

**DILATOMETRIC STUDY OF SOLID/LIQUID
FRACTIONS OF MILKFAT FROM
DIFFERENT SPECIES**

DISSERTATION

SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS
FOR THE DEGREE OF

Master of Science

IN

DAIRYING

(**DAIRY CHEMISTRY**)

TO THE KURUKSHETRA UNIVERSITY
KURUKSHETRA

By

PRITPAL SINGH KALSI

B. Sc.

DIVISION OF DAIRY CHEMISTRY
NATIONAL DAIRY RESEARCH INSTITUTE
(I. C. A. R.)
KARNAL (HARYANA) INDIA

1984

Registration No. 81-DK-34

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FRIEND
LATE S. PARAMJIT SINGH SOHAL

Dr. G.S. SHARMA
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Dated Jan., 30, 1984

I certify that the work reported in this dissertation entitled "DILATOMETRIC STUDY OF SOLID/LIQUID FRACTIONS OF MILKFAT FROM DIFFERENT SPECIES", was carried out by Mr. PRITPAL SINGH KALSI, under my guidance, in partial fulfilment of the requirement for his Degree of Master of Science in Dairying (Dairy Chemistry) of the Kurukshetra University.


(G.S. SHARMA)

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P. Kalsi

(PRITPAL SINGH KALSI)

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INTRODUCTION

(Including literature review)

Ghee, the clarified butterfat is one of the most important sources of oils and fats in the Indian diet. Fat is generally at a premium in the Indian diet and a high fat product is always considered better than a low fat product. Ghee also has an important place in the Ayurvedic and Unani system of medicines, as it is used as a base material in the preparation of several medicines in these systems. It is

used from time immemorial in the religious functions too. Further, a large population in India is vegetarian and to them ghee is the only acceptable fat from animal origin. The current production of ghee is estimated to be 5 lakh tonnes a year (Anaja, 1982). The extent of the level of ghee production in India speaks of the diversion of the major quantity of the milk production towards this product. The reason may be the easy manufacturing method with simple equipments and techniques and also the good shelf life under the tropical conditions of storage and ordinary packing. Also milk being highly perishable item, its conversion into ghee was considered to be the best and safest approach to consume one of the most important and desirable component of milk, that is, milkfat.

Attempts have been made through continuous research in the processing of milk to ghee with special reference to the development of typical ghee flavours and grainy texture in ghee and also its preservation. The aim is to develop a suitable process to make good quality ghee. At present, three conventional methods are followed for the preparation of ghee, namely;

- I. Direct cream method - centrifugal separation of high fat cream and its direct boiling along with the serum solids.

II. Creamry butter method - churning of ripened or unripened cream to white butter and its conversion in ghee.

III. Desi method - preparation of dahi, churning it into white butter and finally conversion in ghee.

The unorganised sector in rural areas follows the desi method, while the commercial scale manufacturing units have the choice for the creamry butter method. It is true that the ghee made by our modern dairies is not as good as the traditionally made desi ghee. The desi ghee sells at higher prices. It is considered superior because it has better grains, it keeps longer and also it has pleasant flavour. These are probably the main aspects for its higher price. Probably the secret lies in the liquid fraction of milkfat. In the traditional process there is natural selection of fat and a large portion of the liquid fat is left out in the butter milk. The solid fraction, thus, recovered has longer life and better grains. In the traditional process, the total recovery of ghee is only 80% of the fat.

The melting and solidification characteristics of milk fat not only are of practical importance to the

processing of dairy products but represent intriguing examples of the physical equilibria that operate in the fat phase. Milk fat is composed principally of mixed triglycerides differing widely in physical characteristics. These triglycerides have most different melting points. Milk fat, therefore, has no sharply well defined melting point but melts over a wide range of temperature called melting interval. Unless precautions are taken to assure that completely liquified fat is cooled rapidly to low temperatures ($\approx -10^{\circ}\text{C}$), variable melting characteristics are noted. Similarly, variations in the solidification temperature, i.e., first appearance of the solid phase, of milk fat reflect the cooling procedure. Rahn and Sharp (1928) reported solidification points of 19.7 and 23.6 $^{\circ}\text{C}$ for samples of the same fat cooled by immersion at 14 and 20 $^{\circ}\text{C}$, respectively. Mulder (1953) cited one example in which the solidification and melting points were separated by approximately 19 $^{\circ}\text{C}$. A number of other workers (Mohr and Baur, 1939; Richardson, 1936) also noted the similar variable melting and solidification characteristics of milk fat. Mulder (1953) concluded that 'the lower the temperature at which the fat is caused to solidify (within the solid-liquid region) the lower is the melting point'. The rate of cooling has, thus, an effect on the melting and solidification points

of milk fat. It was also concluded that liquid fat should be cooled rapidly to a temperature below the solid-liquid region before carrying out measurements of characteristic figures such as melting point, solidification point and melting interval. A possible method for calculating the proportions of solid and liquid fat was also mentioned. Mulder and Klomp (1956) reported the proportion of solid and liquid glycerides at different temperatures in winter and in summer cream after the fat had crystallised at temperature of melting ice. At a temperature of 18°C , about 50% of the winter fat was in the solid state while at the same temperature about 35% of the summer fat was in the solid state. Hennewijk (1955) reported 50 - 70% solid fat in butter fat at 5°C .

Eres'ko and Rabotyakoya (1977) reported that the percent of solid and liquid phase in butter was an important factor indicating the structural, mechanical and physical properties of butter. The dilatometric method was considered the accurate method for the determination of solid and liquid phase in butter fat. Kankare and Antila (1974) observed that the spreadability of butter can be significantly improved by the use of liquid milkfat fractions. During the indoor feeding season (I value of milkfat 29.0 - 30.9 and mp about 32°C), the fraction can be added in non-emulsified form

directly to the cream before churning at a rate of upto 33% of the total cream fat content. The temperature stability of the butter (no oiling off at 20°C for 4 hrs) as well as its keeping quality at 5°C, was good.

Schultz and Timmen (1966) described the experiment on milk fat fractions and its possible application. It was suggested that the admixture of corresponding amounts of separated hard or soft fat fractions to milkfat may serve to standardise the consistency of the butter produced. Fractions of soft milk fat (liquid fractions) with a high content of unsaturated fatty acids may be used in the production of diatic milk products. The use of fractions of hard milk fat was suggested for manufacture of dried butter for tropics. Dacker (1970) carried the study on the temperature crystallisation and separation of butter fat into fractions ranging in mp from 13°C to above 30°C. It was observed that the most liquid fraction is highly coloured and flavoured and should find other use in addition to the improvement of the spreadability of butter.

Norris et al. (1973) determined the liquid fat content at different temperatures from melting curves. Also the correlations between percent of liquid fat and weight % of short chain fatty acids as well as with cis unsaturated

acids were determined. Norris et al. (1971) observed the increased concentration of unsaturated and short chain fatty acids in liquid fraction and that of long chain saturated fatty acids in the solid fraction.

The rheological properties of products with a high content of fat as for example butter and margarine are to a great extent determined by the ratio between solid and liquid fat. This has been shown by using different measuring methods (IDF, 1982). The ratio of solid to liquid fat in butter fat can be used to characterise its physical properties. Mortensen and Denmark (1974) showed that the iodine value of butter fat gives a good measure of the level of solid and liquid fat in it and hence the consistency characteristics.

Deuel et al. (1943) have shown that the digestibility of fats and their melting points are closely related. It was observed that the fats of low melting point are better digested than those with higher melting points.

Thermal expansion, one of the indirect common method for the determination of solid/liquid fraction in milk fat, has also been studied by a few workers in case of cream, owing to its importance in dairy processing. Smith and Doan (1951) determined expansion of the cream volume of pasteurized fluid

milk by the addition of reconstituted superheated skim milk, and utilized the same to detect adulteration of pasteurized milk with the latter. Fernandez-Martin (1970) conducted dilatometric study of cream and observed the three areas of marked crystallization. The most important of those was around 15°C . Gulyaev-Zaitsev (1967) determined thermal dilation coefficient for seven samples of milk fat obtained at different times of the year which ranged from 0.867 to $0.925 \text{ mm}^3/\text{g}/^{\circ}\text{C}$. A comparable result on thermal expansion of liquid butter fat was also reported by De Man and Wood (1959), i.e., $8.5 \text{ mm}^3/10 \text{ g } ^{\circ}\text{C}$ between $40 - 60^{\circ}\text{C}$. The maximum thermal expansion of cream observed by Vandam (1922) was between $12 - 18^{\circ}\text{C}$.

Mulder (1953) reported the maximum increase in the volume of the cream between 10 and 20°C which means that much fat probably changes from the stage of aggregation in this temperature region.

It is ample clear from the preceding paragraphs that the knowledge of the solid and liquid fraction of milk fat is of utmost importance in dairy processing in many aspects. But from the perusal of the literature one could see that the information on this aspect of milk fat is in scanty. Such property of buffalo (Bubalus bubalis) milk fat has yet not been

studied. The same is true in case of milk fat from cow of Indian origin (Bos indicus). The milkfat from goat which may become a significant ingredient of Indian dairy industry in future also needs attention for similar study. Such information on this milkfat is also not available. Further thermal expansion of milkfat (ghee) plays a very important role in its packaging and processing too. But the data on this thermal property of ghee from milk of all the three species (buffalo, cow and goat) are not available in the literature. Also in case of cream, the base raw material for manufacturing ghee, such study needs attention, as a few western workers have attempted the same.

It was, therefore, felt that a comprehensive knowledge of solid and liquid fractions in milkfat and its thermal expansion, a matter being of considerable technological interest, is likely to be of great help to the dairy industry. The well established compositional differences of milkfat from buffalo, cow and goat needs the work for comparative study too. Consequently, a research programme was undertaken to investigate into these aspects of milkfat and cream.

PROGRAMME OF RESEARCH WORK

The project work was taken up in two phases, as outlined below:

PHASE I

1. Fabrication and assembling of the instruments, and standardisation of the same for the determination of thermal expansion of milkfat and cream from different species.

PHASE II

1. Preparation of cream and milk fat (ghee) samples from buffalo, cow and goat milk.
2. Determination of fat in cream samples.
3. Estimation of moisture in ghee samples.
4. Determination of thermal expansion of milkfat from different species (buffalo, cow and goat)
5. Determination of coefficient of thermal expansion of milkfat and cream from three species.
6. Determination of solid/liquid fractions of buffalo, cow and goat milkfat.
7. Statistical analysis of the results obtained.

Available informations on thermal expansion of milkfat and cream and solid/liquid fractions of milkfat are reviewed in the preceding chapter. Materials used and experimental methods followed to study the thermal expansion and solid/liquid fractions of milkfat, results obtained from such studies and critical discussions on these results including statistical analysis of the data are described in chapters 2 and 3. A summary of the results and relevant conclusions obtained from the investigation is presented in chapter 4. A bibliography containing references to various earlier works mentioned in this study followed chapter 4.



MATERIALS AND METHODS

Milk samples

Composite bulk milk samples were collected from the animals (buffalo, cow and goat) of the Institute herd. Animals were under identical conditions of feeding and management throughout the sampling period. All the milk samples were collected with a short span of seven weeks to have the same composition of fat and avoid the seasonal effect on it.

Preparation of milkfat

Cream: - The whole milk was warmed on water bath to about 45°C and skimmed at 1200 r.p.m using a hand operated cream separator. The cream so obtained was collected for further experimental purposes.

Ghee: - Ghee samples were prepared from the milk collected from all the three animals, e.g., buffalo, cow and goat. Creamry butter method was followed for the preparation of ghee.

The cream obtained was pasteurized and cooled to room temperature. It was inoculated with about 2% (by volume) starter culture (DRC_1) obtained from the Dairy Bacteriology Division of the Institute. After mixing cream sample thoroughly it was ripened for overnight in B.O.D. at 30°C , then the cream was churned into butter under standard conditions. The butter was washed with water and the creamry butter so obtained was heated to 112°C in a stainless steel vessel for 3 - 4 minutes until slight brown residue appears. After cooling the melted fat to room temperature, it was filtered through a muslin cloth. The clear filtrate, i.e., ghee was used for the investigation.

Estimation of fat in cream

The fat in cream was estimated by Gerber method (I.S. 1966).

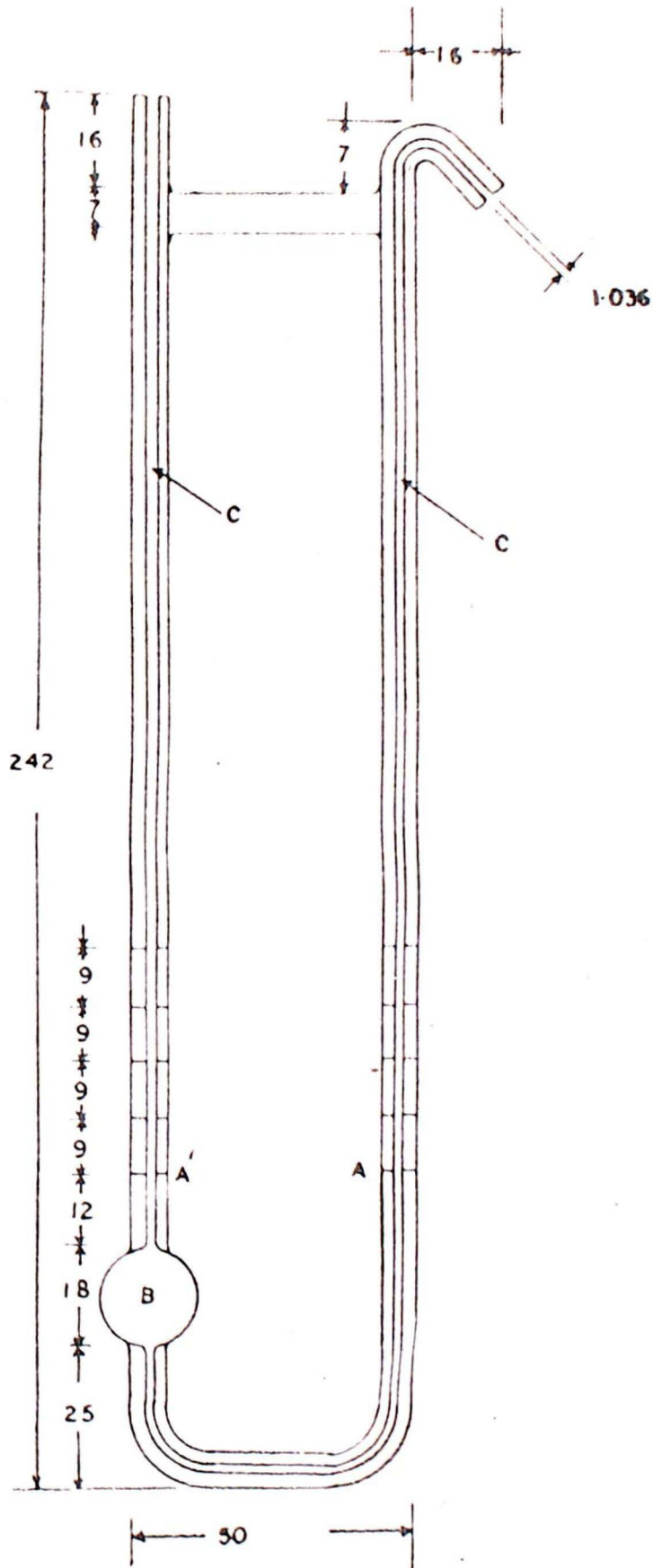
10 ml of sulphuric acid (density, 1.807 - 1.812 g/ml at 27°C) was taken in a calibrated butyrometer and 5.0 g of accurately weighed well mixed cream sample was layered gently on it followed by 1 ml of amyl alcohol (density, 0.803 - 0.805 g/ml at 27°C). Butyrometer reading was taken at $65 \pm 2^\circ\text{C}$. Average of duplicate analyses was recorded in g/100 ml.

Estimation of moisture in ghee

The moisture in ghee samples was determined gravimetrically following the procedure recommended by the Indian Standards Institution (I.S. 1966). The results as average of duplicate observations were expressed in g/100 g.

Determination of thermal expansion

Equipment: - Thermal expansion of the samples was determined with a specially fabricated microdilatometer. It was made of corning glass of known cubical expansion ($0.096 \times 10^{-4}/^\circ\text{C}$.) in the shape of two long armed U, as described through the diagram in Fig. 1. The capillary tube



A,A-GRADUATION B-BULB C,C-CAPILLARY
 ALL DIMENSIONS IN MM

FIG. I. THERMAL EXPANSION DILATOMETER.

used for the dilatometer was tested for uniformity of its bore by the standard method of introducing a known weight of mercury thread in the capillary and measuring its length at different places with a cathetometer. The accurate radius of the capillary (0.0518 cm) was calculated from the experimentally determined average length of the mercury thread inside the capillary and its weight. The volume of the dilatometer upto the two marks AA' in Fig. 1 was determined by completely filling it with mercury upto the marks and subsequently from the weight of mercury needed to fill up and its density at the specific temperature. The volume of the dilatometer at room temperature (20°C) was, thus, calculated to be 1.9468 ml.

A thermostatically controlled water bath maintained with in $\pm 0.05^{\circ}\text{C}$ of any desired temperature was used for equilibrating the dilatometer with sample at desired temperature. The dilatometer was placed vertically dipped inside the water bath with suitable stand, clamp etc. allowing only the tips of the two limbs to protude above the water surface.

The dilatometer was thoroughly cleaned using successively teapole, tap water, warm chromic acid, tap water and distilled water, and was dried with ethanol followed by diethyl ether and stream of dust free air.

The sample was filled upto the marks AA' or near about.

In the later case, that is, liquid level standing above AA', the weight of the column in both the limbs of the dilatometer above or below AA' was measured accurately with cathetometer and the corresponding volume of liquid was calculated and added or subtracted to that upto AA' to get the actual initial volume of the sample. About 20 minutes period was allowed for equilibration at each temperature which ranged from 10 to 80°C at intervals of 5°C. The expansion of the liquid between two successive temperature was calculated from the rise of the milkfat or cream menisci in the two verticle limbs of the dilatometer accurately measured with a cathetometer. The coefficient of thermal expansion of the sample was calculated from the observed expansion, the volume of the sample at the initial temperature and the temperature rise. The correction for the cubical expansion of the glass of the dilatometer was not applied in the results reported.

For measurements of the heights of the menisci in the limbs of the dilatometer a cathetometer with a short focus telescope (Oriental Scientific Apparatus Workshop, India) was used. The cathetometer was fitted with a direct reading micrometer controlling movement of the telescope carriage and gave direct reading upto 0.001 cm within working length of 100 cm.

The thermal expansion was calculated in terms of volume as well as the coefficient of the thermal expansion in the

range of 10° to 80°C .

Ghee samples, before transfer to the dilatometer, were deaerated in a vacuum flask under reduced pressure (5 - 10 mm of Hg) for at least 30 minutes at 90°C with occasional violent shaking. Similar treatment was given to the cream samples for 5 minutes at 40°C . After deaerating the ghee and cream, the samples were brought to room temperature and transferred into the dilatometer for further observation of thermal expansion.

Determination of ratio of solid to liquid fat in ghee.

The determination of the ratio of solid to liquid fat in ghee by dilatometry is based on the change in the volume which occurs when fat goes from a solid to liquid state. The change in the specific volume can be measured accurately in the dilatometer at different temperatures as described in the preceding paragraphs under the heading "Determination of thermal expansion". The specific volume is recorded as a function of temperature on a graph to determine the solid/liquid fat content in ghee at various temperatures as per the standard procedures (IDF, 1982). At low temperature where the fat is almost solidified, a straight line with a small incline is obtained as the result of the expansion of the solid fat, and at high temperature a corresponding straight line with an

incline due to the expansion of melted fat appears, with inbetween a raising curve. The estimation of solid and liquid fractions of fat is made from the extrapolated straight lines.

In the present investigation the curves have been plotted between the volume and temperature, as the mass of the contents in the dilatometer was kept same during the complete observation of thermal expansion of milkfat sample over $10-60^{\circ}\text{C}$, and hence amounting to the same results of solid/liquid fractions .

Statistical analysis

All the results were statistically analysed on a programmable Micro-computer (HCL-2200) and standard error (S.E) in all the cases was estimated according to Snedecor and Cochran (1968).

RESULTS AND DISCUSSION

The dilatometric study was carried out on the thermal expansion of milkfat, its solid/liquid fraction, and thermal expansion of cream from milk of buffalo, cow and goat at various temperatures using the equipments and methodology described in chapter II. Results from such study, statistical analyses and discussion thereon are presented here under different headings.

Thermal expansion of milkfat (Ghee).

Thermal expansion of milkfat made from milk of all the three species, buffalo, cow and goat was measured between 10 and 80°C at an interval of 5°C. In case of milkfat from buffalo milk the average volume in (mm³) with standard error for 8 samples increased from 2.0339 ± 0.00549 at 10°C to 2.1380 ± 0.00437 at 80°C with intermediate values at temperatures over this range (Table 1). The respective values for cow and goat milkfat were observed to change from 2.0325 ± 0.00697 to 2.1494 ± 0.00617 and from 2.0242 ± 0.00442 to 2.1502 ± 0.00283 (average for eight samples in each case), respectively (Table 1) with intermediate values at temperatures between 10 and 80°C. The average values for moisture in milkfat from buffalo, cow and goat milk were 0.379 ± 0.0354, 0.318 ± 0.0233 and 0.270 ± 0.0950, respectively. The respective ranges for all these average moisture values were 0.250 to 0.425, 0.200 to 0.400 and 0.160 to 0.370.

Factors influencing thermal expansion of milkfat (ghee).

To study the effect of temperature and different species (buffalo, cow and goat) on thermal expansion of milkfat the data presented in preceding paragraph (also in Table 1) were statistically analysed and the results there from are presented

TABLE 1

Volume of milkfat at various temperatures

Temperature (°C)	Volume (mm ³)		
	Buffalo milk fat	Cow milkfat	Goat milk fat
10	2.0339 ± 0.00549	2.0325 ± 0.00697	2.0242 ± 0.00442
15	2.0340 ± 0.00549	2.0327 ± 0.00692	2.0245 ± 0.00450
20	2.0343 ± 0.00550	2.0331 ± 0.00696	2.0250 ± 0.00444
25	2.0348 ± 0.00549	2.0356 ± 0.00617	2.0430 ± 0.00378
30	2.0397 ± 0.00560	2.0518 ± 0.00582	2.0602 ± 0.00364
35	2.0550 ± 0.00573	2.0676 ± 0.00605	2.0710 ± 0.00354
40	2.0650 ± 0.00550	2.0779 ± 0.00642	2.0798 ± 0.00323
45	2.0743 ± 0.00518	2.0870 ± 0.00643	2.0888 ± 0.00306
50	2.0860 ± 0.00493	2.0960 ± 0.00639	2.0977 ± 0.00296
55	2.0948 ± 0.00486	2.1062 ± 0.00681	2.1069 ± 0.00290
60	2.1036 ± 0.00474	2.1146 ± 0.00662	2.1157 ± 0.00281
65	2.1129 ± 0.00460	2.1230 ± 0.00659	2.1244 ± 0.00265
70	2.1207 ± 0.00447	2.1327 ± 0.00611	2.1329 ± 0.00269
75	2.1291 ± 0.00441	2.1409 ± 0.00616	2.1416 ± 0.00276
80	2.1380 ± 0.00437	2.1494 ± 0.00617	2.1502 ± 0.00283

Results are average of 8 samples in each category (Buffalo, cow and goat) with average moisture of 0.379 ± 0.0354, 0.318 ± 0.0233 and 0.27 ± 0.0950, respectively.

in Tables 2 and 3. The results were also graphically plotted (Figs. II to IV) to effect visual evaluation of the influence of temperature on thermal expansion of milkfat from buffalo, cow and goat milk.

The experimental data of thermal expansion of milkfat from all the three species could be best fitted to the temperature by regression equations of third order. The regression coefficients for such equations, their variations in regression (R^2) and correlation coefficients (r) are given in Table 2. The regressions correlating thermal expansion of milkfat respectively from buffalo, cow and goat milk to the temperature are given by the following equations:

$$Y = 2.0463 - 2.0671 \times 10^{-3} (t) + 8.3532 \times 10^{-5} (t^2) - 5.4731 \times 10^{-7} (t^3) \quad \text{I}$$

$$Y = 2.0308 - 7.9492 \times 10^{-4} (t) + 6.4466 \times 10^{-5} (t^2) - 4.5598 \times 10^{-7} (t^3) \quad \text{II}$$

$$Y = 2.0033 + 1.2601 \times 10^{-3} (t) + 2.2070 \times 10^{-5} (t^2) - 1.9181 \times 10^{-7} (t^3) \quad \text{III}$$

The values for variation in regression (R^2) for the Equations I, II and III are respectively 99.59, 99.22 and 99.11 percents, which indicate that a very high proportion of the variance of thermal expansion can be attributed to its third order regression on the temperature of milkfat in each category. The correlation coefficients (r) derived from these (R^2) values, between thermal expansion and temperature for milkfat from buffalo, cow and goat milk were 0.9979, 0.9961 and 0.9956, respectively, (Table 2). These values confirmed that the influence of temperature on thermal expansion of milkfat was significant (at $P = 0.01$) for all the three species.

The average results for change in volume at various temperature and the regression lines based on expected values according to the above equations are delineated in Figs. II, III and IV for buffalo, cow and goat milkfat, respectively. It is observed from these figures that inspite of an overall increase in volume of milkfat with rise in temperature between 10 to 80°C, the maximum increase was observed within a specific range of temperature. While in buffalo milkfat, the major increase in the volume was between 25 to 50°C, the same in case of cow and goat milkfat was observed between 22.5 to 45°C and 20 to 35°C, respectively. Towards the lower side of the above temperature ranges, there was almost negligible

TABLE 2

Coefficients for polynomial regression of thermal expansion of various milkfats on temperature¹

$$(\text{Regression equation } Y = b_0 + b_1 t + b_2 t^2 + b_3 t^3)$$

Sr. No.	Sample	Value of b's				Variation in regression (R^2)	correlation coefficient (r)
		b_0	b_1	b_2	b_3		
1.	Buffalo milkfat	2.0463	-2.0671×10^{-3}	8.3532×10^{-5}	-5.4731×10^{-7}	0.9959	0.9979 **
2.	Cow milkfat	2.0308	-7.9492×10^{-4}	6.4466×10^{-5}	-4.5598×10^{-7}	0.9922	0.9961 **
3.	Goat milkfat	2.0033	1.2601×10^{-3}	2.2070×10^{-5}	-1.9181×10^{-7}	0.9911	0.9956 **

¹ Results are for 8 sets of samples of milkfat in each category incorporated in Table 1.

** Significant at $P = 0.01$

FIG. II THERMAL EXPANSION OF SOLID AND LIQUID MILK FAT (BUFFALO).

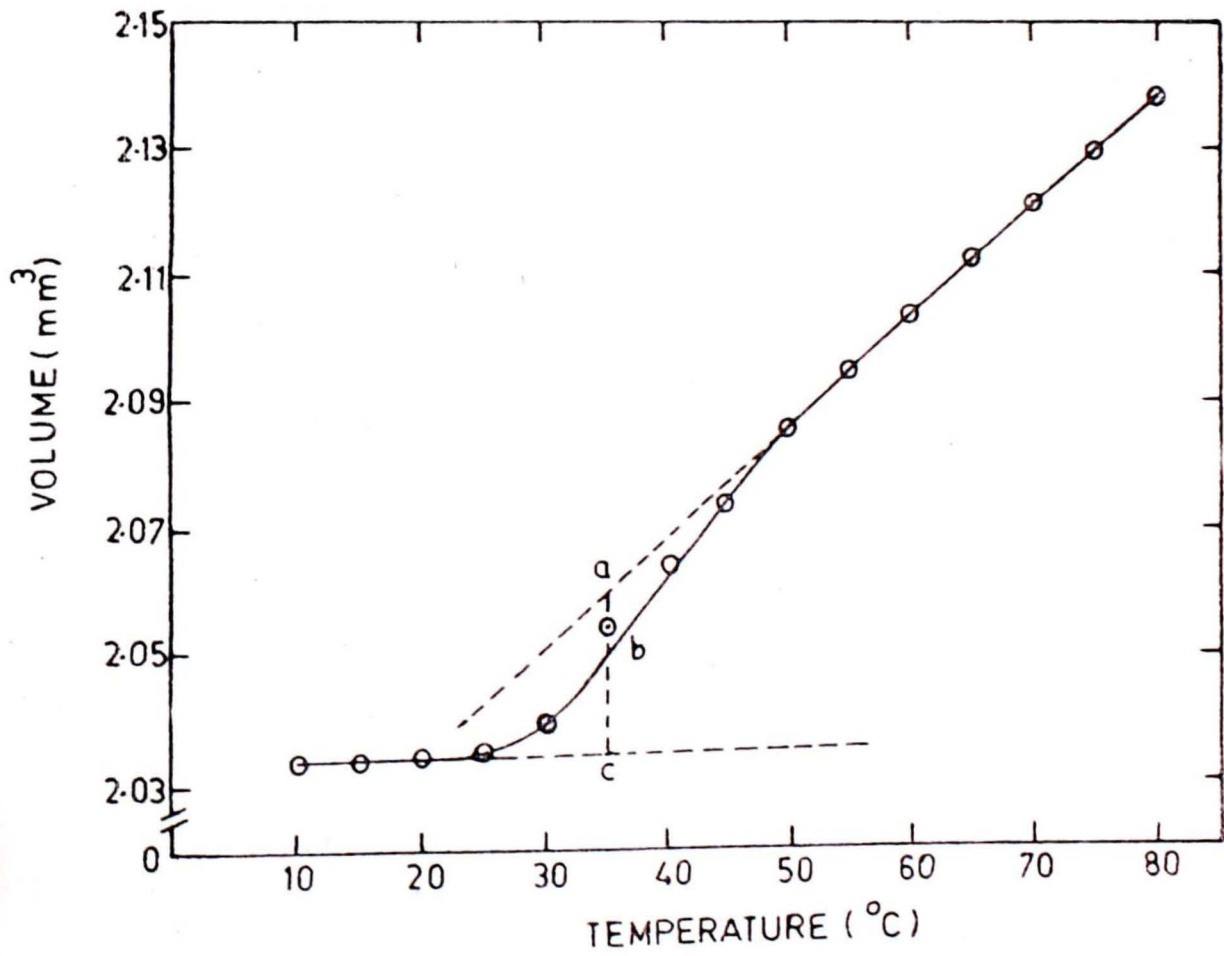


FIG. III THERMAL EXPANSION OF SOLID AND LIQUID MILK FAT (COW).

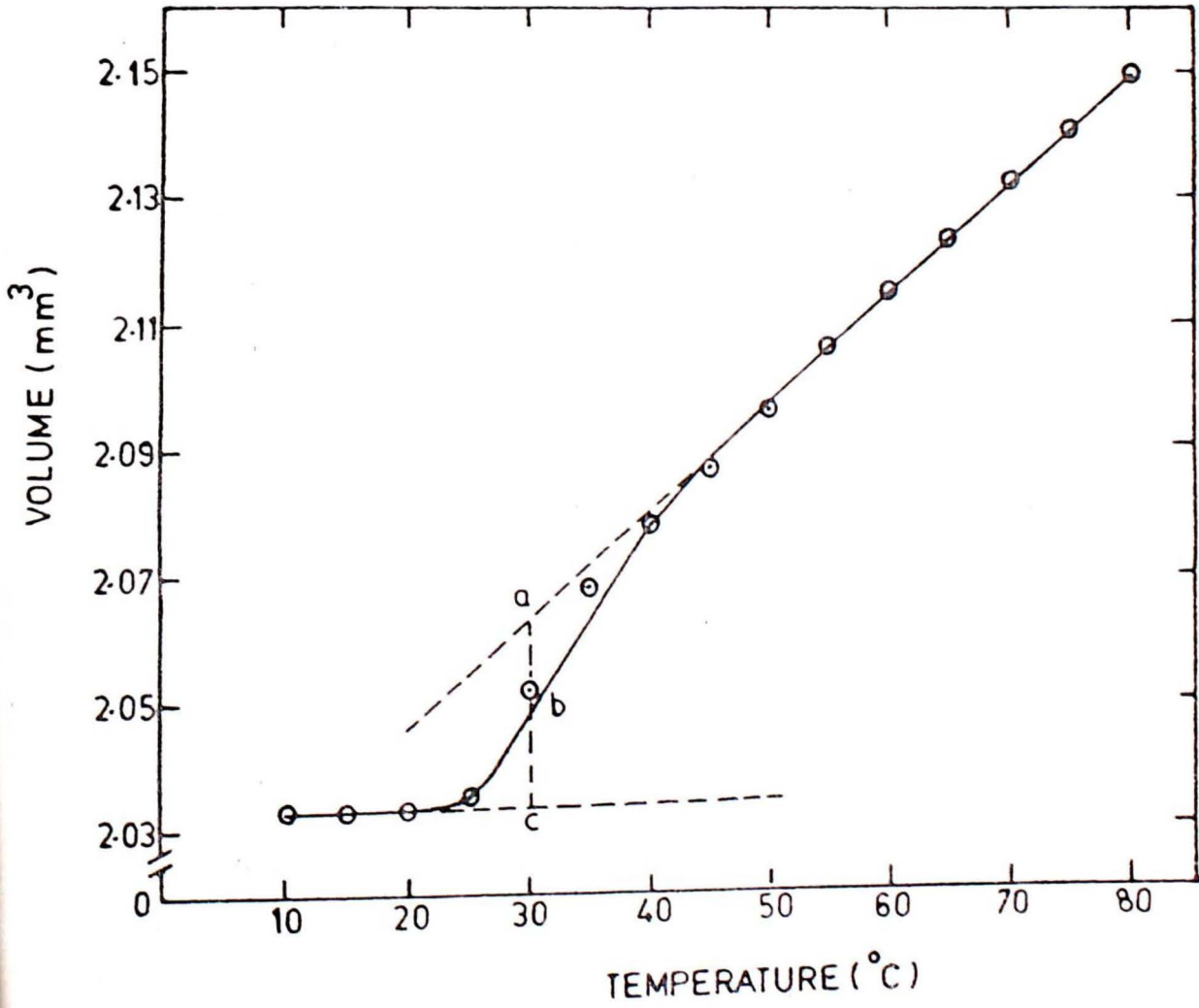
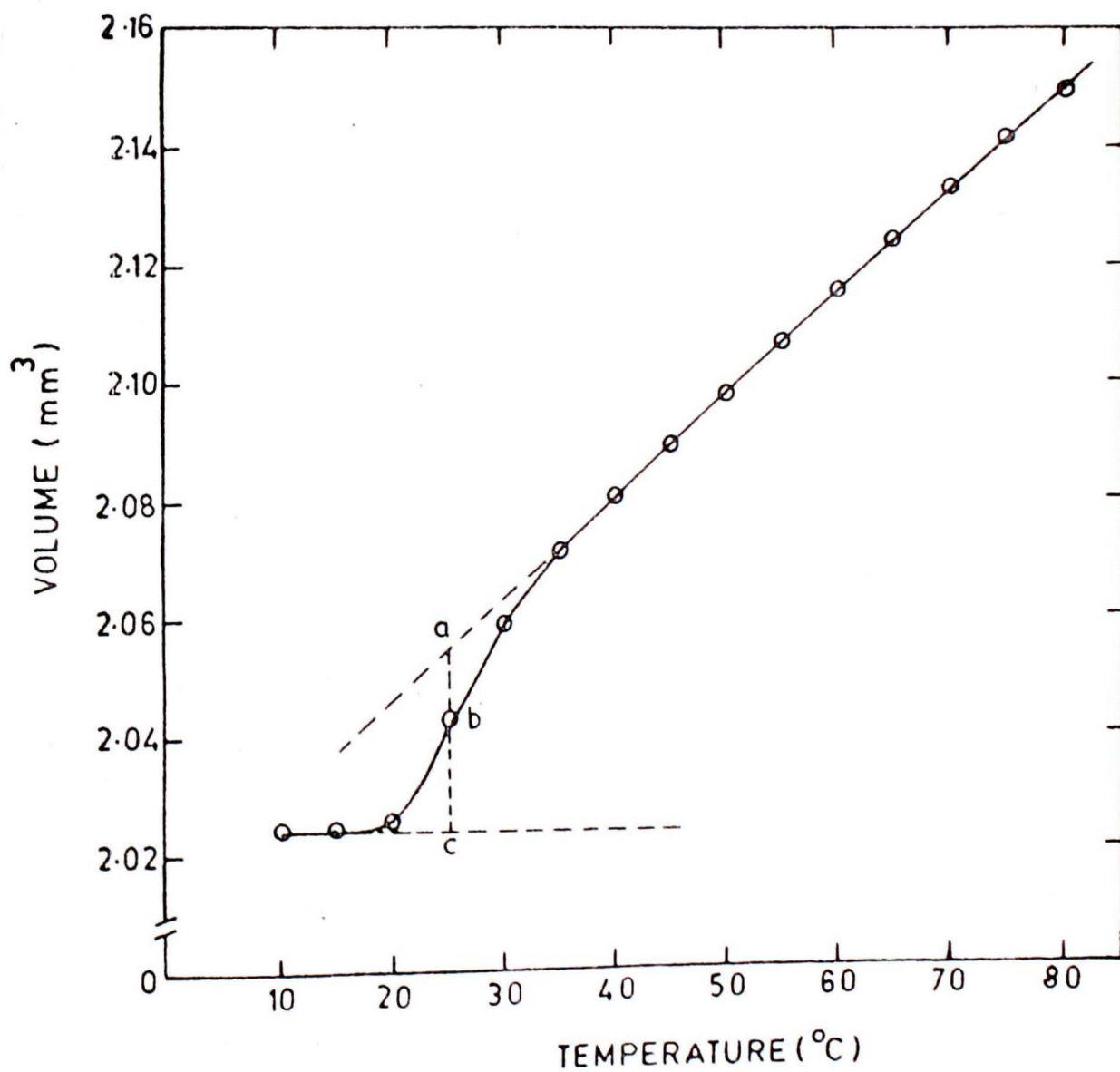


FIG. IV THERMAL EXPANSION OF SOLID AND LIQUID MILK FAT (GOAT).



change in volume of milkfat from all the three species, while on the upperside of these temperature ranges an almost uniform and regular change was observed. A detailed discussion on these results would be presented in the following paragraphs. However, it was concluded from these results that the milkfats from buffalo, cow and goat had different melting intervals, i.e., 28.3°C , 26.0°C and 20.0°C , respectively.

The effect of both, temperature and species on thermal expansion of milkfat is further illustrated by the data recorded in Table 3 through analysis of variance. It is seen from these results that the thermal expansion was significantly ($P = 0.01$) influenced by both, temperature and species. The interaction between the influence of temperature and species on thermal expansion was not significant. However, the variation in thermal expansion due to individual sample was significant which could be attributed to the experimental fact that the original volume of milkfat taken in the dilatometer could not be kept same for all the samples due to technical problems.

Solid and liquid fractions of milkfat (ghae)
at various temperatures.

The values for solid and liquid fractions of milkfat from all the three species were calculated using the curves plotted in figs. II, III and IV according to the procedure

TABLE 3

Analysis of variance for the influence of temperature and species on the thermal expansion of milkfat¹

Sr. No.	Source of variation	df	SS	MSS	F
1.	Between samples	7	0.0114	1.6376×10^{-3}	9.1201**
2.	Between species	2	0.0074	3.6779×10^{-3}	20.4823**
3.	Between temperature	14	0.5527	3.9477×10^{-2}	219.8502**
4.	Interaction between temperature and species	28	0.0064	2.2977×10^{-4}	1.2796 NS
5.	Error	308	0.0553	1.7956×10^{-4}	

1 Results pertain to the data recorded in Table 1.

df Degrees of freedom
 SS Sum of squares
 MSS Mean sum of squares
 ** Significant at P = 0.01
 NS Not-significant
 F Variance ratio

described in I.D.F. (1982). The fraction of solid fat at any temperature is given by ab/ac and the liquid fraction by bc/ac (a, b, c points are marked in figs.). The results so obtained in case of all three species are recorded in Tables 4, 5 and 6.

Buffalo milkfat

The fraction of solid and liquid fat in the milkfat from buffalo were determined at different temperatures between 22.5 to 50.8°C. The results are recorded in Table 4. From the results, it was observed that at 22.5°C there was no liquid fraction present in buffalo milkfat. As the temperature increased the liquid fraction also increased and at 50.8°C (point of inflexion for the curve represented by Eqn. 1 and Fig. II) the fat was almost cent percent liquid. The increase in the magnitude of liquid fraction of fat with increase in temperature was observed irregular as depicted through the data recorded in Table 4 at a temperature interval of 1°C. The interesting observation in buffalo milkfat was that at 33°C the liquid and solid fractions existed almost in equal magnitude. The melting interval for this milkfat was calculated 28.5°C.

Cow milkfat

The results recorded in Table 5 depict the pattern of solid and liquid fractions of cow milk fat present at various

TABLE 4

Solid/liquid fractions of Milkfat (Buffalo) at various temperatures

Sr. No.	Temperature (°C)	Solid (%)	Liquid (%)	Temperature (°C)	Solid (%)	Liquid (%)
1	22.5	100	0	36	35.3	64.7
2	24	99.0	1.0	37	31.7	68.3
3	25	88.5	11.5	38	25.7	74.3
4	26	87.0	13.0	39	21.6	78.4
5	27	85.7	14.3	40	18.3	81.7
6	28	84.4	15.6	41	15.2	84.8
7	29	82.1	17.9	42	13.1	86.9
8	30	70.6	29.4	43	10.7	89.3
9	31	64.2	35.8	44	9.0	91.0
10	32	57.1	42.9	45	5.4	94.6
11	33	50.3	49.7	46	4.5	95.5
12	34	45.3	54.7	47	2.7	97.3
13	35	41.2	58.8	50.8	0	100

Results pertain to the data plotted in Fig. II and recorded in Table 1.

TABLE 5

Solid/liquid fractions of Milkfat (cow) at various temperatures

Sr. No.	Temp. (°C)	Solid (%)	Liquid (%)	Temp. (°C)	Solid (%)	Liquid (%)
1	21	100	0	33	30.1	69.9
2	24	91.8	8.2	34	26.7	73.3
3	25	88.9	11.1	35	22.7	77.3
4	26	82.0	18.0	36	18.8	81.2
5	27	74.8	25.2	37	14.5	85.5
6	28	66.4	33.6	38	11.4	88.6
7	29	57.2	42.8	39	9.0	91.0
8	30	49.8	50.2	40	6.6	93.4
9	31	42.9	57.1	41	3.9	96.1
10	32	35.3	64.7	47	0	100

Results pertain to the data plotted in Fig. III, and recorded in Table 1.

TABLE 6

Solid/liquid fractions of Milkfat (goat) at various temperatures

Sr. No.	Temp. (°C)	Solid (%)	Liquid (%)	Temp (°C)	Solid (%)	Liquid (%)
1	18.5	100	0	27	29.2	70.8
2	19	96.1	3.9	28	22.8	77.2
3	20	92.3	7.7	29	17.9	82.1
4	21	85.9	14.1	30	12.0	88.0
5	22	76.5	23.5	31	9.7	90.3
6	23	67.7	32.3	32	7.0	93.0
7	24	55.7	44.3	33	4.7	95.3
8	25	44.6	55.4	34	2.2	97.8
9	26	35.7	64.3	38.5	0	100

Results pertain to the data plotted in Fig. IV and recorded in Table 1.

temperatures. It was observed in case of cow milk fat that at 21°C the milkfat was almost in solid phase and no liquid fraction was present. As the temperature increased above 21°C the liquid fraction also increased in non-uniform fashion. At 47°C , which was the point of inflexion for the curve represented by Eqn. II and Fig. III, the whole fat was present in liquid phase. Similar to the case of buffalo milkfat a temperature of 30°C was noticed at which the milk fat existed in solid and liquid fractions approximately in equal ratio. The melting interval for such milkfat was recorded to be 26°C .

Goat milkfat

The experimental data obtained for solid and liquid fractions of goat milkfat at various temperatures were tabulated in Table 6. In this case the liquid fraction of fat was observed negligible at 16.5°C and the solid fraction ceases to exist at a temperature equal to 38.5°C , which is the point of inflexion of the curve in Fig. IV and expressed through Eqn. III. The melting interval for this milkfat was found 20°C . The solid and liquid fractions of this milkfat was almost present in equal ratio at about 24.5°C .

Comparison of solid/liquid fractions in three milkfats.

From the above results it was clear that all the three milkfats differed significantly in their melting behaviour. The buffalo milkfat contained almost 100% liquid fraction at 50.8°C which is the highest in all three milkfats. This was observed lowest in case of goat milkfat (38.5°C) with intermediate value of 47°C for cow milkfat. Such difference in melting behaviour of these three milkfats could be explained on the basis of the fact that the buffalo milkfat differ in its fatty acid composition from that of cow milk (Ramamurthy and Narayanan, 1971). The same may be true in case of the goat milkfat. However, Mulder (1955) also reported that butterfat in most cases melts completely at temperatures below 40°C although compounds are present in it which do not melt upto 50°C in the pure state.

The goat milkfat starts melting above 18.5°C , the lowest in all the three milkfats under investigation. The same is observed above 22.5°C and 21°C in case of buffalo and cow milkfat, respectively. Such differences can be attributed to the possible dominant role of low melting acids in goat milkfat and those of high melting acids in buffalo milkfat.

Such study of solid/liquid fractions in milkfats has

not been reported so far and the results by other workers are not available in the literature for comparison. However, a few workers have reported the melting point of milkfat ranging between 30- 41°C (Janness and Patton, 1959), which is most probably for cow milkfat, determined by capillary method. This method may not be as accurate as the one used in the present investigation.

Coefficient of thermal expansion of milkfat (ghee) from buffalo, cow and goat.

Coefficient of thermal expansion of milkfat from buffalo, cow and goat (8 samples in each category) was determined between 10 to 80°C with uniform interval of 5°C, using the data recorded in Table 1. The average coefficient of thermal expansion (hereafter to be called as thermal expansion) for buffalo milkfat at 10 - 15°C was 2.0 ($\times 10^{-5}/^{\circ}\text{C}$) which increased to 72.0 ($\times 10^{-5}/^{\circ}\text{C}$) at 10-80°C. The average values with ranges for thermal expansion at the intermediate temperature intervals between 10 and 80°C are recorded in Table 7. The respective values for cow and goat milkfats were 2.0 ($\times 10^{-5}/^{\circ}\text{C}$) and 3.0 ($\times 10^{-5}/^{\circ}\text{C}$) at 10 - 15°C, and 82.0 ($\times 10^{-5}/^{\circ}\text{C}$) and 89.0 ($\times 10^{-5}/^{\circ}\text{C}$) between 10 and 80°C. The values at the intermediate temperature intervals for these milkfats are also recorded in Table 7.

TABLE 7

Coefficient of thermal expansion of milkfat (ghee)
from different species at different temperatures

Sr. No.	Temp. (°C)	Thermal expansion coefficient $\times 10^{-5}/^{\circ}\text{C}$					
		Buffalo		Cow		Goat	
		Range	Average	Range	Average	Range	Average
1	10-15	1-5	2	1-4	2	1-5	3
2	10-20	1-6	3	1-7	3	2-5	4
3	10-25	2-4	3	4-36	10	49-72	62
4	10-30	7-32	14	30-65	48	81-97	89
5	10-35	37-56	41	59-86	69	86-100	93
6	10-40	48-61	51	67-87	75	85-101	92
7	10-45	53-65	57	69-88	77	84-101	91
8	10-50	58-69	61	71-88	78	85-101	91
9	10-55	61-72	64	73-91	81	85-99	91
10	10-60	64-72	66	73-90	81	85-98	91
11	10-65	66-76	68	74-88	81	84-99	90
12	10-70	67-78	69	75-95	82	84-97	89
13	10-75	68-79	70	75-94	82	85-96	89
14	10-80	69-80	72	76-93	82	85-95	89

Results pertain to the average for 8 sets of samples in each category of buffalo, cow and goat milkfat with respective moisture content of 0.379 ± 0.0354 , 0.318 ± 0.0233 and 0.270 ± 0.0950 respectively.

Effect of temperature

To study the effect of temperature on coefficient of thermal expansion of three milkfats the data presented in Table 7 were statistically analysed and results therefrom are described here and also through Table 8 and Figs. V, VI and VII.

The experimental results could be best fitted in third order regressions for all the three milkfats. The regression coefficients for such equations, their variations in regression (R^2) and correlation coefficients (r) are recorded in Table 8. The regressions for buffalo, cow and goat milkfat, respectively are given by the following equations:

$$10^4 \cdot Y = -1.5141 + 0.1863 (t) + 1.7703 \times 10^{-3} (t^2) - 3.0756 \times 10^{-5} (t^3) \quad \text{IV}$$

$$10^4 \cdot Y = 3.6470 + 0.5607 (t) - 8.2886 \times 10^{-3} (t^2) + 3.7803 \times 10^{-5} (t^3) \quad \text{V}$$

$$10^4 \cdot Y = 5.3605 + 1.0411 (t) - 2.3036 \times 10^{-2} (t^2) + 1.5919 \times 10^{-4} (t^3) \quad \text{VI}$$

The respective levels of variations in regression equations were 94.96, 93.30 and 91.60% indicating a close cubic

TABLE 8

Coefficients for polynomial regression of coefficient of thermal expansion of Milkfat on temperature¹

$$\text{(Regression equation } 10^4 \cdot Y = b_0 + b_1 t + b_2 t^2 + b_3 t^3 \text{)}$$

Sr. No.	Samples	Value of b's				Variation in regression (R^2)	Correlation coefficient (r)
		b_0	b_1	b_2	b_3		
1.	Buffalo milkfat	-1.5141	0.1863	1.7703×10^{-3}	-3.0756×10^{-5}	0.9496	0.9745**
2.	Cow milkfat	-3.6470	0.5607	-8.2886×10^{-3}	3.7803×10^{-5}	0.9330	0.9659**
3.	Goat milkfat	-5.3605	-1.0411	-2.3036×10^{-2}	1.5919×10^{-4}	0.9160	0.9571**

¹ Results pertain to the data recorded in Table 7

** Significant at $P = 0.01$

FIG. V VARIATION IN COEFFICIENT OF THERMAL EXPANSION OF MILK FAT (BUFFALO) DUE TO TEMPERATURE.

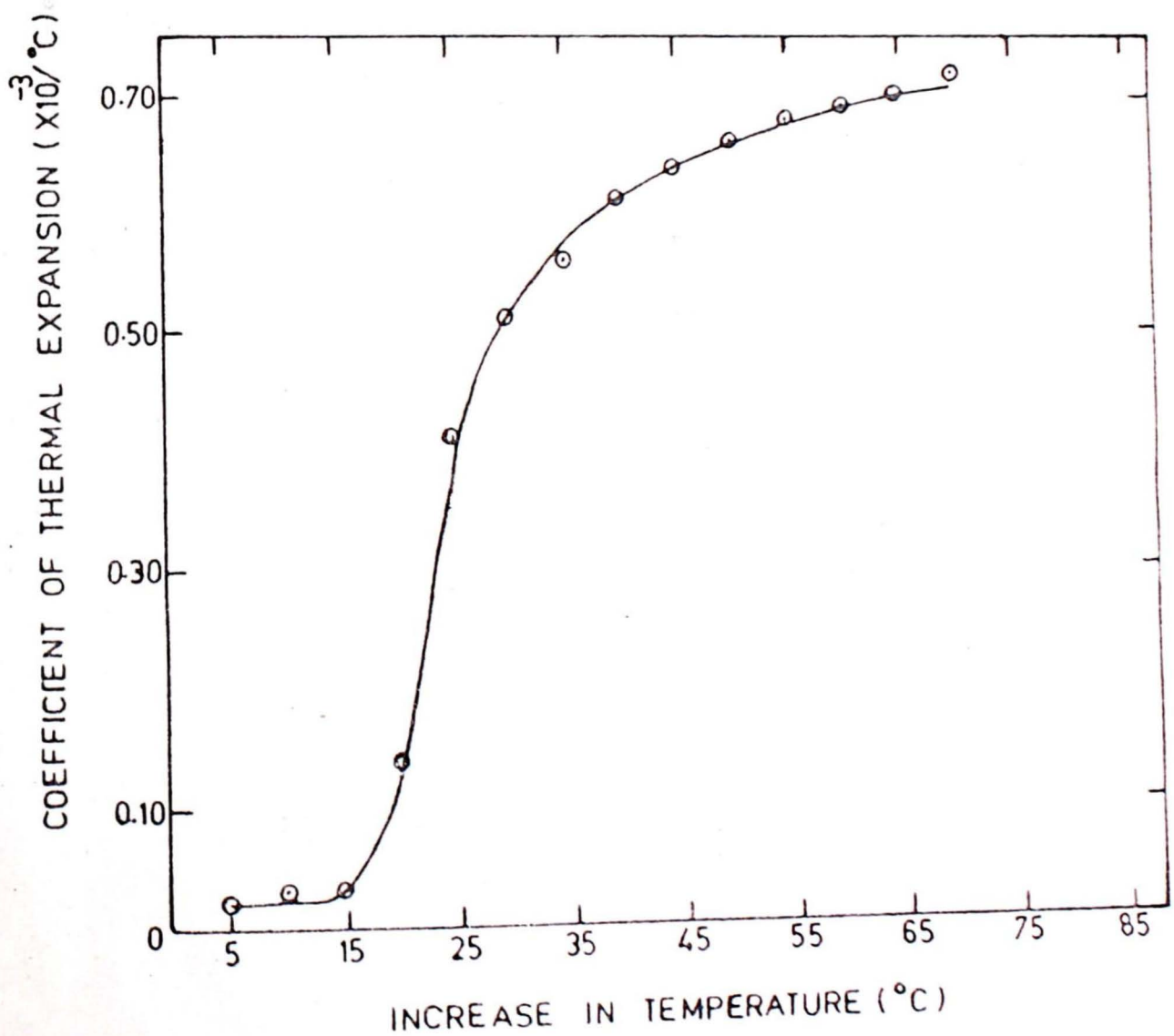


FIG. VI VARIATION IN COEFFICIENT OF THERMAL EXPANSION OF MILK FAT (COW) DUE TO TEMPERATURE.

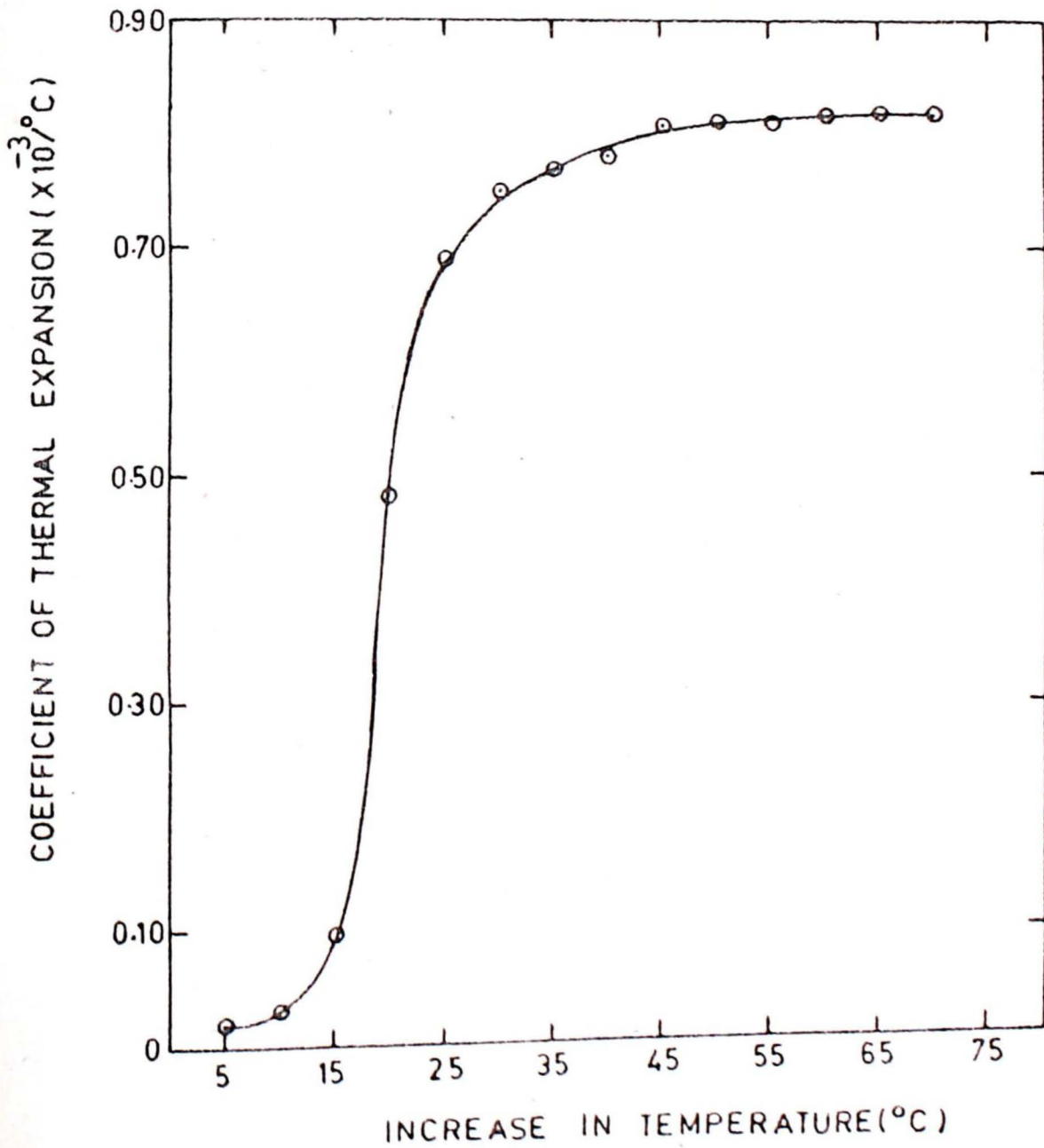
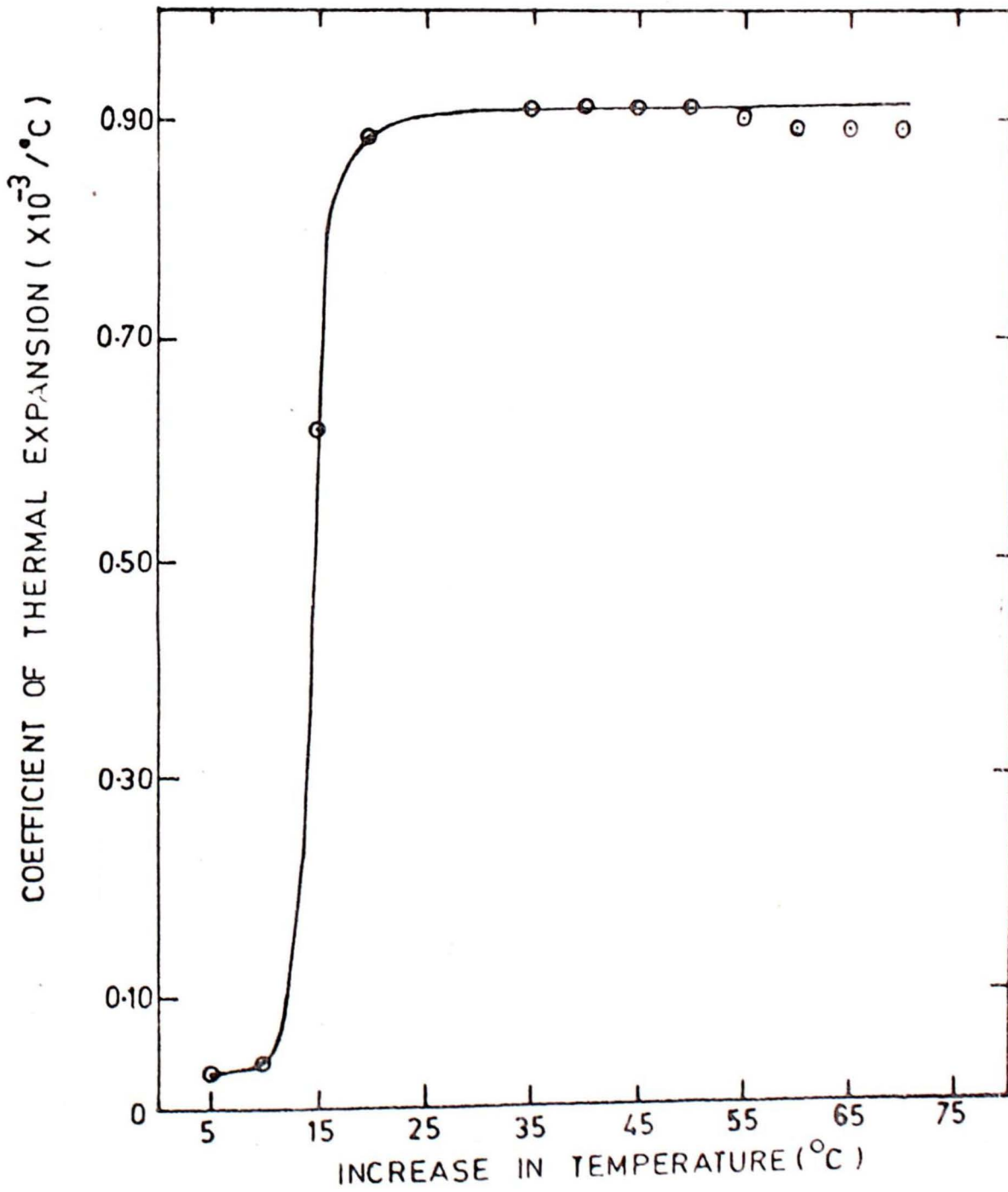


FIG. VII VARIATION IN COEFFICIENT OF THERMAL EXPANSION OF MILK FAT (GOAT) DUE TO TEMPERATURE.



relation between thermal expansion and temperature in all the three milk fats. The correlation coefficients (r), deduced from these variations in regression are 0.9745, 0.9659 and 0.9571 for buffalo, cow and goat milkfat, respectively. From statistical significance of these ' r ' values it could be concluded that the influence of temperature on coefficient of thermal expansion was significant at ($P = 0.01$) for all the three milkfats. Besides the gradual increase in thermal expansion of all the three milkfats with rise in temperature (Figs. V, VI and VII), it is seen from the plots of regression line and observed values that there is a tendency for steep rise in thermal expansion within a specific temperature range in case of all the three milkfats. This region in case of buffalo milkfat falls for a temperature increase between 15 to 30°C above 10°C which is the initial temperature of the samples. The same range of increase in temperature for cow and goat milk fats were 10-25°C and 10-20°C, respectively (Figs. VI and VII). From these figures (V, VI and VII), it is concluded that above these ranges of increase in temperature, the coefficient of thermal expansion in milkfats does not increase by significant amount and remains almost stable, excepting in buffalo milkfat wherein it keeps rising for some more rise in temperature. Such behaviour could be explained on the basis of the fact that these three milkfats differ significantly in the composition of triglycerides with different

melting point range. The goat milkfat seems to have more low melting fatty acids as compared to buffalo and cow milkfats. Buffalo milk fat must be having greater proportion of high melting acids giving rise to a high melting interval, with intermediate values in case of cow milk fat. Such study on coefficient of thermal expansion of milkfat from buffalo, cow and goat milk have not been reported so far and as such no comparison can be described with the present data. Also it was reported that different cooling treatments given to the milkfat gave rise to the different melting points as well as to the different solid fat content (De Man and Wood, 1959). However, some workers have reported the thermal dilation coefficient for liquid and solid butter fat without mentioning the temperature (Hannewijk and Haighton 1957; Gulyaev-Zaitsev 1967), excepting De Man and Wood (1959) who reported the thermal dilation over 40 - 60°C temperature. But these results can not be compared with those obtained from the present investigation.

Thermal expansion of cream

Coefficient of thermal expansion of cream from buffalo, cow and goat milk was determined over the range of 10 to 80°C. The average values (8 samples in each category) along with ranges of coefficient of thermal expansion are recorded in Table 9. The expansion in cream between 10 - 15°C was observed

TABLE 9

Coefficient of thermal expansion of cream from different species at different temperatures

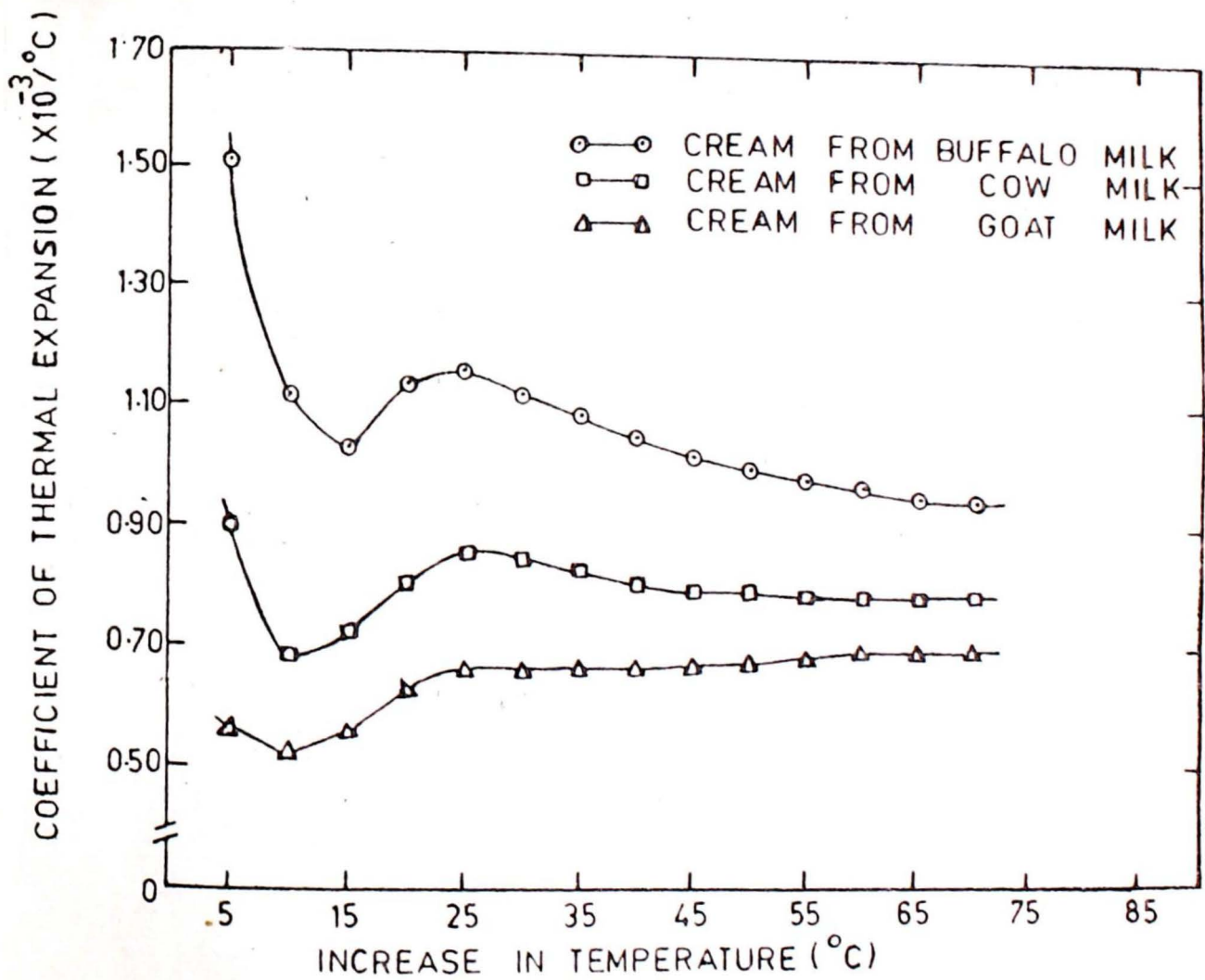
Sr. No.	Temp. ($^{\circ}\text{C}$)	Thermal expansion coefficient $\times 10^{-5}/^{\circ}\text{C}$					
		Buffalo		Cow		Goat	
		Range	Av.	Range	Av.	Range	Av.
1	10-15	115-165	151	66-126	91	54-61	57
2	10-20	90-126	112	56-88	69	51-57	53
3	10-25	90-118	103	62-87	73	56-58	57
4	10-30	96-125	114	72-95	81	62-66	64
5	10-35	100-124	116	77-96	86	64-69	67
6	10-40	99-121	112	77-93	85	65-70	67
7	10-45	93-116	109	76-91	83	65-70	67
8	10-50	91-111	105	75-90	81	66-68	67
9	10-55	90-108	102	75-88	80	66-69	68
10	10-60	88-105	100	74-86	80	66-70	68
11	10-65	87-104	98	74-85	79	68-71	69
12	10-70	87-102	97	75-84	79	68-71	70
13	10-75	86-100	95	75-84	79	68-71	70
14	10-80	85-99	95	76-84	79	69-72	71

Results are average of 8 samples in each category i.e. buffalo, cow and goat cream with average values of fat percent 65.69 ± 0.123 , 60.50 ± 0.142 and 54.41 ± 0.250 , respectively.

highest ($151 \times 10^{-5}/^{\circ}\text{C}$) in case of buffalo while the same in goat cream ($57 \times 10^{-5}/^{\circ}\text{C}$) were lowest with intermediate value of 91 ($\times 10^{-5}/^{\circ}\text{C}$) in case of cow cream. As the temperature further increased from $10 - 25^{\circ}\text{C}$ there was overall decrease in thermal expansion of buffalo and cow cream but an increase in case of goat cream. In the latter case, it kept on rising upto 35°C after which it became almost stable and what so ever the change in thermal expansion was there, it was negligible. However, in case of buffalo and cow cream, it increased from 25 to 35°C after which there was a fall in the coefficient of thermal expansion and than it became stable. Such non-uniform expansion of the cream is the resultant of the thermal expansion of the cream plasma, the fat and the expansion produced by the melting of fat. It could be concluded from the above results that the volume of the cream increased most between $10 - 15^{\circ}\text{C}$ in case of buffalo and cow which means that much fat probably changes from the stage of aggregation in this temperature range. Mulder (1953) reported such observation in cream between $10 - 20^{\circ}\text{C}$. While, De Man and Wood (1959) observed the same fact between $12 - 18^{\circ}\text{C}$. Such observation in the present study is shown closely in Fig. VIII where the average values for the coefficient of thermal expansion (Table 9) have been graphically plotted. However, in case of goat cream the same phenomenon was not observed between $10 - 15^{\circ}\text{C}$.



FIG. VIII VARIATION IN COEFFICIENT OF THERMAL EXPANSION OF CREAM DUE TO TEMPERATURE.



The data in table 9 were statistically analysed to elucidate further the effect of temperature and species on the thermal expansion of cream. The results from such analyses are presented in Table 10. It is seen that coefficient of thermal expansion of cream was significantly ($P = 0.01$) influenced by temperature and species. The interaction between the influence of temperature and species on thermal expansion was also significant ($P = 0.01$). However, the variation in thermal expansion due to individual samples was non-significant.

TABLE 10

Analysis of variance for the influence of temperature and species on the thermal expansion of cream¹

Sr. No.	Source of variation	df	SS	MSS	F
1.	Between samples	7	0.0016	2.2736×10^{-2}	1.6990 NS
2.	Between species	2	0.0076	3.7811×10^{-3}	28.2549**
3.	Between temperature	14	0.4238	3.0275×10^{-2}	226.2305**
4.	Interaction between temperature and species	28	0.0092	3.2982×10^{-4}	2.4646**
5.	Error	308	0.0412	1.3382×10^{-4}	

1. Results pertain to the data recorded in Table 9.

df Degrees of freedom
 SS Sum of squares
 MSS Mean sum of squares
 NS Not-significant
 ** Significant at P = 0.01
 F Variance ratio.

2. The solid fraction in buffalo, cow and goat milkfats was 38.5% at 22.5°C, 35.5% at 21.0°C and 32.5% at 18.5°C.

3. The solid fraction in buffalo, cow and goat milkfats was 38.5% at 22.5°C, 35.5% at 21.0°C and 32.5% at 18.5°C.

4. The solid fraction in buffalo, cow and goat milkfats was 38.5% at 22.5°C, 35.5% at 21.0°C and 32.5% at 18.5°C.

5. The solid fraction in buffalo, cow and goat milkfats was 38.5% at 22.5°C, 35.5% at 21.0°C and 32.5% at 18.5°C.

6. SUMMARY

7. The results obtained from the present investigation are summarised hereunder:

1. The liquid fraction in buffalo, cow and goat milkfats (ghee) was almost negligible at 22.5, 21.0 and 18.5°C, respectively.

2. The entire milkfat existed in liquid phase at 50.8, 47.0 and 38.5°C, respectively in buffalo, cow and goat milkfat.
3. The milkfat was present in almost same ratio in two fractions (solid and liquid) in buffalo, cow and goat milkfat at 33.0, 31.0 and 24.5°C, respectively.
4. The melting intervals for buffalo, cow and goat milkfat were 28.3, 26.0 and 20°C, respectively.
5. Thermal expansion of all the three milkfats was positively and significantly ($P = 0.01$) influenced by the temperature of measurements, following a cubical relation of the general form:

$$Y = b_0 + b_1 t + b_2 t^2 + b_3 t^3$$

6. The thermal expansion of milkfat was also significantly ($P = 0.01$) influenced by the species.
7. Thermal expansion of cream from buffalo, cow and goat milk containing fat 65.69 ± 0.123 (S.E), 60.50 ± 0.142 , $54.41 \pm 0.250\%$, respectively was significantly influenced by temperature and species.
8. In case of buffalo and cow cream, the maximum expansion was observed between 10 - 15°C, but similar phenomenon was

not observed in case of goat cream.

The above observations made during the present investigation have been discussed in Chapter III and also the explanation where ever could be possible, has been described on these observations.

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