolysis using stainless steel electrodes and the results on the electrolytically settled starches in three types of water from different sources are outlined in this section (Appendix K to L).

4.3.2.1 Borewell water

a. Viscosity

The viscosity of the electrolysed starch sample in bore well water was found to be lower than that of control at all the treatments (Fig.4.20). However, among the treatments, viscosity values were slightly higher for 10 V/cm compared to 6.7 V/cm treated samples. At the beginning of the heating cycle, viscosity dropped from 0.56 Pas for control to 0.43, 0.29 and 0.35 Pas for 5, 6.7and 10 V/cm, respectively. At the end of heating cycle at 95°C,

Table 4.15 Differential scanning calorimeter (DSC) gelatinization parameters of cassava starch electroflocculated in water from different sources

Treatments	Gelatinization temperatures, °C			Gelatinization enthalpy, J/g
	T_o	T_e	ΔT	ΔH
Control (Native starch)	61.05	79.90	18.85	12.02
Bore well water				
5.0 V/cm	61.90	78.75	16.85	12.39
6.7 V/cm	62.03	79.93	17.90	12.00
10.0 V/cm	61.90	80.05	18.15	13.02
Dam water				
13.3 V/cm	62.40	79.20	16.80	12.63
16.70 V/cm	62.50	80.90	18.40	12.33
20.0 V/cm	61.10	78.35	17.25	10.32
Distilled water				
13.3 V/cm	61.00	78.80	17.80	12.36
16.70 V/cm	61.05	78.30	17.25	12.16
20.0 V/cm	60.95	78.90	17.95	13.32

T_0 = Onset gelatinization temperature, T_e = Final gelatinization temperature, ΔT =Gelatinization interval (T_0-T_e)

they were reduced to 0.47, 0.23, 0.19 and 0.19 Pas and when cooled to 35°C, the viscosity values were 0.36, 0.20, 0.18 and 0.22 Pas for the control, 5, 6.7 and 10 V/cm, respectively Culson and Richardson (1980) and Fukui and Yuu (1984) reported that due to electrolysis, oxidative reaction of the organic material took place. Oxidative reaction is responsible for reduction in viscosity (see section 4.2.3.2.3). The higher viscosity at 10 V/cm compared to 6.7 V/cm may be attributed to the disassociation of the electrode material during electrolysis which provide salts which increase the viscosity, but the oxidative action was more predominant than disassociation as the viscosity values were still lower than control. Wurzburg (1989) also reported a reduced viscosity for the starch suspension electrolysed in presence of chloride ions.

b. Dynamic Modulus

The elastic and viscous modulus also followed the same trend as viscosity showing lower values for all treated samples than control, but higher values at 10 V/cm compared to 5 and 6.7 V/cm. The percent decrease in G' values from control was 65.7, 81.8 and 77% at 75°C and 77.2, 85.4 and 76.7% at 95°C for 5, 6.7 and 10 V/cm, respectively. When the starch samples were allowed to cool to 35°C, about 73.0, 77.6 and 71.2% reduction were observed at the above condition of treatments.

The per cent drop in G" values from control were 23.2, 47.0 and 36.7% at 75°C; 50.2, 60.0 and 51.2% at 95°C and 44.7, 56.4 and 42% at 35°C for 5, 6.7 and 10 V/cm treated samples, respectively. When the sample was held at 35°C for one hour, G" values were reduced by 35.9, 19.4 and 13.1%, respectively for the above conditions.

The results indicated that G' had undergone higher reduction than G" at all treatments reflecting the tendency of the gel to shift from elastic to viscous.

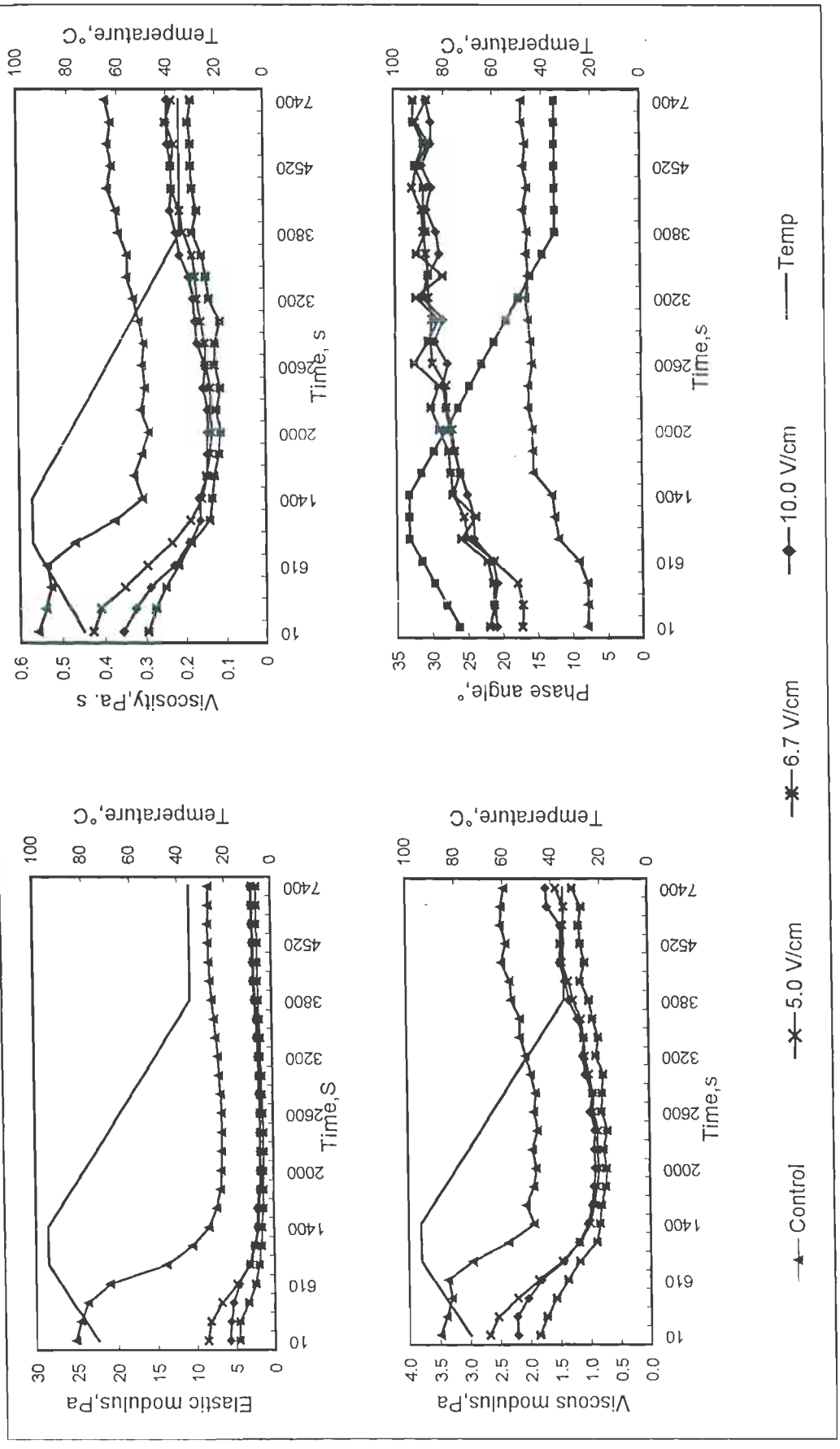


Fig. 4.20 Rheological properties of cassava starch electroflocculated in borewell water

c. Phase angle

Phase angle values followed an increasing trend compared to control, But as against other rheological parameters, it was found to be slightly lower at 10 V/cm compared to 6.7 V/cm. The phase angle values gradually increased from 7.8 to 16.9° for control whereas change in the treated sample showed an irregular values ranging from 17.1 - 32.50, 21.2-32.40 and 20.8-31.2° for 5, 6.7 and 10 V/cm, respectively. Maximum of 2-3 fold enhancement in phase angle was observed which confirmed the thinning out of the suspension. This is in agreement with higher reduction in G' and not much change for G".

4.3.2.2 Dam water

The rheological parameters of the starch sample electrolysed in dam water are depicted in Fig.4.21 and Appendix L. The absence or presence of small amount of salt present in this water caused a delay in initiating the electrolysis and hence higher potential levels were applied in this case and distilled water treatment.

a. Viscosity

The viscosity values at 13.3 V/cm were nearly equal to that of control during heating and cooling cycle except at holding for one hour where the values were slightly higher than the control. But for 16.7 and 20 V/cm, viscosity values registered a gradual decrease as voltage increased and was lower than that of control. Unlike borewell water, the gradual reduction in viscosity with increase in voltage indicated that oxidative effect due to electrolysis is more pronounced than the effect of disassociation of electrode material. The salt content in dam water is less compared to borewell water, which lead to low rate of electrolysis. The reduced oxidative action lead to higher viscosity of the sample than that of borewell water.

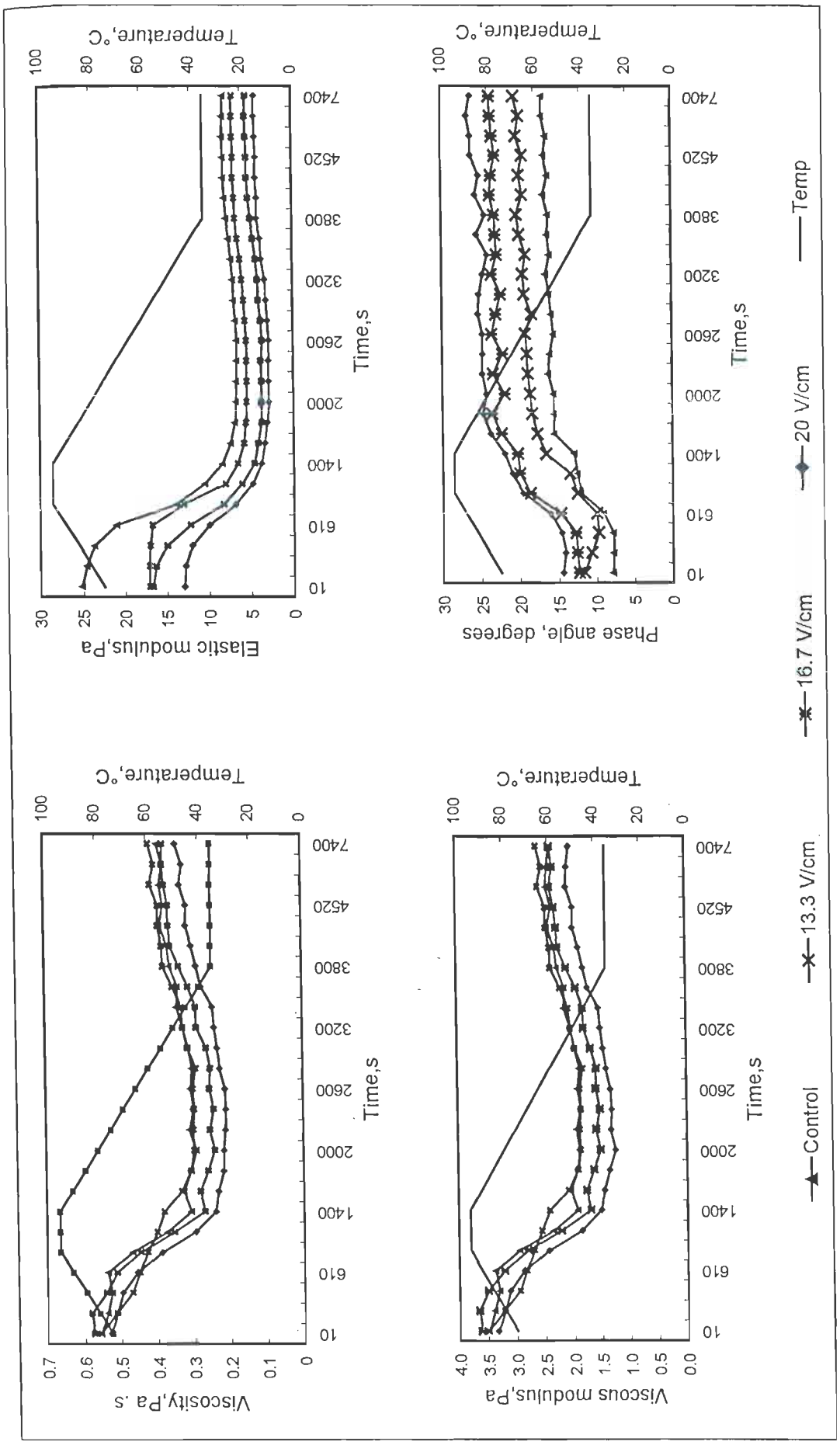


Fig. 4.21 Rheological properties of cassava starch electroflocculated in dam water

b. Dynamic modulus

Figure.4.21 indicated that G' values followed the similar pattern as control, giving rise to lower values as the voltage increased. G' values dropped from 25.2 Pa for the control to 13.0 Pa at the beginning of the heating cycle at 75°C; from 13.8 for the control to 6.86 Pa at 95°C; from 7.73 for the control to 4.15 Pa at 35°C when held for one hour at 20 V/cm. At maximum applied voltage of 20 V/cm , G' values were reduced by 48.4% at 75°C, 50.3% at 95°C, 46.3 % at cooling 35°C and 45.9% at holding 35°C. This values showed that change in G' was nearly constant.

G" values followed the same pattern as viscosity and slightly higher values were recorded at the cooling cycle for 13.3 V/cm. For the treatment with maximum voltage of 20 V/cm, G" values were reduced by 4.6% at 75°C; 17.3% at 95°C, 20.4% at 35°C cooling and 14.3% at holding at 35°C. These lower reduction in G" values indicated that viscous nature was not very much affected by the treatment. The higher level of decrease for G' values demonstrated the comparatively higher shift towards the viscous nature of the gels formed.

c. Phase angle

Unlike the treatment with borewell water, the phase angle values for the starch electrolysed in dam water followed the same trend as control, giving higher but consistent values at all the treatments. The phase angle values ranged from 7.8-16.90 for control, 9.8 to 20.5 for 13.3 V/cm, 12.3-23.7 for 16.7 V/cm and 14.1-26.2 for 20 V/cm, but these values were slightly lower than those of the borewell water even at high voltage. The higher and regular increase in phase angle values indicated a phase shift from elastic to viscous nature, but the change is not that much pronounced.

4.3.2.3 Distilled water

The visco-elastic character of the starch sample electrolysed in distilled water is depicted in Fig.4.22 and Appendix M.

a. Viscosity

Viscosity values followed the same trend as that of borewell water *i.e.*, values were lower than that of control, but they registered a minor increase at 20 V/cm compared to 16.7 V/cm. The viscosity values for control, 13.3, 16.7, and 20 V/cm were 0.56, 0.55, 0.53 and 0.55 Pas at 75°C; 0.36, 0.34, 0.31 and 0.31 Pas at 35°C cooling; 0.39, 0.40, 0.34 and 0.37 Pas at 35°C holding. These values were found to be higher than that of borewell water, but nearly equal to that of dam water. It indicated that even at low voltage, borewell water having more salt content permits higher rate of electrolysis, which led to more oxidation of starch and thereby lowered the viscosity of the starch samples.

b. Dynamic Modulus

G' values followed the same trend as control and resembled dam water treated samples. However, at 20 V/cm, there was an increase in G' during the heating cycle compared to 16.7 V/cm and then remained steady. At the maximum applied voltage of 20 V/cm, G' values were reduced by 34.9% at 75°C, 33.8% at 95°C, 37.3% at 35°C cooling, 31.7% at 35°C holding and the changes followed a constant pattern.

G" values were found to be lower than that of control at all the conditions of the experiments except at the last part of the cooling cycle. At the highest voltage 20 V/cm, the G" values were reduced from control by about 3.4% at 75°C, 13.6% at 95°C, 13.7% at 35°C cooling, and 5.5% at 35°C holding.

When compared to G' values, where the drop from the control was about 30-37%, G" values were reduced only by 3-15% showing more effect on the elastic nature and undergoing shift towards viscous characteristics.

c. Phase angle

Phase angle values changed in the same fashion as that of control, but with increased values. Unlike borewell water, nearly consistent values were recorded for the samples electrolysed with distilled water. The phase angle values ranged from 7.8-16.90, 9.6-20.4,

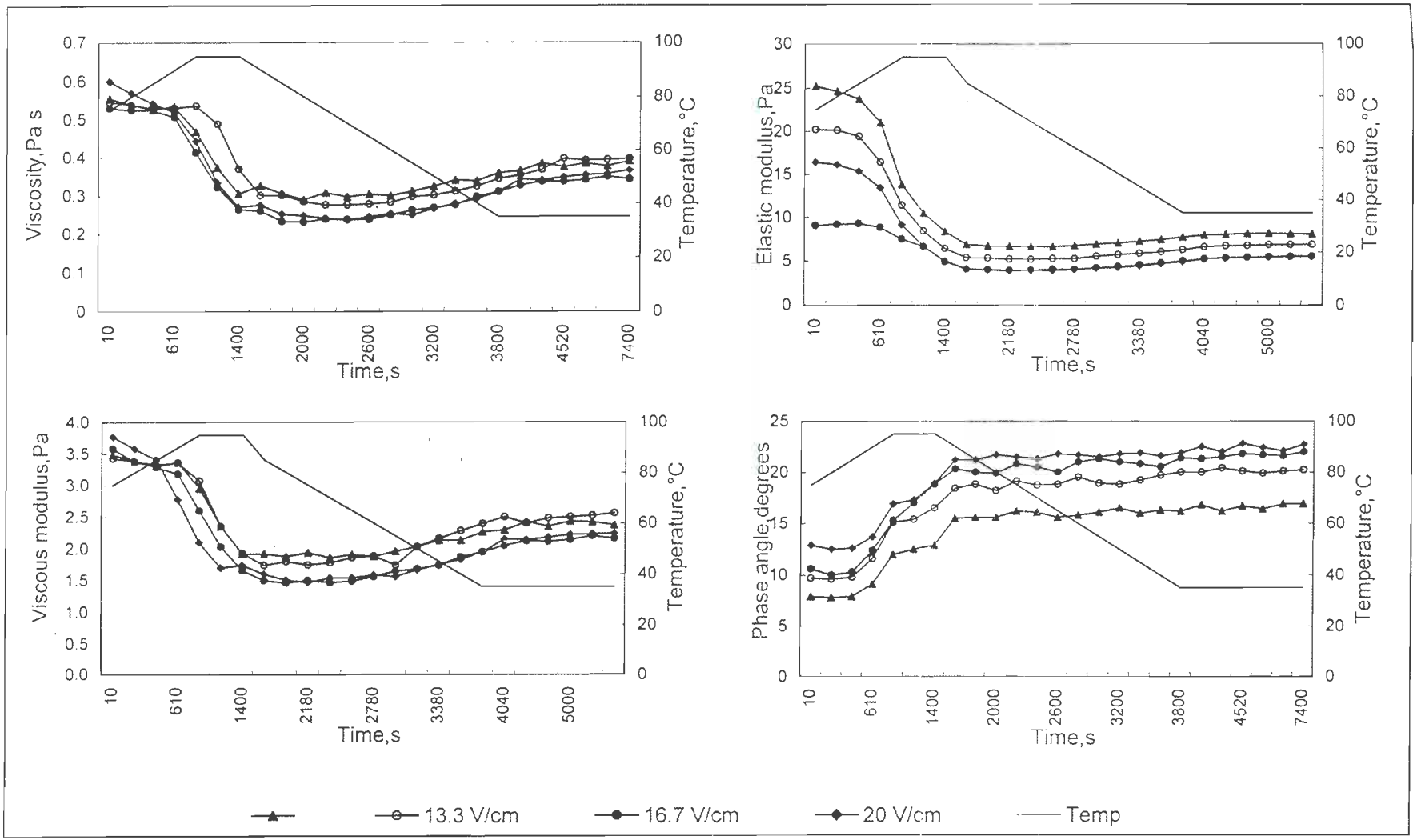


Fig.4.22 Rheological properties of cassava starch electroflocculated in distilled water

10-22, 12.5–22.8° for control, 13.3, 16.7 and 20 V/cm treated samples, respectively. This is slightly lower than that of dam water and borewell water. The higher value of phase angle by the treatment confirmed that phase transition has occurred due to the process.

4.4 Effect of Inclined Column on Settling

Effect of inclination of the bottom surface of the container on settling of cassava starch was studied for different angle (0-40°) and feed concentrations (2-8%). As settling progressed, a gradation of concentration occurred in the settling column with depth and hence per cent of starch retained in supernatant suspension (50 ml) was measured to compare the results which is presented in Fig.4.23 and Appendix N.

From the figure, it is clear that as inclination increased, the amount of starch retained in the supernatant was reduced, indicating more settling of starch. For higher concentration, effect of inclination was found to be more. When the inclination was raised from 0 to 40°, per cent starch in supernatant was decreased from 5.62 to 3.33, 6.5 to 4.0, 8.5 to 5.5 and 16.5 to 9.5% for 2, 4, 6 and 8% suspension concentration. The graph showing the effect of inclination could be divided into three segments *i.e.*, upto 20°, there was a progressive drop, from 20 to 30° values are slightly reduced or almost constant and at 40°, a sharp decrease started.

With increase in starch concentration, per cent starch retained was also increased due to more interaction between the cohesive starch particles causing reduced settling velocity of particles. This was observed for all the angles whereas effect of inclination was found to be more pronounced only at the higher concentrations.

When a suspension is taken in a vertical column, particles move downward under gravity and settle at the bottom with its settling velocity. If the bottom surface is inclined, particle have a tendency to settle on the downward facing surface, along with the bottom surface due to the velocity component of the particle in horizontal and vertical directions.

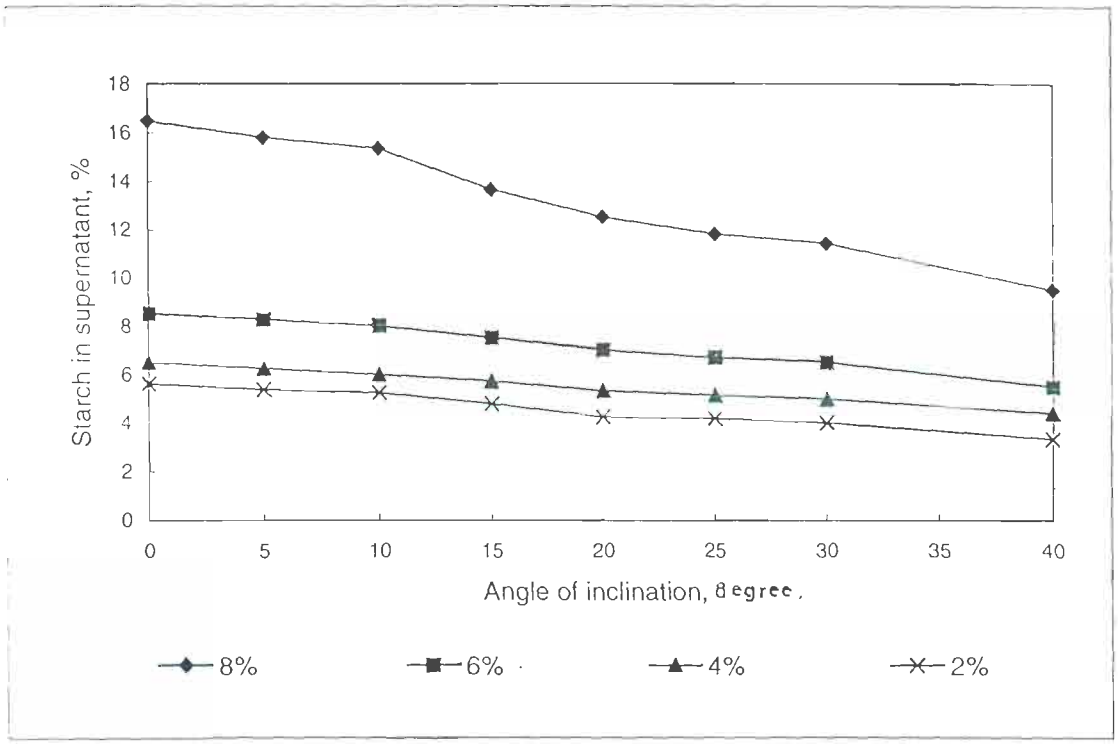


Fig.4.23 Effect of inclined column on settling of cassava starch

When particle comes in contact with the inclined surface, naturally they slide down to achieve a stable position at the bottom. So this settling along with the natural settling at the bottom account for the higher settling rate or less amount of starch in the supernatant liquid over an inclined bottom surface. Pearce (1962) also observed an accelerated rate of settling in an inclined tank by inserting a series of inclined plates.

ANOVA table (Table 4.16) clearly indicated that angle, concentration and their interaction have significant effect on the starch settling ($P \leq 0.01$).

4.4.1 Mathematical model

The graph relating the angle of inclination and starch in supernatant water almost followed a straight line relationship and hence the data was fitted into an equation of the form $S = A + B\theta$ and for different concentrations, they are as follows:

For 2% concentration:	$S = 5.7 - 0.059\,\theta$	($r^2 = 0.98$)	....4.14
For 4% concentration:	$S = 6.4 - 0.053\,\theta$	($r^2 = 0.99$)	....4.15
For 6% concentration:	$S = 8.6 - 0.076\,\theta$	($r^2 = 0.99$)	....4.16
For 8% concentration:	$S = 16.66 - 0.181\theta$	($r^2 = 0.98$)	....4.17

where,

S = starch in supernatant, %

θ = angle of inclination of floor, degrees

From the above equations, the value of A and B was found to be increased with increase in concentration and could be correlated with concentration (C, per cent) as :

$$A = [-0.0023\,(C-0.832)^2 + 0.179]^{-1} \qquad (r^2 = 0.99) \qquad ....4.18$$

$$B = [-0.60\,(C-3.33)^2 + 18.28]^{-1} \qquad (r^2 = 0.98) \qquad ....4.19$$

The general equation governing the effect of inclined column on settling of cassava starch was :

$$S = A - B\theta \qquad \ldots.4.20$$

where, $A = [-0.0023\ (C-0.832)^2 + 0.179]^{-1}$

$B = [-0.60\ (C-3.33)^2 + 18.28]^{-1}$

Table 4.17 gives the percent deviation of the predicted values from the observed values using the above said equation. The equation could very well be suited to model the effect of inclination on settling with in an error of ±5%

4.5 Centrifugal Settling of Cassava Starch Suspension

The results of centrifugal force on cassava starch settling at various concentrations are collected and depicted in Fig.4.24 and Appendix O.

When the centrifuge is rotated at 500, 1000 and 1500 rpm , the peripheral speed (tangential velocity) of the rotor was found to be 7.72, 15.44 and 23.16 m/s by considering the average radial distance of the particle from the rotor's axis as 14.74 cm (Appendix P).

4.5.1 Settling characteristics at 7.72 m/s peripheral speed (500 rpm)

From Fig.4.24, it is clear that percent starch settled increased with increase in concentration and speed. At a rotor peripheral speed of 7.72 m/s , the rate of settling was gradually increased upto 45 s and then registered a sharp increase upto 90 s and at the latter stages, it gives almost constant values except at 2% concentration where it progressively increased upto 120 s. After first 15 s under centrifugal field, about 89.13, 93.33, 94.41 and 95.26% of the starch present in the feed with 2, 4, 6 and 8% concentrations were settled. It was slightly increased to 91.23, 94.50, 95.25 and 96.44% after 45 s for 2, 4, 6 and 8% concentrations, respectively . After 120 s it was raised to 96.50, 97.56, 98.13, 98.44%, respectively and after 180 s, *i.e.,* at the end of the process, the quantum of starch settled was 97.25, 97.81, 98.45 and 98.59% for 2, 4, 6 and 8%, respectively.

Table 4.16 ANOVA table for the effect of inclined column on starch settling

SV	DF	SS	MS	F
Treatment	31	849.27	27.40	71.12**
Angle, A	7	85.59	12.23	31.74**
Concentration, C	3	735.68	245.23	636.62**
A x C	21	28.00	1.33	3.46**
Error	32	12.38	0.39	
Total	63	861.60		

** Significant at 1% level

Table 4.17 Per cent deviation of predicted values from observed values using model for inclined column settling.

Angle of inclination, degree	Per cent starch in supernatant							
	Concentration, %							
	2		4		6		8	
	O	P	O	P	O	P	O	P
0	5.62	5.7	6.5	6.43	8.5	8.55	16.5	16.72
	(1.40)		(1.08)		(0.59)		(1.33)	
5	5.38	5.4	6.25	6.15	8.21	8.20	15.81	15.77
	(0.37)		(1.60)		(0.12)		(0.25)	
10	5.25	5.11	6.0	5.87	8.0	7.84	15.35	14.82
	(2.66)		(2.17)		(2.00)		(3.40)	
15	4.8	4.82	5.73	5.59	7.52	7.49	13.65	13.87
	(0.42)		(2.44)		(0.40)		(1.60)	
20	4.25	4.53	5.34	5.37	7.0	7.13	12.51	12.92
	(6.50)		(0.56)		(1.80)		(3.28)	
25	4.19	4.24	5.15	5.03	6.7	6.78	11.8	11.97
	(1.19)		(2.33)		(1.19)		(1.44)	
30	4.0	3.95	5.0	4.75	6.5	6.42	11.42	11.02
	(1.25)		(5.00)		(1.27)		(3.50)	
40	3.3	3.36	4.4	4.19	5.5	5.71	9.5	9.12
	(1.80)		(4.70)		(3.82)		(4.17)	

O = Observed value P = Predicted value

* The values in paranthesis gives the % deviation

4.5.2 Settling characteristics at 15.44 m/s peripheral speed (1000 rpm)

When the tangential velocity was increased to 15.44 m/s, there was a sharp increase in the percent settling of starch upto 120 s. About 94.75, 95.33, 96.42, 97.03% of the starch was settled after 15 s; after 120 s, 97.88, 98.25, 98.52, 98.6%; and after 180 s, 98.43, 98.5, 98.65, 98.62% starch was settled in 2, 4, 6 and 8% concentrations, respectively.

4.5.3 Settling characteristics at 23.16 m/s peripheral speed (1500 rpm)

Unlike other speeds, for 23.16 m/s, the values of percent starch settled registered a sharp increase upto 60 s and then gradual increase upto 120 s and almost constant values thereafter. After 15 s of settling, 95.25, 95.94, 97.08 and 97.60% starch was settled for 2, 4, 6 and 8% concentrations and after 180 s, this quantity was 98.43, 98.56, 98.79 and 98.82%, respectively for the above concentrations.

Amount of starch settled *i.e.,* settling rate was found to increased with increase in peripheral speed of the rotor and concentration. Settling velocity of a spherical particle in centrifugal field is given by the equation (Earle,1985):

$$V_f = \frac{D^2 n^2 R(\rho_s - \rho_f)}{1640\mu_f} \qquad \ldots.4.21$$

where, D = diameter of particle, m; n= speed of rotation, rpm, R = radius of rotation, m; ρ_s = density of particle, kg/m^3 ; ρ_s = density of fluid, kg/m^3; μ_f = viscosity of fluid, Pa.s. By substituting the values in the above equation, settling velocity of the particle in centrifugal field for different speeds of rotation was obtained as 0.007, 0.028 and 0.063m/s for 500,1000 and 1500 rpm, respectively. From the equation, as speed increased, angular velocity of the particle increased and inturn settling velocity.

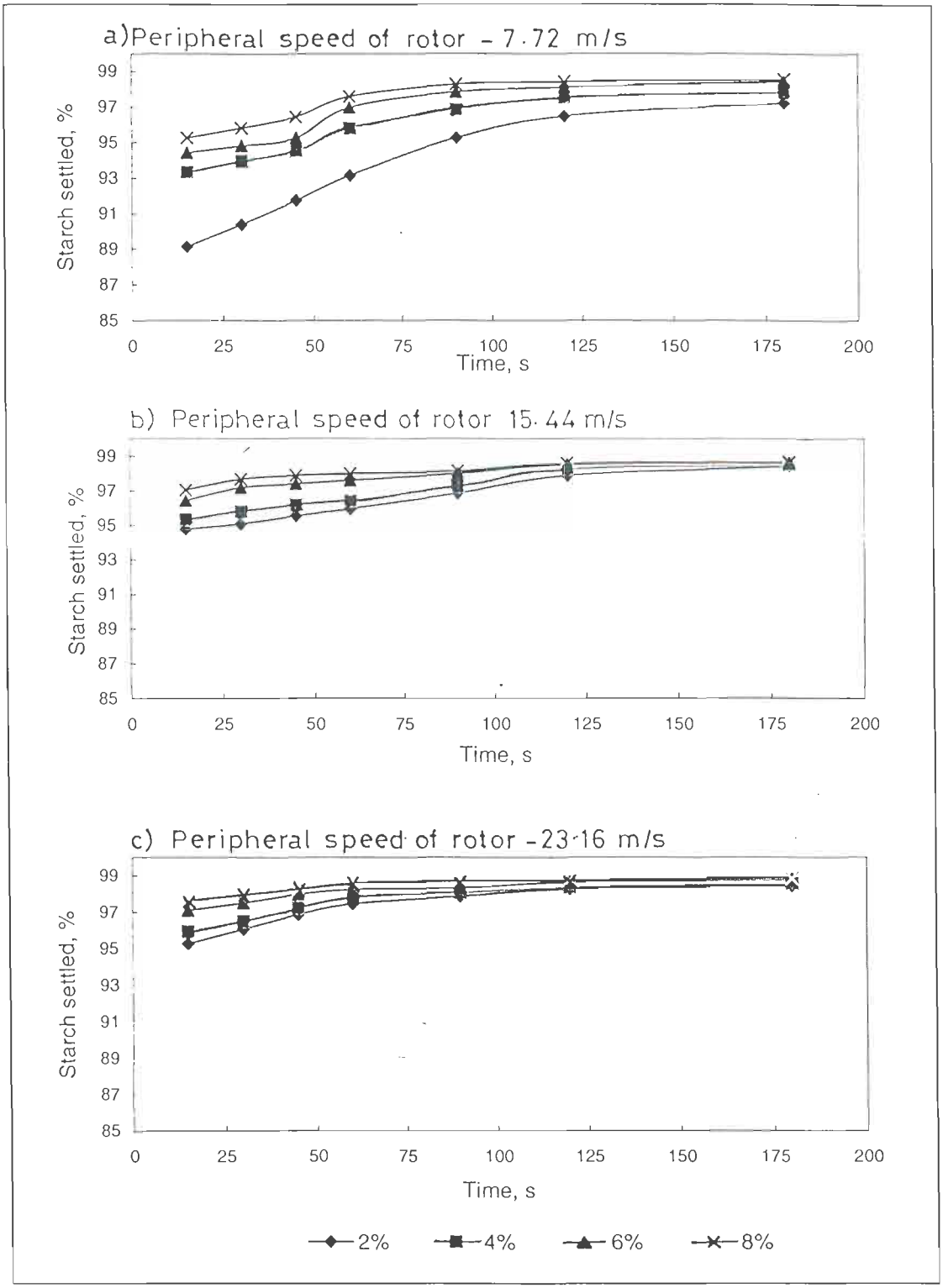


Fig.4.24 Effect of centrifugal force on settling characteristics of cassava starch

From Fig.4.24, it is seen that as the velocity increased, there was a progressive increase in quantity of starch settled upto15.44 m/s and after that, the increase was not that much pronounced especially at lower concentrations. At 2% concentrations, when the speed was increased from 7.72 to 15.44 m/s after 15 s of settling, the quantity of starch settled was increased from 89.13 to 94.75% whereas at 23.16 m/s peripheral speed, it was increased to 95.25% only. Similar trends were also observed for other concentrations, but the variation was less. This may be due to difference in settling velocity experienced by the particles in the centrifugal field. When the peripheral speed increased from 7.72 to 15.44 m/s, there was about 4 time increase in the settling velocity whereas when it changed from 15.44 to 23.16 m/s , the increase was about 2.25 times. This decreased rate in increasing settling velocity is attributed to the above pattern of settling.

When the duration of settling was increased, there was not much variation in the total amount of starch settled with respect to speed and concentration. After a lapse of 180 s settling time, the quantity of starch settled was 97.25, 97.81, 98.45 and 98.59% at a speed of 7.72 m/s; 98.43, 98.50, 98.62 and 98.67% at 15.44 m/s and 98.43, 98.56, 98.79 and 98.82% at 23.16 m/s for 2, 4, 6 and 8% concentrations, respectively.

The higher percentage of starch settled at higher concentration is due to the presence of more numbers of starch particles per unit volume leading to more interaction between the particles causing them to form aggregates of increased size. As size of the particle increased, obviously settling velocity increases and more amount of starch will settle. At higher concentration combined with high speed, most of the starch was found to be settled in the initial phase of settling itself and further increase was very less *i.e.*, for 6% concentration for the last 120 s, the increase was only 0.58% and for 8%,it was only 0.24% This indicated the more influence of speed than concentration.

Table 4.18. ANOVA table for centrifugal settling of cassava starch

SV	DF	SS	MS	F
Treatment	83	587.18	7.07	24.96**
Time (T)	6	225.73	37.62	132.73**
Concentration (C)	3	124.07	41.36	145.91**
Speed (S)	2	127.35	63.67	224.64**
T x C	18	26.93	1.50	5.28**
T x S	12	42.18	3.51	12.40**
C x S	6	33.57	5.59	19.74**
T x C x S	36	7.36	0.20	<1
Error	84	23.81	0.28	
Total	167	610.99		

** Significant at 1% level

ANOVA table revealed that concentration, settling time and speed of rotation had higher influence on centrifugal settling of cassava starch ($P \leq 0.01$) and so is the case with their interactions.

4.5.4. Centrifugal settling of fresh starch

Experiments were conducted to compare the effects of centrifugal force on fresh and dried starch settling and no noticeable change in settling characteristics were observed as indicated from the percent of starch settled after different time intervals (Fig.4.25 and Appendix Q).

It was seen that fresh and dried starch followed the same trend and at higher velocities, the percent starch settled curves overlap with each other indicating no difference in settling velocity. After 15 s under the action of centrifugal force (7.72 m/s peripheral velocity) about

93.33 and 95.26% of dried starch particles were settled in 4 and 8% concentrations whereas for fresh starch, it was about 93.25 and 95.18%, for 3.92 and 8.10% concentrations, respectively. At the end of the experiments *i.e.*, after 180 s, 97.81% of dried starch present in the 4% concentration and 97.89% of fresh starch present in the 3.92% concentrations was settled out, whereas at 8% concentration, 98.59% of settling was completed for dried starch and 98.42% for raw starch having 8.1% concentration.

For 15.44 m/s, about 95.33% and 97.03% starch was settled after 15 s for dried starch at 4 and 8% concentrations , and for fresh starch, these figures amount to be 95.09 and 97.09%, respectively for 3.92 and 8.10% concentrations . After 180 s about 98.25 and 98.67% of the fresh starch was settled in 3.92 of 8.10% suspensions where as for dried starch, it was 98.50 and 98.67% for 4 and 8% concentrations, respectively.

For 23.16 m/s, from the graph, it was clear that the curves for raw and dried starch at both concentrations were overlapping each other indicating absolutely no difference in settling behavior of these starches at this velocity. After 15 s, 95.94% of the dried starch was settled where as for fresh starch, it was 95.95% for about 4%. For 8% concentration, the amount of starch settled was 97.60 and 97.65% for dried and fresh starch, respectively. At the end of the experiment, *i.e.* after 180 s, for about 4% concentration, 98.56% of dried starch and 98.60% of the fresh starch was settled where as for 8% concentration, it amount to be 98.82 and 98.80% for dried and fresh starch.

The data on centrifugal settling of fresh and dried starch showed that both starches behaved in the same manner in a centrifugal field and at higher velocity, the settling behaviour is more or less same for fresh and dried starches of same concentration. This is contradictory to the observations made in gravitational settling, where raw starch took more time for settling due to the hydration-repulsion effect. But in centrifugal field, it is possible for the centrifugal force to overcome all these effects of dispersive forces and very

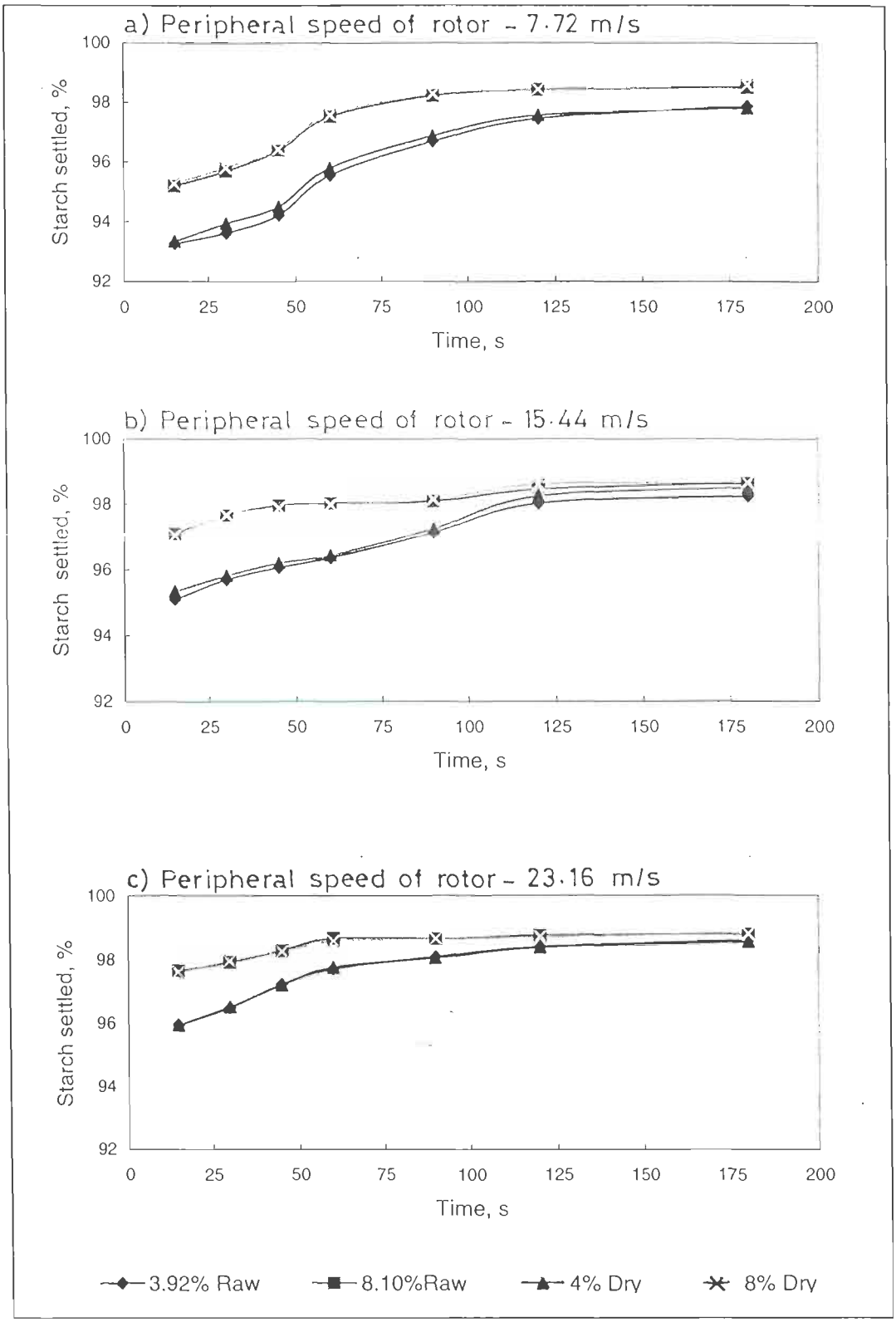


Fig.4.25 Comparison of settling characteristics of fresh and dried cassava starch under the influence of centrifugal force

compact cake was formed by centrifugal action as surface tension forces in the fine pores which causes retention of moisture was overcame by the centrifugal force. This will not be possible by gravitational forces (Coulson and Richardson, 1980).

4.6 Hydrocyclones

The hydrocyclones were fabricated with designed dimensions using 22 gauge galvanized iron sheet and the inlet, vortex finder and lid were soldered to the main body. They were tested at various feed concentrations and pressures; readings on underflow (U/F) rate and concentration; overflow (O/F) rate and concentration were noted. The hydrocyclone performance represented by underflow volume split (U_{VS}) , increase in U/F concentration with respect to feed (C_i), total efficiency (E_T), and reduced efficiency (E_R) were calculated for each hydrocyclone and depicted in Figs. 26 to 29. The performance parameters of hydrocyclones are also given in Appendix Q and R.

4.6.1 30 mm hydrocyclone

4.6.1.1 Underflow volume split (U_{VS})

U_{VS} is measured as the ratio of the flow rate of the U/F stream to that of feed stream and it gives an idea about quantum of overflow stream coming out of the hydrocyclone which can be reused for pulping operation provided if it contains minimum solids. The present study showed that feed concentration and pressure had very little effect on Uvs for the operating range tested (Fig 4.26a). The U_{VS} data ranged from 5.06 to 6.13 for 2% , 4.95 to 7.41 for 3.5% , 6.50 to 7.36 for 5% , 6.98 to 8.92 for 6.5% and 7.77 to 9.09% for 8% concentrations, respectively. From these values, it is clear that about 90-95% of the suspension will be coming out as overflow stream and good amount of this water can be saved and recycled provided the overflow contains minimum starch content. The results further revealed that , in general, increase in feed pressure and concentration decreased the over flow, however, the effects were very less when compared to the other performance parameters .

4.6.1.2 Increase in underflow concentration (C_i)

Increase in underflow concentration (C_i) was found to be increased with increase in feed pressure but followed a decreasing trend with increase in feed concentration (Fig.26b). A maximum of 11.12 fold increase in concentration was obtained with 2% feed concentration fed at 3kg/cm^2 operating pressure where as minimum of 1.91 times increase in concentration was recorded for 8% concentration at 0.6 kg/cm^2 feed pressure.

Though C_i was enhanced with pressure, the change was less marked at high pressures. For 2% feed concentration, the U/F concentration was increased by 4.00, 6.83, 9.26, 10.66 and 11.12 times at 0.6, 1.2, 1.8, 2.4 and 3 kg/cm^2 operating pressures. It also showed that for the increase in feed pressure from 0.6 to 2.4 kg/cm^2, the C_i increased at a decreasing rate at all pressures. But when the feed pressure increased from 2.4 to 3.0 kg/cm^2, the increase in C_i value was only 0.46 times more than at 2.4 kg/cm^2 feed pressure *i.e.,* the rate of decrease was much higher than the other values. The U/F concentration was enhanced by 3.65, 4.95, 5.95, 6.79 and 7.28 times for 3.5% feed concentration, 3.10, 3.86, 5.40, 6.28 and 6.60 times for 5%, 2.22, 3.51, 4.33, 5.03 and 5.49 times for 6.5% and 1.91, 2.98, 3.53, 4.07 and 4.18 times for 8% concentrations for the operating pressures of 0.6, 1.2, 1.8, 2.4 and 3.0 kg/cm^2, respectively. In all these cases, increase in U/F concentration was pronounced upto 2.4 kg/cm^2 and thereafter pressure effect was less marked.

When the concentration was increased, C_i value showed a declining trend. At the lowest operating pressure of 0.6 kg/cm^2 studied , U/F was increased by 4.00, 3.65, 3.10, 2.22 and 1.91 times and at maximum pressure of 3 kg/cm^2, the increase was 11.12, 7.28, 6.60, 5.49 and 4.18 folds at 2, 3.5, 5, 6.5 and 8 % feed concentrations, respectively.

When compared the effect of feed pressures and concentration, the pressure had a pronouncing role in deciding the U/F concentration than that of concentration, though their effect was entirely different from each other. Feed pressure had a direct effect on U/F concentration whereas feed concentration had an inverse relationship with U/F concentration.

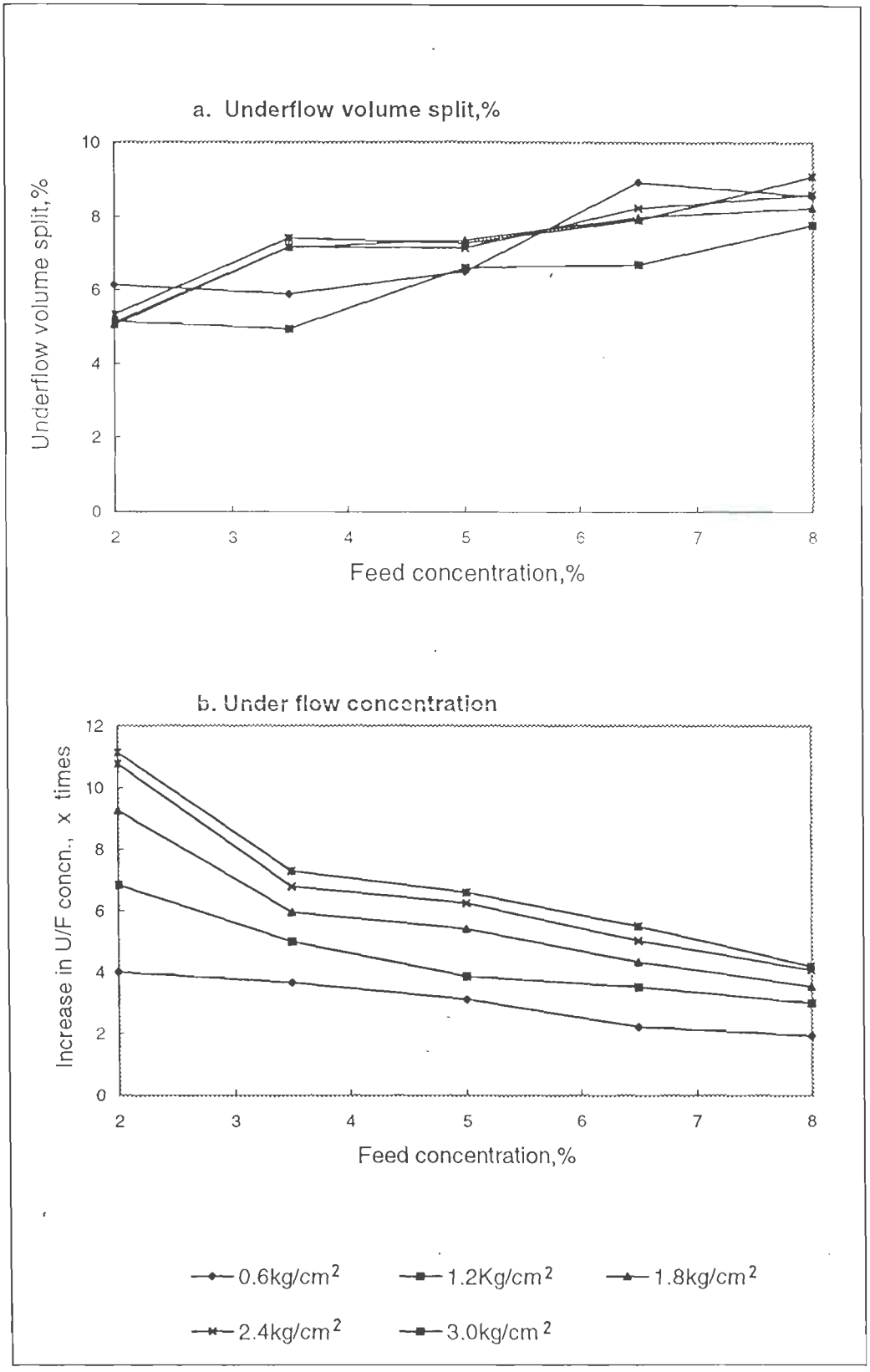


Fig.4.26 Performance of 30 mm diameter hydrocyclone operated at different feed pressures

4.6.1.3 Efficiency

It was found that both total and reduced efficiencies were increased with increase in feed pressures and decrease in concentrations (Fig 4.27). Here, only E_R values are discussed as it is the more realistic term to characterise the hydrocyclone performance. E_R values were calculated as 26.13, 6.98, 49.33, 57.42 and 62.65% for 2% concentration; 22.83, 25.98, 45.97, 52.40 and 58.24% for 3.5% concentration; 21.56, 27.30, 43.24, 48.06, 51.73 for 5% concentration; 21.73, 26.35, 37.49, 45.17 and 47.62% for 6.5% concentration and 17.87, 25.10, 31.68, 38.32, 41.79% for 8% concentration at 0.6, 1.2, 1.8, 2.4 and 3 kg/cm^2 operating pressures, respectively.

When the concentration was increased from 2 to 8%, E_R values were reduced from 26.13 to 17.87% at 0.6 kg/cm^2, 36.98 to 25.10 % at 1.2 kg/cm^2, 49.33 to 31.68% at 1.8 kg/cm^2, 57.42 to 38.32 % at 2.4 kg/cm^2 and 62.65 to 41.79 % at 3 kg/cm^2. From these values of E_R, it is clear that the effect of feed pressure on E_R is more than that of concentration.

4.6.2 50mm hydrocyclone

The performance of 50 mm diameter hydrocyclone was similar to that of 30 mm hydrocyclone but with decreased values for all parameters studied, showed a retarding influence of diameter on hydrocyclone performance.

4.6.2.1 Underflow volume split (U_{VS})

During the experiment the U_{VS} values varied from 6.53 to 7.12, , 6.30 to 6.97 , 6.34 to 7.31, 6.56 to 7.14, 6.28 to 7.57% at 2 , 3.5, 5, 6.5 and 8% feed concentrations, respectively for the range of operating pressures studied. The results are depicted in Fig.28a and it clearly indicated that U_{VS} was not much affected either by pressure or by concentration of the feed suspension.

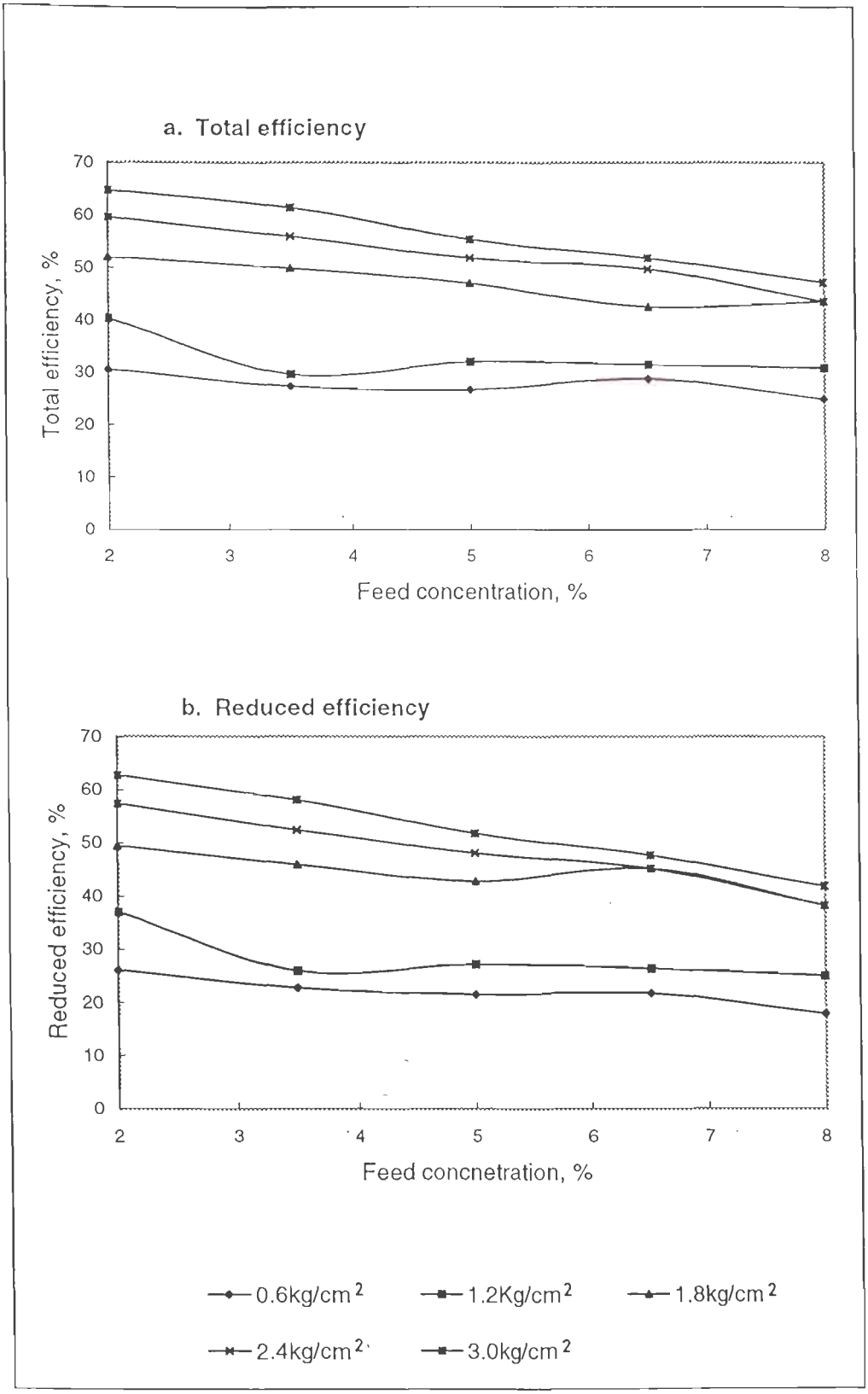


Fig.4.27 Efficiency of 30 mm hydrocyclone operated at different feed pressures

4.6.2.2 Underflow concentration

C_i increased with increasing pressures and decreased with feed concentrations as in the case of 30 mm system (Fig.28b). At 2% concentration, the U/F concentration was increased by 1.6 times at 0.6 kg/cm^2 and 3.63 times at 3kg/cm^2. The C_i value was increased from 1.29 to 3.44 for 3.5%, 1.19 to 2.36 for 5%, 1.03 to 2.02 for 6.5% and 0.98 to 1.75 for 8% feed concentrations when the feed pressures were varied from 0.6 to 3.0 kg/cm^2 When the feed concentration was increased from 2 to 8%, C_i was found to be decreased from 1.60 to 0.98, 2.34 to 1.13, 3.00 to 1.37, 3.33 to 1.51 and 3.63 to 1.75 for 0.6, 1.2, 1.8, 2.4 and 3.0 kg/cm^2, respectively. The variation in increase in U/F concentration was more with feed pressures than with feed concentration.

4.6.2.3 Efficiency

The total and reduced efficiencies were also found to be increased with pressures and decreased with concentrations. But the values were very low when compared to that of 30 mm hydrocyclone (Fig.29a and b). The E_T values were changed from 18.46 to 31.09, 15.94 to 28.36, 13.90 to 24.53, 13.33 to 21.59 and 12.43 to 18.99% at 2.0, 3.5, 5.0, 6.5 and 8% feed concentrations when the operating pressure was increased from 0.6 to 3 kg/cm^2 respectively. In each case E_R value was found to be varied from 12.21 to 26.12, 9.69 to 23.43, 8.06 to 18.58, 7.25 to 15.56 and 6.57 to 12.98% at the above mentioned operating conditions. The very low values of the E_T and E_R showed the poor separation of the solid particles into U/F stream which is in agreement with the low increase in U/F concentration.

4.6.3 Discussion

The effect of concentration of the feed on hydrocyclone performance was studied by many researchers and reported a decrease in separation efficiency with increase in solid concentration (Molyneaux, 1962; Bloor and Ingham, 1983; Schwallbach, 1988 and Trim and Marder, 1995 and Thangavel et al., 1998[a]). Trim and Marder (1995) obtained an

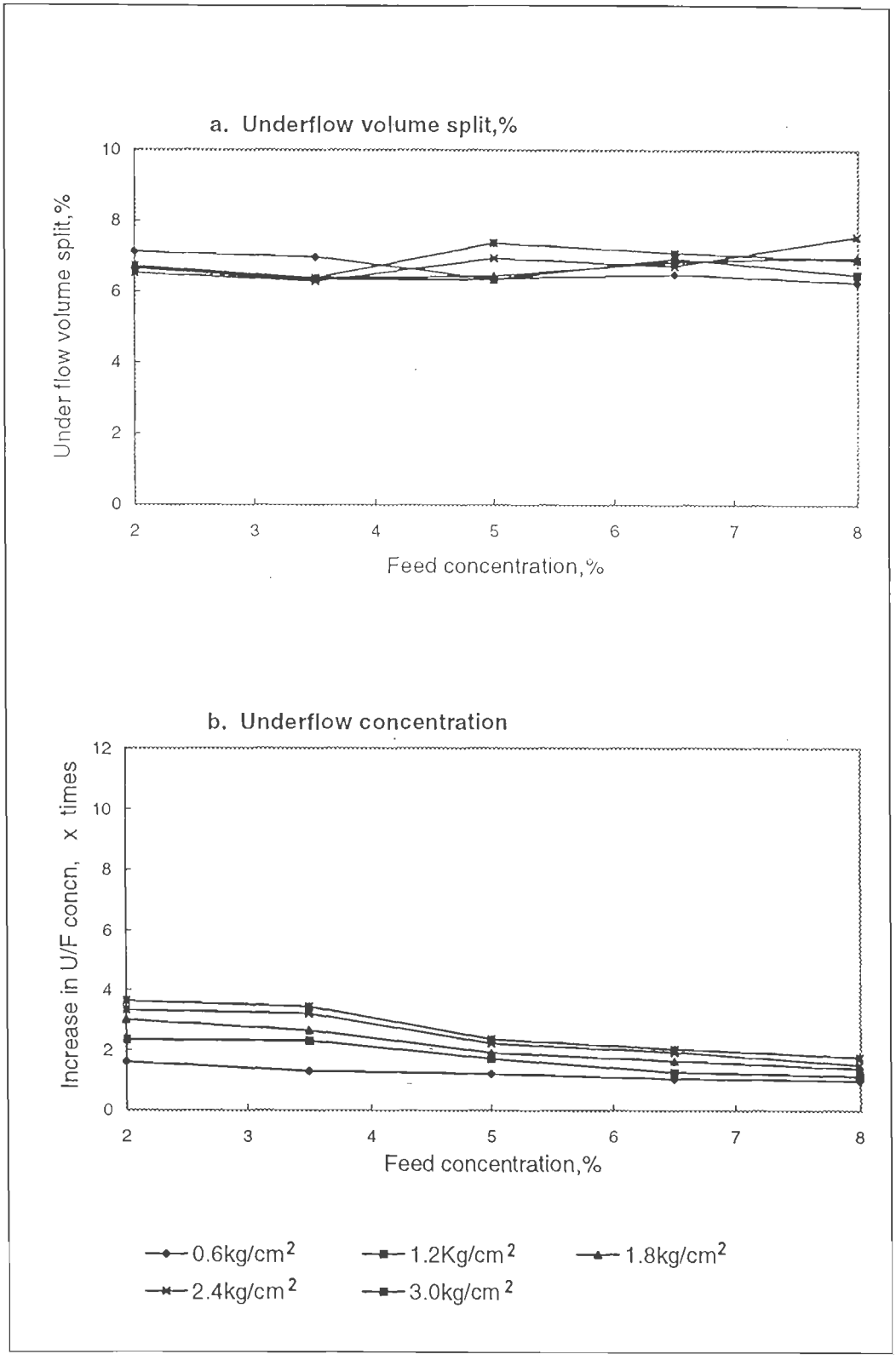


Fig.4.28 Performance of 50 mm diameter hydrocyclone operated at different feed pressures

efficiency of about 68-79% for cassava starch using a 25 mm diameter hydrocyclone having 2.2 and 3 mm underflow and overflow diameter, respectively. In another study, they got 68-84% reduced efficiency for 50 mm hydrocyclone with 6.4 and 8 mm underflow and overflow diameter, respectively for an operating pressures ranging from 140 to 350 kPa with 4.5% feed concentration. The comparative lower value for E_R in the present study may be due to larger sized underflow and overflow diameters. They also reported an increase in feed pressure resulted in increased efficiency but the influence was less marked at higher pressure level. The underflow volume split obtained in the present study was found to be almost constant with change in feed concentration which confirmed the earlier findings of Trim and Marder (1995). Thangavel et al.(1998[a]) reported a 206% increase in underflow concentration for 5% cassava starch feed using 100 mm hydrocyclone operated at 49 kPa with inlet diameter 19 mm and orifice diameter 5 mm. These values were lower than that obtained in the present investigation perhaps due to the lower geometrical dimensions.

When compared the performance of 30 and 50 mm hydrocyclones, it is clear that separation efficiency was markedly affected by the size of hydrocyclone. When diameter was increased, the efficiency got decreased due to low radial acceleration produced on the particles. The centrifugal force produced on the particle in a rotating motion in a hydrocyclone is inversely proportional to the radius of rotation and hence lower radius of rotation causes to produce high centrifugal force, producing higher effect on separation. Banerjee and Dey (1993) reported that increase in hydrocyclone diameter, with other conditions constant, reduced the efficiency of separation. Van Esch (1991) recommended an E_R value of greater than 50% to wash starch economically. In the present study, E_R was recorded as 62.68, 58.30 and 51.76 % for 30 mm and 26.06, 24.02 and 18.55 % for 50 mm hydrocyclones at 2, 3.5 and 5 % feed concentrations, respectively, when it was operated at 3 kg/cm^2. This clearly indicates that use of 50 mm diameter hydrocyclone may

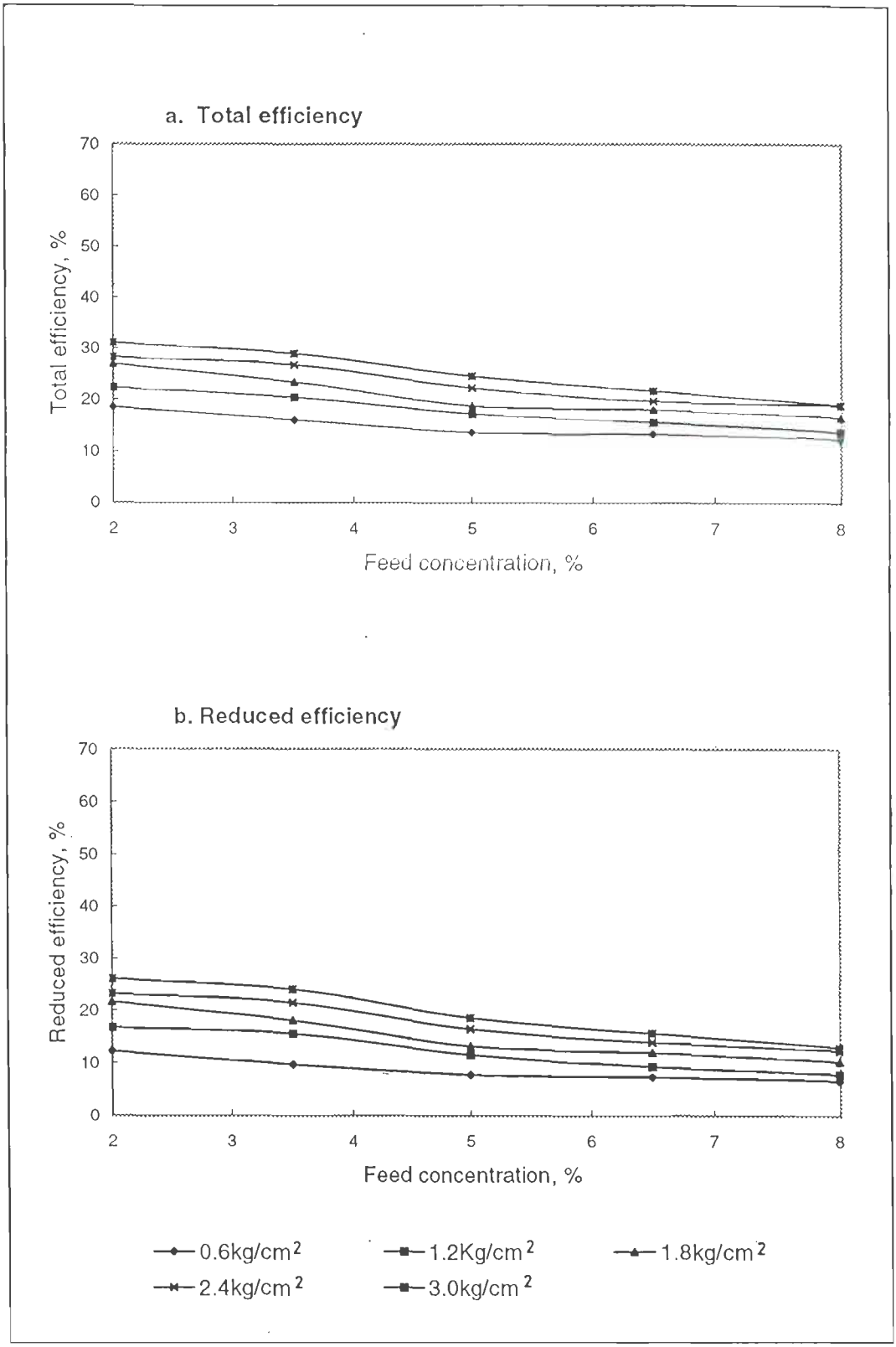


Fig.4.29 Efficiency of 50 mm hydrocyclone operated at different feed pressures

not be economical for concentrating cassava starch milk, whereas 30 mm could be used. But very low U_{vs} values resulted in high overflow rate having high amount of starch which necessitated further recycling of overflow to reduce the starch content in it.

4.6.4 Overflow recycling

As seen in Section 4.6.1.1, less than 8 % of the feed was collected as underflow and the remaining quantity is going as overflow stream. But the high amount of starch in the overflow made it unsuitable for the direct reuse in rasping operation and hence overflow was channeled for gravitational settling again. An attempt was made to examine the effect of recycling of overflow on the concentrations and to see the number of passes required to reduce the starch content in the overflow stream to a minimum level and the data for the same for 30 and 50mm hydrocyclones were collected and presented in Table 4.19.

4.6.4.1 Overflow concentration

The feed was introduced at 3.5% concentration to the 30mm hydrocyclone with an operating pressure of 1.8 kg/cm^2. There was about 2.67 and 22.53% starch concentration in the overflow and underflow, respectively. This overflow stream with 2.67% concentration was collected and again passed through the hydrocyclone, giving about 2.25% starch content in the overflow. Recycling efficiency was found to be 35.71% after first pass. Similarly, the overflow coming out from each stage was recycled through the hydrocyclone and the O/F concentration was found to be reduced to 1.93, 1.68, 1.28, 1.23, 1.06, 0.87, 0.72, 0.69, 0.70 and 0.68% after successive recycling till 11th pass. The corresponding recycling efficiencies were 44.86, 52.00, 63.34, 64.83, 69.71, 75.14,9.42,, 80.28, 80.00 and 80.59 % when compared to original feed concentration. It is observed from the table that after 9th pass, the recycling efficiency was almost constant and concluded that 9 passes were enough to reduce the starch content in O/F from 3.5% for feed to 0.69%. This value of concentration was found to be on par with the solid content of waste water generated from the starch factories *i.e.*, 0.32 - 0.96% (Balagopalan *et al.*, 1987).

The underflow volume split was found to be about 7.17% at 1.8 kg/cm^2 for 3.5% solid concentration. It meant that about 92% of the feed suspension volume was diverted as overflow. When the overflow was recycled again and again, 43.43% suspension was coming out as overflow after 9th pass. Since this much quantity of water was immediately available with less than 0.70% starch, it could be safely used for rasping operation. The results on the recycling of overflow in 30 mm hydrocyclone showed a net saving of 43% fresh water and same level of reduction in the effluent too. The study also revealed that the underflow concentration had been increased to 10.80 times as that of feed stock. This in turn helped to reduce the size of the settling tank for further settling of starch.

Experiments with 50 mm hydrocyclone showed a less reduction in starch content in the overflow after successive passes with the obvious reasons as explained in Section 4.6.2. Starch content in the overflow was reduced to 1.15% after 9th passes, giving only about 67.14% recycling efficiency. To further reduce the starch content in the overflow, more number of passes are required which in turn reduces the availability of final volume of overflow stream to be used for recycling operation. The experimental results also revealed that after 9th pass, the underflow concentration was increased only 3.9 times as compared to the feed suspension which is much less when compared to the results of 30 mm hydrocyclone for the same number of passes.

4.6.4.2 Underflow concentration

The underflow concentration was found to be reduced steadily after each recycling as the overflow from the previous pass was used as feed for the next stage, due to this , the concentration of the feed goes on decreasing. The underflow concentration was increased as the number of passes increased which is in tune with the observation made for individual hydrocyclone that increase in U/F concentration was increased with decrease in solid content in the feed. For 30 mm hydrocyclone, the value was increased by 5.43 times

Table 4.19 Effect of recycling on the O/F and U/F concentrations during cassava starch milk concentration

Properties	No. of recycling											
		I	II	III	IV	V	VI	VII	VIII	IX	X	XI
30 mm hydrocyclone												
Feed concentration, %	3.50	2.67	2.25	1.93	1.68	1.28	1.23	1.06	0.87	0.72	0.69	0.70
Overflow concentration, %	2.67	2.25	1.93	1.68	1.28	1.23	1.06	0.87	0.72	0.69	0.70	0.68
Recycling efficiency, %	23.71	35.71	44.86	52.00	63.34	64.83	69.71	75.14	79.42	80.28	80.00	80.59
Underflow concentration, %	22.53	20.78	17.51	15.92	13.93	12.78	12.55	12.17	11.48	8.50	9.25	9.56
Increase in underflow concentration x times	5.43	6.78	6.78	7.24	7.29	8.98	9.20	10.48	12.19	10.80	12.40	12.65
50 mm hydrocyclone												
Feed concentration, %	3.5	2.73	2.48	2.16	1.85	1.37	1.36	1.30	1.28	1.22	1.15	1.10
Overflow concentration, %	2.73	2.48	2.16	1.85	1.37	1.36	1.30	1.28	1.22	1.15	1.10	0.99
Recycling efficiency, %	22	29.41	38.28	47.14	60.86	61.11	62.85	63.42	65.14	67.14	68.57	71.17
Underflow concentration, %	13.06	10.88	10.42	9.55	8.66	6.82	6.80	6.25	6.00	5.98	6.56	6.73
Increase in underflow concentration x times	2.73	2.99	3.20	3.42	3.68	3.98	4.00	4.23	4.00	3.90	4.70	4.75

when it first passed and increased to 10.80 times after 9th recycling. Similarly for 50mm, it was about 2.73 times after first pass and increased to about 3.90 times after 9th pass, indicating lower rate of increase in U/F concentration as compared to 30 mm system.

4.6.5 Battery of hydrocyclones

4.6.5.1 Battery of 50 and 30 mm hydrocyclones

During the operation of hydrocyclones, it was expected to get an overflow stream with smaller particles/lesser concentration and underflow stream having comparatively larger sized particles or higher concentration than the feed. The hydrocyclones were designed mainly by considering the particle size, and smaller the size of the particle, smaller would be the diameter of hydrocyclone. If overflow with smaller sized particles is fed to a smaller diameter hydrocyclone, it is expected to give a higher recovery factor. This was tested by connecting 50 mm and 30 mm hydrocyclones in series and the U/F and O/F concentration were recorded.

When the units were connected in series, the overflow from the 50 mm unit was directed to 30 mm unit and the U/F from each unit and overflow from the 30 mm unit were collected separately for their volume and concentration. The results showed an increasing trend for U/F concentration with increasing pressures. For 2% feed concentration, increase in U/F concentration was 3.36, 4.93 and 6.24 times for 1.2, 1.8 and 2.4 kg/cm^2 operating pressures, respectively for 30 mm hydrocyclone where as for 50 mm unit, it was increased by about 0.52, 0.63 and 0.88 times only for the same operating conditions. These values were comparatively much lower when compared to the values obtained when the units were operated as individual units. When 30 mm unit was operated alone, about 6.83, 9.26 and 10.76 times increase in U/F concentration was recorded. For 50 mm unit, it was 2.34, 3.00, and 3.33 times than the feed. However, the combined units recorded a lower O/F concentration as compared to 50 mm unit.

When they are operated individually, the O/F concentration lies between 1.6 and 1.53% for 50 mm and 1.28 to 0.90% for 30 mm whereas the combined unit had recorded an O/F concentration of 1.57 to1.36 at the above operating pressures.

For 3.5% feed concentration, U/F concentration was increased by 3.5, 4.59 and 4.99 times for 30 mm and 0.7, 0.93 and 0.85 times for 50 mm hydrocyclones in the battery at 1.2, 1.8 and 2.4 kg/cm^2, respectively showing lower values than the individual units. But overflow concentration was reduced to 2.34 where as for individual unit, it was reduced to 2.76 for 50 mm and 1.6% for 30 mm units. The overflow concentration from the combined unit was lower than the 50 mm unit as expected, since the overflow from this unit was again experienced a radial acceleration causing more separation in the 30 mm unit. This reduction in the increase in U/F concentration may be attributed to the back pressure developed by the second unit, which may reduce the separating efficiency.

4.6.5.2 Battery of 30 mm hydrocyclones

The overflow from a single hydrocyclone unit had lower solid content than the feed, and if the units are connected in series, more washing effect may be expected. This was tested by connecting five units of 30 mm hydrocyclones of designed dimensions in series. Though the U/F concentration from each unit had higher values than that of feed, increase was significantly low when compared to the individual units in operation. Only 0.46 and 0.8 time increase was observed for the first unit at 2 and 3 kg/cm^2 operating pressures. The overflow was reduced to 2.28% at 3 kg/cm^2. When they were connected in series, the overflow from the first unit was going as inflow to the second unit. The diameter of the overflow pipe is higher than that of inlet pipe. The volume of the O/F from the larger diameter pipe was going through a smaller pipe to the second unit, which may cause back flow of the suspension to the first unit, retarding the separation effect.

The high back pressure developed in the units might also be responsible for the drastic reduction in the increase in concentration and hence a modification was made to reduce the back flow or back pressure effect and the same is discussed below.

4.6.5.3 Modified battery of hydrocyclones

Modified battery was made against the design principle by making the inlet and overflow pipe having same dimensions so that backflow/pressure could be reduced to a certain extent. These modified units were connected in series and U/F and O/F concentration were noted from each units . Increase in U/F concentration from each unit was found to be decreased giving 9.54 from the first unit to 2.18 times for 5^{th} unit. The feed for the first unit contained 3.5% solid content where as the feed for the last unit was obtained from the overflow after passing through four units which eventually reduced the feed concentration to that unit. The increase in U/F concentration for the first unit in the battery was 1.73 times which was lesser than that of individual unit, again due to the effect of back pressure though the extent was less when compared to the assembly described in Section 4.6, 5.2, where only 0.8 time increase was observed. The overflow from this battery contained only 1.56% starch content.

4.6.5.4 Efficiency of the battery

The volumetric flow rate of the U/F and O/F streams was noted and mass flow rate of solid in total U/F stream collected was found out. When the units were connected in series prior to modification, the E_T and E_R were found to be 63.35 and 32.13% respectively. The modified system gave E_T and E_R values as 88.74% and 76.04%, respectively. When a single unit was operated, its E_T and E_R were 61.38 and 58.30%, respectively. There was an increase of 27.36 and 17.74% for E_T and E_R in the modified system when compared to the single unit, perhaps due to the enhanced quantity of underflow from the battery.

4.6.5.5 Effect of recycling of O/F and U/F stream of the modified battery

4.6.5.5.1 Overflow recycling

The overflow from the first unit contained about 1.56 % starch and it was recycled to the system and both U/F and O/F concentrations were noted. The O/F concentration was found to be about 0.65% and underflow from all the units had an effective concentration of 2.38% after first recycling as shown in Fig.4.30. It was again recycled to give 0.44% O/F concentration and 1.08% U/F concentration. Again passing through the battery two times gave about 0.40 and 0.37% O/F concentration and 0.74 and 0.44% U/F concentrations.

4.6.5.5.2 U/F recycling

The underflow from the 1st battery contain about 5.86% starch content, it was again fed to the battery to give 2.24 and 9.12 %, O/F and U/F concentrations, respectively. This overflow after recycling 4 times gave about 1.09 and 3.46%, 0.63 and 1.48%, 0.46 and 0.84% and 0.43 and 0.71% O/F and U/F concentrations, respectively after each pass (Fig.4.30).

4.6.6 Particle size distribution

Particle size analysis was done microscopically with 400X magnification factor and the data for various configuration of hydrocyclone are represented in Table 4.20.

4.6.6.1 30 mm hydrocyclone

Table 4.20 gives the average size of the particles in the U/F and O/F streams for hydrocyclone operating individually at various pressures and feed concentrations. For each concentration, feed particle size was measured separately. In all the cases, the average particle seize in U/F stream was found to be higher than that of O/F stream. However, as the concentration increases, the gradation range become narrow. Influence of pressure is more noticeable at 2% concentration only. The size of particle in U/F stream increased

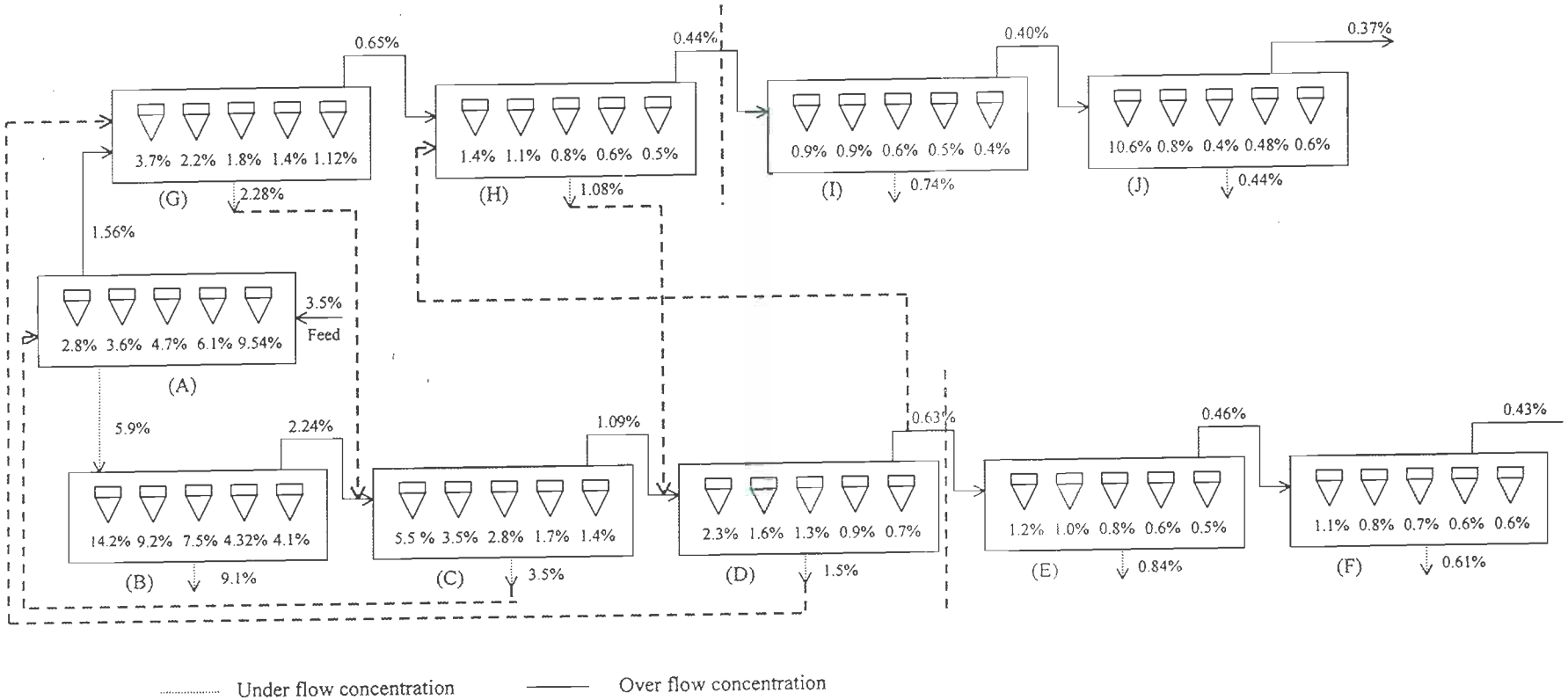


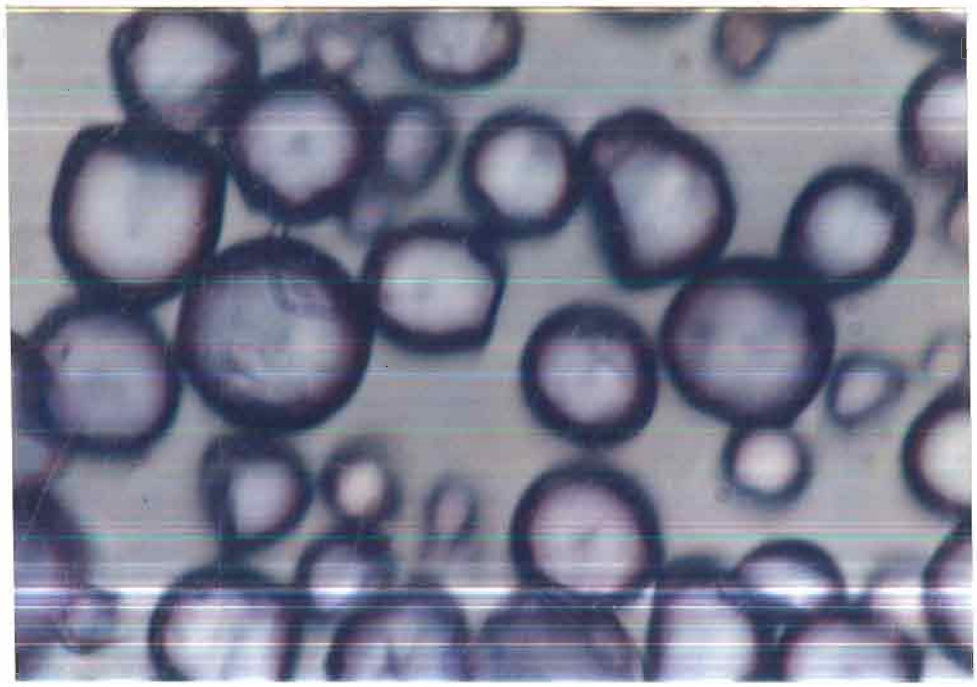
Fig. 4.30 Schematics of the recycling of overflow and underflow streams of the modified battery of hydrocyclones

from 15.5 to 20.2 μm for 2%, 14.40 to 16.8 μm for 3.5%, 13.3 to 17.7 μm for 5% and 14.3 to 15.3 μm for 6.5% feed concentration when the operating pressure was increased from 0.6 to 1.8 kg/cm^2. For the same range of pressures, the average particle size in O/F stream was 10 to 12.6 μm for 2%, 12.6 to 15.3 μm for 3.5%. 12.1 to 13.3 μm for 5% and 9.9 to 13.7 μm for 6.5% feed concentration. The microphotograph of the particles in feed, underflow and overflow streams having 3.5 feed concentration and 1.8kg/cm^2 are given in Plate 4.1

At higher concentration, the particle-particle interaction may be more and this may affect the particle flow behaviour inside the hydrocyclone and finally the average particle size distribution in U/F and O/F. But at 2%, there is a marked difference in particle size due to the negligible interaction between particles. Particle in the U/F stream has larger diameter than that of O/F stream which was in tune with basic principle of centrifugal separation that larger particle will experience more radial acceleration and move towards the U/F stream.

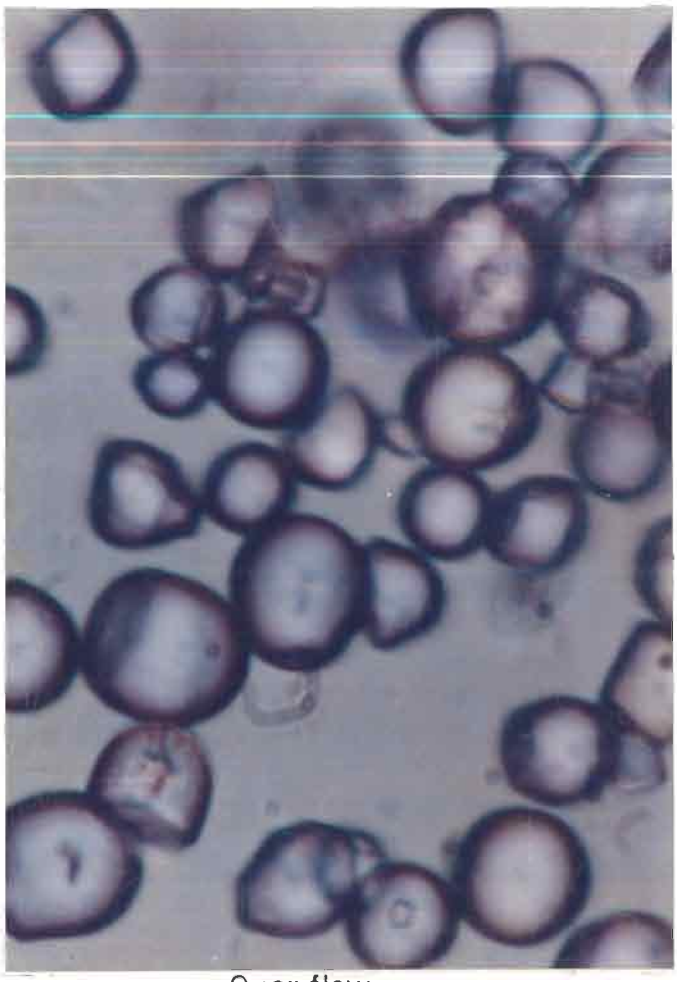
4.6.6.2 Battery of hydrocyclone

The particle size in U/F stream of the battery was found to be decreased towards the end Table 4.21. It gives about 15.4 μm for 1st unit and then gradually reduced to 10.1, 10.3, 10.0 and 9.3 μm for 2nd, 3rd, 4th and 5th unit, respectively where as feed has an average particle size of 11.7 μm . The particle size in O/F stream was only 5.6 μm. This difference in particle size is due to the feed characteristics. The overflow from the 1st unit is taken to second unit and overflow from the second is taken to third unit and so on. Naturally overflow from a hydrocyclone contain particle of lower size than the underflow and thus go on decreasing as the number of stages increasing and hence the final over flow stream contains very minute particles. The photomicrograph of the starch particles in the feed , U/F (1st unit) and overflow streams are given in Plate 4.2.



a. Feed

b. Under flow



c. Over flow

Plate. 4.1 Particle size distribution in underflow and overflow stream of the single hydro cyclone

Table 4.20 Particle size distribution in the O/F and U/F streams of a hydrocyclone of 30 mm diameter

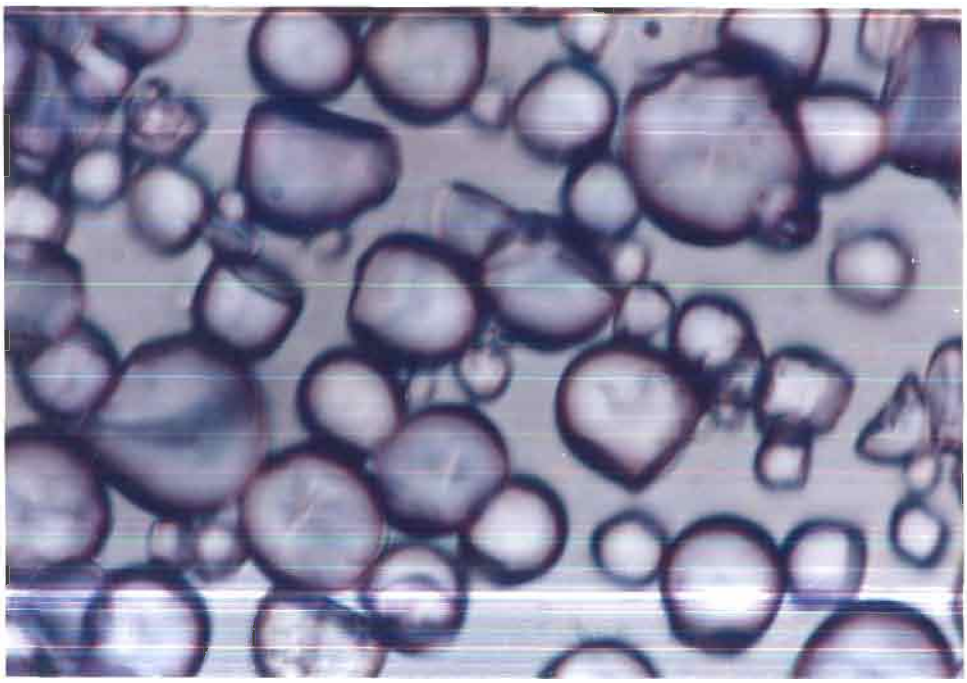
Feed pressure, kg/cm^2	Particle size, μm											
	2.0 % concentration			3.5 % concentration			5.0 % concentration			6.5 % concentration		
	Feed	U/F	O/F	Feed	U/F	O/F	Feed	U/F	O/F	Feed	U/F	O/F
0.60		15.5±5.9	10.0±4.5		14.4±6.7	12.6±4.8		13.3±4.6	12.1±4.2		14.3±5.0	9.9±4.1
1.20	13.1±7.2	17.1±5.0	10.5±4.7	13.7±6.5	16.5±6.7	13.0±6.2	11.6±4.3	16.1±5.3	13.1±5.0	10.9±5.8	15.3±4.2	13.5±5.8
1.80		20.2±6.9	12.6±6.0		16.8±5.2	15.3±7.0		17.7±4.7	13.3±5.3		15.3±4.7	13.7±5.0

U/F - Underflow stream, O/F - Overflow stream

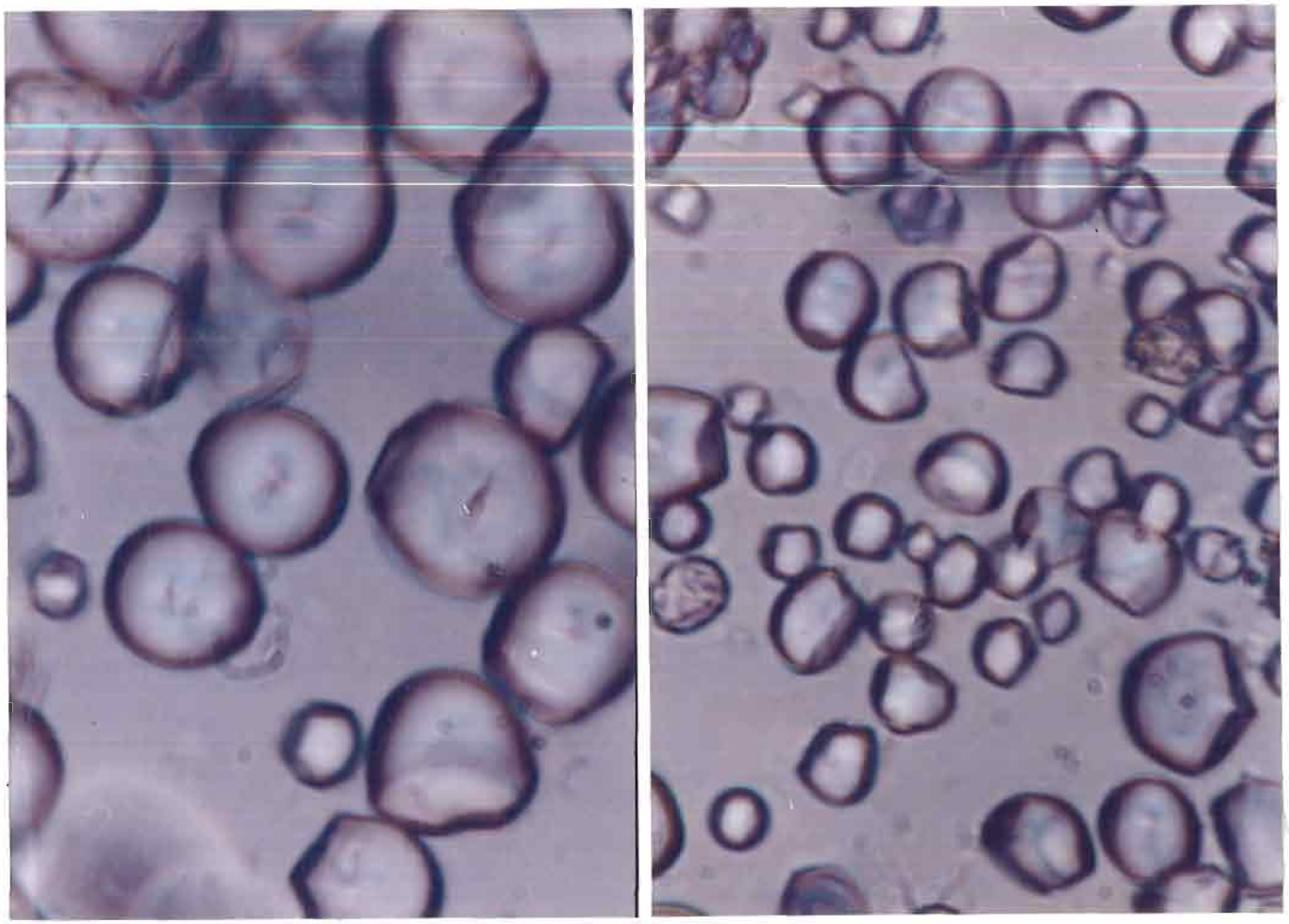
Table 4.21 Particle size distribution in the O/F and U/F streams of a battery of 5 hydrocyclones

Starch suspension	Particle size, μm
Feed	11.7±3.4
Underflow stream	
Ist unit	15.4±4.3
IInd unit	10.1±3.5
IIIrd unit	10.3±3.5
IVth unit	10.0±2.6
Vth unit	9.3±3.2
Overflow stream	5.6±1.8

Hydrocyclone diameter - 30 mm, Feed pressure - 3 kg/cm^2, Feed concentration - 3.5%



a. Feed



b. Under flow (1st)

c. Over flow

Plate 4.2 Particle size distribution in underflow and overflow stream of the modified battery of hydro cyclone

4.7 Techno - economic feasibility of the battery of hydrocyclones

The cost analysis of the modified battery of the hydrocyclone is given in Appendix T. The total operational cost of the system was found to be Rs.18.3/h. When a single battery of five hydrocyclones (30 mm diameter) is operated at 3 kg/cm^2 at 3.5% feed concentration, an overflow stream of 1.56% concentration was obtained. The feed rate of the unit is 0.84 m^3/h. Hence, to concentrate one cubic meter of starch milk to give an overflow concentration of 1.56% the operating cost was 21.79 ≈ Rs.22/-.

A system of six batteries of hydrocyclones (A, B, C, D, G and H) can be operated simultaneously as shown in Fig.4.30. The overflow and underflow streams of the batteries can be looped in such a way that finally there will be one underflow and one overflow stream to come out from the system. The underflow stream from G and H will diverted to C and D, respectively. Underflow from C and D will be going to feed tank and G, respectively. The overflow from D is going to H so that it will give only one overflow at H having 0.44% concentration and one underflow (9.1%) from B. By following this model, the volume of overflow stream coming out from the battery H was calculated to be 47% of the feed stream which could be directly reused for rasping operations as it contained the minimum starch content *i.e.*, only 0.44%.

The above model has the following advantages. The volume of settling tank could be reduced to a great extent as the stream coming to the tank is concentrated (9.1%) and the volume to be handled will be reduced to half of the original feed stream. The amount of effluent coming out from the factory will be reduced and hence the operating cost of the effluent treatment plant. As the overflow stream is reused for the rasping operation, whatever starch present in it, could be saved. Also, the fresh water consumption in the factory could be reduced to half.

SUMMARY AND CONCLUSIONS

CHAPTER V

SUMMARY AND CONCLUSIONS

Cassava (*Manihot esculenta* Crantz) being a rich source of starch (25-30%) finds extensive use in food, feed and industrial sector. They are mainly processed for starch and sago and there are about 800 cassava processing units in Tamil Nadu alone, producing starch and sago in cottage and medium scale sector. Extraction of starch from raw tubers involves various unit operations viz., washing, peeling, washing of peeled tubers, rasping, screening, settling, purification, pulverisation and drying.

The major technological problems associated with cassava starch industry in India or elsewhere are high water consumption, more space requirement, larger starch settling time and weather dependent drying which adversely affect the product quality. The longer detention of starch with fruit water in the settling tanks causes fermentation, producing alcohol and organic acid, which give foul smell polluting the entire atmosphere. Hence, separation of starch from water, immediately after screening will reduce the quantity of effluent, volume of settling tank and fresh water requirement by reusing fruit water.

Cassava starch suspension in water can be considered as a coarse and freely dispersed solid-liquid system. Settling of starch make use of the principle of gravitational settling. But the wide range and rather smaller particles in suspension cause them to behave in a different manner than that of discrete particle during settling. Hence, the fundamental principle governing the settling of two phase disperse system was extended to study the kinetics of starch granule settling in water. The data on the settling characteristics of starch obtained from the tests could be used for designing settling basins or tables.

Speed of settling and compactness of settled starch determine the efficiency of settling process. Various chemicals are reported to be added by the starch industries during settling to improve the whiteness of the starch. But any detrimental effect by these chemicals on the rheological and thermal properties of starch can lead to the lowering of starch value. Hence, it was decided to compare the effect of some of the easily and commonly available chemicals on settling of starch and also the effect of these chemicals on the rheological and thermal properties of starch.

Starch particles having wide range of sizes and non-uniform shapes will be subjected to random motion during settling and larger particles attain higher terminal velocity compared to smaller ones. The random motion and velocity gradient of particles lead to both perikinetic and/or orthokinetic flocculation to occur during settling. Because of these, particles travel at different velocities and collide to form aggregates and thus the increased mass of the particles enhance the settling rate. The collision frequency can be increased by producing gas bubbles electrolytically within the starch milk. The gas bubbles of smaller sizes during their upward movement come in contact with the starch particles and cause frequent collision producing flocculation at faster rate there by enhance the settling rate. A preliminary investigation to extent the principle of electroflocculation in starch settling was made and its effect on the rheological properties of starch was studied. Settling of cassava starch in inclined column was also undertaken to know the effect of the inclined floor surface on settling of cassava starch.

Settling is the physical separation of solid particle in water under the action of gravitational force. It is a slow process, but the velocity of settling can be enhanced by applying centrifugal force where settling takes place by a force which is several hundred times greater than the gravitational force. Centrifugal settling of cassava starch was analysed using a special gadget (rotor) fitted to a laboratory centrifuge.

Hydrocyclone is a solid-liquid separation device, functions based on the principle of centrifugal separation. It has been suggested as a practical alternative in solid - liquid separation involving biological material and suspension. Though they have been used in large-scale starch manufacturing since long to separate starch from impurities and to concentrate starch milk from different crops, the selection is usually according to manufacture's specification. But to obtain maximum separation efficiency, they are to be designed based on the properties of solid and liquid involved in the disperse system. A hydrocyclone was designed, developed and tested by changing feed pressures and concentrations of cassava starch.

Hydrocyclone, while in operation divide the feed stream into overflow stream with the bulk of fluid together with very finer particle and underflow with concentrated suspension having coarser particles. If the overflow contain starch particle at a minimum level, it can be reused for rasping operations so that starch, if any, in the stream will not go as a waste. Battery of five hydrocyclones was connected in series with one inlet and was tested. Recycling of overflow stream was carried out to estimate the amount of water saved by the use of hydrocyclones.

Salient findings which are drawn from the present investigations are listed below.

1. All four types of settling viz. discrete, flocculent, hindered and compression settling are found to be occurred during starch settling. Starch suspension of low concentration (≤6%) were characterised by discrete or flocculent settling whereas in high concentrated suspension (≥10%), hindered or compression settling was predominant.
2. The settling velocity of particle in suspension was found to be lower than the free falling velocity of starch particle in water in isolation (0.169 mm/s) and it was reduced significantly with concentration and height of suspension, but diameter of settling column did not produce any effect.

3. Settling velocity of cassava starch particle in concentrated suspension can be derived using the equation

$$\frac{v_c}{v_o} = A\varepsilon^{(B.\frac{H}{D})/\varepsilon}$$

where,

v_c = Settling velocity, mm/s

v_o = Free falling velocity, mm/s

ε = Fluid volumetric concentration, decimal

H = Height of suspension, mm

D = Diameter of settling column, mm

A = Constant $= 3.06 - 0.28\frac{H}{D} + 0.03\left[\frac{H}{D}\right]^2$

B = Constant $= [0.079\ \frac{H}{D} - 0.046]^{-1}$

4. Fresh starch took more time for settling than dried starch due to the hydration repulsion effect arose by the surface charge of the granule. Based on the design curves obtained from the batch settling tests, 0.001 m^3/s loading rate of the suspension was calculated as optimum for a settling table of 100 m length, 50 cm width and 30 cm depth.

5. Final water content in the settled starch (M) cake could be correlated with time of settling (t) as M = 94.5 – 7.4 ln (t) with in ± 1.5% deviation. Three hours of settling produced moderately compact starch cake as increased settling time did not produce much reduction in moisture content.

6. Acids and sodium metabisulphite aided settling in more effective way than sodium hypochlorite and alum. However, more compacted starch cake was

obtained with sodium hypochlorite, followed by sulphuric acid, sodium metabisulphite, hydrochloric acid and alum.

7. DSC analysis showed the gelatinization temperature of cassava starch as 66.9-85.07°C and gelatinization enthalpy as 15.5 J/g. But these values were enhanced for the starch settled in the presence of chemicals; the acid treatment had more pronounced effect. Gelatinization enthalpy was not changed significantly except for hydrochloric acid treatment showing most of the changes are taking place in the amorphous region only during heating.

8. Viscosity of the native starch was found to have undergone breakdown during heating. Elastic modulus values were higher than viscous modules during heating and cooling of starch showing the starch paste had a tendency to behave like solid, which was confirmed by the low phase angle values.

9. Settling in the presence of acids or alum lowered the rheological parameters of the starch; effect of hydrochloric acid was not much severe as compared to sulphuric acid. Sodium metabisulphite did not produce any noticeable detrimental effect on the starch quality. When it was required for low viscosity applications, acid modified or alum treated starch could be used.

10. Settling rate was enhanced by electroflocculation of cassava starch and at 8.33 V/cm; amount of starch in the supernatant was 0.12% only. Temperature of suspension was found to be increased with increase in applied potential and reached to 68.5°C for 13.33 V/cm.

11. The gelatinization properties of the starch is not affected by electroflocculation in different sources of water. Rheological parameters of electroflocculated starch were lower than that of control except, the phase angle showing the thinning out of starch suspension during heating.

12. Disassociation of the electrode material was a major problem in electroflocculation causing the fruit water to become more coloured, which

necessitate the selection of suitable inert electrode material to apply this technology into practice.

13. Equation of the form $S = A+B\,\theta$ could be used for predicting the effect of inclined column on settling of cassava starch where S = per cent starch in supernatant, θ = angle of inclination, degrees. The constant A and B could be correlated with concentration (C) as $A = [-0.0023\ (C-0.832)^2 + 0.179]^{-1}$ and $B = [0.60(C-3.33)^2 + 18.28]^{-1}$. Settling tanks having bottom floor surface inclined about 20-30° recorded faster settling rate.

14. Settling velocity of starch particle in a centrifugal field can be increased to 373 times than the gravitational settling velocity by keeping the peripheral speed of the modified rotor as 23.16 m/s.

15. Concentration of the underflow stream from a 30 mm hydrocyclone, designed based on the properties of starch and water was found to be increased with increase in feed pressure and decreased with increase in feed concentration.

16. Total and reduced efficiencies showed an increasing trend with increasing pressures but decreased with increasing concentration. Reduced efficiency of 62.68% and 41.87% was obtained at 3 kg/cm^2 pressure for 2 and 8% concentrations, respectively. Underflow volume split was found to be affected very little by both feed pressure and concentration and the value ranged from 5-8%.

17. 50 mm hydrocyclones gave lower values for all the performance parameters when compared to 30 mm unit. Battery of 50 and 30 mm hydrocyclones in series did not produce any noticeable reduction in overflow concentration due to the backpressure developed in the units.

18. The overflow stream from 30 mm hydrocyclone operated at 1.8 kg/cm^2 with 3.5% feed concentration was recycled and found that the overflow concentration was reduced to 0.69% after 9^{th} recycling and about of 43% of feed stream with 0.69% starch could be reused or diverted back for rasping operation. Recycling of over flow

stream from 50 mm hydrocyclones reduced the starch concentration to only 1.15% after 9th recycling and more passes are required to reduce the starch content to such a level for reuse, which eventually reduced the volume of overflow.

19. Battery of five hydrocyclones of 30 mm diameter (with other dimensions as per design) gave about 2.28% starch in overflow when operated at 3 kg/cm^2 with 3.5% feed concentration. Modified battery (with equal inlet and overflow diameter) reduced the overflow concentration to about 1.56% for the same operating conditions.

20. Experiments with recycling of overflow from the modified battery of hydrocyclones gave about 0.40% starch in the overflow stream after 3 passes whereas underflow recycling yielded 0.46% starch after 4 passes. About 47% of water can be saved if the overflow stream from the battery is recycled.

21. Particle size distribution was found to be higher in underflow stream and low in overflow stream. When 30 mm hydrocyclone was operated at 1.8 kg/cm^2, underflow particle size was 20.2 and 15.3 μm for 2 and 6.5% feed concentration, respectively, where as overflow contained 12.6 and 13.7 μm sized particle at the above conditions. Modified battery of hydrocyclones operated at 3 kg/cm^2 at 3.5% feed concentration permitted only 5.6 μm sized particle in the overflow stream.

22. The total operational cost of the modified battery of hydrocyclone was found to be Rs.22/- to concentrate one cubic meter of starch milk to give an overflow concentration of 1.56%.

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APPENDICES

Appendix A Effect of low concentration on settling characteristics of cassava starch in different column diameter

Time, min	Sediment height, mm								
	Column diameter, 37 mm			Column diameter, 28 mm			Column diameter, 22 mm		
	Concentration, %			Concentration, %			Concentration, %		
	2	4	6	2	4	6	2	4	6
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
5	3.50	8.50	12.00	6.00	9.00	15.00	6.00	12.50	19.00
10	6.00	14.50	22.00	8.15	15.00	28.00	9.00	18.25	30.00
15	6.50	15.00	27.00	8.35	15.50	30.00	10.00	18.50	33.00
20	6.75	14.25	25.00	8.50	14.75	27.50	11.00	17.75	30.00
30	6.75	13.75	22.00	8.50	14.25	25.50	11.00	17.00	27.50
40		13.75	21.00		14.25	24.50		17.00	26.50
50			20.50			24.00			26.50
60			20.50			24.00			

Appendix B Effect of high concentration on settling of cassava starch in 37 mm column diameter for different heights of suspensions

Time, min	Height of interface, mm											
	Concentration, %											
	10			12			14			16		
	Height of suspension, mm			Height of suspension, mm			Height of suspension, mm			Height of suspension, mm		
	100	200	300	100	200	300	100	200	300	100	200	300
0	100.00	200.00	300.00	100.00	200.00	300.00	100.00	200.00	300.00	100.00	200.00	300.00
5	65.00	159.50	245.50	75.00	166.50	252.00	82.50	173.00	268.50	88.00	182.00	283.00
10	29.50	117.50	201.00	47.50	131.00	214.00	60.50	147.00	238.50	72.50	160.00	260.00
15	23.50	79.50	162.50	33.00	99.00	177.00	40.50	119.00	208.00	58.50	138.00	234.00
20	21.25	53.00	122.50	28.00	71.00	139.00	33.50	92.00	172.00	48.00	115.00	208.00
30	19.25	46.50	82.00	25.50	56.00	96.00	30.25	69.50	136.50	40.00	93.00	179.00
40	18.25	44.00	61.25	23.50	49.50	70.50	27.75	59.00	99.50	35.50	75.00	152.00
50	17.75	41.25	56.50	22.25	46.00	64.80	26.50	54.75	85.00	32.00	64.00	128.00
60	17.75	39.25	53.25	22.00	41.50	60.80	25.50	51.50	78.50	30.00	59.50	108.00
70		36.00	50.75	22.00	38.75	58.00	25.00	49.25	73.75	29.00	56.50	95.00
80		34.00	48.75		37.75	56.00	25.00	47.75	70.75	28.00	54.50	90.00
90		33.00	48.00		37.50	54.50		46.25	67.50	28.00	53.80	86.50
100		33.00	47.00		37.50	53.50		45.50	65.25		52.50	84.50
110			46.50			52.00		45.00	64.50		52.00	81.50
120			46.50			51.50		44.50	63.50		51.50	78.50
130						51.00		44.25	62.50		51.00	75.50
140						51.00		44.00	61.50		50.00	74.50
150								44.00	61.00		49.50	72.50
160									61.00		49.00	70.50
170											49.00	68.50
180												68.50

Appendix C Effect of high concentration on settling of cassava starch in 49 mm diameter column for different heights of suspension

Time, min	Height of interface, mm											
	Concentration, %											
	10			12			14			16		
	Height of suspension, mm			Height of suspension, mm			Height of suspension, mm			Height of suspension, mm		
	100	200	300	100	200	300	100	200	300	100	200	300
5	60.00	164.50	246.00	73.00	168.00	259.00	77.5	173.50	269.50	84.00	185.00	282.00
10	28.75	120.50	210.50	46.50	128.00	224.00	54.00	147.50	239.50	68.00	160.00	256.00
15	22.25	78.50	168.00	31.25	128.00	190.00	37.75	121.00	210.00	53.00	140.00	230.00
20	20.00	53.00	130.00	27.25	88.50	149.00	32.00	92.00	168.00	45.00	120.00	205.00
30	17.75	41.00	93.00	23.50	65.00	105.00	27.25	66.50	135.00	39.00	102.00	180.00
40	16.75	36.50	71.50	21.25	49.00	80.50	24.75	56.26	98.00	35.50	91.00	155.00
50	16.25	34.50	62.50	20.25	43.00	70.50	23.75	51.25	82.25	34.40	81.00	132.50
60	16.25	32.75	59.50	19.75	41.50	63.75	22.95	48.00	75.50	32.50	75.00	117.50
70		32.00	55.75	19.75	39.00	62.00	22.75	46.00	71.25	31.50	69.00	105.00
80		31.25	53.50		38.25	58.00	22.75	44.25	68.00	29.50	63.50	95.00
90		31.00	51.75		37.00	56.50		42.75	65.25	29.00	61.00	88.50
100		31.00	50.50		36.25	55.50		42.50	63.50	29.00	58.80	84.50
110			50.50		36.00	54.00		41.50	62.25		56.00	80.50
120					36.00	53.75		41.50	61.50		55.50	78.50
130						53.00		41.25	59.50		54.00	76.50
140						53.00		41.25	58.00		53.00	74.75
150									58.00		52.00	72.00
160											52.00	69.00
170												68.00
180												66.00
190												65.5
200												65.5

Appendix D Effects of low and high concentrations on settling characteristics of fresh cassava starch in 37 mm column diameter with 200 mm height of suspension

Settling time, min	Sediment height, mm				Height of interface, mm			
	Low concentration, %				High concentration, %			
	4.04	6.45	7.92	9.10	13.43	15.85	16.97	20.01
0	0	0	0	0	200	200	200	200
10	2.00	3.00	3.00	4.00	181.50	182.00	184.50	188.00
20	4.00	6.50	7.50	10.00	162.50	163.50	165.50	172.50
30	6.50	11.00	13.50	15.55	141.00	142.50	146.50	157.50
40	8.50	15.25	22.50	22.00	120.00	125.50	129.50	142.50
50	10.25	17.50	24.50	25.75	97.50	106.50	110.00	126.00
60	11.50	19.00	24.50	27.50	77.50	88.50	92.50	111.00
70	12.50	19.50	23.75	27.25	60.75	72.50	75.50	96.75
80	12.75	19.75	23.25	26.25	57.00	60.00	65.00	84.50
90	12.75	20.00	22.75	25.50	55.00	57.00	61.50	78.50
100		20.25	22.25	24.50	53.25	54.50	59.75	74.00
110		20.25	22.00	24.25	52.00	53.50	57.75	70.50
120			22.00	24.00	50.75	52.50	55.75	67.50
130				24.00	49.75	50.50	54.25	65.50
140				24.00	48.00	49.75	53.25	63.25
150					46.50	48.50	52.25	61.75
160					45.25	48.00	51.25	60.50
170					44.50	48.00	51.00	59.25
180					44.50	47.50	50.75	58.00
190						47.50	50.50	57.00
200							50.00	56.50
210							50.00	56.25
220								56.0
230								56.0

Appendix E Effect of settling time on final water content of the starch cake

Settling time, h	Water content, %		
	Concentration, %		
	5	15	25
3	86.54	86.31	86.23
4	84.74	84.53	83.82
5	83.82	83.69	83.32
6	81.42	81.18	81.12
7	80.18	80.00	79.15
8	80.20	79.80	79.20

Appendix F Rheological properties of starch settled in presence of sulphuric acid

Time s	Temp °C	Phase angle, °						Viscosity, Pa.s					
		Contol	5 mM	10 mM	20 mM	30 mM	40 mM	Contol	5 mM	10 mM	20 mM	30 mM	40 mM
10	75	20.3	41.4	27.6	85.2	81.4	71.9	0.78	0.11	0.00	0.01	0.01	0.00
190	80	20.0	40.8	54.4	72.5	87.2	78.7	0.77	0.10	0.00	0.01	0.00	0.01
370	85	19.5	38.8	86.6	61.3	69.3	87.1	0.73	0.10	0.00	0.01	0.03	0.01
610	90	19.4	40.1	89.6	69.2	80.3	76.7	0.70	0.10	0.01	0.00	0.01	0.00
790	95	19.7	37.3	89.7	72.7	78.4	70.4	0.67	0.10	0.02	0.01	0.00	0.01
1090	95	20.0	42.0	87.3	52.5	69.0	53.8	0.61	0.10	0.01	0.00	0.00	0.01
1390	95	20.0	40.9	53.1	64.1	89.0	80.3	0.57	0.08	0.01	0.01	0.01	0.02
1400	95	20.0	47.3	40.2	78.3	89.3	80.9	0.57	0.09	0.01	0.00	0.00	0.01
1580	90	20.9	40.6	40.3	76.6	61.6	67.8	0.56	0.08	0.00	0.01	0.01	0.02
1820	85	20.7	38.6	49.5	85.8	58.9	83.4	0.50	0.08	0.02	0.01	0.00	0.00
2000	80	21.3	40.8	80.7	84.2	83.1	89.5	0.50	0.08	0.01	0.01	0.01	0.01
2180	75	21.0	48.6	38.3	82.3	55.0	77.3	0.50	0.08	0.00	0.00	0.01	0.02
2420	70	21.5	45.4	66.9	59.8	42.0	48.5	0.50	0.10	0.01	0.00	0.00	0.00
2600	65	21.8	47.7	56.7	74.3	88.0	65.3	0.55	0.11	0.00	0.01	0.01	0.01
2780	60	21.6	43.6	62.8	67.2	55.0	89.5	0.55	0.10	0.00	0.01	0.01	0.01
3020	55	21.2	41.6	86.1	62.6	53.3	81.0	0.58	0.11	0.00	0.00	0.01	0.02
3200	50	20.8	37.0	72.8	69.0	77.1	77.0	0.59	0.11	0.01	0.01	0.01	0.03
3380	45	20.4	40.8	56.3	64.7	69.0	73.9	0.59	0.12	0.01	0.01	0.00	0.01
3560	40	20.4	39.6	46.5	70.1	80.8	74.9	0.61	0.12	0.01	0.00	0.00	0.00
3800	35	19.7	39.8	58.7	70.3	80.4	81.5	0.63	0.13	0.01	0.01	0.01	0.01
4280	35	19.8	41.9	46.8	71.9	77.7	83.3	0.71	0.14	0.01	0.01	0.01	0.01
4880	35	19.7	42.0	75.8	70.1	73.9	88.5	0.76	0.14	0.02	0.01	0.01	0.00
5660	35	19.8	39.6	55.7	84.2	89.7	86.3	0.84	0.14	0.01	0.01	0.02	0.01
6560	35	20.1	40.4	54.2	69.6	88.7	86.9	0.89	0.13	0.01	0.01	0.01	0.02
7400	35	20.4	39.4	41.9	56.6	78.2	83.1	0.93	0.14	0.01	0.01	0.01	0.01

Time s	Temp °C	Elastic modulus(G'), Pa						Viscous modulus(G''), Pa					
		Contol	5 mM	10 mM	20 mM	30 mM	40 mM	Contol	5 mM	10 mM	20 mM	30 mM	40 mM
10	75	13.75	0.77	0.03	0.03	0.01	0.03	4.90	0.68	0.02	0.04	0.05	0.03
190	80	13.60	0.70	0.03	0.03	0.04	0.03	4.87	0.61	0.01	0.04	0.01	0.05
370	85	13.45	0.80	0.05	0.05	0.06	0.05	4.61	0.65	0.01	0.07	0.17	0.03
610	90	12.70	0.77	0.00	0.04	0.01	0.01	4.41	0.65	0.05	0.01	0.04	0.03
790	95	11.90	0.80	0.00	0.03	0.03	0.02	4.20	0.61	0.10	0.07	0.00	0.03
1090	95	10.75	0.67	0.00	0.05	0.02	0.08	3.87	0.60	0.07	0.02	0.00	0.04
1390	95	9.93	0.60	0.02	0.04	0.00	0.06	3.60	0.52	0.03	0.08	0.05	0.09
1400	95	9.82	0.49	0.13	0.03	0.05	0.02	3.58	0.54	0.11	0.02	0.00	0.06
1580	90	9.65	0.59	0.14	0.06	0.02	0.13	3.54	0.51	0.00	0.04	0.04	0.12
1820	85	8.84	0.59	0.09	0.00	0.00	0.02	3.16	0.47	0.10	0.05	0.02	0.03
2000	80	8.71	0.55	0.01	0.01	0.07	0.01	3.14	0.47	0.01	0.09	0.07	0.05
2180	75	8.68	0.45	0.03	0.02	0.04	0.05	3.12	0.51	0.02	0.01	0.05	0.11
2420	70	9.00	0.61	0.03	0.05	0.02	0.02	3.23	0.62	0.06	0.02	0.02	0.05
2600	65	9.33	0.61	0.01	0.02	0.00	0.02	3.44	0.67	0.00	0.05	0.06	0.05
2780	60	9.84	0.68	0.01	0.06	0.02	0.03	3.48	0.65	0.01	0.08	0.03	0.05
3020	55	10.47	0.81	0.04	0.02	0.05	0.03	3.66	0.71	0.04	0.02	0.07	0.14
3200	50	10.91	0.94	0.01	0.02	0.01	0.03	3.70	0.70	0.03	0.05	0.03	0.05
3380	45	11.37	0.90	0.04	0.04	0.00	0.04	3.74	0.77	0.06	0.08	0.02	0.09
3560	40	11.83	0.94	0.06	0.02	0.04	0.05	3.85	0.78	0.04	0.03	0.02	0.02
3800	35	12.60	1.00	0.03	0.02	0.01	0.04	3.93	0.83	0.09	0.05	0.03	0.08
4280	35	13.80	1.00	0.08	0.04	0.03	0.03	4.44	0.90	0.12	0.04	0.04	0.04
4880	35	14.75	0.97	0.03	0.03	0.02	0.01	4.77	0.87	0.07	0.04	0.06	0.01
5660	35	15.45	1.04	0.05	0.04	0.09	0.06	5.27	0.86	0.08	0.05	0.12	0.03
6560	35	15.65	0.95	0.06	0.01	0.04	0.02	5.60	0.81	0.09	0.03	0.04	0.09
7400	35	15.80	1.04	0.10	0.05	0.01	0.01	5.80	0.86	0.10	0.08	0.01	0.05

Appendix G Rheological properties of starch settled in presence of hydrochloric acid

Time	Temp	Phase angle, °						Viscosity, Pa.s					
s	° C	Control	15 mM	30 mM	60 mM	90 mM	120 mM	Control	15 mM	30 mM	60 mM	90 mM	120 mM
10	75	20.3	20.0	48.8	50.0	76.8	62.1	0.78	0.68	0.07	0.01	0.01	0.02
190	80	20.0	19.6	48.6	49.6	87.3	88.8	0.77	0.66	0.06	0.00	0.00	0.03
370	85	19.5	18.1	47.6	42.8	74.3	80.4	0.73	0.61	0.06	0.01	0.00	0.03
610	90	19.4	18.2	54.1	44.5	67.7	80.0	0.70	0.57	0.06	0.00	0.01	0.02
790	95	19.7	18.0	50.3	80.6	34.1	83.1	0.67	0.55	0.06	0.01	0.00	0.03
1090	95	20.0	18.3	52.8	41.9	60.0	74.8	0.61	0.52	0.05	0.01	0.00	0.04
1390	95	20.0	18.4	50.9	80.2	78.3	51.8	0.57	0.50	0.05	0.01	0.00	0.01
1400	95	20.0	18.8	46.5	64.7	59.7	88.6	0.57	0.50	0.04	0.01	0.01	0.00
1580	90	20.9	19.6	53.4	41.6	84.2	84.4	0.56	0.50	0.04	0.01	0.01	0.03
1820	85	20.7	19.6	52.1	75.9	86.4	67.6	0.50	0.52	0.05	0.01	0.00	0.03
2000	80	21.3	20.1	46.6	65.8	83.3	36.6	0.50	0.52	0.03	0.01	0.00	0.01
2180	75	21.0	20.7	58.1	77.8	84.4	69.2	0.50	0.54	0.05	0.01	0.01	0.05
2420	70	21.5	21.3	59.5	63.1	83.8	81.2	0.50	0.58	0.04	0.01	0.01	0.03
2600	65	21.8	21.4	57.1	62.4	89.4	84.2	0.55	0.61	0.05	0.01	0.01	0.04
2780	60	21.6	21.8	48.8	67.8	84.0	79.5	0.55	0.64	0.05	0.01	0.01	0.01
3020	55	21.2	22.0	50.8	88.8	56.7	86.4	0.58	0.69	0.06	0.01	0.01	0.01
3200	50	20.8	22.2	50.7	87.4	88.3	88.4	0.59	0.74	0.06	0.01	0.01	0.00
3380	45	20.4	22.6	54.1	68.8	81.1	73.6	0.59	0.78	0.07	0.00	0.01	0.01
3560	40	20.4	22.1	52.3	62.9	60.1	72.6	0.61	0.80	0.08	0.01	0.00	0.03
3800	35	19.7	21.9	52.3	71.8	65.6	82.8	0.63	0.89	0.09	0.01	0.01	0.02
4280	35	19.8	21.6	52.4	60.8	79.5	70.8	0.71	0.95	0.09	0.00	0.00	0.02
4880	35	19.7	21.8	52.7	76.9	66.7	73.4	0.76	0.95	0.10	0.01	0.07	0.03
5660	35	19.8	21.7	52.4	50.2	85.6	63.9	0.84	0.96	0.09	0.01	0.01	0.03
6560	35	20.1	21.8	52.0	55.6	89.2	89.1	0.89	0.90	0.09	0.01	0.02	0.03
7400	35	20.4	21.8	51.5	60.5	77.7	85.2	0.93	0.96	0.09	0.01	0.03	0.01

Time	Temp	Elastic modulus(G'), Pa						Viscous modulus(G''), Pa					
s	° C	Control	15 mM	30 mM	60 mM	90 mM	120 mM	Control	15 mM	30 mM	60 mM	90 mM	120 mM
10	75	13.75	11.85	0.07	0.03	0.02	0.07	4.90	4.29	0.42	0.03	0.09	0.12
190	80	13.60	11.65	0.06	0.04	0.06	0.05	4.87	4.13	0.37	0.01	0.01	0.19
370	85	13.45	11.65	0.06	0.03	0.01	0.14	4.61	3.80	0.36	0.05	0.01	0.23
610	90	12.70	10.98	0.06	0.02	0.02	0.22	4.41	3.59	0.37	0.02	0.05	0.17
790	95	11.90	10.63	0.06	0.01	0.02	0.13	4.20	3.48	0.36	0.06	0.02	0.10
1090	95	10.75	10.22	0.05	0.04	0.09	0.31	3.87	3.30	0.30	0.06	0.03	0.11
1390	95	9.93	9.52	0.05	0.08	0.04	0.05	3.60	3.14	0.27	0.07	0.02	0.23
1400	95	9.82	9.47	0.04	0.14	0.05	0.14	3.58	3.14	0.25	0.05	0.07	0.16
1580	90	9.65	9.29	0.04	0.16	0.02	0.10	3.54	3.15	0.27	0.05	0.03	0.25
1820	85	8.84	9.16	0.05	0.02	0.03	0.23	3.16	3.24	0.30	0.05	0.02	0.12
2000	80	8.71	9.32	0.03	0.01	0.02	0.06	3.14	3.29	0.22	0.06	0.02	0.05
2180	75	8.68	9.38	0.05	0.05	0.03	0.17	3.12	3.41	0.30	0.03	0.03	0.23
2420	70	9.00	9.58	0.04	0.04	0.04	0.10	3.23	3.62	0.26	0.05	0.04	0.13
2600	65	9.33	9.82	0.05	0.03	0.05	0.26	3.44	3.83	0.36	0.06	0.07	0.09
2780	60	9.84	10.25	0.05	0.00	0.04	0.13	3.48	4.02	0.30	0.04	0.04	0.15
3020	55	10.47	10.87	0.06	0.01	0.06	0.14	3.66	4.33	0.36	0.06	0.05	0.07
3200	50	10.91	11.40	0.06	0.03	0.06	0.19	3.70	4.60	0.44	0.05	0.04	0.06
3380	45	11.37	12.00	0.07	0.04	0.03	0.09	3.74	4.87	0.43	0.06	0.03	0.05
3560	40	11.83	12.80	0.08	0.02	0.01	0.22	3.85	5.20	0.48	0.03	0.02	0.19
3800	35	12.60	13.85	0.09	0.05	0.01	0.12	3.93	5.61	0.54	0.08	0.06	0.05
4280	35	13.80	14.95	0.09	0.05	0.02	0.07	4.44	5.99	0.59	0.06	0.03	0.18
4880	35	14.75	15.15	0.10	0.05	0.03	0.15	4.77	5.98	0.62	0.03	0.23	0.28
5660	35	15.45	15.15	0.09	0.05	0.15	0.06	5.27	6.02	0.58	0.05	0.03	0.32
6560	35	15.65	15.10	0.09	0.02	0.09	0.27	5.60	5.99	0.57	0.04	0.09	0.06
7400	35	15.80	15.10	0.09	0.02	0.06	0.07	5.80	5.97	0.57	0.05	0.09	0.12

Appendix H Rheological properties of starch treated with sodium hypochlorite

Time s	Temp °C	Phase angle, °						Viscosity, Pa.s					
		Control	0.17 mM	0.35 mM	0.70 mM	1.06 mM	1.41 mM	Control	0.17 mM	0.35 mM	0.70 mM	1.06 mM	1.41 mM
10	75	20.3	19.9	19.9	19.9	22.7	24.0	0.78	0.83	0.68	0.59	0.46	0.36
190	80	20.0	19.3	19.6	19.3	21.5	23.6	0.77	0.80	0.67	0.57	0.42	0.34
370	85	19.5	19.5	19.8	20.0	22.2	24.0	0.73	0.78	0.63	0.57	0.44	0.34
610	90	19.4	19.8	19.4	20.3	22.5	25.5	0.70	0.76	0.62	0.55	0.42	0.33
790	95	19.7	20.5	20.4	20.7	23.0	23.4	0.67	0.76	0.63	0.54	0.41	0.29
1090	95	20.0	20.3	19.8	20.8	23.6	24.4	0.61	0.72	0.60	0.53	0.41	0.29
1390	95	20.0	19.9	19.9	20.3	23.5	26.0	0.57	0.68	0.58	0.49	0.39	0.30
1400	95	20.0	20.2	19.6	20.5	23.3	26.8	0.57	0.68	0.56	0.49	0.38	0.29
1580	90	20.9	20.7	21.5	22.5	24.5	26.4	0.56	0.68	0.59	0.51	0.37	0.28
1820	85	20.7	20.8	22.5	22.2	25.4	27.5	0.50	0.65	0.57	0.47	0.36	0.28
2000	80	21.3	20.9	21.0	22.1	25.1	25.9	0.50	0.64	0.56	0.46	0.35	0.26
2180	75	21.0	21.2	23.0	22.4	25.4	25.3	0.50	0.65	0.57	0.47	0.36	0.25
2420	70	21.5	21.5	22.0	22.8	26.1	26.9	0.50	0.66	0.58	0.48	0.37	0.27
2600	65	21.8	21.6	22.2	22.9	25.5	28.1	0.55	0.66	0.60	0.51	0.36	0.28
2780	60	21.6	21.8	23.0	23.4	26.1	27.9	0.55	0.67	0.63	0.50	0.38	0.28
3020	55	21.2	22.3	23.4	23.8	26.7	27.6	0.58	0.70	0.66	0.53	0.40	0.30
3200	50	20.8	22.5	24.1	24.4	26.4	29.7	0.59	0.73	0.71	0.56	0.40	0.32
3380	45	20.4	23.0	24.3	25.7	27.5	28.6	0.59	0.76	0.75	0.61	0.43	0.32
3560	40	20.4	22.6	25.1	25.6	28.8	29.5	0.61	0.78	0.80	0.64	0.47	0.35
3800	35	19.7	22.6	25.6	26.5	27.7	29.4	0.63	0.80	0.85	0.71	0.48	0.37
4280	35	19.8	24.2	25.5	26.9	29.0	29.2	0.71	0.94	0.99	0.79	0.54	0.40
4880	35	19.7	24.7	25.2	26.9	29.1	29.7	0.76	1.09	1.01	0.81	0.57	0.42
5660	35	19.8	24.4	24.9	26.4	28.9	29.6	0.84	1.11	1.00	0.81	0.57	0.42
6560	35	20.1	23.8	24.7	26.7	28.7	30.0	0.89	1.10	0.99	0.81	0.56	0.43
7400	35	20.4	23.8	24.9	26.4	28.6	30.3	0.93	1.10	1.01	0.81	0.56	0.44

Time s	Temp °C	Elastic modulus(G'), Pa						Viscous modulus(G''), Pa					
		Control	0.17 mM	0.35 mM	0.70 mM	1.06 mM	1.41 mM	Control	0.17 mM	0.35 mM	0.70 mM	1.06 mM	1.41 mM
10	75	13.75	14.45	11.90	10.23	6.97	5.06	4.90	5.22	4.30	3.69	2.91	2.25
190	80	13.60	14.35	11.85	10.18	6.97	4.85	4.87	5.03	4.20	3.56	2.74	2.12
370	85	13.45	13.90	11.50	9.77	6.73	4.74	4.61	4.91	4.14	3.56	2.74	2.11
610	90	12.70	13.25	11.00	9.39	6.31	4.40	4.41	4.76	3.88	3.47	2.62	2.10
790	95	11.90	12.50	10.60	9.01	6.09	4.25	4.20	4.74	3.94	3.41	2.57	1.84
1090	95	10.75	12.30	10.50	8.73	5.84	4.15	3.87	4.53	3.77	3.34	2.55	1.84
1390	95	9.93	11.75	9.99	8.29	5.53	3.80	3.60	4.26	3.61	3.07	2.42	1.85
1400	95	9.82	11.70	9.89	8.21	5.54	3.64	3.58	4.29	3.51	3.06	2.38	1.84
1580	90	9.65	11.25	9.45	7.68	5.13	3.58	3.54	4.25	3.72	3.17	2.34	1.78
1820	85	8.84	10.80	8.65	7.31	4.76	3.38	3.16	4.11	3.58	2.98	2.27	1.76
2000	80	8.71	10.56	9.20	7.17	4.76	3.31	3.14	4.03	3.51	2.91	2.22	1.61
2180	75	8.68	10.45	9.00	7.18	4.71	3.26	3.12	4.08	3.60	2.96	2.24	1.54
2420	70	9.00	10.37	8.98	7.18	4.72	3.35	3.23	4.13	3.66	3.02	2.25	1.70
2600	65	9.33	10.40	9.08	7.30	4.78	3.32	3.44	4.15	3.77	3.08	2.29	1.77
2780	60	9.84	10.45	9.33	7.35	4.85	3.41	3.48	4.24	3.96	3.17	2.38	1.80
3020	55	10.47	10.63	9.63	7.53	4.97	3.57	3.66	4.42	4.17	3.33	2.50	1.87
3200	50	10.91	10.83	10.12	7.73	5.12	3.54	3.70	4.60	4.46	3.51	2.53	2.02
3380	45	11.37	11.03	10.21	7.99	5.18	3.72	3.74	5.05	4.73	3.85	2.69	2.03
3560	40	11.83	11.45	10.80	8.37	5.36	3.89	3.85	5.93	5.05	4.01	2.55	2.20
3800	35	12.60	12.13	11.70	8.94	5.74	4.08	3.93	6.10	5.62	4.46	3.02	2.30
4280	35	13.80	13.29	13.05	9.84	6.18	4.53	4.44	6.50	6.23	4.98	3.41	2.53
4880	35	14.75	15.04	13.40	10.15	6.39	4.58	4.77	6.83	6.32	5.09	3.55	2.62
5660	35	15.45	15.65	13.45	10.30	6.46	4.61	5.27	6.98	6.26	5.09	3.56	2.61
6560	35	15.65	15.80	13.60	10.15	6.45	4.62	5.60	6.90	6.26	5.11	3.53	2.67
7400	35	15.80	15.85	13.55	10.25	6.48	4.68	5.80	6.91	6.32	5.09	3.53	2.73

Appendix I Rheological properties of starch treated with sodium metabisulphite

Time s	Temp °C	Phase angle, °						Viscosity, Pa.s					
		Control	1.56 mM	3.12 mM	4.68 mM	6.24 mM	7.80 mM	Control	1.6 mM	3.1 mM	4.7 mM	6.2 mM	7.8 mM
10	75	20.3	23.1	23.2	23.5	24.7	25.7	0.78	0.77	0.73	0.73	0.53	0.48
190	80	20.0	23.1	22.6	23.2	25.3	25.8	0.77	0.74	0.70	0.71	0.52	0.47
370	85	19.5	23.1	22.2	23.1	23.7	25.1	0.73	0.71	0.66	0.69	0.48	0.45
610	90	19.4	23.1	22.2	22.9	23.7	25.0	0.70	0.67	0.61	0.64	0.45	0.42
790	95	19.7	23.1	22.3	23.4	24.0	24.7	0.67	0.64	0.60	0.61	0.43	0.40
1090	95	20.0	22.6	22.5	23.5	24.7	25.5	0.61	0.61	0.57	0.56	0.41	0.37
1390	95	20.0	22.2	22.2	23.3	25.3	25.6	0.57	0.57	0.53	0.51	0.38	0.35
1400	95	20.0	22.3	22.1	23.6	25.6	25.7	0.57	0.56	0.52	0.51	0.38	0.34
1580	90	20.9	22.7	23.2	24.8	25.9	25.8	0.56	0.57	0.53	0.51	0.36	0.34
1820	85	20.7	23.8	23.5	25.3	26.6	26.2	0.50	0.57	0.52	0.50	0.36	0.32
2000	80	21.3	23.9	24.1	25.5	26.5	26.1	0.50	0.57	0.54	0.51	0.36	0.32
2180	75	21.0	24.2	24.8	25.8	27.3	27.7	0.50	0.59	0.57	0.52	0.38	0.34
2420	70	21.5	24.8	25.1	26.0	27.3	27.2	0.50	0.62	0.59	0.55	0.38	0.32
2600	65	21.8	25.0	25.4	26.0	27.7	27.1	0.55	0.65	0.61	0.55	0.40	0.33
2780	60	21.6	25.0	25.6	26.4	28.6	27.3	0.55	0.68	0.65	0.58	0.43	0.34
3020	55	21.2	25.1	25.7	26.7	29.1	27.8	0.58	0.73	0.69	0.63	0.47	0.35
3200	50	20.8	24.9	26.0	26.7	29.6	28.0	0.59	0.77	0.74	0.66	0.50	0.36
3380	45	20.4	24.6	26.0	26.3	29.0	27.9	0.59	0.81	0.79	0.69	0.52	0.38
3560	40	20.4	24.3	25.9	26.6	29.1	27.9	0.61	0.86	0.84	0.75	0.56	0.38
3800	35	19.7	24.1	25.5	26.3	29.1	28.5	0.69	0.92	0.90	0.82	0.62	0.40
4280	35	19.8	23.2	24.9	25.5	28.4	30.1	0.71	0.97	0.96	0.87	0.67	0.46
4880	35	19.7	23.0	24.7	25.6	28.0	30.2	0.76	0.97	0.96	0.88	0.66	0.54
5660	35	19.8	23.2	24.8	25.7	28.1	30.0	0.84	0.98	0.96	0.88	0.67	0.55
6560	35	20.1	23.1	24.9	25.5	27.9	30.1	0.89	0.95	0.96	0.88	0.67	0.56
7400	35	20.4	22.9	24.8	25.5	28.3	30.0	0.93	0.95	0.96	0.88	0.67	0.55

Time s	Temp °C	Elastic modulus(G'), Pa						Viscous modulus(G''), Pa					
		Control	1.56 mM	3.12 mM	4.68 mM	6.24 mM	7.80 mM	Control	1.6 mM	3.1 mM	4.7 mM	6.2 mM	7.8 mM
10	75	13.75	10.90	10.10	10.73	7.20	6.32	4.90	4.84	4.58	4.59	3.31	3.04
190	80	13.60	10.80	10.00	10.52	6.94	6.15	4.87	4.64	4.38	4.44	3.27	2.97
370	85	13.45	10.50	9.71	10.25	6.92	6.05	4.61	4.47	4.17	4.32	3.03	2.83
610	90	12.70	9.78	9.00	9.50	6.45	5.71	4.41	4.19	3.89	4.04	2.83	2.66
790	95	11.90	9.33	8.70	8.95	6.06	5.43	4.20	4.04	3.75	3.85	2.69	2.50
1090	95	10.75	8.95	8.11	8.09	5.57	4.94	3.87	3.84	3.56	3.51	2.55	2.35
1390	95	9.93	8.52	7.63	7.43	5.06	4.54	3.60	3.57	3.31	3.21	2.45	2.17
1400	95	9.82	8.35	7.37	7.29	5.01	4.52	3.58	3.49	3.28	3.18	2.39	2.17
1580	90	9.65	8.11	7.12	6.94	4.69	4.38	3.54	3.60	3.30	3.20	2.28	2.11
1820	85	8.84	7.91	7.15	6.68	4.51	4.12	3.16	3.59	3.29	3.15	2.26	2.03
2000	80	8.71	7.92	7.31	6.69	4.54	4.07	3.14	3.55	3.40	3.19	2.26	1.99
2180	75	8.68	8.04	7.40	6.73	4.57	4.02	3.12	3.70	3.55	3.24	2.36	2.11
2420	70	9.00	8.32	7.65	6.93	4.68	3.93	3.23	3.91	3.69	3.37	2.41	2.03
2600	65	9.33	8.56	7.87	7.11	4.83	3.99	3.44	4.06	3.86	3.47	2.53	2.04
2780	60	9.84	8.93	8.18	7.42	4.95	4.07	3.48	4.26	4.06	3.67	2.70	2.12
3020	55	10.47	9.64	8.75	7.85	5.28	4.19	3.66	4.60	4.37	3.92	2.94	2.23
3200	50	10.91	10.20	9.20	8.35	5.54	4.22	3.70	4.84	4.65	4.17	3.14	2.28
3380	45	11.37	10.90	9.69	8.94	5.91	4.37	3.74	5.08	4.97	4.40	3.28	2.24
3560	40	11.83	11.60	10.40	9.47	6.38	4.47	3.85	5.39	5.26	4.72	3.54	2.41
3800	35	12.60	12.10	11.30	10.45	7.05	4.72	4.10	5.78	5.66	5.14	3.91	2.50
4280	35	13.80	13.20	12.50	11.35	7.75	5.09	4.44	6.06	6.03	5.46	4.18	2.88
4880	35	14.75	13.85	12.60	11.50	7.87	5.93	4.77	6.08	6.06	5.56	4.17	3.41
5660	35	15.45	13.90	12.40	11.60	7.92	6.07	5.27	6.14	6.06	5.56	4.21	3.46
6560	35	15.65	13.90	12.40	11.65	7.92	6.08	5.60	5.99	6.06	5.52	4.20	3.49
7400	35	15.80	13.80	12.40	11.60	7.86	6.05	5.80	5.95	6.03	5.52	4.22	3.46

Appendix J Rheological properties of starch trated with alum

		Phase angle, °						Viscosity, Pa.s					
Time s	Temp °C	Control	16.70 mM	28.50 mM	37.50 mM	44.40 mM	50.00 mM	Control	16.7 mM	28.5 mM	37.5 mM	44.4 mM	50.0 mM
10	75	20.3	34.1	33.2	50.0	38.0	40.8	0.78	0.12	0.10	0.09	0.07	0.07
190	80	20.0	32.3	35.6	47.0	30.5	48.0	0.77	0.12	0.11	0.08	0.06	0.08
370	85	19.5	32.3	30.6	49.2	36.5	50.3	0.73	0.12	0.09	0.07	0.06	0.07
610	90	19.4	30.3	33.2	47.7	25.6	44.2	0.70	0.11	0.10	0.06	0.06	0.06
790	95	19.7	32.8	31.8	48.3	32.4	48.1	0.67	0.11	0.08	0.06	0.06	0.06
1090	95	20.0	30.0	39.6	43.1	37.4	48.7	0.61	0.08	0.07	0.04	0.06	0.04
1390	95	20.0	34.7	41.8	69.6	37.4	54.5	0.57	0.08	0.06	0.03	0.04	0.03
1400	95	20.0	35.8	41.1	51.5	58.7	56.3	0.57	0.07	0.05	0.03	0.05	0.03
1580	90	20.9	37.5	38.1	67.1	45.7	40.7	0.56	0.06	0.04	0.02	0.04	0.02
1820	85	20.7	39.9	51.0	64.5	64.3	61.1	0.50	0.05	0.04	0.03	0.03	0.02
2000	80	21.3	46.5	48.4	68.9	58.3	69.0	0.50	0.06	0.04	0.04	0.04	0.03
2180	75	21.0	38.5	60.7	62.3	61.5	59.5	0.50	0.05	0.05	0.03	0.02	0.02
2420	70	21.5	44.2	51.6	82.0	62.5	58.0	0.50	0.06	0.04	0.03	0.04	0.03
2600	65	21.8	41.4	53.2	64.7	62.3	66.5	0.55	0.06	0.05	0.04	0.03	0.03
2780	60	21.6	40.8	57.4	67.4	66.9	64.5	0.55	0.05	0.06	0.03	0.04	0.03
3020	55	21.2	44.6	60.5	68.6	80.2	66.2	0.58	0.06	0.06	0.04	0.04	0.04
3200	50	20.8	46.2	55.2	68.6	45.2	63.1	0.59	0.07	0.06	0.05	0.04	0.04
3380	45	20.4	49.6	49.9	71.7	63.3	67.8	0.59	0.07	0.06	0.05	0.05	0.04
3560	40	20.4	50.4	53.3	66.6	54.7	60.8	0.61	0.08	0.07	0.05	0.05	0.05
3800	35	19.7	49.5	61.0	71.2	40.4	58.0	0.63	0.09	0.09	0.06	0.07	0.06
4280	35	19.8	48.7	55.3	70.0	55.1	64.6	0.71	0.09	0.08	0.06	0.07	0.06
4880	35	19.7	50.3	55.2	67.5	55.7	65.8	0.76	0.10	0.09	0.06	0.07	0.06
5660	35	19.8	51.6	59.7	69.0	64.1	65.7	0.84	0.10	0.10	0.05	0.07	0.06
6560	35	20.1	49.6	57.6	68.4	54.7	68.9	0.89	0.10	0.10	0.05	0.07	0.05
7400	35	20.4	50.7	53.8	58.0	60.1	58.8	0.93	0.06	0.09	0.05	0.07	0.06

		Elastic modulus(G'), Pa						Viscous modulus(G''), Pa					
Time s	Temp °C	Control	16.70 mM	28.50 mM	37.50 mM	44.40 mM	50.00 mM	Control	16.7 mM	28.5 mM	37.5 mM	44.4 mM	50.0 mM
10	75	13.75	1.16	1.00	0.45	0.41	0.54	4.90	0.78	0.66	0.54	0.46	0.43
190	80	13.60	1.19	0.93	0.44	0.38	0.46	4.87	0.75	0.67	0.48	0.37	0.49
370	85	13.45	1.15	0.96	0.37	0.38	0.38	4.61	0.72	0.57	0.43	0.41	0.42
610	90	12.70	1.20	0.94	0.33	0.34	0.41	4.41	0.70	0.68	0.36	0.37	0.39
790	95	11.90	1.03	0.80	0.31	0.35	0.33	4.20	0.66	0.50	0.35	0.40	0.36
1090	95	10.75	0.89	0.56	0.20	0.35	0.22	3.87	0.52	0.54	0.22	0.37	0.24
1390	95	9.93	0.69	0.39	0.06	0.22	0.16	3.60	0.47	0.35	0.16	0.23	0.22
1400	95	9.82	0.63	0.38	0.14	0.27	0.11	3.58	0.44	0.33	0.18	0.29	0.16
1580	90	9.65	0.52	0.35	0.06	0.15	0.16	3.54	0.38	0.27	0.14	0.23	0.13
1820	85	8.84	0.40	0.12	0.08	0.10	0.07	3.16	0.33	0.25	0.17	0.18	0.12
2000	80	8.71	0.34	0.23	0.08	0.10	0.07	3.14	0.35	0.26	0.23	0.19	0.17
2180	75	8.68	0.42	0.17	0.08	0.13	0.07	3.12	0.32	0.31	0.18	0.13	0.14
2420	70	9.00	0.38	0.18	0.02	0.11	0.13	3.23	0.37	0.23	0.18	0.23	0.20
2600	65	9.33	0.40	0.23	0.10	0.12	0.09	3.44	0.35	0.30	0.22	0.19	0.20
2780	60	9.84	0.34	0.22	0.09	0.15	0.07	3.48	0.29	0.35	0.21	0.24	0.17
3020	55	10.47	0.39	0.23	0.09	0.18	0.11	3.66	0.38	0.40	0.23	0.25	0.26
3200	50	10.91	0.39	0.26	0.12	0.22	0.13	3.70	0.40	0.37	0.30	0.27	0.27
3380	45	11.37	0.39	0.36	0.10	0.21	0.11	3.74	0.46	0.43	0.32	0.30	0.27
3560	40	11.83	0.39	0.34	0.11	0.22	0.17	3.85	0.47	0.46	0.30	0.32	0.27
3800	35	12.60	0.51	0.32	0.11	0.25	0.19	3.93	0.59	0.57	0.35	0.45	0.30
4280	35	13.80	0.52	0.34	0.06	0.25	0.17	4.44	0.58	0.48	0.39	0.45	0.35
4880	35	14.75	0.51	0.37	0.15	0.25	0.16	4.77	0.61	0.53	0.35	0.43	0.36
5660	35	15.45	0.48	0.35	0.14	0.27	0.16	5.27	0.60	0.60	0.39	0.41	0.35
6560	35	15.65	0.51	0.41	0.13	0.25	0.13	5.60	0.60	0.65	0.23	0.41	0.34
7400	35	15.80	0.55	0.39	0.21	0.29	0.22	5.80	0.67	0.53	0.34	0.46	0.35

Appendix K Rheological properties of starch electofloculated in borewell water

Time, s	Temp.,°C	Phase Angle, °				Viscosity, Pa.s			
		Contr	5V/cm	6.7V/cm	10V/cm	Control	5V/cm	6.7V/cm	10V/cm
10	75	7.90	17.20	21.90	20.90	0.56	0.43	0.29	0.35
190	80	7.80	17.10	21.20	21.20	0.54	0.41	0.28	0.32
430	85	7.90	17.90	21.40	20.80	0.53	0.35	0.25	0.29
610	90	9.10	21.20	22.20	21.40	0.54	0.30	0.22	0.23
850	95	12.00	25.00	25.90	24.10	0.47	0.23	0.19	0.19
1090	95	12.50	25.50	23.80	24.10	0.37	0.19	0.14	0.16
1400	95	12.90	27.10	27.10	25.00	0.31	0.16	0.13	0.17
1580	90	15.50	26.10	27.40	26.00	0.33	0.15	0.13	0.15
1820	85	15.60	26.80	27.70	26.90	0.31	0.14	0.12	0.15
2000	80	15.60	27.10	28.90	27.50	0.29	0.14	0.11	0.15
2180	75	16.20	27.90	30.10	28.00	0.31	0.14	0.12	0.14
2360	70	16.10	27.90	28.90	28.40	0.30	0.14	0.11	0.16
2600	65	15.60	29.80	32.40	27.70	0.30	0.15	0.13	0.15
2780	60	15.80	30.10	30.40	29.50	0.30	0.15	0.13	0.17
3020	55	16.10	29.90	28.90	28.30	0.31	0.16	0.11	0.17
3200	50	16.50	30.30	32.10	31.20	0.32	0.17	0.14	0.18
3380	45	16.00	30.30	28.30	30.30	0.34	0.17	0.15	0.19
3560	40	16.30	30.60	31.90	28.70	0.34	0.18	0.16	0.21
3800	35	16.20	31.00	30.60	29.90	0.36	0.20	0.18	0.22
4040	35	16.80	31.20	30.90	30.50	0.37	0.21	0.17	0.23
4280	35	16.20	32.50	30.90	29.80	0.39	0.23	0.18	0.23
4520	35	16.70	31.80	32.10	31.10	0.38	0.23	0.18	0.23
5000	35	16.40	30.20	30.80	29.90	0.38	0.22	0.18	0.24
6800	35	16.90	32.00	32.30	29.80	0.38	0.24	0.19	0.24
7400	35	16.90	30.40	32.30	30.50	0.39	0.23	0.18	0.24

Time, s	Temp., °C	Elastic modulus, Pa				Viscous modulus, Pa			
		Control	5 V/cm	6.7 V/cm	10 V/cm	Control	5 V/cm	6.7 V/cm	10 V/cm
10	75	25.20	8.64	4.59	5.79	3.49	2.68	1.85	2.21
190	80	24.60	8.22	4.58	5.70	3.39	2.53	1.73	2.22
430	85	23.70	6.81	3.36	5.34	3.30	2.20	1.57	2.03
610	90	21.00	4.79	2.43	4.61	3.36	1.86	1.37	1.81
850	95	13.80	3.14	2.01	3.22	2.95	1.47	1.18	1.44
1090	95	10.50	2.48	1.65	2.57	2.35	1.18	0.89	1.19
1400	95	8.37	1.95	1.57	2.11	1.92	1.00	0.84	1.03
1580	90	7.36	1.90	1.40	1.92	2.05	0.93	0.81	0.97
1820	85	6.87	1.73	1.30	1.78	1.92	0.88	0.74	0.93
2000	80	6.71	1.66	1.32	1.71	1.88	0.85	0.72	0.91
2180	75	6.68	1.63	1.27	1.69	1.94	0.87	0.77	0.92
2360	70	6.60	1.63	1.25	1.73	1.86	0.86	0.70	0.91
2600	65	6.61	1.62	1.34	1.72	1.91	0.93	0.80	0.98
2780	60	6.74	1.63	1.37	1.75	1.88	0.94	0.79	0.94
3020	55	6.91	1.73	1.38	1.76	1.96	1.00	0.76	1.06
3200	50	7.02	1.81	1.56	1.86	2.03	1.06	0.88	1.09
3380	45	7.23	1.85	1.49	1.96	2.13	1.08	0.84	1.08
3560	40	7.42	1.90	1.66	2.05	2.13	1.12	0.93	1.18
3800	35	7.73	2.09	1.73	2.23	2.26	1.25	0.98	1.31
4040	35	7.91	2.27	1.76	2.39	2.29	1.32	1.13	1.37
4280	35	8.02	2.25	1.87	2.41	2.42	1.43	1.05	1.46
4520	35	8.11	2.34	1.81	2.47	2.35	1.45	1.12	1.44
5000	35	8.13	2.40	1.91	2.52	2.43	1.41	1.14	1.44
6800	35	8.07	2.43	1.87	2.53	2.42	1.39	1.10	1.83
7400	35	8.04	2.43	1.87	2.56	2.37	1.52	1.91	2.06

Appendix L Rheological properties of starch electofloculated in dam water

Time, s	Temp.,°C	Phase Angle, °				Viscosity, Pa.s			
		contro	13.3V/cm	16.7V/cm	20V/cm	Control	13.3V/cm	16.7V/cm	20V/cm
10	75	7.90	11.60	12.30	14.40	0.56	0.56	0.55	0.53
190	80	7.80	10.70	12.60	14.10	0.54	0.51	0.53	0.51
430	85	7.90	9.80	12.80	14.60	0.53	0.47	0.52	0.50
610	90	9.10	9.95	14.70	16.00	0.54	0.45	0.51	0.46
850	95	12.00	12.50	18.60	19.60	0.47	0.43	0.45	0.39
1090	95	12.50	13.50	20.00	21.00	0.37	0.40	0.35	0.30
1400	95	12.90	16.60	20.30	21.90	0.31	0.38	0.27	0.24
1580	90	15.50	17.70	22.30	23.70	0.33	0.33	0.28	0.23
1820	85	15.60	18.40	23.60	24.30	0.31	0.31	0.26	0.22
2000	80	15.60	18.60	21.90	24.30	0.29	0.30	0.24	0.22
2180	75	16.20	18.90	23.40	24.80	0.31	0.30	0.25	0.21
2360	70	16.10	19.00	22.20	24.80	0.30	0.30	0.24	0.21
2600	65	15.60	19.20	23.60	24.80	0.30	0.30	0.25	0.21
2780	60	15.80	18.40	23.00	25.30	0.30	0.29	0.25	0.22
3020	55	16.10	19.30	22.40	25.20	0.31	0.31	0.26	0.23
3200	50	16.50	19.50	23.60	24.70	0.32	0.32	0.29	0.24
3380	45	16.00	19.10	22.90	24.10	0.34	0.33	0.29	0.24
3560	40	16.30	19.90	23.00	25.40	0.34	0.35	0.31	0.27
3800	35	16.20	20.30	23.20	24.40	0.36	0.38	0.33	0.29
4040	35	16.80	19.50	23.70	25.60	0.37	0.38	0.36	0.30
4280	35	16.20	19.90	23.50	25.10	0.39	0.39	0.36	0.31
4520	35	16.70	19.50	23.10	26.20	0.38	0.39	0.36	0.31
5000	35	16.40	20.30	23.40	26.20	0.38	0.41	0.37	0.33
6800	35	16.90	19.90	23.60	26.20	0.38	0.40	0.38	0.32
7400	35	16.90	20.50	23.70	26.20	0.39	0.41	0.38	0.34

Time, s	Temp., °C	Elastic modulus, Pa				Viscous modulus, Pa			
		Contr	13.3V/cm	16.7V/cm	20V/cm	Control	13.3V/cm	16.7V/cm	20V/cm
10	75	25.20	17.10	16.70	13.00	3.49	3.50	3.42	3.33
190	80	24.60	17.10	16.30	12.80	3.39	3.23	3.36	3.21
430	85	23.70	17.00	15.00	12.00	3.30	2.95	3.50	3.12
610	90	21.00	16.70	12.20	9.96	3.36	2.83	3.21	2.86
850	95	13.80	13.00	8.30	6.86	2.95	2.69	2.80	2.44
1090	95	10.50	8.06	6.10	4.83	2.35	3.37	2.22	1.85
1400	95	8.37	6.52	4.58	3.73	1.92	2.41	1.69	1.50
1580	90	7.36	5.76	4.10	3.31	2.05	2.09	1.77	1.45
1820	85	6.87	5.53	3.72	3.01	1.92	1.91	1.63	1.36
2000	80	6.71	5.48	3.75	2.87	1.88	1.86	1.51	1.25
2180	75	6.68	5.42	3.67	2.88	1.94	1.88	1.59	1.33
2360	70	6.60	5.39	3.73	2.86	1.86	1.86	1.52	1.32
2600	65	6.61	5.46	3.64	2.88	1.91	1.87	1.59	1.33
2780	60	6.74	5.61	3.73	2.97	1.88	1.82	1.58	1.41
3020	55	6.91	5.74	3.99	3.10	1.96	1.96	1.68	1.46
3200	50	7.02	5.95	4.13	3.26	2.03	2.03	1.80	1.50
3380	45	7.23	6.11	4.30	3.63	2.13	2.06	1.81	1.53
3560	40	7.42	6.42	4.57	3.75	2.13	2.21	1.94	1.72
3800	35	7.73	6.73	4.88	4.15	2.26	2.37	2.10	1.80
4040	35	7.91	6.78	5.11	4.12	2.29	2.38	2.24	1.88
4280	35	8.02	6.91	5.23	4.20	2.42	2.45	2.27	1.98
4520	35	8.11	6.92	5.31	4.22	2.35	2.45	2.72	1.97
5000	35	8.13	6.95	5.35	4.30	2.43	2.57	2.37	2.08
6800	35	8.07	6.97	5.42	4.35	2.42	2.51	2.33	2.07
7400	35	8.04	6.97	5.37	4.35	2.37	2.60	2.37	2.03

Appendix M Rheological properties of starch electoflocculated in distilled water

Time, s	Temp., °C	Phase Angle, °				Viscosity, Pa.s			
		Control	13.3 V/cm	16.7 V/cm	20 V/cm	Control	13.3 V/cm	16.7 V/cm	20 V/cm
10	75	7.90	9.70	10.60	12.90	0.56	0.55	0.53	0.55
190	80	7.80	9.60	10.00	12.50	0.54	0.54	0.53	0.53
430	85	7.90	9.80	10.30	12.60	0.53	0.53	0.52	0.54
610	90	9.10	11.60	12.40	13.70	0.54	0.53	0.51	0.52
850	95	12.00	15.10	15.30	16.90	0.47	0.46	0.41	0.44
1090	95	12.50	15.40	17.00	17.30	0.37	0.49	0.32	0.33
1400	95	12.90	16.50	18.80	18.90	0.31	0.37	0.26	0.27
1580	90	15.50	18.40	20.30	21.20	0.33	0.30	0.26	0.28
1820	85	15.60	18.80	20.00	21.20	0.31	0.30	0.23	0.25
2000	80	15.60	18.20	19.90	21.70	0.29	0.29	0.23	0.25
2180	75	16.20	19.10	20.80	21.50	0.31	0.28	0.24	0.24
2360	70	16.10	18.70	20.50	21.32	0.30	0.28	0.24	0.24
2600	65	15.60	18.80	20.00	21.80	0.30	0.28	0.24	0.25
2780	60	15.80	19.50	21.00	21.70	0.30	0.28	0.25	0.25
3020	55	16.10	18.90	21.30	21.50	0.31	0.30	0.26	0.25
3200	50	16.50	18.80	21.00	21.80	0.32	0.30	0.27	0.27
3380	45	16.00	19.20	20.80	21.90	0.34	0.31	0.28	0.28
3560	40	16.30	19.70	20.50	21.60	0.34	0.32	0.30	0.29
3800	35	16.20	20.00	21.40	21.90	0.36	0.34	0.31	0.31
4040	35	16.80	20.00	21.30	22.50	0.37	0.35	0.33	0.34
4280	35	16.20	20.40	21.50	22.00	0.39	0.37	0.34	0.34
4520	35	16.70	20.10	21.80	22.80	0.38	0.40	0.34	0.35
5000	35	16.40	19.90	21.70	22.40	0.38	0.39	0.34	0.35
6800	35	16.90	20.10	21.60	22.10	0.38	0.39	0.35	0.36
7400	35	16.90	20.20	22.00	22.70	0.39	0.39	0.34	0.37

Time,s	Temp.,°C	Elastic modulus, Pa				Viscous modulus, Pa			
		Control	13.3 V/cm	16.7 V/cm	20 V/cm	Control	13.3 V/cm	16.7 V/cm	20 V/cm
10	75	25.20	20.20	9.10	16.40	3.49	3.43	3.48	3.37
190	80	24.60	20.10	9.20	16.10	3.39	3.38	3.38	3.26
430	85	23.70	19.40	9.30	15.30	3.30	3.33	3.29	3.21
610	90	21.00	16.40	8.85	13.40	3.36	3.36	3.18	2.78
850	95	13.80	11.40	7.50	9.14	2.95	2.95	2.60	2.55
1090	95	10.50	8.44	6.63	6.71	2.35	2.35	2.03	1.70
1400	95	8.37	6.40	4.89	4.98	1.92	1.93	1.66	1.74
1580	90	7.36	5.80	4.44	4.47	2.05	1.98	1.64	1.59
1820	85	6.87	5.35	4.06	4.10	1.92	1.74	1.17	1.60
2000	80	6.71	5.29	4.02	3.92	1.88	1.80	1.46	1.51
2180	75	6.68	5.19	3.97	3.84	1.94	1.75	1.50	1.47
2360	70	6.60	5.15	3.99	3.84	1.86	1.78	1.47	1.54
2600	65	6.61	5.23	4.10	3.85	1.91	1.86	1.49	1.54
2780	60	6.74	5.25	4.08	4.00	1.88	1.89	1.56	1.59
3020	55	6.91	5.51	4.22	4.13	1.96	1.74	1.65	1.56
3200	50	7.02	5.70	4.39	4.20	2.03	2.03	1.68	1.67
3380	45	7.23	5.84	4.57	4.37	2.13	2.16	1.74	1.75
3560	40	7.42	6.02	4.76	4.62	2.13	2.28	1.87	1.83
3800	35	7.73	6.27	5.01	4.85	2.26	2.39	1.95	1.95
4040	35	7.91	6.57	5.22	5.17	2.29	2.50	2.05	2.15
4280	35	8.02	6.71	5.30	5.37	2.42	2.40	2.12	2.14
4520	35	8.11	6.72	5.38	5.35	2.35	2.48	2.11	2.18
5000	35	8.13	6.83	5.43	5.39	2.43	2.50	2.14	2.22
6800	35	8.07	6.81	5.45	5.51	2.42	2.52	2.20	2.23
7400	35	8.04	6.84	5.48	5.49	2.37	2.56	2.16	2.24

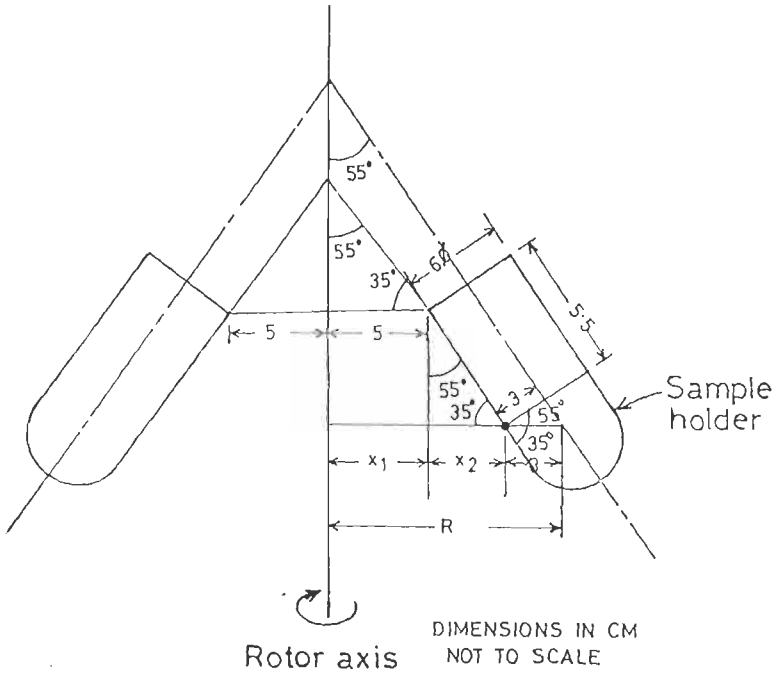
Appendix N Effect of inclined column on settling of cassava starch

Angle of inclination, degree	Starch in supernatant, %			
	Concentration, %			
	2	4	6	8
0	5.62	6.50	8.50	16.50
5	5.38	6.25	8.270	15.81
10	5.25	6.00	8.00	15.35
15	4.80	5.73	7.52	13.65
20	4.25	5.34	7.00	12.51
25	4.190	5.15	6.70	11.80
30	4.00	5.00	6.50	11.42
40	3.33	4.00	5.50	9.50

Appendix O Centrifugal settling of cassava starch

Time, s	Per cent starch settled											
	Peripheral speed of rotor, m/s											
	7.72				15.44				23.16			
	Concentration, %				Concentration, %				Concentration, %			
	2	4	6	8	2	4	6	8	2	4	6	8
15	89.13	93.33	94.41	95.25	94.75	95.33	96.42	97.03	95.25	95.94	97.08	97.60
30	90.38	93.94	94.80	95.81	95.08	95.81	97.16	97.66	96.05	96.50	97.50	97.96
45	91.23	94.50	95.25	96.44	95.53	96.19	97.38	97.87	96.85	97.19	97.95	98.25
60	93.13	95.80	96.98	97.59	95.95	96.43	97.58	97.97	97.44	97.80	98.21	98.58
90	95.25	96.87	97.85	98.28	96.85	97.25	97.99	98.15	97.85	98.05	98.32	98.65
120	96.50	97.56	98.13	98.44	97.88	98.25	98.52	98.60	98.25	98.36	98.62	98.72
180	97.25	97.81	98.45	98.59	98.43	98.50	98.62	98.67	98.43	98.56	98.79	98.82

Appendix P. **Diagramatic representation of a rotor with sample holder employed in centrifugal settling**



Calculation of tangential velocity

From figure, $R = x_1 + x_2 + x_3 = 14.74$ cm

where

R = average radial distance from rotor axis,

$x_1 = 5$ cm, $x_2 = 5.5 \text{ Sin } 55° = 4.51$ cm, $x_3 = 3 \text{ Cos } 55° = 5.23$

Tangential velocity, $V = 2\pi R N/60$ where N = rpm of the rotor

For

500 rpm, V = 7.72 m/s

1000 rpm, V = 15.44 m/s

1500 rpm, V = 23.16 m/s

Appendix Q Comparison of centrifugal settling of dried and fresh starch

Time, sec	Per cent starch settled											
	Peripheral speed of rotor, m/s											
	7.72				15.44				23.16			
	Concentration, %				Concentration, %				Concentration, %			
	Dried starch		Fresh starch		Dried starch		Fresh starch		Dried starch		Fresh starch	
	4	8	3.92	8.10	4	8	3.92	8.10	4	8	3.92	8.10
15	93.33	95.26	93.25	95.18	95.33	97.03	95.09	97.09	95.94	97.60	95.95	97.65
30	93.94	95.81	93.62	95.69	95.81	97.66	95.69	97.65	96.51	97.96	96.49	97.92
45	94.50	96.44	94.21	96.38	96.19	97.87	96.05	97.96	97.19	98.25	97.21	98.27
60	95.80	97.59	95.55	97.53	96.43	97.97	96.35	98.02	97.75	98.58	97.70	98.67
90	96.87	98.28	96.68	98.21	97.25	98.15	97.13	98.09	98.05	98.65	98.09	98.65
120	97.56	98.44	97.45	98.42	98.25	98.60	98.03	98.45	98.38	98.74	98.40	98.75
180	97.81	98.59	97.89	98.42	98.50	98.67	98.25	98.65	98.56	98.82	98.60	98.80

Appendix R Performance of 30 mm hydrocyclone for cassava starch milk concentration

Concentration, %	Feed pressure, kg/cm^2	Feed rate, l/s	Underflow		Overflow		U_{vs}	C_i	E_T, %	E_R, %
			Rate, l/s	Concn., %	Rate, l/s	Concn., %				
2.0	0.6	0.212	0.013	10.00	0.199	1.49	6.13	4.00	30.66	26.13
	1.2	0.292	0.015	15.66	0.277	1.28	5.14	6.83	40.22	36.98
	1.8	0.336	0.017	20.51	0.319	1.02	5.06	9.26	51.89	49.33
	2.4	0.352	0.018	23.31	0.334	0.90	5.11	10.66	59.60	57.42
	3.0	0.375	0.020	24.24	0.355	0.81	5.33	11.12	64.64	62.65
3.5	0.6	0.238	0.014	16.29	0.224	2.75	5.88	3.65	27.37	22.83
	1.2	0.283	0.014	20.97	0.269	2.61	4.95	4.95	29.64	25.98
	1.8	0.265	0.019	24.33	0.246	2.39	7.17	5.95	49.84	45.97
	2.4	0.307	0.022	27.26	0.285	1.60	7.17	6.79	55.81	52.40
	3.0	0.324	0.024	28.98	0.300	1.49	7.41	7.28	61.33	58.24
5.0	0.6	0.246	0.016	20.50	0.230	4.00	6.50	3.10	26.66	21.56
	1.2	0.303	0.020	24.32	0.283	3.63	6.60	3.86	32.10	27.30
	1.8	0.299	0.022	32.01	0.277	2.93	7.36	5.40	47.10	43.21
	2.4	0.336	0.024	36.24	0.312	2.72	7.14	6.25	51.77	48.06
	3.0	0.330	0.024	37.98	0.306	2.48	7.27	6.60	55.24	51.73
6.5	0.6	0.213	0.019	20.92	0.194	5.06	8.92	2.22	28.71	21.73
	1.2	0.315	0.022	29.31	0.293	4.82	6.98	3.51	31.49	26.35
	1.8	0.326	0.026	34.62	0.300	4.16	7.98	4.33	42.48	37.49
	2.4	0.340	0.028	39.21	0.312	3.66	8.24	5.03	49.68	45.17
	3.0	0.351	0.028	42.21	0.323	3.45	7.98	5.49	51.80	47.62
8.00	0.6	0.245	0.021	23.25	0.223	6.61	8.57	1.91	24.91	17.87
	1.2	0.309	0.024	31.85	0.285	6.00	7.77	2.98	30.92	25.10
	1.8	0.340	0.028	36.24	0.312	5.63	8.24	3.53	37.31	31.68
	2.4	0.337	0.029	40.56	0.308	5.19	8.61	4.07	43.63	38.32
	3.0	0.308	0.028	41.43	0.280	4.92	9.09	4.18	47.08	41.79

U_{vs} = Underflow volume split C_i = Increase in U/F concentration x times E_T = Total efficiency E_R = Reduced efficiency

Appendix S Performance of 50 mm hydrocyclone for cassava starch milk concentration

Concentration, %	Feed pressure, kg/cm^2	Feed rate, l/s	Underflow		Overflow		U_{vs}	C_i	E_T, %	E_R, %
			Rate, l/s	Concn., %	Rate, l/s	Concn., %				
2.0	0.6	0.379	0.027	5.20	0.352	1.75	7.12	1.60	18.46	12.21
	1.2	0.571	0.038	6.68	0.533	1.60	6.65	2.34	22.23	16.69
	1.8	0.625	0.042	7.99	0.583	1.51	6.72	3.00	26.85	21.58
	2.4	0.674	0.044	8.65	0.630	1.53	6.53	3.33	28.23	23.22
	3.0	0.729	0.049	9.25	0.680	1.47	6.72	3.63	31.09	26.12
3.5	0.6	0.459	0.032	8.03	0.427	3.20	6.97	1.29	15.99	09.69
	1.2	0.583	0.037	11.50	0.546	2.95	6.35	2.29	20.85	15.49
	1.8	0.673	0.043	12.72	0.630	2.90	6.39	2.63	23.22	17.98
	2.4	0.730	0.046	14.69	0.684	2.76	6.30	3.20	26.45	21.50
	3.0	0.800	0.051	15.54	0.749	2.68	6.38	3.44	28.31	23.43
5.0	0.6	0.425	0.027	10.94	0.398	4.61	6.35	1.19	13.90	08.06
	1.2	0.599	0.038	13.49	0.561	4.43	6.34	1.70	17.12	11.51
	1.8	0.682	0.044	14.49	0.638	4.30	6.45	1.90	18.70	13.09
	2.4	0.720	0.050	15.98	0.670	4.16	6.94	2.20	22.19	16.38
	3.0	0.725	0.053	16.78	0.672	4.02	7.31	2.36	24.53	18.58
6.5	0.6	0.427	0.028	13.21	0.399	6.04	6.56	1.03	13.33	07.25
	1.2	0.562	0.039	14.59	0.523	5.89	6.94	1.24	15.58	9.28
	1.8	0.686	0.047	17.02	0.639	5.72	6.85	1.62	17.94	11.90
	2.4	0.728	0.049	18.96	0.679	5.62	6.73	1.92	19.64	13.84
	3.0	0.728	0.052	19.65	0.676	5.48	7.14	2.02	21.59	15.56
8.0	0.6	0.478	0.030	15.85	0.448	7.46	6.28	0.98	12.43	06.57
	1.2	0.631	0.041	17.00	0.590	7.37	6.50	1.13	13.81	07.82
	1.8	0.659	0.046	18.98	0.613	7.10	6.98	1.37	16.56	10.30
	2.4	0.700	0.053	20.09	0.647	6.82	7.57	1.51	19.01	12.38
	3.0	0.796	0.055	22.00	0.741	6.96	6.91	1.75	18.99	12.98

U_{vs} = Underflow volume split C_i = Increase in U/F concentration, E_T = Total efficiency E_R = Reduced efficiency

Appendix T. Cost analysis of modified battery of hydrocyclones

Materials required	Quantity	Rate, Rs.	Cost, Rs.
GI sheet (22 Gauge)	60 x 30 cm	20/ft^2	40.00
Flexible hose ¾"	15 m	10/m	150.00
MS pipe ¼"	1.25 m	150/m	188.00
Coupling ¼"	9 no.	4/no.	36.00
Bent ¼"	10 no.	4/no.	40.00
Elbow ½"	1 no.	15/no.	15.00
Pressure gauge	1 no.	300/no.	300.00
Valve ½"	1 no.	75/no.	75.00
Shellak	1 no.	20/no	20.00
Reducer ½ - ¼"	1 no.	15/no.	15.00
Strainer	1 no.	75/no.	75.00
Hose clamp	2 no.	4/no	8.00
Pistom pump with motor- 3hp	I no	10000	10000.00
		Total cost of the material	= 10962.00

Actual labour charge	=	Rs. 600/-
Overhead charges	=	Rs. 200/-
Total cost of the unit	=	**Rs. 11762/-**

Assumptions made

Life of hydrocyclone	-	5 yrs
Life of pump	-	10 yrs
Salvage value	-	10% cost of the unit
Interest on investment	-	13%
Total working hours/yr	-	1000
Repairs and maintenance (no moving parts)	-	2%
Tax/insurance	-	2% cost of unit

Fixed cost of hydrocyclone unit (excluding pump)

Initial cost of unit	=	Rs. 1762/-
Salvage value	=	$1762 \times 0.10 \approx$ Rs. 177/-
Depreciation	=	$\frac{1762 - 177}{5}$ = Rs. 317
Interest	=	$\frac{(1762+177) \times 0.13}{2}$ = Rs. 126
Tax/Insurance	=	1762×0.02 = Rs. 36
Fixed cost of hydrocyclone	=	Rs. 480/-

Fixed cost of pump

Useful life	=	10 year
Cost of pump	=	Rs. 10000/-
Salvage value	=	$\frac{10000 \times 10}{100}$ = Rs. 1000/-
Depreciation	=	$\frac{10000 - 1000}{10}$ = Rs. 900/-

Interest on investment = $\frac{10000 + 1000 \times 0.13}{2}$

= Rs. 715/-

Tax/Insurance = 10000 x 0.02 = Rs. 200/-

Fixed cost of pump = Rs.1815/-

Variable cost of the unit

Labour charge @ 75/day (8 h) = 75/8 = Rs.9.4/h

Electricity charge @ 2.5/kw = 3 hp = Rs.5.6/h

Repairs/maintenance @ 10% for pump and 2% for hydrocyclone. = $\frac{10000 \times 0.10 + 1762 \times 0.02}{1000}$

= Re. 1/h

Total variable cost/h = Rs.16/-

Total fixed cost/yr = fixed cost of hydrocyclone + pump

= Rs. 480+1815 = 2295

Fixed cost/h = Rs.2.3/-

Total operational cost = 16+2.3 = Rs.18.3/h

Feed rate of the unit = 14 l/min

= **840 l/h = 0.84 m^3/h**

The total operational cost of the system was found to be Rs.18.3/h. When a single battery of five hydrocyclones (30 mm diameter) is operated at 3 kg/cm^2 at 3.5% feed concentration, an overflow stream of 1.56% concentration was obtained. Feed rate of the unit is 0.84 m^3/h Hence, to concentrate one cubic meter of starch milk to give an overflow concentration of 1.56% the operating cost was 21.79 ≈ Rs.22/-.