DIELECTRIC PROPERTIES AND ELECTRICAL CONDUCTIVITY OF EDIBLE OILS

Thesis

Submitted to the Punjab Agricultural University
in partial fulfilment of the requirements
for the degree of
MASTER OF TECHNOLOGY
in
AGRICULTURAL ENGINEERING
(Minor : Computer Sc. & Electrical Engg.)

by

DILIP KUMAR
(L-94-AE-101-M)

Department of Processing & Agricultural Structures
College of Agricultural Enginneering
PUNJAB AGRICULTURAL UNIVERSITY
LUDHIANA-141004
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CERTIFICATE I

This is to certify that this thesis entitled, ***"Dielectric Properties and electrical conductivity of edible oils"***, submitted for the degree of Master of Technology, in the subject of Processing and Agricultural Structures (Minor: Computer Science and Electrical Engineering), of the Punjab Agricultural University, Ludhiana is a bonafied research work carried out by ***Dilip Kumar (L-94-AE-101-M)*** under my supervision and that no part of this thesis has been submitted for any other degree.

The assistance and help received during the course of investigations have been fully acknowledged.

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CERTIFICATE-II

This is to certify that the thesis entitled, ***"Dielectric Properties and electrical conductivity of edible oils"*** submitted by ***Dilip Kumar (L-94-AE-101-M)*** to the Punjab Agricultural University, in partial fulfilment of the requirements for the degree of Master of Technology, in the subject of Processing and Agricutlural Structures (Minor: Computer Science and Electrical Engineering), has been approved by the student's Advisory Committee after an oral examination on the same, in collaboration with an External Examiner.

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ABSTRACT

Electrical properties provide excellent opportunities for non-destructive sensing of quality characteristics of edible oils hence a study was conducted to determine the dielectric properties and electrical conductivity of four edible oils (sunflower, groundnut, mustard, cottonseed) and correlate them with viscosity and FFA content.

Data for dielectric constant, loss tangent and electrical conductivity as a function or temperature and FFA content were taken. Viscosity of oils at different temperatures (20-100°C) and dielectric constant at different frequencies (1-100 KHz) was also determined. Results showed that for all the four oils the increase in temperature led to a general increase in their dielectric properties and electrical conductivity.

Several correlations were fitted correlating viscosity and FFA content with loss tangent and electrical conductivity. Correlation constants for the best fit were presented. It was suggested that loss tangent and electrical conductivity could be used to determine viscosity and FFA content of oils with fair degree of accuracy within temperature range 20-100°C.

Amarjit Singh

Signature of Major Advisor

Kumar

Signature of student

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Kumar

Date: 11-6-1997

(Dilip Kumar)

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LIST OF SYMBOLS AND ABBREVIATIONS

ASTM	American Society for Testing Mechinery
OIP	Oxidation Induction Period
PAU	Punjab Agricultural University
ISI	Indian Standard Institution
FFA	Free Fatty Acids
°C	Degree Centigrade
N	Normality
ml	millilitre
rpm	revolution per minute
cp	centi poise
d	loss angle
Ic	Charging current
Khz	Kilo hertz
a.c	alternating current
D.C	Direct Current
K	Cell constant
pf	pico farad
exp	exponential

ln	logrithm
r^2	correlation coefficient
et al.	and others
w.r.t.	with respect to
g\l	gram\litre
τ	electrical conductivity
μ	Viscosity
$\in$	Dielectric constant
R_1, R_2, R_3	Replications

CHAPTER I

INTRODUCTION

The technology of oil processing has changed completely in the last twenty years in terms of scale and sophistication. Oil properties play an important role in oil processing. In our quest to gain a mechanistic understanding of the changes occurring during oil processing, the knowledge of oil properties is essential. Quantitative and qualitative information on the oil properties is necessary in the design and operation of oil processing equipment and the quality of the end product.

As the new uses of electric energy are developed and as new methods, processes and devices came into being which utilize or are influenced by the electrical nature of biological materials, the knowledge about the electrical properties of edible oils assume great significance. Electrical properties include conductivity, resistivity, dielectric and electrostatic properties.

Some of the factors which influence the electrical properties of agricultural materials are applied a.c. frequency temperature, moisture content, density, chemical composition and non-homogenity of materials.

Electrical properties are being used in the determination of water content, drying, heating, quality detection, electrical treatment, pest control and sorting and grading.

As manufacturing equipment becomes more sophisticated, raw materials and services more expensive and customer expectations more acute, then it is only by harnessing and developing the most modern monitoring and process control techniques that we can rise to these challenges and provide a consistent and high quality product at the lowest possible cost.

The quality standards of edible oils can be tested by various methods. Chemically, it can be tested by neutralization, saponification, iodine value, FFA content etc. Chemical methods have the disadvantage that they cannot be performed by untrained people. A well equipped chemical laboratory is required for the evaluation. The conventional techniques besides being time consuming and destructive in nature results in variation in the test data on the account of faulty handling of oil samples and use of different measuring equipments with different specifications. Thus, there is specific need to evolve accurate techniques to assess the condition and quality of oil and to find method/methods which are non-destructive, quick, reliable and can be used for on-line measurement in oil processing industry for effective process monitoring systems.

Viscosity is an important physical property of fatty oils and of products made from them. In the drying oil industries, the process of heat-bodying, blowing and other methods of modifying oils cause large changes in viscosity. Determination of viscosity in these cases provides an excellent test for process control. To an engineer viscosity of the oil to be handled is, of course, a fundamental factor in the design of pumps, piping, stills and other equipments used for oil processing and to the formulator of products containing fat, its viscosity may be a very important consideration (Eckey, 1954).

FFA content is an important chemical parameter for assessing the quality and condition of edible oils (Raghav, 1996).

Researches by Risman and Bengtsson (1971), Pace et al. (1968) and El-Shami et al. (1992) have indicated that electrical properties such as loss tangent, resistivity, conductivity may be used as true indicators of conditions and quality of edible oils.

Against this background, the determination of dielectric properties and electrical conductivity has assumed paramount importance because of their potential application in handling, processing and quality evaluation of edible oils.

It is, therefore, proposed to conduct investigation with following objectives:

1. Measuring of dielectric properties of cottonseed oil, sunflower oil, groundnut oil and mustard oil at different frequencies and different temperatures.

2. Measurement of electrical conductivity of above edible oils at different temperatures.

3. To establish correlation between FFA content and viscosity with dielectric property and electrical conductivity.

CHAPTER II

REVIEW OF LITERATURE

The information regarding the viscosity, dielectric properties and electrical conductivity and their utilization in quality evaluation of edible oils has been presented in this chapter. The review has been presented under following sections:

1. Viscosity
2. Dielectric properties
3. Electrical conductivity

2.1 Viscosity

Gouw et al.(1966) measured the viscosities of saturated fatty acids methyl esters at 20°C, 40°C and 70°C. The measurements were carried out with Ostwald viscometers. A new empirical equation relating viscosity to temperature was developed which accurately described the viscosity temperature relationships for a large variety of liquids over a wide temperature range.

Reid et al.(1977) described viscosity as a measure of internal fluid friction which tends to oppose any dynamic change in the fluid motion. Most of liquids obey Andrade correlation, hence an approximately linear relationship exists between the logrithm of viscosity and reciprocal of the absolute temperature.

Higher viscosity of oils as quoted by Formo et al. (1979) was due to the intermolecular attractions of the long chains of their glyceride molecules. Increase in the degree of unsaturation of oils was generally accompanied by decrease in viscosity. Thus viscosity of oil increased slightly with hydrogenation. Oil containing fatty acids of low molecular weight were slightly less viscous than oils of equivalent degree of unsaturation containing only high molecular weight acids.

Kubota et al.(1982) measured the viscosity and density of eight vegetable oils (soybean, rapeseed, corn, groundnut, sesame, coconut, cottonseed and olive) and salad and frying oils at 10-60°C. The dependence of viscosity and density on temperature was expressed by the empirical equations. The equations had high correlation coefficients.

Noureddini et al.(1992) measured the viscosity of a number of vegetable oils (crambe, rapeseed, corn, soybean, milkweed, coconut, tesquerella) and eight fatty acids as a function of temperature from 84°C to 110°C. Viscosity measurements were performed according to American Society for Testing Materials (ASTM) test methods D_{445} and D_{446}. Several correlations were fitted to experimental data and correlation constants for best fit were presented which are valid for temperature range 24°C to 110°C. Correlation constants are valuable for designing or evaluating chemical processing equipments such as heat exchangers, reactors, distillation columns, mixing vessels and process piping.

Toro-Vazquez and Infante-Guemero (1993) obtained equations and correlation constants which described the temperature-dependent variation of the absolute viscosity (μ) of 21 oils (sunflower, corn, rapeseed, sesame etc.) and oil liquid fat (melted butter) mixtures. Regressional analysis were based on two approaches fitting the oil and oil fat mixture viscosity profiles to a quadratic extension of the Andrade equation; a multiple variable regressional modelling. The former provided the best predictive models but the coefficient associated with temperature effect had no physical-chemical meaning. The multiple regressional approach fitted in a equation included parameters such as saponification value and iodine value of fatty acids of acylglycerols. Regressional models described effects of *cis* double bonds and fatty acid chain length on interactions of acylglycerols which determine μ of the system. It was suggested that multiple variable regressional analysis may be an excellent tool for better understanding the quantitative structure functional property relationships in liquid systems.

2.2 Dielectric Properties

Pace et al. (1968) measured the dielectric constant and loss tangent of 11 commercial fats and oils at three different temperatures and at three different frequencies in the microwave region. Measurements were made using the precision slotted line technique. The difference in dielectric properties among fats and oils appeared to be dependent on the phase (solid vs liquid) of the material and generally correspond to degree of unsaturation as evidenced by iodine values. The differences in loss factors among fats and oils at any given temperature and frequency were too small to be of any practical importance.

Risman and Bengtsson (1971) reported the dielectric properties of fats and oils obtained by the cavity perturbation method. Solid fats showed lower loss factor as compared to liquid fats which may be due to restricted movement of molecules in the solid state.

Belyaev et al. (1977) studied the dielectric properties of two types of deep frying fat and sunflower oil in view of the increased use of fat in combination with high frequency heat for heating foods. The permitivity, energy absorption coefficient and loss tangent of the products were measured at 22°C and 45°C. Results showed that for all the three fats the increase in temperature led to a general increase in their dielectrical properties.

Dielectric constant of most oils was in the range of about 3.0-3.2 as quoted by Formo et al.(1979). Oxidation increases the dielectric constant by the introduction of polar groups. Dielectric constants of free fatty acids were not greatly different from those of the parent glycerides. The dielectric constant increased with the number of double bonds present in fatty acids.

Tareev (1979) described oils as non-polar or very weak polar substances. Dielectric properties of oils were affected by the extent of impurities, pores filled with air, hygroscopic nature etc. Dielectric constant initially showed an increase with increase in temperature because orientation of dipoles are facilitated with increase in temperature but further increase in temperature led to intensification of chaotic thermal oscillations of molecules which resulted in decrease in degree of orderliness of their orientation hence drop in dielectric constant.

Fritsch (1981) studied the possibility of rate of deterioration of oils used for deep frying by measuring total polar materials, change in dielectric constant and iodine value. A soybean oil, an animal vegetable shortening and a hydrogenated vegetable shortening oil were tested. Measurements were taken after heating oil at 190°C for 8 hour each day. For monitoring a frying operation, in which there was no dilution of the frying fat by the fat in the food being fried. The change in the dielectric constant was the simplest of the methods judged reliable. Free fatty acid determination by titration and peroxide value were not recommended procedures for measuring frying fat deterioration.

Yoon et al.(1985) carried out investigations for the determination of lipid oxidation in rice bran and palmolein oil using electrical, optical, spectroscopic and extraction methods. Dielectric constant of oils were measured by using food oil sensor N1-22. The dielectric constant of both oils were about 2.1 which increased to about 9.0 after 50 hour of heating. In case of rice bran oil polar components, dielectric constant, refractive index and polymer content were shown to have high correlation.

Wu and Nawar (1986) carried out investigation to develop a practical method for monitoring the quality of corn oil during frying process. A special effort was made to find a technique that would not be affected by dilution, since replenishment with fresh oils to varying degree is a frequent necessity. Nine analytical methods i.e. measurement of viscosity, polymers, change in dielectric constant, polar compounds, free fatty acids, smoke point etc. were used. Since each single method was influenced by the replenishment with fresh oil combinations of two methods were studied in an attempt to produce a single value unaffected by dilution. The ratio of polymer to dielectric constant proved not only to be adequate for monitoring the quality of used oils but also was affected minimally by replenishment.

Wilhelm et al.(1988) examined butter oil, sunflower oil,olive oil and a blend containing equal proportions of the three oils used for potato frying for oxidative stability by measuring changes in dielectric constant and volatile compounds. The blend exhibited greater stability than expected.

Nelson (1991) in his review paper on dielectric properties of agricultural products, measurements and applications had discussed the nature of dielectric properties variation with frequency, temperature, and product density, techniques for measurement of dielectric properties, models for estimation of dielectric properties of grain and soybeans as a function of moisture content, frequency temperature and bulk density, applications of dielectric properties in drying, seed treatment, moisture determination, quality measurement, pest control etc.

El-Shami et al.(1992) compared the dielectric properties to conventional methods of analysis (viscosity, refractive index, iodine value, peroxide value and free fatty acids) for evaluating the frying quality of a blend of cottonseed and sunflower oils. The apparent relaxation time, the activation energy and the entropy changes for dielectric relaxation of heated oil samples were calculated. Results indicated that dielectric constant and dielectric loss are useful tools for predicting deterioration occurring during heating of the oil.

2.3 Electrical Conductivity

Effendiev et al.(1977) studied the effect of substances accompanying vegetable oil on the electrical conductivity of miscelles. It was established that electrical conductivity was dependent above all on phosphatides. When the concentration limits approached the content in the industrial miscelles the electrical conductivity was approximately two orders higher than that of miscelles without phosphatides. The electrical conductivity of phosphatides allowed the assumption that chemical changes that occur during electrochemical reactions by the action of electrostatic field, waxy substances, primary and secondary oxidation products, saponifiable materials, free fatty acids and mechanical impurities did not have significant effect on the electrical conductivity of the miscelles.

Shopov et al.(1978) measured electrical conductivity of 90 samples of alkaline soapstocks during neutralization of sunflower seed oil. The concentration of free NaOH in the samples ranged from 0 to 10 g/l and the concentration of soap was 4.5-12.6%. The investigations were carried out at 65°C, 70°C and 75°C using conductometry. Mathematical model of electrical conductivity was established as linear dependence of the concentration of NaOH and the concentration of soap.

Martin and Sanz (1985) measured electrical conductivity of milk concentrate at 1 KHz in temperature range 5-75°C. Viscosity was the major cause of conductivity.

Variation and a relationship between both physical properties was established. This investigation was used to correlate viscosity with conductivity in our study.

Fardiaz (1994) used electrical conductivity meter to determine the oxidation induction period (OIP; a measure of shelf life) of soybean, palm and peanut oil. Oil was heated to 98°C and a stream of air was blown through the oil. Volatile oxidation products were collected in vials containing water and the conductivity of water was measured and plotted against time. The OIP for soybean, peanut and palm oils were 4, 7 and 35hr respectively. Oil containing more polyunsaturated fatty acids had a shorter OIP than oil containing less polyunsaturated fatty acids.

CHAPTER III

MATERIALS AND METHODS

This chapter deals with the methodology and procedures followed and the equipment and materials used during the course of this study. Viscosity of oils were determined in the Dairy Technology Laboratory using viscometer available in the Department of Food Science and Technology, PAU, Ludhiana. Chemical analysis for the determination of FFA content of oils was done in the Chemical Analysis Laboratory of the Department of Processing and Agricultural Structures, PAU, Ludhiana. Dielectric properties and electrical conductivity of oils was determined in the Material Science Laboratory of the Department of Mechanical Engineering, Thapar Institute of Engineering and Technology, Patiala. The information has been presented under the following sections:

1. Selection of edible oils
2. Preparation of oil samples with different FFA content
3. Measurement of FFA content of oil samples
4. Measurement of viscosity of oils
5. Measurement of dielectric properties and electrical conductivity with temperature

6. Measurement of dielectric constant with frequency
7. Measurement of loss tangent and electrical conductivity of oil samples with different FFA content
8. Data Analysis

3.1 Selection of Edible Oils

Four popular edible oils which are commonly consumed by the people of India were selected for this study. These were cottonseed, groundnut, mustard and sunflower oil. It was found by the study of Singh and Mulukutla (1996) that groundnut oil was most preferred oil for everyday in most of the Western, Southern and Central Indian States. In eastern India expeller mustard oil was relatively more favoured by the consumers. In urban India in view of the medically established close relationship between the incidence of cardiovascular diseases and the intake of saturated fats, poly-unsaturated oils, like those of sunflower, cottonseed, sesame, soybean etc. were preferred.

Freshly extracted mustard oil (gobhi sarson) was procured from a local oil expeller. Sunflower (PSFH-67) oil was extracted with German made mini screw oil expeller (CA59) available in the Department of Processing and Agricultural Structures. As no particular variety of cottonseed and groundnut was available, commercially available refined cottonseed oil and groundnut oil was used in this study which were obtained from a local oil dealer. Details are given in Table 3.1.

Table 3.1. Description of oils used

S.No.	Oil	Variety	Descritpion	FFA content %,oleic acid
1.	Cottonseed	Commercial	Refined, bleached and deodorized	-
2.	Groundnut	Commercial	Refined, bleached and deodorized	-
3.	Mustard	*Gobhi sarson*	Freshly extracted	0.35
4.	Sunflower	PSFH-67	Freshly extracted	0.56

3.2 Preparation of Oil Samples with Different FFA Content

Freshly extracted mustard and sunflower oils were used for the preparation of samples of different FFA content. 500 ml oil was heated at constant temperature between 100-110°C in a constant temperature oil bath for different durations to alter the rate of hydrolysis and enzymatic activity. Details of sample, temperature, duration etc. are given in Table 3.2. Chemical analysis was done after four weeks to ascertain the FFA content Of the samples.

Table 3.2. Treatments of different oil samples of mustard and sunflower

Sample NO.	Mustard and sunflower treatments		
	Temperature	Duration	Others, if any
I	110°C	60 min	
II	110°C	30 min	
III	100°C	50 min	
IV	100°C	20 min	
V	-	-	Filtered sample kept at room temperature 15-20°C
VI	-	-	Unfiltered samples kept at room temperature(15-20°C) with varying solid particles
VII	-	-	

3.3 Measurement of FFA Content

A known amount (1.0 g) of oil sample was taken in 250 ml flask. 20 ml of absolute alcohol and 20 ml of benzene was added to it and was titrated with aqueous solution of N/40 NaOH after adding few drops of phenolphthalein as an indicator to the first pink colour which persists for 30 seconds. Free fatty acids of the sample was computed using the following expression:

$$\text{FFA (oleic acid)}\% = \frac{V\ N\ M}{W} \qquad ..(3.1)$$

$$= \frac{V \times 1 \times 282}{40 \times 10 \times 1} = .705\ V \qquad \ldots(3.2)$$

where

V = Volume of NaOH used, ml
N = Normality of NaOH used (.025N)
W = Weight of oil sample (1.0 g)
M = Molecular weight of oil, oleic acid predominating (282)

3.4 Measurement of Viscosity

Viscosity is a measure of internal friction in molecules. The coefficient of viscosity is defined as force per unit area required to maintain a unit difference of velocity between two parallel layers that are unit distance apart.

Brookfield Dial Reading Viscometer was used to measure viscosity at different temperatures. The principle used in Brookfield viscometer is to measure the torque necessary to overcome the viscous resistance of the fluid by rotating a sensing element called spindle through a beryllium copper spring in a fluid. The degree to which the spring is wound is indicated by the red pointer is proportional to the viscosity of the fluid. For a given spring deflection the actual viscosity depends on spindle speed and spindle size and shape.

The spindle used in this study was of largest size and the speed of rotation selected after measuring viscosity at different speeds was 60 rpm. 250 ml oil was heated in a beaker 2-4°C above the temperature at which readings are to be taken. The beaker was mounted such that the spindle was immersed into the oil at the indicated level. The dial readings at 60 rpm was taken which was equal to the viscosity of oil at that temperature in centi poise (cp). The temperature range used was 20°-100°C. The limitations of this instrument were; it is generally used for routine tests, dial readings below 10 units are not accurate and there was no inbuilt provision for heating the sample within the instrument.

3.5 Measurement of Dielectric Properties and Electrical Conductivity of Oils at Different Temperature

An Oil Test System developed by Sivananda Electronics, Devlali was used for measuring dielectric parameters such as dielectric constant and loss tangent and electrical conductivity of edible oils used for this study. Various components and their working is presented under this sub-section.

3.5.1 Three terminal oil test cell

Three terminal oil cell (Fig.3.1) was used for measuring the dielectric properties and electrical conductivity of oils. The oil cell used was in accordance with the drawing given in IS: 6103 and 6262 - 1971 recommended by ISI for research tests. The fabrication of the cell was such that it can be assembled and dismantled with ease to facilitate easy cleaning of its component part (Appendix A). Electrodes were of stainless steel. The terminals were separated by teflon rings. Guard terminal was used to shield the measuring electrode to

Fig.3.1.Three terminal oil test cell

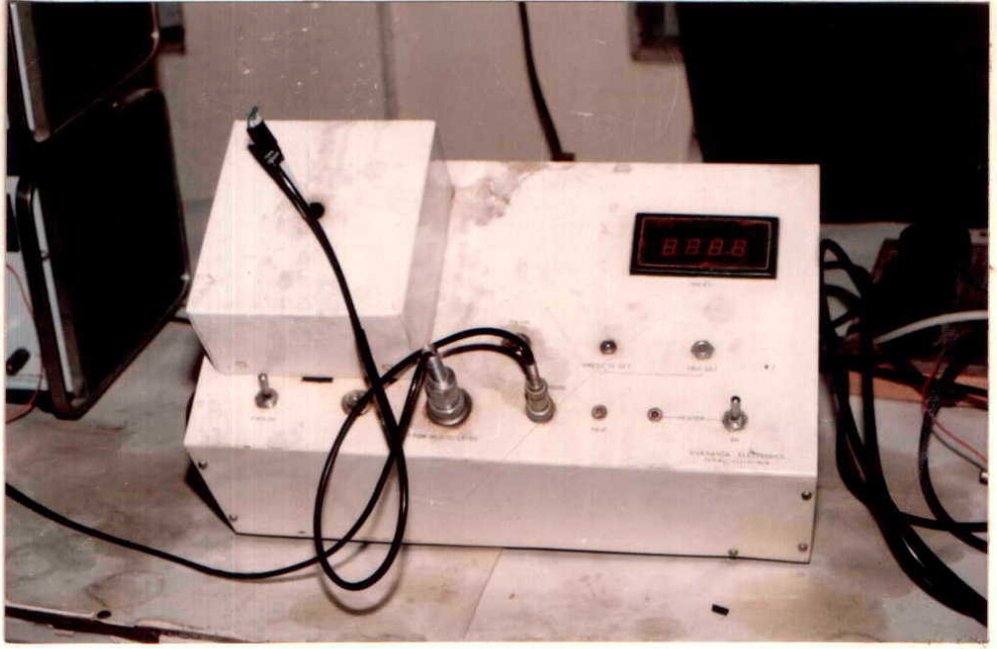

Fig.3.2.Heating chamber

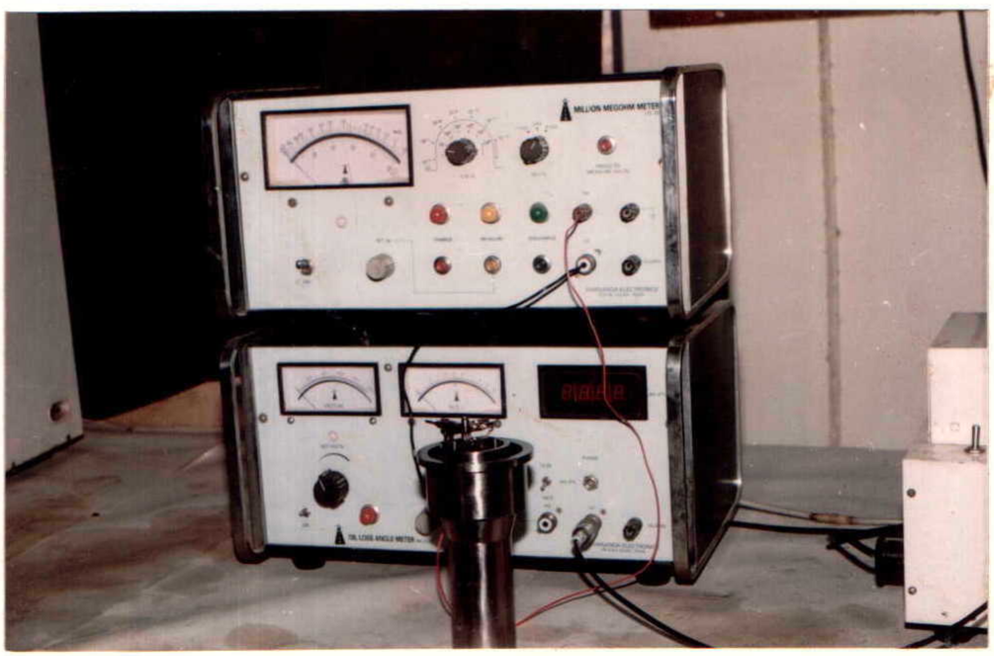

Fig.3.4.Set up for measuring dielectric properties and electrical conductivity

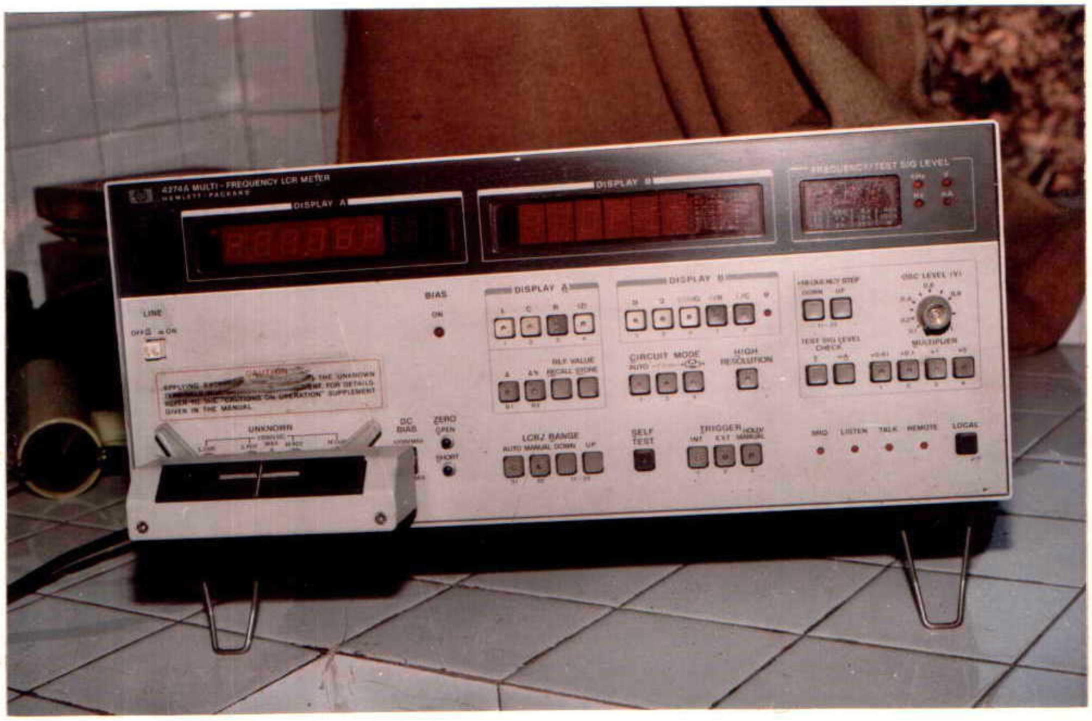

Fig.3.5.Multi Frequency LCR Meter

CHAPTER IV

RESULTS AND DISCUSSION

The results obtained from the investigation have been presented and discussed in this chapter under the following sub-sections:

1. Effect of temperature on viscosity, dielectric properties and electrical conductivity
2. Effect of frequency on dielectric constant
3. Effect of FFA content on tangent loss angle and electrical conductivity

Four edible oils namely sunflower oil, mustard oil, groundnut oil and cottonseed oil were selected for this study. The properties selected were viscosity, dielectric constant, tangent loss and electrical conductivity. The temperature range selected was 20°-100°C.

Results of variation in the selected properties with temperature, frequency and FFA content are discussed in this sub-section. The observed values of different parameters are given in Appendix E. Computed average values for all properties are summarized in Tables 4.1-4.6. Results obtained for each properties and their relationships has been discussed in the following sub-sections:

4.1 Effect of Temperature on Viscosity, Dielectric Properties and Electrical Conductivity

4.1.1 Viscosity

It was evident from the data presented in Table 4.1 and Fig.4.1 that viscosity of all the oils showed a non-linear decreasing trend with increase in temperature. This was due to the fact that viscosity of a liquid is determined by the force of cohesion between its molecule which decreases with increase in temperature because the kinetic energy of the molecule is increased by the rise in temperature. At 20°C the cottonseed oil exhibited higher viscosity while the viscosity of groundnut oil was least. The fall in the viscosity was rapid at lower temperature range. Also the difference in the viscosities among the oils was more at lower temperature than at higher temperature range. A regression equation relating viscosity to the temperature was developed of the form:

$$Y = a \exp(b/T^3) \qquad \ldots(4.1)$$

taking logrithm of both sides

$$\ln y = \ln a + \frac{b}{T^3} \qquad \ldots.(4.2)$$

where

Y = viscosity, cp

T = Absolute temperature

a, b = Constants

The above equation had good correlation coefficient for all the four oils (R^2>.99). The values of constants a,b and correlation

Table 4.1. Variation of viscosity of oils with temperatures

Temperature (°C)	Viscosity (cp)			
	Cotton seed	Groundnut	Mustard	Sunflower
20	77.0	48.0	71.0	64.0
40	36.5	26.5	37.5	31.5
60	19.5	15.0	25.0	17.5
80	14.5	10.5	16.0	11.5
100	10.0	8.0	13.0	9.0

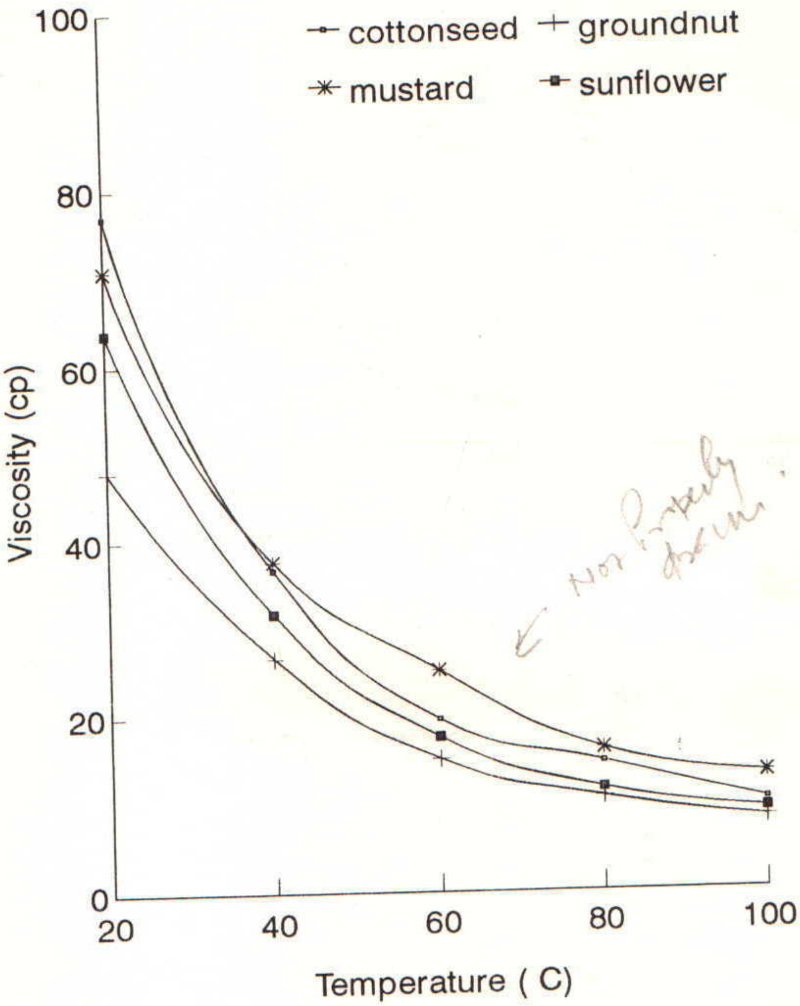

Fig.4.1.Variation of viscosity of oils with temperature

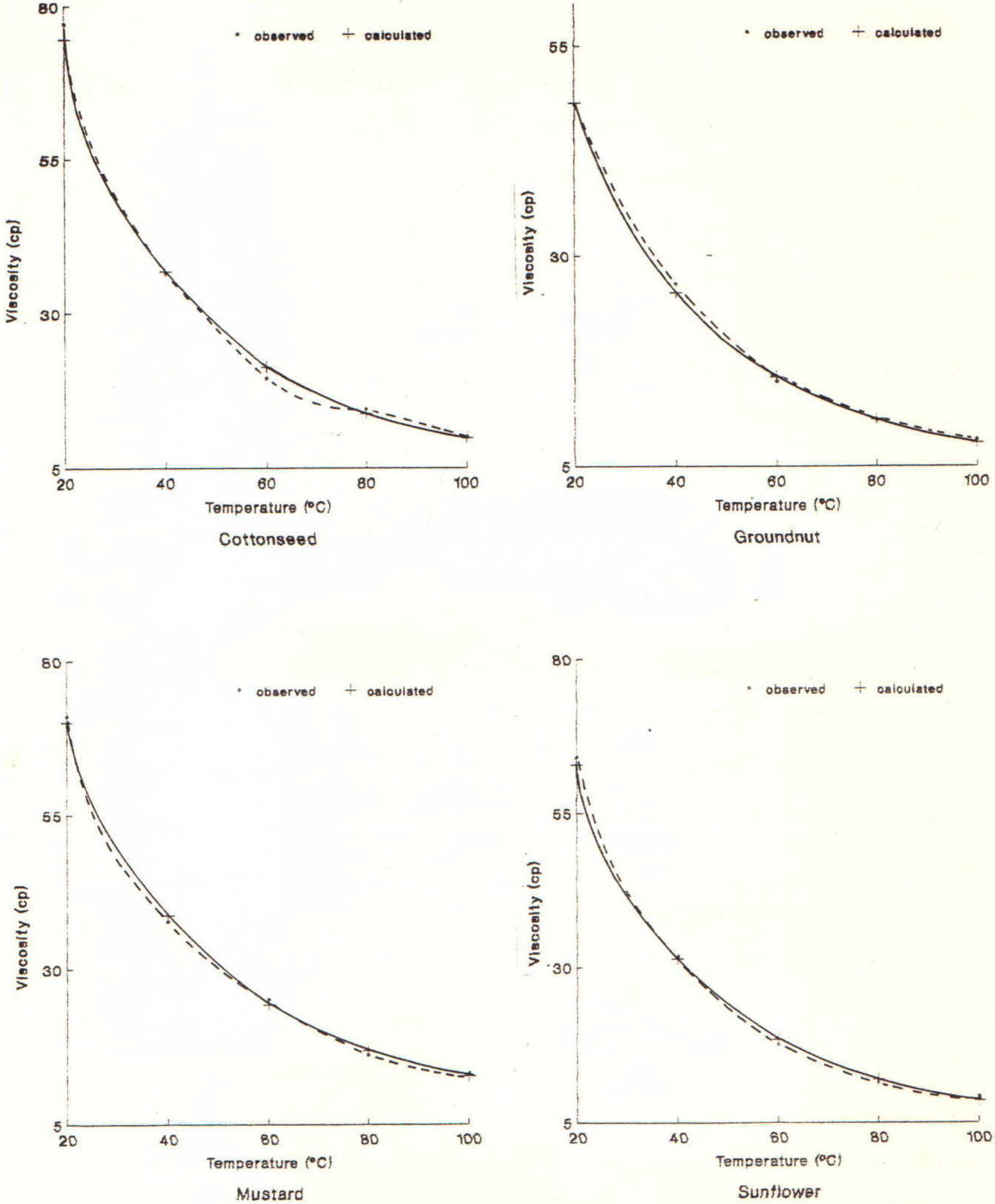

Fig 4.2. Variation of viscosity of oils with temperature. the dotted lines are experimental data and firm lines are correlation derivatives

coefficient are given in Table F.1.

4.1.2 Dielectric constant

Dielectric constant of the oils was in the range 2.7 to 2.9 at 20°C (Table 4.2). It was evident that dielectric constant of all the oils remained almost constant upto one derived placed but only increased a little in the temperature range 70-100°C (Fig.4.3).

The effect of temperature on dielectric constant was explained by Fuoss (1943) that with the increase In temperature the dielectric constant goes through a sigmoidal increase. At low temperature, high internal viscosity produces long relaxation times and hence little dipole orientation which resulted in low values of dielectric constant. As the temperature was increased lower viscosity resulted in decrease of relaxation time which permitted partial orientation of molecules hence dielectric constant increases and approaches maximum when polarization approaches synchrony with the field. Further increase In temperature witnessed decrease in dielectric constant due to unsynchronization of polarization with the field.

The increase In the dielectric constant over the temperature range 20-80°C was not distinctly visible as within this temperature range the change In dielectric constant was mostly confined to second or third decimal place (Pace, 1968) and the instrument used for this study had least count of 0.1. Initially the dielectric constant of refined oils was less than

Table 4.2. Variation of dielectric constant of oils with temperature at constant frequency (50 Hz)

Temperature (°C)	Dielectric constant			
	Cotton seed	Groundnut	Mustard	Sunflower
20	2.8	2.7	2.9	2.9
30	2.8	2.7	2.9	2.9
40	2.8	2.7	2.9	2.9
50	2.8	2.7	2.9	2.9
60	2.8	2.7	2.9	2.9
70	2.8	2.7	2.9	2.9
80	2.9	2.7	2.9	2.9
90	2.9	2.8	3.0	2.9
100	3.0	2.9	3.0	3.0

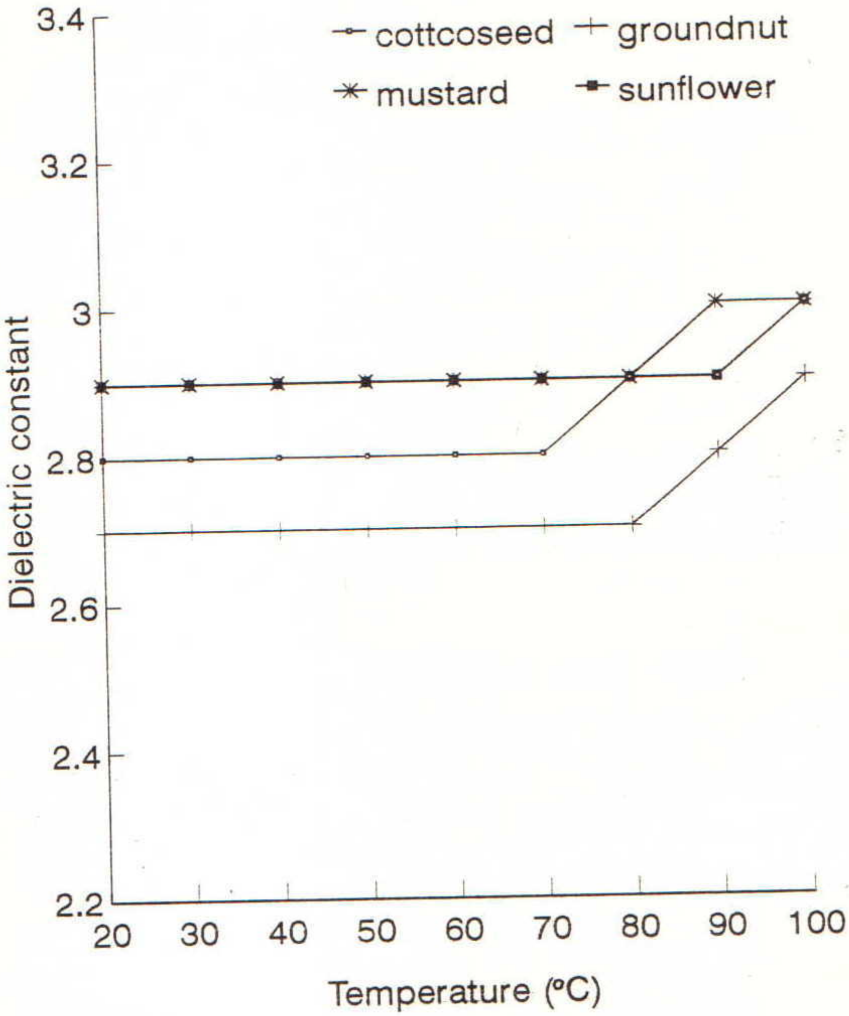

Fig.4.3. Variation of dielectric constant of oils with temperature at constant frequency(50Hz)

freshly extracted oils (Table 4.2) which suggests that electronic polarization was facilitated by the presence of extent of impurities (solid particles, pores filled with air etc.).

4.1.3 Loss tangent

The loss tangent increased appreciably with the increase in temperature (Table 4.3). The increase was non-linear (Fig.4.4). Loss tangent at 20°C of cottonseed, groundnut, mustard and sunflower oil were 0.5, 1.5, 18.8 and 14.2 (expressed in percentage) respectively which increased to 11.0, 15.2, 83.5 and 72.0 respectively at 100°C. Tareev (1979) explained the phenomenon of increase in loss tangent with temperature that when the temperature was increases viscosity decreases which results in diminishing the amount of losses due to friction of rotating dipoles hence loss tangent increased with temperature.

A regression equation relating viscosity with loss tangent was developed of the form:

$$y = ax + b \qquad \ldots(4.3)$$

where

$y = 1/\mu$

x = Loss tangent, %

μ = Viscosity, cp

a, b = constants

The values of constants a,b and correlation coefficient for all the four oils are tabulated in Table F.2. This equation has

Table 4.3. Variation of Loss Tangent with temperature at constant frequency (50 Hz)

Temperature (°C)	Loss Tangent			
	Cotton seed	Groundnut	Mustard	Sunflower
20	0.5	1.5	18.8	14.2
30	1.4	2.0	28.9	21.1
40	2.6	2.9	38.5	27.4
50	3.2	3.9	48.2	35.1
60	4.7	5.6	56.4	39.2
70	5.8	7.7	64.2	46.0
80	6.7	10.8	71.1	54.4
90	8.7	12.3	77.7	63.5
100	11.0	15.2	83.5	72.0

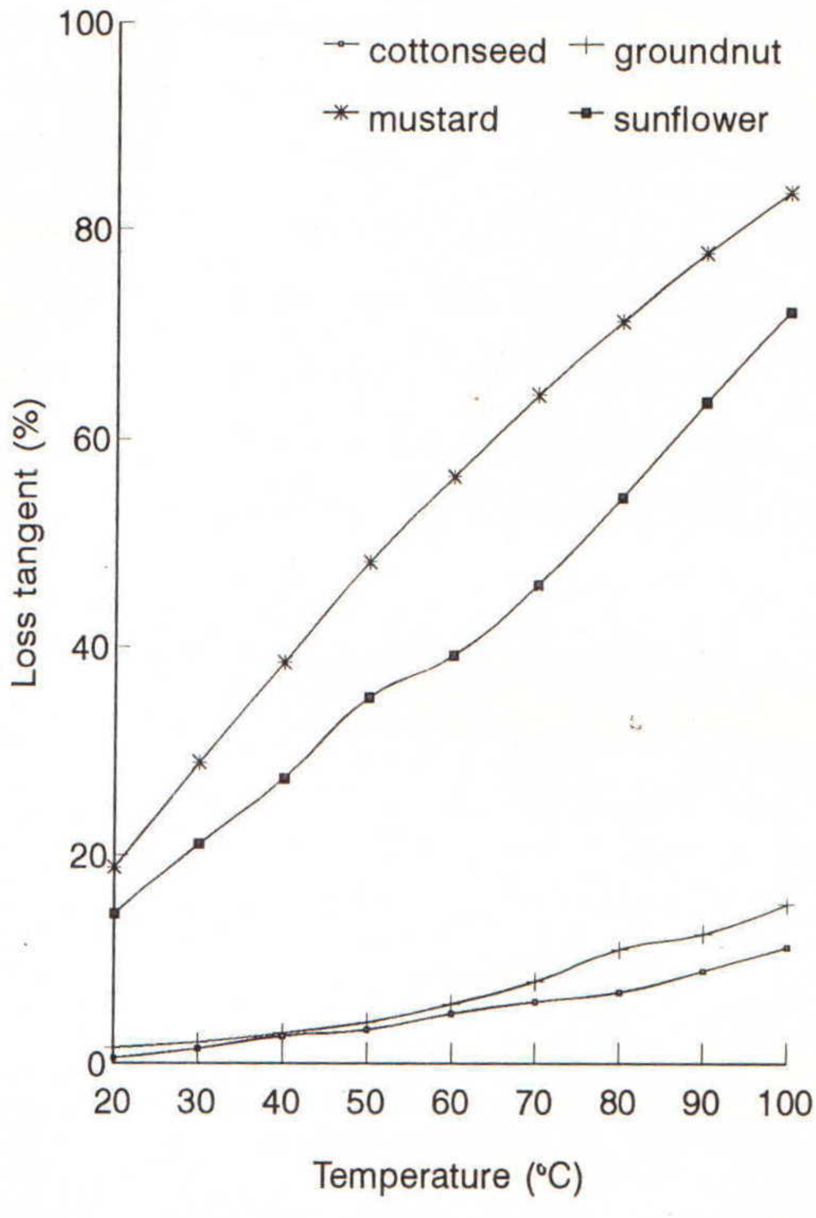

Fig.4.4.Variation of loss tangent of oils with temperature at constant frequency(50Hz)

good correlation coefficient (r^2 >.96) for all the four oils.

4.1.4 Electrical conductivity

The electrical conductivity of all the oils increased with the rise in temperature (Table 4.4). The increase was non-linear (Fig.4.5). The initial electrical conductivity of cottonseed was least ($4x10^{-13}$ mho cm^{-1}) while it was highest for sunflower oil ($91x10^{-13}$ mho cm^{-1}). The increase in the electrical conductivity was due to fact that with the increase in temperature the viscosity decreases which results in decrease in resistance due to decrease in intermolecular attraction and hence electrical conductivity increases (Tareev, 1979).

A regression equation relating viscosity with electrical conductivity was developed of the form:

$$y = ax + b \qquad \ldots(4.4)$$

where

$y = \ln(\mu)$

$x = \ln(\tau)$

μ = viscosity, cp

τ = conductivity, mho cm^{-1}

This equation had good correlation coefficient (r^2>.97). the values of constants a, b and correlation coefficients are tabulated in Table F.3.

4.2 Effect of Frequency on Dielectric Constant

At constant temperature of 15°C, the dielectric constants of all the oils were practically constant over the

Table 4.4. Variation of electrical conductivity of oils with temperature

Temperature (°C)	Conductivity (mho $cm^{-1}x10^{-13}$)			
	Cotton seed	Groundnut	Mustard	Sunflower
20	4	12	54	91
30	10	17	118	131
40	18	26	170	194
50	24	39	340	272
60	62	60	504	378
70	78	90	648	425
80	92	136	756	524
90	106	158	890	619
100	121	206	908	716

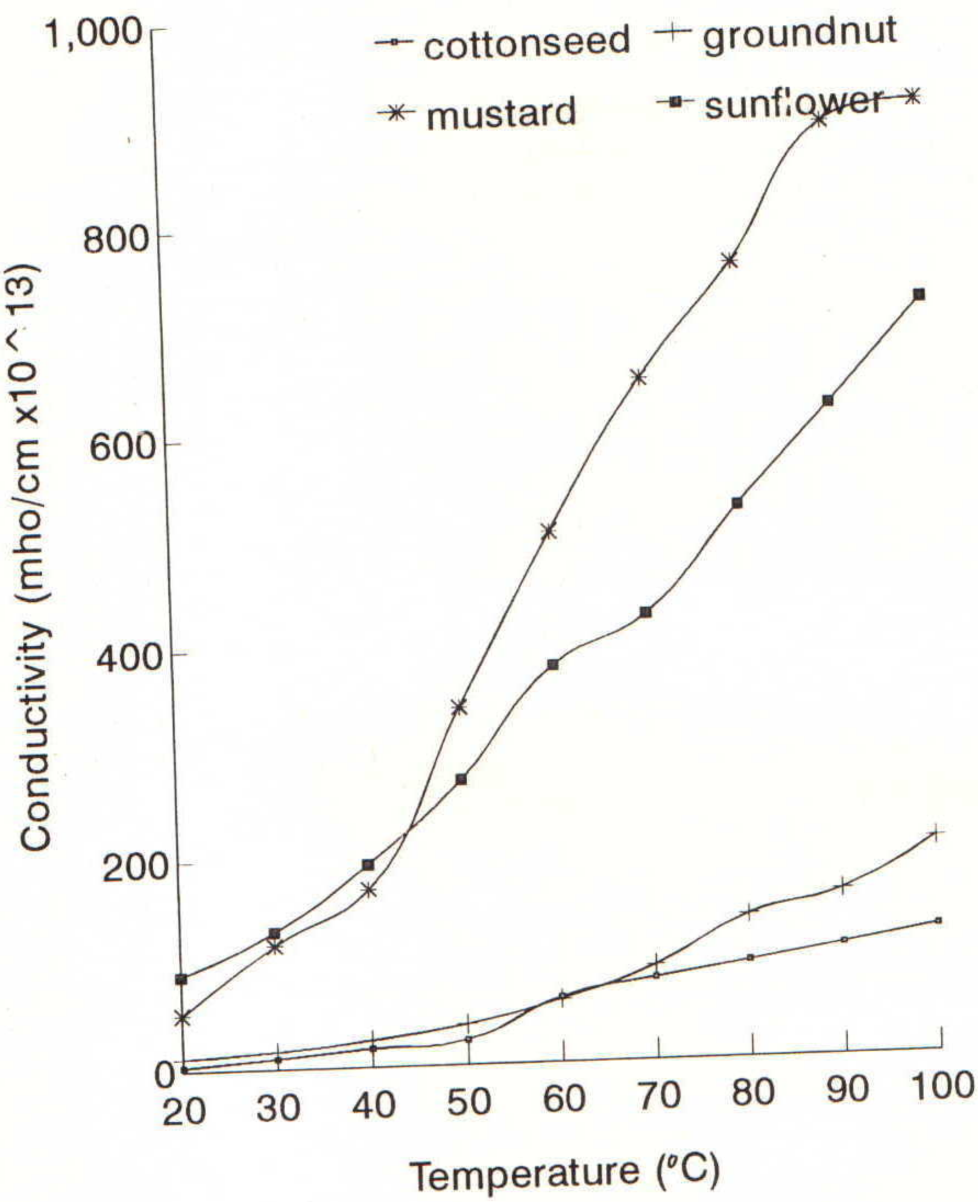

Fig.4.5.Variation of electrical conductivity of oils with temperature at constant frequency(50Hz)

Table 4.5. Variation of dielectric constant of oils with frequency at constant temperature (15°C)

Frequency (KHz)	Dielectric constant			
	Cotton seed	Mustard	Sunflower	Groundnut
1	2.91	2.88	2.92	2.98
2	2.91	2.88	2.92	2.98
4	2.91	2.88	2.92	2.98
10	2.90	2.88	2.90	2.98
20	2.90	2.88	2.90	2.98
40	2.89	2.88	2.90	2.97
100	2.89	2.88	2.90	2.97

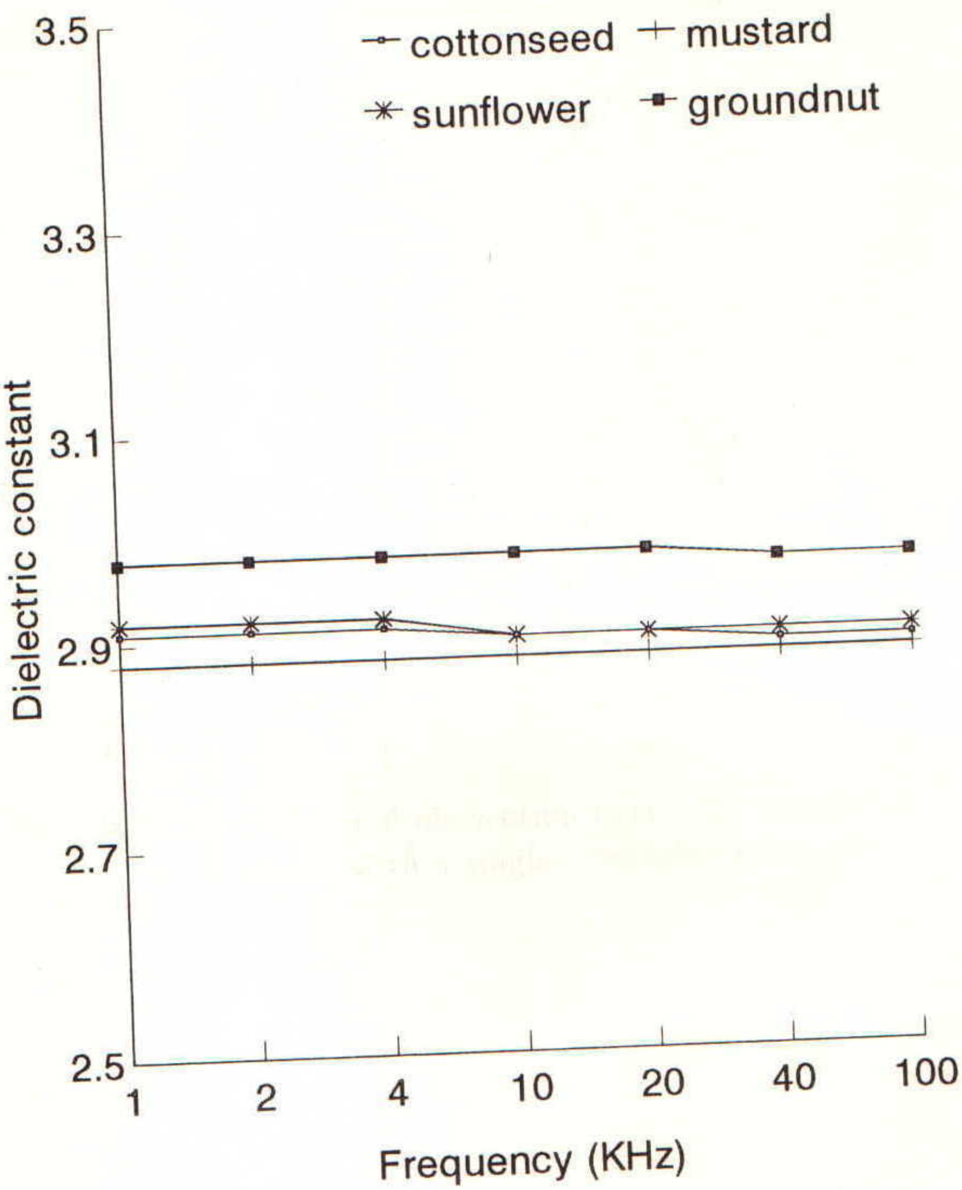

Fig.4.6.Variation of dielectric constant of oils with frequency at constant temperature(15°C)

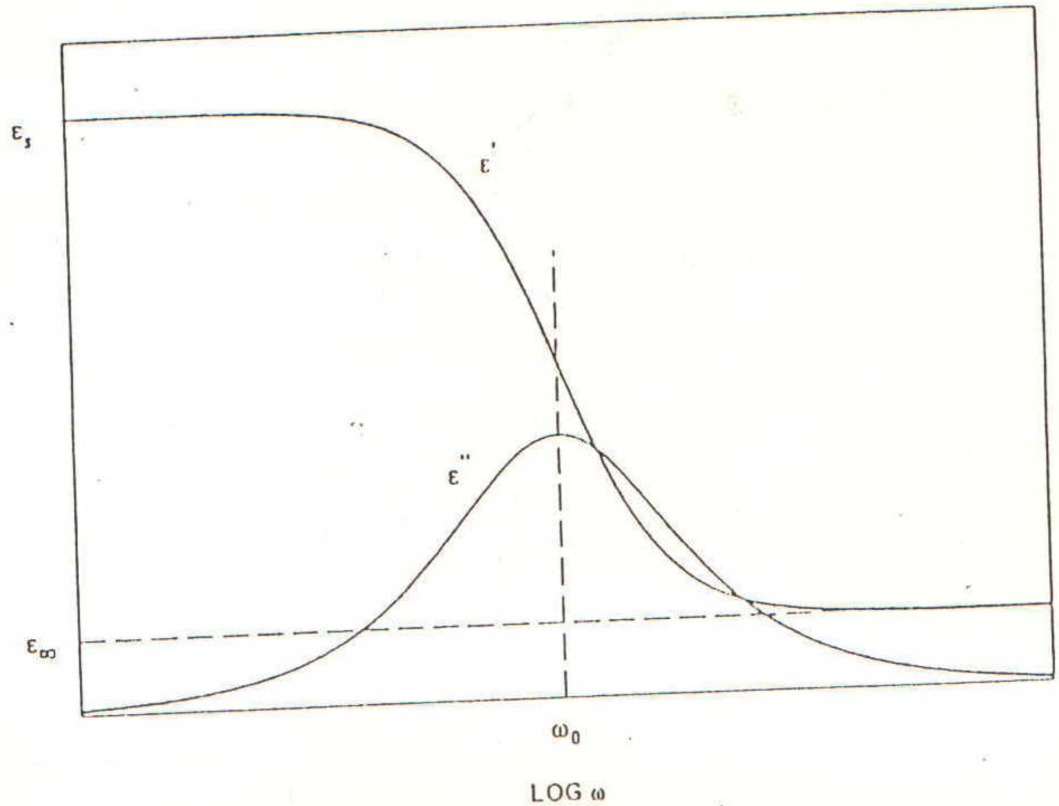

Fig. 4.7 Dispersion and absorption curves for the Debye model for a polar substance with a single relaxation time.

entire range of frequency used but showed a little decrease at 40 KHz and 100 KHz (Table 4.5 and Fig.4.6). The decrease was not much significant as the frequency range available was very less. The dependence of dielectric constant on frequency was explained by Debye (1929) that dielectric constant decreased from ϵ_s to ϵ_∞ as frequency is increased (Fig.4.7). ϵ_s is the maximum static dielectric constant and ϵ_∞ is the optical dielectric constant. As the frequency is increased it becomes more difficult for the molecules to reorient themselves against the viscosity of the medium hence dielectric constant decreased with frequency.

4.3 Effect of FFA Content on Loss Tangent and Electrical Conductivity

It was evident from the data presented in Table 4.6 and Fig.4.9-4.10 that loss tangent and electrical conductivity increased with increase in FFA content. This phenomenon of increase in loss tangent and electrical conductivity with FFA content may be postulated that in oil samples the hydroxyl ion (OH^-) increases with hydrolysis of the oil (enzymatic or non-enzymatic) in presence of water molecule both in glycerol as well as in free fatty acid molecule as per reaction given in Fig.4.8. This increase in hydroxyl ion increases the number of polar molecules as well as ionic conductivity as compared to the large fat molecule which is basically non-polar. Hence there was increase in loss tangent and electrical conductivity.

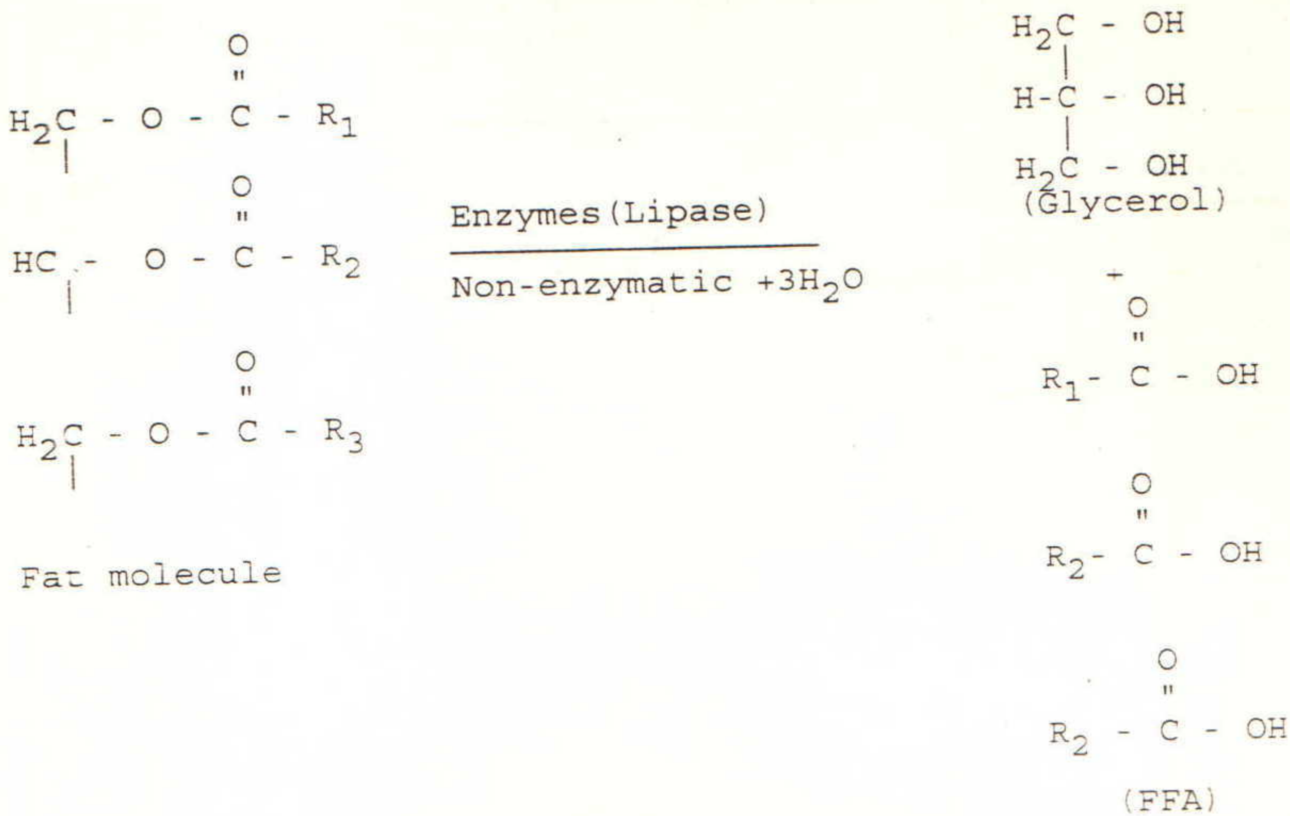

Fig.4.8. Hydrolysis of a triglyceride molecule

Table 4.6. Variation of electrical conductivity and loss tangent with FFA content

Sample No.	Mustard			Sunflower		
	FFA content, oleic acid(%)	Conductivity mhocm^{-1}x10^{-13}	Loss tangent (%)	FFA content, oleic acid(%)	Conductivity (mho cm^{-1}x10^{-13})	Loss tange: (%)
I	0.42	46.0	6.1	1.13	29.0	3.0
II	0.71	51.0	6.7	1.48	30.0	3.2
III	0.85	53.0	7.4	1.62	32.0	3.4
IV	0.92	57.0	7.8	1.83	34.0	3.7
V	1.06	67.0	8.3	2.26	40.0	4.1
VI	1.27	69.0	9.0	2.40	4.1	4.2
VII	1.56	89.0	10.5	2.61	50.0	4.7

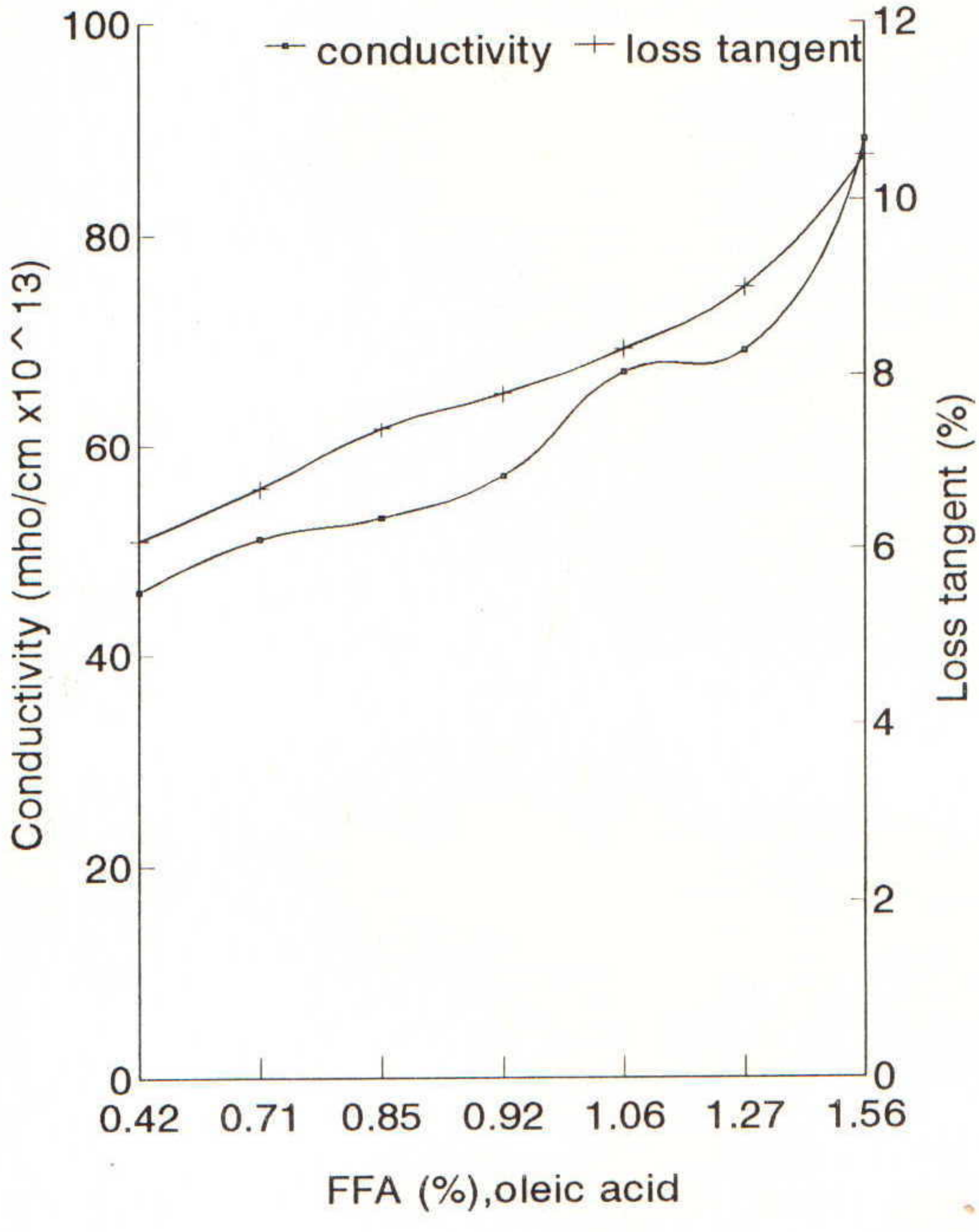

Fig.4.9.Variation of electrical conductivity and loss tangent of mustard oil with FFA content

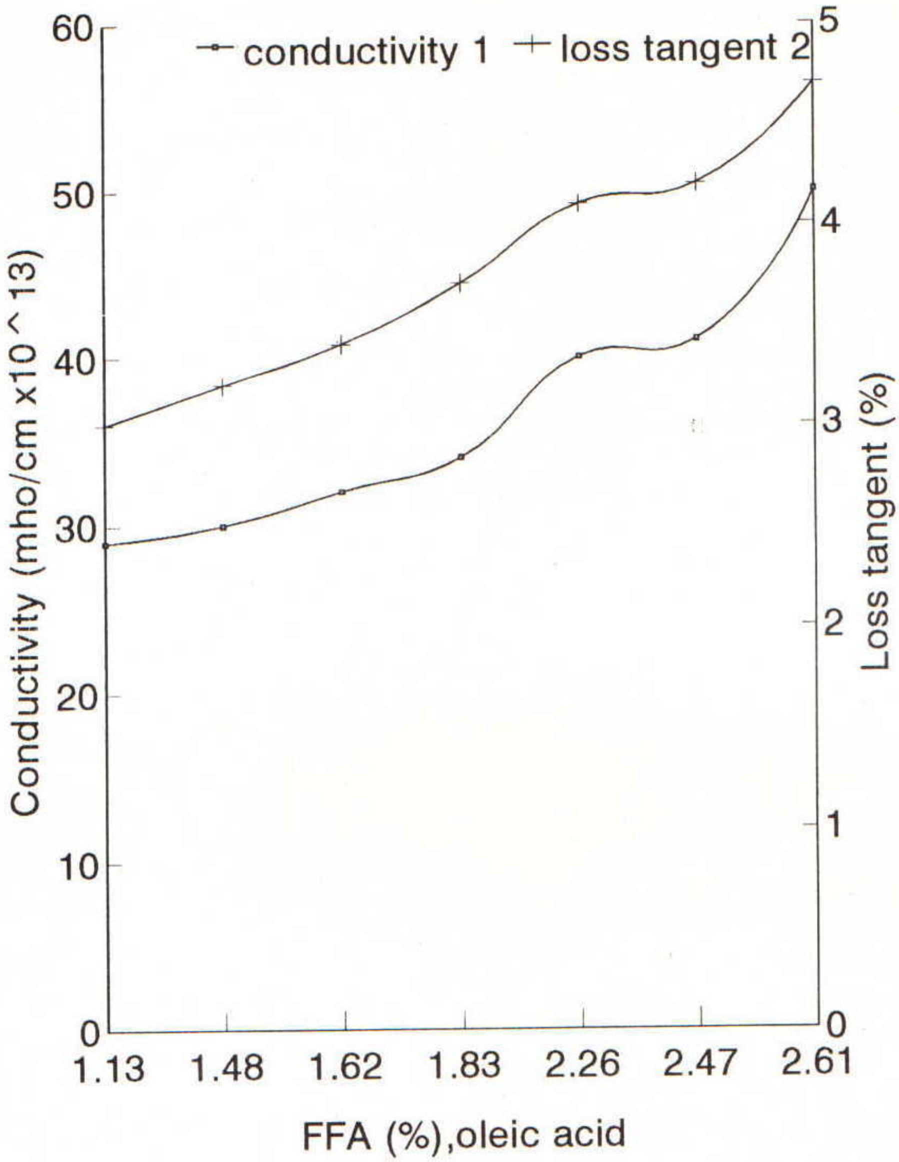

Fig.4.10.Variation of electrical conductivity and loss tangent of sunflower oil with FFA content

A linear regression equation relating FFA with loss tangent was established of the form:

$$y = a \ln x + b \qquad ..(4.5)$$

where

y = FFA, %

x = Loss tangent, %

a,b = Constants

The correlation coefficient (r^2) for both the oils was greater than 0.98. The value of constants a,b and correlation coefficients are tabled in Table F.4.

CHAPTER V

SUMMARY AND CONCLUSIONS

Electrical properties which include dielectric properties provide opportunities for non-destructive sensing of quality characteristics in agriculture and food products where those quality characteristics can be well correlated with the electrical properties. If good correlations exist, these electrical properties can be sensed with appropriately designed instruments which can be calibrated to provide the desired information about the quality of the product. Interest in the potential use of electrical properties of edible oils for non-destructive quality evaluation is more recent.

It was, therefore, decided to conduct investigations on selected edible oils. With the objective of measuring their dielectric properties and electrical conductivity at different temperatures and to establish correlation between FFA content, viscosity with dielectric properties and electrical conductivity.

Four edible oils namely cotton seed, groundnut, mustard and sunflower were selected for the study. Viscosity of all the oils was measured with Brookfield Dial reading viscometer. Dielectric properties and electrical conductivity was measured with Oil Loss Angle Meter MLO-1D and Million Megohm Meter LS-3D respectively. These equipments were supplied by Sivananda Electronics. Dielectric constant at different frequencies (1 KHz-100 KHz) at constant temperature of 15°C was determined with Model 4274A multifrequency LCR Meter. Seven samples each for sunflower oil and mustard oil with different FFA contents were prepared and their tangent loss and electrical conductivity was measured.

Viscosity of all the oils showed a non-linear decreasing trend with temperature. At 20°C the viscosity of cottonseed was highest (77 cp) while it was lowest for groundnut (48 cp). Linear regression equation were developed relating viscosity with temperature, loss tangent and electrical conductivity respectively. All the regression equations had regression coefficient greater than .96 for all the oils. The dielectric constant of the oils varied between 2.7-2.9 at 20°C. It remained nearly constant throughout the temperature range. The loss tangent values (in percentage) were in the range 0.5-14.2 at 20°C which increased with the increase in temperature. The electrical conductivity of all the oils also increased with the increase in temperature. It varied between 4-91x10^{-13} mho cm^{-1} for all the oils. Cottonseed oil was least conductive while sunflower was most among all oils. Loss tangent and electrical conductivity showed an increasing trend with the increase in FFA content of the oil samples. From the experimental investigations, the following conclusions could be drawn:

1. Viscosity of oils varied non-linearly with the increase in temperature. Initially (20°C) the viscosity of cottonseed oil was highest (77 cp) followed by mustard (71 cp), sunflower (64 cp) and groundnut oil (48 cp).
2. Viscosity could be well correlated with temperature. The regression equation developed had high correlation coefficient (r^2>.995) for all the four oils between temperature 20°C-100°C.
3. Dielectric constant of edible oils remained almost constant over the entire temperature range (20°C-100°C) with a little increase (0.2-0.3 units).
4. Initially the dielectric constant of refined oils (2.8 for cottonseed and 2.7 for groundnut) was less than that of freshly extracted oils (2.9 for mustard and sunflower oil).
5. Loss tangent increased appreciably with the increase in temperature. Initially the loss tangent of cottonseed oil was least (0.5%) followed by groundnut (1.5%), sunflower (14.2%) and mustard (18.8%).
6. The increase in loss tangent with temperature interval was less (1-3%) in case of cottonseed and groundnut while it was more (8-10%) for mustard and sunflower.
7. The regression equation developed relating viscosity with loss tangent had good correlation coefficient (r^2>.96) for all the four oils within temperature range

20°C-100°C.

8. Electrical conductivity of all the oils increased with the increase in temperature. Initially cottonseed oil was least conductive ($4x10^{-13}$ mho cm^{-1}) followed by groundnut (12^{-13} mho cm^{-1}), mustard (54^{-13} mho cm^{-1}) and sunflower (91^{-13} mho cm^{-1}) .

9. Cottonseed oil was least conductive over the entire temperature range (20°C-100°C).

10. The regression equation relating viscosity with electrical conductivity had high correlation coefficient (r^2>.98) over the temperature (20°C-100°C).

11. Dielectric constant remained practically constant over frequency range (1-100 KHz) at 15°C.

12. There was increase in loss tangent with the increase in FFA content of oil samples.

13. The electrical conductivity increased from 46 to 89×10^{-13} mho cm^{-1} when FFA content was increased to 0.42 to 1.56 for mustard oil.

14. For sunflower oil the electrical conductivity varied from 29 to $50x10^{-13}$ mho cm^{-1} when FFA content increased from 1.13 to 2.61%.

Suggestions For Future Studies

1. More number of crude oils should be taken.
2. Experiments should be carried out over a wide range of temperature and frequency to obtain a general trend in dielectric properties and electrical conductivity of edible oils.
3 Wider range and more number of oil samples of varying FFA content should be taken to get a clear view of its effect on dielectric properties and electrical conductivity.
4. The experiment should be conducted to optimize loss tangent and electrical conductivity w.r.t. FFA content by varying temperature,applied a.c. frequency and voltage.
5. Effect of physical and chemical parameters like colour,carbon chain,peroxide value,iodine value etc. on the dielectric properties and electrical conductivity should also be studied.
6. Replications should be taken for same oil sample(used for taking measurements of selected properties) to check the validity of the regression equations.

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APPENDIX-A

Cleaning Procedure for the Test Cell
(Catalogue SHIVA INSTRUMENTS)

1. Use carbon tetrachloride for cleaning the oil Test Cell.

2. Separate the component parts of three terminal oil test cell i.e., outer case and inner electrode and keep them on the clean ceramic tile.

3. Fill the outer case, partially, with cleaning agent

4. Assemble the component parts with solvent contained n outer case.

5. Shake the cell assembly vigorously and separate the parts and throw the cleaning agent.

6. Repeat the above procedure twice or thrice with fresh solvent.

7. Dry the cell to around 60°C

8. The cleaned dry empty cell should show resistance above 10^9 megohms. If not, repeat the above cleaning process again.

9. The cleaning of the cell should be done before and after every test.

APPENDIX-B

Procedure for Measurement of Dielectric Constant
(catalogue SHIVA INSTRUMENTS)

Before proceeding to make this test, clean the oil cell thoroughly. This may be ensured by testing empty clean cell on Million Megohm Meter. The resistance should be practically infinite.

1. Connect the Oil Loss Angle Meter to 230 V AC, 50 Hz power supply and switch on the equipment.

2. Keep 'OPERATE' switch in 'SET ZERO' position and voltage at zero.

3. Connect the empty cell to the Oil Loss Angle Meter as follows:

   a) Terminal marked 'H.V' should be connected to outer case terminal of the three terminal oil cell.

   b) Terminal marked 'L.V.' should be connected to the inner terminal of the oil cell.

   c) Terminal marked 'GUARD' should be connected to the middle terminal of the oil cell.

4. Put the 'OPERATE' switch in 'HV ON' position.

5. Gradually raise the voltage to about 500V

6. With the help of 'NULL' control adjust the null meter to read 1.

7. Put 'OPERATE' switch in 'SET ZERO' position.

8. Pour required quantity of oil in the cell.

9. Now put the 'OPERATE' switch in 'HV ON' position.

10. Null Meter will show a different reading. This new reading directly shows the Dielectric constant of the sample under test.

11. Heat the oil cell with oil in it to the desired temperature in the heating chamber.

12. Take measurement of Dielectric constant on Null Meter.

APPENDIX-C

Procedure for Measurement of loss tangent
(Catalogue SHIVA INSTRUMENTS)

1. Clean the Oil Cell thoroughly, this can be ensured by measuring on Million Megohm Meter which should be practically infinite.
2. Pour oil to test in the oil cell gradually, the oil enters the space cavity in between the two electrodes of cell through holes provided.
3. Keep Oil Cell in Heating Chamber.
4. insert the sensing probe in the oil cell.
5. Switch on the Heater, the meter on Heating Chamber will indicate the temperature of the oil in cell.
6. Connect Oil Loss Angle Meter to 230V AC, 50Hz power supply switch on the instrument.
7. Keep the voltage zero and 'OPERATE' switch in 'SET ZERO' position.
8. Connect the three Terminal Oil Test Cell to the instrument as explained in Appendix B.
9. Slowly raise the voltage to 500V to create desired stress level.
10. Put the 'operate' switch in 'HV ON' position
11. When the desired temperature is reached with the help of 'NULL' control adjust the null indicator to read 5.
12. Again put the 'OPERATE' switch in 'SET ZERO'. Set the zero on loss tangent meter carefully with the help of 'SET ZERO' control.
13. Put 'OPERATE' switch in 'HV ON' position and read the value of loss tangent on the DPM directly in percentage.
14. Repeat steps 3-13 for verification

APPENDIX-D

Procedure for Resistivity Measurement
Catalogue SHIVA Instruments

1. Clean the oil cell thoroughly.

2. Connect the Million Megohm Meter LS-3D to the mains supply. Switch on the instrument.

3. Select range required.

4. Push its 'MEASURE' push button control. Set meter to read infinity, when test terminals are open.

5. Push 'DISCHARGE ' push button and connect the empty oil cell to the instrument as follows:

   a) H.V. Terminal to the outer terminal Oil cell.

   b) L.V. Terminal to the innermost terminal of the Oil Cell.

   c) Guard terminal to the middle terminal of the Oil Cell.

   d) Terminal marked earth is connected to guard terminal by a Short Link.

6. Push 'MEASURE' push button and read the value of resistance, it should show infinity. If not remove the test cell after pushing the 'DISCHARGE' push button. Then clean the cell again and repeat the procedure till resistance is infinite.

7. Pour the required quantity of oil in the test cell.

8. Connect the cell to the instrument.

9. Put voltage control to +500V.

10. Push 'CHARGE' push button, after one minute of charging push the 'MEASURE' button and note the reading on meter.

11. Push 'DISCHARGE' push button and keep the cell in this position for 5 minutes.

12. Total resistance is:
    Meter reading x range multiplier.

13. Calculate the Resistivity
    Resistivity = Resistances (ohm) x cell constant (K)

APPENDIX-E

Table E.1. Viscosity of oils at 60 rpm

VISCOSITY (CP)

Temp. °C	Cottonseed				Groundnut				Mustard				Sunflower			
	R_1	R_2	R_3	Avg.	R_1	R_2	R_3	Avg.	R_1	R_2	R_3	Avg.	R_1	R_2	R_3	Avg.
20	79	76	76	77	48	48	48	48	72	70	71	71	66	65	61	64
40	37	36	36.5	36.5	28	26	25.5	26.5	40	39.5	39	39.5	33	31	32.5	31.5
60	20	20.5	18	19.5	18	16	11	15	28	24	23	25	18	17.5	17	17.5
80	16	13	14.5	14.5	12	10.5	9	10.5	0	15	13	16	14	10.5	10	11.5
100	11	10	9	10	10	9	7	8	17	12	10	13	12	8	7	9

Table E.2. Loss tanget of oils with temperature

Temp.	Tangent loss(tan d)%															
	Cotton seed				Groundnut				Sunflower				Mustard			
°C	R_1	R_2	R_3	Avg.	R_1	R_2	R_3	Avg.	R_1	R_2	R_3	Avg.	R_1	R_2	R_3	Avg.
20	.5	.5	.5	.5	1.5	1.5	1.5	14.0	14.2	14.3	14.2	14.2	18.6	18.7	19.0	18.8
30	1.4	1.4	1.4	1.4	1.9	2.0	2.0	2.0	21.0	21.1	21.2	21.1	28.4	29.0	29.2	28.9
40	2.5	2.6	2.7	2.6	2.9	2.9	3.0	2.9	27.2	27.6	27.4	27.4	38.0	38.5	39.0	38.5
50	3.1	3.2	3.4	3.2	3.7	3.9	4.1	3.9	34.8	35.0	35.5	35.1	47.8	48.3	48.3	48.2
60	4.6	4.7	4.8	4.7	5.6	5.6	5.6	5.6	39	39.	39.5	39.2	56.2	56.4	56.6	56.4
70	5.8	5.8	5.8	5.8	7.7	7.7	7.7	7.7	45.8	46.1	46.1	46.0	63.8	64.3	64.5	64.2
80	6.4	6.7	7.1	6.7	10.8	10.9	11.0	10.9	54.0	54.6	54.8	54.4	70.9	71.2	71.3	71.1
90	8.6	8.7	8.7	8.7	12.0	12.3	12.6	12.3	63.1	63.6	63.8	63.5	77.4	77.6	78.1	77.7
100	10.8	11.0	11.2	11.0	15.0	15.2	15.4	15.2	71.6	72.2	72.2	72.0	83.3	83.6	83.7	83.5

Table E.3. Calculation of electrical conductivity of edible oils

Temp. °C	Resistance x 10^6 R_1	R_2	R_3	Average $R \times 10^6$	Conductivity $\times 10^{-13}$
		Sunflower			
20	150	145	155	150	91
30	106	104	105	105	131
40	70	70	71	70	194
50	50	50	50	50	272
60	37	36	35	36	378
70	32	32	32	32	425
80	25	24	28	26	524
90	23	22	22	22	619
100	19	19	19	19	716
		Groundnut			
20	1145	1145	1135	1140	12
30	800	810	790	800	17
40	516	516	512	514	26
50	350	360	340	350	39
60	225	230	220	225	60
70	150	150	150	150	90
80	100	100	100	100	136
90	86	86	86	86	158
100	66	66	66	66	206

Temp. °C	R_1	Resistance$\times 10^6$ R_2	R_3	Average $R\times 10^6$	Conductivity $\times 10^{-13}$
		Cottonseed			
20	3600	3400	3200	3400	4
30	1500	1300	1200	1260	10
40	800	750	700	760	18
50	600	570	550	570	24
60	250	230	210	220	62
70	180	170	160	170	78
80	150	150	150	150	92
90	130	130	130	130	106
100	110	110	110	110	121
		Mustard			
20	270	245	240	252	54
30	125	115	110	115	118
40	85	80	75	80	170
50	40	40	40	40	340
60	28	27	26	27	504
70	22	21	21	21	648
80	18	18	17	18	756
90	15.5	15.5	15.0	15.3	890
100	15	15	15	15	908

APPENDIX-F

Table F.1. Values of constants and correlation coefficient for regression equation between viscosity and temperature

	Cotton seed	Groundnut	Mustard	Sunflower
a	.37622	.3432	.9191	.2623
b	99024318.78	88872424.07	83758988.31	97555479.06
r^2	.996	.998	.997	.997

Table F.2. Values of constants and correlation coefficient for regression equation between viscosity and loss tangent

	Cotton seed	Groundnut	Mustard	Sunflower
a	9.898	.007	.009	.002
b	-.009	.016	-.009	-.009
r^2	.966	.981	.993	.989

Table F.3. Values of constants and correlation coefficient for regression equation between viscosity and electrical conductivity

	Cotton seed	Groundnut	Mustard	Sunflower
a	-.523	-.621	-.532	-.949
b	-10.578	-13.231	-9.532	-19.983
r^2	.997	.992	.976	.996

Table F.4. Values of constants and correlation coefficient for regression equation between FFA content and loss tangen

	Cotton seed	Groundnut	Mustard	Sunflower
a			.486	3.311
b			1.590	-2.441
r^2			.991	.984