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Aging Characteristics of Polytetrafluoroethylene (PTFE)

**By
Hasan Zia Syed**

**A Thesis submitted to
The Faculty of Graduate Studies and Research through
The Department of Electrical & Computer Engineering
in partial fulfillment of the requirements for the
Degree of Master of Applied Science**

**University of Windsor,
Windsor, Ontario, Canada
2002**



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ABSTRACT

The aging characteristics of polytetrafluoroethylene (PTFE) are studied. The effects of salinity and temperature on the surface properties of PTFE are reported. To investigate the phenomenon of the loss and recovery of hydrophobicity, the surface of PTFE was subjected to various wetting conditions. The contact angle is a good indication of hydrophobicity and is used to determine the state of the surface of PTFE. It is observed that the hydrophobicity of PTFE decreased with increasing salinity, temperature and the duration of exposure. The surface free energies due to London dispersion (γ_{SD}) and hydrogen bonding (γ_{SH}) forces on the surface of PTFE are calculated from the contact angle measurements using both water and its components.

A study is performed to measure the coefficient of diffusion of water into PTFE for different salinities and temperatures. The increase in the weight of PTFE with increasing and methyl iodide. A computer program in "C" has been developed to calculate the surface free energy time of immersion is measured. The recovery of hydrophobicity, after aging, up to 5000 h is studied.

The average surface roughness and peak surface roughness is also measured during the aging and correlated with the loss of hydrophobicity.

The effect of RF discharges on the hydrophobicity is also investigated.

The effect of aging on the ac and dc flashover voltage at 10mS/cm saline solution at 0 ± 1.5 , 22 ± 4 , 44 ± 2 , $73\pm2^\circ$ and $99\pm2^\circ\text{C}$ shows a decreasing trend with increasing temperature. The decrease in ac and dc flashover voltage is correlated with the loss of

hydrophobicity of PTFE and surface roughness.

In order to study the changes due to the loss of hydrophobicity on the PTFE surface, a Fourier transform infrared spectroscopy (FTIR) is carried out from which a mechanism responsible for the loss of hydrophobicity is suggested.

Dedicated to

My mother, brother Syed A. Zia, wife Nighat, daughter
Sairah, Sons Sulman Zia and Nauman Zia, for their love,
inspiration and support.

Acknowledgements

Praise be to Allah (The God), The Almighty Who gave me the opportunity to undertake this task.

I wish to thank Dr. R. Hackam, my thesis advisor, for his supervision, guidance, support and encouragement throughout the course of this research work.

I would like to thank my department reader, Professor P. H. Alexander and my external reader, Dr. M. K. S. Madugula, for their valuable advice toward the fulfillment of the thesis work.

I would also like to thank Dr. Barbara Zielinski of the Biology department for her cooperation and help for microtoming of PTFE specimens in order to improve FTIR spectra.

I would also like to thank Dr. Ricardo Aroca and Dr. James Green of the Chemistry and Biochemistry department for their cooperation and help for the FTIR spectra of PTFE specimens.

Thanks are also due to Mr. John Robinson of Mechanical, Automotive and Materials Engineering department for carrying out the Scanning Electron Microscopy (SEM), of PTFE specimens.

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List of Abbreviations

ASR	Average Surface Roughness
C	Concentration (g/cm^3)
Cal	Calorie
D	Diffusion Coefficient (m^2/sec)
Ea	Activation Energy (J/mole or kcal/mole)
F	Time rate of transfer of matter per unit area ($\text{g}/\text{sec}\cdot\text{m}^2$)
FTIR	Fourier transform infrared spectroscopy
G	Gamma
IR	Infrared spectroscopy
L	Length (meter)
MSR	Maximum surface roughness
PTFE	Polytetrafluoroethylene
R	Universal gas constant ($8314.3\text{J}/\text{k}\cdot\text{moleK}$)
RF	Radio frequency
t	Time (sec or hour as defined)
T	Absolute temperature (K)

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Chapter 1

Properties of PTFE

1.0 Introduction

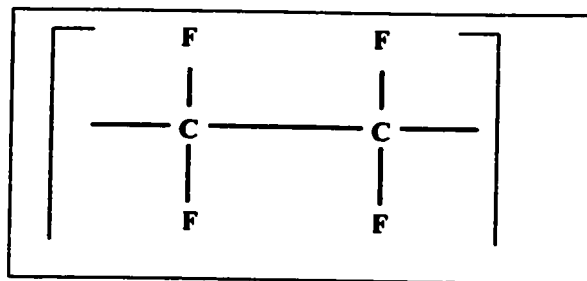
Polymer materials are widely used for electrical insulation in the wire and cable industry for transporting power and for communications. Polymers have been used widely in place of inorganic porcelain and glass for external housing of high voltage (HV) and low voltage (LV) outdoor devices since 1960's. Ceramic and glass insulators are highly wettable, when exposed to fog, dew, or rain, because of their high surface energy [1], and thus are hydrophilic as they possess a low contact angle. In the presence of natural and industrial contaminants such as dust, salt, cement and other industrial pollutants, leakage current develops on the surface, which may cause a flashover on the insulator and consequently an outage of the power system. Polymers offer significant advantages over porcelain and glass, such as lightweight, easy installation, reduced breakage and high impact resistance [1]. Polymer materials have low surface free energy and are hydrophobic. Hydrophobicity is defined as the surface property of a solid material, which provides the resistance to form a continuous film of water. A surface which has an inherent ability to maintain a high level of hydrophobicity in the presence of moisture would have a low leakage current, much reduced dry band arcing and therefore a better overall electrical performance and hence a longer life time [2]. The measurement of the contact angle of a water droplet on the polymer surface provides an indication of the hydrophobicity of the surface [1,3].

In this research the aging characteristics of polytetrafluoroethylene (PTFE) is reported and co-related with ac and dc surface flashover voltage.

The PTFE provides excellent resistance to almost all known chemicals and solvents. It has very low tendency to absorb moisture and has excellent dielectric properties. Its normal operating temperature is up to 260°C. Due to these excellent properties, teflon is widely used as electrical insulation and bushing, particularly for space and aeronautical industries [4].

1.1 Polytetrafluoroethylene

The polytetrafluoroethylene was first discovered at Du Pont in 1938 and was commercially available after 1946 [6]. The molecular structure of PTFE is as follows,



Fluorocarbon materials all have in common the widest operating temperature ranges of all the plastic, outstanding resistance to attack by chemicals, excellent dielectric properties and minimal tendency to absorb water. These characteristics are attributed to the strong carbon-fluorine bond. [5, 6 and 21].

PTFE is widely used for electrical wire and cable insulation, in applications at voltage up to 1kV and extremely high temperatures of up to 260°C (Federal USA specifications; MIL-W-168 78) and also where a resistance to abrasive environment is required at low voltages up to 600 V (MIL-W-22759). Additionally it is also employed in special applications where no other materials suffice including fire alarm cables, communication wires in low-flame-spread, coaxial cables and sensors in hostile environment [5,6]. The

low dielectric loss of PTFE over a wide spectrum of frequencies further extends its engineering utility. There has been much work published on PTFE including high field conduction and charge stability, but a study on its surface properties in the presence of moisture at various salinity and at different temperatures has not been reported yet.

1.2. Properties of PTFE

Polytetrafluoroethylene is a crystal-containing polymer whose structure is dependent on the technique used to cool it from a high temperature melt to room temperature. The physical and mechanical properties of tetrafluoroethylene polymer are affected by its crystalline structure [6]; some of the general properties of PTFE are given in table 1.A.

Table 1

General Properties of PTFE

Specific Gravity	2.1-2.3
Melting point, °C	327
Minimum service temperature, °C	-55
Maximum service temperature, °C	300
Flammability	None
Tensile Strength, psi	$1.8 - 4 \times 10^3$
Modulus of elasticity, tension, psi	58×10^3
Stiffness, psi	60×10^3
Elongation, %	100 – 800
Dielectric constant, 60 cps and 10^6 cps	2 – 2.2
Volume resistivity, ohm. cm	$10^{15} - 10^{17}$
Water absorption	< 0.01 %
Surface resistance, ohms	3.5×10^3

1.3 Research Objectives

1. To determine the hydrophobic behavior of PTFE after immersion in a saline solution at different temperatures and different conductivity levels over an extended period of time.
2. To study the effect of dc stress on the hydrophobic behavior of PTFE.
3. To study the effect of RF discharges on the loss and recovery of the hydrophobicity of PTFE.
4. To study the change in the surface roughness of PTFE during aging and recovery.
5. To study the change in the weight of PTFE during aging in different salinity of water and temperatures.
6. To determine the coefficient of diffusion of saline water into PTFE for different conductivity levels at different temperatures.
7. To determine the surface free energies (and its components) of PTFE at different stages of aging.
8. To study the recovery of hydrophobicity of PTFE at room temperature after aging at different temperatures in saline solutions of different conductivity levels.
9. To study the effect of high temperatures on the hydrophobic behavior of PTFE
10. To understand the mechanism of loss and recovery of hydrophobicity of PTFE insulation under the stress of heat and salinity.
11. To study the effects of temperature, salinity and the duration of exposure of water on the ac and dc flashover voltages.

Chapter 2

Experimental Method

2.1 Introduction

Experiments to determine the loss and the subsequent recovery of hydrophobicity have been carried out to study the aging characteristic of PTFE. The PTFE material was subjected to various tests and combined aging stresses of salinity and heat, which covers, to a great extent, the range of PTFE insulation material that is used in practice for cable and wire insulations in industry. PTFE is used as cable insulation in low voltage applications. [6]

2.2 PTFE Specimens

A length of 9.8 ± 0.3 mm PTFE specimens was cut from a 19 ± 0.1 mm diameter rod. The initial contact angle of a 4~5 μ l distilled water droplet of 7 ± 2 μ S/cm conductivity was $90.5 \pm 3.5^\circ$. The initial low contact angle was also observed in [7] and it is due to presence of dust, and other contaminants on the surface of virgin specimens. The specimens were washed in a 5% acetic acid in an ultrasonic vibrator for two minutes and then followed by washing in distilled water as described in [3, 7, 8, 9, 10 and 11]. The mild acetic acid does not react with polymer chemically and removes the usual contaminants from the surface of the polymer [8, 9]. Before measuring the contact angle the specimens were dried by

contact angle increased to $108 \pm 5^\circ$. This agrees reasonably with the previously reported value of 113° [21]. Two specimens were used at each temperature and conductivity level so as to ensure the reproducibility of the results [7, 9, 10, and 11].

2.3 Conductivity and Temperature of Saline Solutions

The saline solutions were prepared by adding sodium chloride (NaCl) to distilled water at room temperature and the conductivity of the solution was measured by a conductivity meter called (Horizon Ecology Co, type 1484). The range of this conductivity meter is $1.5 \mu\text{S/cm}$ to 500 mS/cm . Four conductivity levels 0.005 ± 0.002 , 1 ± 0.02 , 10 ± 0.1 and $100 \pm 0.1 \text{ mS/cm}$ were used. The conductivity of the saline solutions was checked intermittently during the aging process to ensure stable values of the conductivity. Five different temperatures of 0 ± 1.5 , 22 ± 4 , 44 ± 2 , 73 ± 2 and $99 \pm 2^\circ\text{C}$ were used. The specimens were also aged in air at these four temperatures.

2.4 Aging Process

The virgin PTFE specimen was immersed in different saline solutions of conductivity level of 0.005 mS/cm , 1 , 10 and 100 mS/cm and kept under thermal stress at temperatures of 0 ± 1.5 , 22 ± 4 , 44 ± 1.5 , 73 ± 2 and $99.5 \pm 2^\circ\text{C}$ for different durations. The aged specimen was taken out of the oven or the refrigerator after set interval of time and allowed to reach room temperature ($22 \pm 4^\circ\text{C}$) naturally before measuring the contact angle. The specimen was first washed in distilled water for 2 minutes and then dried using an air dryer at room temperature. The same aged specimen was then weighed, using a high

dryer at room temperature. The same aged specimen was then weighed, using a high precision (0.0001 g) scientific balance (Sartorius Ag Gottingen- Model BP 110 S), to observe the increase in the weight. The contact angle, which is a good measure of hydrophobicity, was then measured by using a Goniometer. The same aging process was employed by [7, 9].

The average surface roughness (ASR) was measured before immersion in the saline solution using a precision surface roughness detector (type Surftest 212 Mitutoyo). The same measurements were repeated in air at room temperature, for the aged specimen at all the temperatures and salinity levels. Two specimens were used at a fixed conductivity level and temperature. Clean glass containers were used to house the saline solutions and the specimens. The contact angle, weight and surface roughness were measured during aging of PTFE until a saturated value of the contact angle was reached.

2.5 Recovery Process

The “recovery process” started immediately after completion (saturation) of the loss of hydrophobicity. The saturated contact angle was also used as the initial reading for the recovery process. After immersion of about 4,000 h in saline solutions at different temperatures, saturation was reached in the values of the contact angle. The aged specimens were then removed from the saline solutions and kept in clean and dry glass containers in air at room temperature 22 ± 4 °C and 55 ± 15 % humidity for up to 5,000 h. The weight, the surface roughness and the contact angle of the aged specimens were recorded and used as the initial value. During the recovery, the changes in the contact angle, the weight and the average surface roughness (ASR) were measured intermittently.

2.6 Goniometer

The contact angle was measured after placing the specimen on a stable horizontal surface of the goniometer as given in [3]. This is a simple set up comprising two stands. One holds a horizontal platform and the other one holds a vertical syringe. One ml syringe was used so as to control the size of the water droplet to within 4~5 μl in order to maintain uniformity throughout the course of the experiment. A small table lamp (40 W) was used to illuminate the droplet clearly through the eyepiece of the goniometer. The contact angle was read within 30 s after placing the droplet of water on the specimen in order to eliminate the effect of evaporation of water on the surface of the specimen.

2.7 DC Stress

The effect of dc stress on the behavior of the contact angle was studied over a period of 500 h. A uniform dc stress of 7.5 and 12.5 kV/cm was applied to virgin specimens of PTFE at 22 ± 4 $^{\circ}\text{C}$. The specimens were placed between two parallel planar stainless steel electrodes having a diameter of 150 mm. The duration of the test was extended up to 500 h and the contact angle was measured intermittently. A Brandenburg regulated high voltage power supply (0 - 30 kV dc) was used.

2.8 RF Discharges

Radio frequency (RF) discharges were applied to virgin specimens of PTFE at 22 ± 4 $^{\circ}\text{C}$ for duration of 10, 15 and 20 minutes using a high frequency RF generator. The frequency of the discharge was approximately 250 kHz. The current flowing at the

virgin specimens were measured before and after their exposure to the RF discharge. The high frequency (HF), tester of Edwards High Vacuum Ltd was used. Fig. 2.1 shows the schematic diagram of the RF discharge measurement.

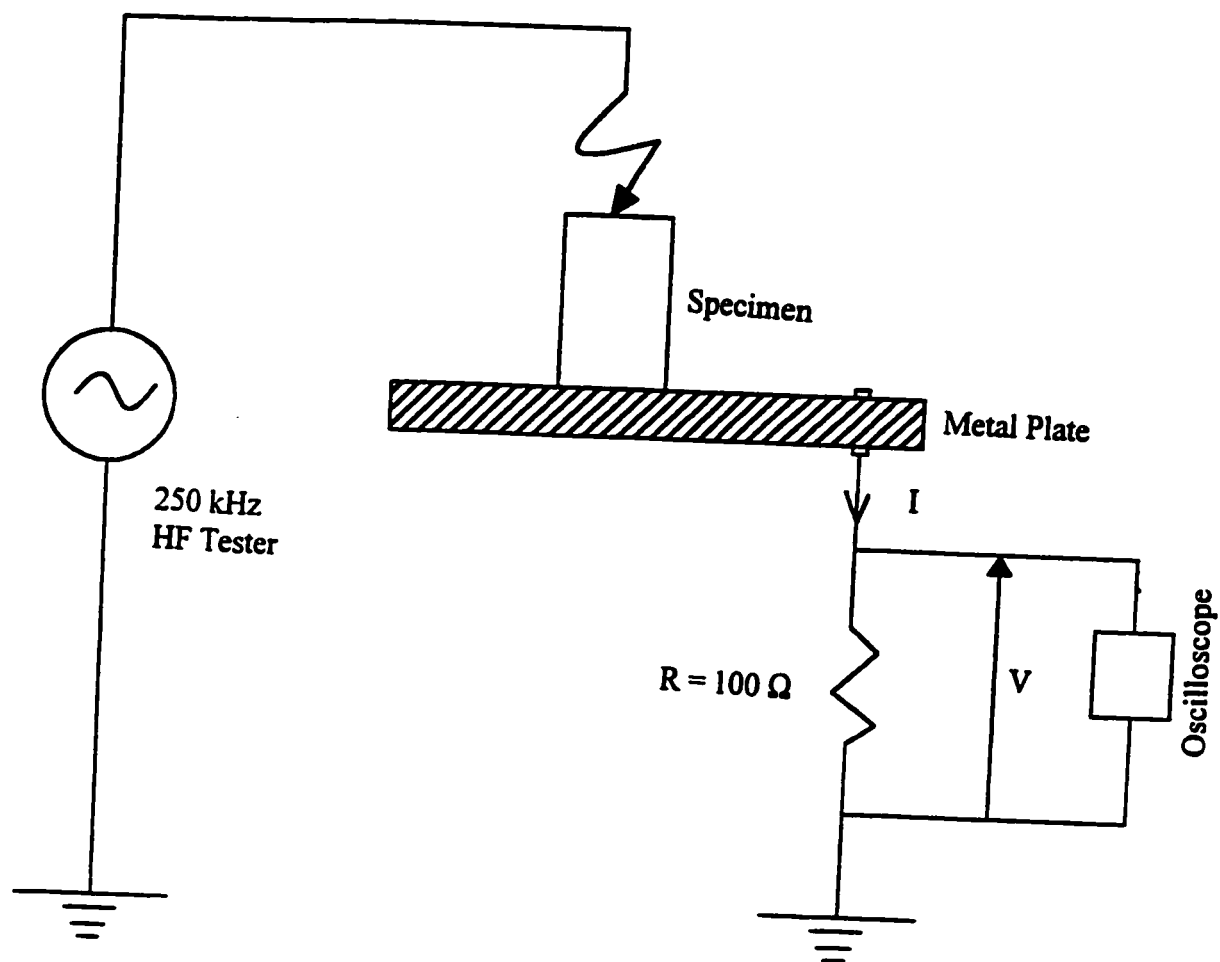


Fig. 2.1. Arrangement to apply RF discharge to polyethylene specimen.

Chapter 3

Measurement and Diagnostic Techniques

3.1. Contact Angle

3.1.1 Determination of Contact Angle

The contact angle is a measure of the surface hydrophobicity of the solid surface. The contact

angle of a liquid droplet on a solid surface is defined as the angle formed by the tangent to the droplet at the three phases (liquid, solid and air) junction and the horizontal surface [1, 13].

Static Contact Angle: If the system is at rest, the contact angle obtained is called the static contact angle.

Dynamic Contact Angle: The contact angle measured when the system is dynamic is called a dynamic contact angle.

The contact angle is a measure of the ability of the solid surface (hydrophobicity) to reject the formation of a continuous film of water. The higher the value of the contact angles, the more hydrophobic the solid surface is said to be. The contact angle depends on the surface tension (surface free energy) of the liquid and that of the solid surface on which the contact angle is measured. Young's equation describes the relationship among the contact angle, surface tension of the liquid and surface tension of the solid [1, 13 and 14].

$$\gamma_S = \gamma_{SL} + \gamma_L \cos\theta \quad \text{-----} \quad (3.1) \text{ Young's Equation}$$

where γ_S = Surface tension of the solid, J/m^2
 γ_L = Surface tension of the liquid, J/m^2

γ_{SL} = Surface tension of the solid / liquid interface, J/m²
 θ = Contact angle of the solid surface.

From equation (3.1) it can be observed that the higher energy solid surfaces, have smaller contact angle, therefore less hydrophobic and wet more readily.

According to [1], surfaces are classified into two categories: high energy and low energy surfaces.

High surface energy materials These include metals, metal oxides and inorganic compounds such as glass, silica, sapphire, porcelain and diamond. Their surface free energy is in the range 200~5000 mJ/m².

Low surface energy materials: These are organic compounds and polymers. They are usually soft, have low melting points and light in weight.

The contact angle of a liquid depends on the temperature of the liquid. All the measurement of the contact angles in the present work were taken at 22±4 °C. The overall change in the contact angle due to the change in the ambient temperature within the range of 22±4 °C is negligible [1]. The contact angle is independent of the conductivity of the droplet liquid [3, 8]. Distilled water of conductivity 7±2 µS/cm was used throughout the course of this study. The contact angle of a liquid droplet depends on the time lapse between placing the sessile droplet on the solid surface and taking the measurement. After 20 minutes the contact angle of droplet of 4~5 µl decreased from 108±5° to 92±1° on the PTFE surface. The variation of the contact angle with time after placing a droplet of water on the PTFE is shown in Fig.3.1. The measurement of the contact angle was completed within 30 s of placing the sessile droplet of water on the horizontal polymer surface.

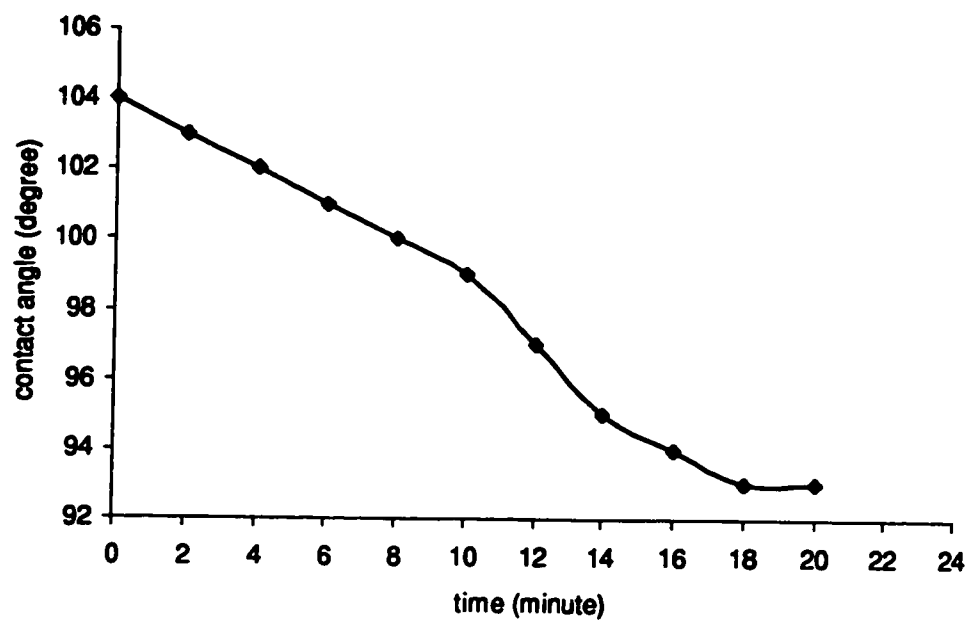


Fig. 3.1 Dependence of the contact angle of water on time after placing the droplet on the specimen. Conductivity of water, 0.005 m S/cm; Volume of droplet, 4~5 μ l.

The contact angle depends on the temperature, near the boiling point the liquid surface tension decreases rapidly, and the contact angle approaches zero [1]. The variation $(d(\theta)/d(T))$ in the contact angle due to temperature is $0.05^\circ/\text{C}$ [1]. The room temperatures at which the contact angle was measured, were in the range of $22\pm 4^\circ$.

Up to a $30\ \mu\text{l}$ volume of the droplet of water, the contact angle was reported to be independent of volume [8, 9]. Khan [7] has extended this limit up to $50\ \mu\text{l}$.

The size of the droplet used throughout this work was approximately $4\sim 5\ \mu\text{l}$ in order to maintain a good consistency of the experimental results.

3.1.2 Measurement of contact angle

The accuracy of the contact angle measurements can be judge by the reproducibility of the measured data of the surface investigated. Several methods to measure the contact angle for liquids on a solid surface in air are available, namely: Drop-bubble Method (Direct Observation-Tangent Method), Reflected Light Method, Interference Microscopy Method and Determination of Contact Angle from Drop Dimension [1].

3.1.2.1 Drop-Bubble Method

The method uses liquid drops or air bubbles on a plane solid surface. This is also known as the sessile drop method or Tangent Method or Direct Observation Method. A sessile liquid drop is placed on the surface of the solid and the contact angle is taken by measuring the angle made between the tangent to the drop profile at the point of contact

with the solid surface. The contact angle can be measured directly using a telescope fitted with goniometer eyepiece, or on a projected image or a photograph of the drop profile. The contact angle can be measured with an accuracy of $\pm 1\sim 2^\circ$. The probability of achieving an accuracy of $\pm 1\sim 2^\circ$ for angles less than 10° or more than 160° is highly uncertain due to the difficulty in locating the point of the contact of the tri-phase junction point [1]. This method has been used in previous studies by [7, 15], and also employed in this study. According to previous workers [1, 3, 8 and 9], it is a very reliable technique for measuring the contact angle in the range $10\sim 160^\circ$.

3.1.2.2 Reflected Light Method

The contact angle can also be measured by using a reflected beam from the surface of the drop of liquid, which is (beam) emitted by a microscope and it at right angle to the drop surface. This method can't be valid for a contact angle higher than 90° .

is

3.1.2.3 The Interference Microscopy Method

The Interference Microscopy Method overcomes the limitations of both the Tangent Method and the Reflected Light Method. In this method, a monochromatic light (a green mercury line) is reflected with a beam splitter onto the reflecting substrate to form interference bands parallel to the drop edge. The contact angle is calculated from the spacing of the interference bands, the refractive index of the liquid and the wavelength of the light [1].

3.1.2.4 Drop Dimensions Method

The contact angle can also be determined by the Drop Dimension Method. This method utilized the trigonometric relationships of the drop geometry to calculate the contact angle [1].

3.2 Surface Roughness:

Good [13] established a mathematical relationship, which showed the dependence of the contact angle on the surface roughness of a solid insulating material.

According to Good [13] if the contact angle is less than 90° , it is decreased by increasing surface roughness, and if the contact angle is greater than 90° , it is increased by surface roughness [16]. Therefore surface wettability depends on the surface roughness. The contact angle hysteresis increases with increasing surface roughness [1, 13, 14 and 16]. According to [8], the leakage current increases with the increase in the surface roughness. Therefore, to correlate the loss of hydrophobicity, a study of the change in the surface roughness of a polymeric insulator as a function of aging time is carried out. In this work, the surface roughness has been determined by Surface Detector (Surfetest 212 Mitutoyo, Japan).

3.2.1 Average Surface Roughness (ASR)

Average Surface Roughness is the mean value of the surface roughness profile over a certain length of the sample.

3.2.2 Maximum Surface Roughness (MSR)

Maximum surface roughness is the arithmetic mean value measured between the

highest peak and the lowest valley. To study the hydrophobic behavior of solid insulating materials Deng, [8] has studied the ASR and correlated it with the loss of hydrophobicity. ASR was measured in this work to determine the changes in the surface roughness of PTFE specimens. The sampling length was adjusted to be 10 mm longitudinally along the specimen rod. The surface roughness was measured 10~14 times on the same specimen at different locations and then an average of these values is reported.

3.3 Measurement of change in weight to calculate the diffusion coefficients

To calculate the diffusion coefficient of PTFE, the PTFE specimen of 19 mm diameter 0.88 ± 4 mm thick were immersed in 0.005, 1, 10 and 100 mS/cm saline solution at the different temperature of 0, 23, 44, 73, and 98°C. The change in the weight of specimens were measured intermittently for a very long period of time till the saturation was obtained. The change in the weight of the PTFE specimen was determined using a sensitive (0.0001 g) balance (Sartorius BP110S).

The diffusion coefficient was calculated by applying equation (4.8) and the time when $\Delta M(t) / M_{\infty} = 0.5$

Chapter 4

Surface Properties of Polytetrafluoroethylene

4.1 Introduction:

Polytetrafluoroethylene (PTFE) is widely employed in electrical insulation systems, which demand a high stability against degradation due to the effects of salinity and oxidation in the presence of moisture. PTFE can be used at high temperatures up to 250°C. In this chapter a study on the loss of hydrophobicity of PTFE in the presence of moisture, different salinities and temperatures is reported. The hydrophobicity is a property of the surface, which gives an indication of the degree of resistance to the formation of a continuous film of water. The loss of hydrophobicity is due to the degradation of the material in wet and polluted conditions. PTFE specimens were immersed in saline solutions where the conductivity was varied from 5 $\mu\text{S}/\text{cm}$ to 100 mS/cm . The temperature of the solutions were 0 ± 1.5 , 22 ± 4 , 44 ± 2 , 73 ± 2 , and $99\pm 2^\circ\text{C}$. The loss of hydrophobicity was measured by determining the contact angle of a droplet of distilled water on the PTFE surface. It was observed that the decrease in the contact angle depended on the combined stress of salinity, temperature and duration of immersion. The influence of a radio frequency (RF) discharge and dc stress on the loss of hydrophobicity was studied. The increase in the weight and the change in the surface roughness and their correlation with hydrophobicity of the specimens are also reported.

The percentage (%) increased in the weight of PTFE was measured using a high precise balancing machine and the coefficients of diffusion of saline water into PTFE specimen were calculated at different temperatures and salinities.

The surface free energy and its components are calculated for 0.005, 1, 10 and 100 mS/cm saline solution at 98°C, and observed that the surface free energy increased with increasing water salinity and its duration of application. To study the effect of temperature on the surface free energy, it was also calculated at 10 mS/cm saline solution at 0, 23, 44, and 73°C. It was observed that the surface free energy increased with increasing temperature and the duration of immersion [15].

Hydrophobicity is the ability of the surface to resist the formation of a continuous film of water. The aging process normally starts from the surface of the insulating material, in the form of degradation due to oxidation. This degradation causes a loss of hydrophobicity on the surface of polymers, which is accelerated with increasing stresses of temperature and salinity. Water can readily cover the surface and permeate into the bulk, after the loss of hydrophobicity.

This chapter covers the effects of various parameters such as water, salinity, heat, ac and dc stresses and RF discharges on the contact angle of a water droplet on the surface of the PTFE specimen. The effects of these stresses on the weight, coefficient of diffusion, surface free energy and surface roughness of PTFE are also discussed.

4.2. Experimental procedure

PTFE cylindrical rods of 9.0 ± 0.6 mm in length and 19 mm in diameter were immersed in saline solutions, at various temperatures and for extended duration. Four conductivities of the saline solutions of 0.005, 1, 10 and 100 mS/cm were used. The required conductivity

was obtained by adding table salt (NaCl) to distilled water, which had a conductivity of 5 $\mu\text{S}/\text{cm}$. The saline solutions and the PTFE specimens were kept at $0\pm1.5^\circ\text{C}$, $22\pm4^\circ\text{C}$, $44\pm2^\circ\text{C}$, $73\pm2^\circ\text{C}$ and $99\pm2^\circ\text{C}$ for duration of up to 4000 hours. The PTFE specimens were also left in air at these temperatures. The specimens were intermittently removed from the saline solution for the measurements of the contact angle θ . The contact angle between the tangent of a droplet of distilled water on the surface of PTFE and the horizontal surface was measured with a goniometer to an accuracy of $\pm0.5^\circ$ [18]. The volume of the droplet of water was about 5 μl . Previously the contact angle was found to be independent of the volume of the droplet for up to 50 μl [19], in agreement with other studies at varying volumes [3, 7].

The contact angle θ at the triple junction of air, water and PTFE, was initially measured before cleaning and was found to be 92° . After washing in a diluted acetic acid (5%, vinegar) and distilled water with the aid of an ultrasonic vibrator and dried at $22\pm4^\circ\text{C}$, the contact angle increased to $108\pm5^\circ$. This value is in good agreement with the previously reported values of 108° [1], 109° [20] and 113° [21] at 20°C .

The contact angle was determined at $22\pm4^\circ\text{C}$ and humidity of, $50\pm15\%$. After removal of the specimens from the saline solutions at 0, 22, 44, 73 and 99°C , they were allowed to cool down or warm up before the contact angle, the surface roughness and the weight were measured. Typically it took 20 to 30 min for the specimens to cool down and the excess water to dry before commencing the measurements of θ . The contact angle was determined within 30 s after placing the droplet of water on the specimen. The contact angle was measured in 6 different locations on each specimen. Two specimens were

employed for each condition in order to determine the reproducibility of the results. The reported values of θ are the averages of 12 readings for each condition.

4.2.1 Measurement of Surface Tension of PTFE during Aging:

Young's equation describes the relationship between the contact angle and the surface free energies. According to equation 3.1 (Young's equation)

$$\gamma_S = \gamma_{SL} + \gamma_L \cos\theta \quad \text{-----} \quad (3.1)$$

where γ_S = surface tension of the solid, J/m²

γ_L = surface tension of the liquid, J/m²

γ_{SL} = Surface tension of the solid / liquid interface, J/m²

θ = Contact angle of the solid surface.

Also the surface tension or the surface free energy can be divided into a dispersion (non-polar) component due to the London dispersive force and a polar component due to the polar force and therefore can be represented by

$$\gamma_S = \gamma_{SD} + \gamma_{SH} \quad \text{-----} \quad (4.1)$$

where suffix

D denotes the dispersion components

and H denotes the polar components

S for solid

and γ_{SD} and γ_{SH} are the surface free energies per unit area due to the dispersion

forces and the hydrogen bonding forces, respectively for the solid. From equation (3.1),

the higher the value of γ_s , the lower the value of contact angle (θ) and material is said to be hydrophilic. For PTFE γ_{SD} and γ_{SH} are responsible for the hydrophobicity and hydrophilicity, respectively.

For a liquid equation (4.2) can be written as

$$\gamma_L = \gamma_{LD} + \gamma_{LH} \quad \text{-----(4.2)}$$

where suffix L is for liquid.

The surface tension of a solid polymer cannot be measured directly. Many indirect methods have been developed to determine the surface tension of solid polymers. This includes the polymer melt methods, liquid homolog methods, equation of state methods and harmonic mean methods, all of these give consistent and reliable results. The geometric mean method, critical surface tension and other methods are less reliable [1].

To study the variation in surface tension due to the aging of the PTFE surface, the Harmonic mean method is employed for this research.

The harmonic mean methods require the measurement of the contact angle of two liquids of known surface tensions and their hydrogen bonding and dispersion force components γ_{SD} and γ_{SH} , respectively. The harmonic mean approximation is applicable for the system of saline water and PTFE as the polarizabilities of the interacting elements are not too different. Then according to [1, 25].

$$\gamma_{SD} = \gamma_s + \gamma_L - (4 \gamma_{SD} \gamma_{LD} / \gamma_s + \gamma_{LD}) - (4 \gamma_{SH} \gamma_{LH} / \gamma_{SH} + \gamma_{LH}) \quad \text{-----(4.3)}$$

Substituting the value of γ_{SL} from equation (3.1) into equation (4.3)

$$\gamma_L (1 + \cos \theta) = (4 \gamma_{SD} \gamma_{LD} / \gamma_s + \gamma_{LD}) + (4 \gamma_{SH} \gamma_{LH} / \gamma_{SH} + \gamma_{LH}) \quad \text{-----(4.4)}$$

Writing the equation (4.4) for water and methylene iodide and solving simultaneously for two unknown quantities γ_{SH} γ_{SD} . The sum of γ_{SH} and γ_{SD} according to (4.1) will give

the value of γ_s . A computer program in “C” has been written to calculate the surface free energies using the mathematical model per equation (4.4) for water and methylene iodide, as shown in Appendix A. Using this program, we can calculate the surface free energies for any solid material by measuring the contact angle with water and methylene iodide and substituting the values of contact angles into the program. The program has been verified by using the literature value of contact angle to calculate the surface free energies as per [1]. For θ_w of (108°) and θ_M of (77°) on PTFE surface, the calculated values of γ_s , γ_{SH} and γ_{SD} are

$$\gamma_s = 0.022545 \text{ J/m}^2$$

$$\gamma_{SH} = 0.002007 \text{ J/m}^2$$

$$\gamma_{SD} = 0.020538 \text{ J/m}^2$$

which agrees with the values reported in [1].

4.2.2 Measurement of Diffusion Coefficient

The coefficients of diffusion of water into PTFE specimen at different salinities and temperatures were investigated and are reported. The PTFE specimen of 19 mm diameter and 9.4 ± 0.6 mm and 0.92 ± 0.06 mm thickness were immersed in saline solutions of salinity of 0.005, 1, 10 and 100 mS/cm. The specimens were kept under heat stress at different temperature of 0°C, 23°C, 44°C, 73°C and 98°C. The increase in the weight of the PTFE specimen was measured regularly until saturation in the weights were reached. Two specimens were used at each salinity and temperature. This method of finding the diffusion of coefficient have previously been reported by [3, 7, 24].

The saline solution were prepared by adding table salt (sodium chloride) to distilled water giving the desired salinity (0.005, 1, 10 and 100 mS/cm).

Crank [26] first develop the relationship describing the rate of absorption of liquid into a membrane. According to Crank when the diffusion is driven by concentration gradient then the rate of absorption of the liquid into the membrane will initially be linear with $t^{0.5}$, provided that there is no chemical reaction between the liquid and the membrane which result in weight changes, then

$$\Delta M(t) / \Delta M_{\infty} = 2(Dt/l^2)^{0.5} \{ (1/\lambda)^{0.5} + 2 \sum_{n=1}^{\infty} [(-1)^n \text{ierfc}(nl/2(Dt)^{0.5})] \} \quad (4.5)$$

where $\Delta M(t) \Rightarrow$ is the increase in the weight ($M(t) - M(0)$) of the membrane (kg) due to the absorption of liquid at time t .

$M(t) \Rightarrow$ is the weight at time t ,

$M(0) \Rightarrow$ is the initial weight at time $t = 0$.

$\Delta M_{\infty} \Rightarrow$ is the saturated increase in the weight (in kg) at infinity time ($t = \infty$) or at very long time

$D \Rightarrow$ is the diffusion coefficient in m^2/s

$l \Rightarrow$ is the thickness of the membrane (in m) and

$\text{ierfc} \Rightarrow$ is a mathematical function, which is defined as

$$\text{ierfc}(x) = (\pi \exp(-x^2))^{1/2} - x * \text{erfc}(x) \quad \text{--- (4.6)}$$

where erfc is a complementary error function

and when $x \rightarrow 0$, $\text{ierfc}(x) \rightarrow \pi^{-1/2}$

and when $x \rightarrow \infty$, $\text{ierfc}(x) \rightarrow 0$

The equation (4.6) is for one-dimensional diffusion and since the diffusion occurs from both ends of the disc, for two-dimensional diffusion, then l (thickness of PTFE disc) is replaced by L , where $L = 2l$.

For the early stages of absorption, the second term in equation (4.6) may be neglected, as D is very small, leading to an error function approaching to zero.

$$\text{Equation (4.6)} \Rightarrow \Delta M(t) / \Delta M_{\infty} = 4(D / \pi L^2)^{0.5} * t^{0.5} \quad \text{----- (4.7)}$$

From equation (4.7), D can be determined from the linear dependence of the plot of $\Delta M(t) / \Delta M_{\infty}$ against $t^{0.5}$, or in the absence of linear dependence D may be determined from the following equation when $\Delta M(t) / \Delta M_{\infty} = 0.5$

$$0.5 = 4(D / \pi L^2)^{0.5} * t^{0.5} \text{ or}$$

$$D = 0.49 (L^2)^{0.5} / t^{0.5} \quad \text{----- (4.8)}$$

4.3 Results and discussion

4.3.1 Effect of water salinity, temperature, and duration of immersion on the hydrophobicity of PTFE

Fig. 4.1 shows the contact angle as a function of immersion time at $0 \pm 1.5^\circ\text{C}$ and for varying conductivity of the solutions, in the range of 0.005 to 100 mS/cm. It can be observed from Fig. 4.1 that the decrease in the contact angle is very slow. At 100 mS/cm the contact angle decreased from 108° to 92° after 5000h of immersion.

Fig. 4.2 shows the contact angle as a function of immersion time at $22 \pm 4^\circ\text{C}$ and for varying conductivity of the solution, in the range of 0.005 to 100 mS/cm. Fig. 4.3 shows that θ decreased steadily with increasing time of immersion for all conductivity levels.

The reduction in θ was larger with increasing salinity. After 4,000 h of immersion at 23°C, θ decreased to 104, 100, 92, and 86°, respectively for the solutions having 0.005, 1, 10, and 100 mS/cm (Fig. 4.2). The contact angle on the specimens left in air remained essentially constant at $105 \pm 3^\circ$, after 4,000 h (Fig. 4.2). The larger reduction in θ at higher conductivity values (depicted in Fig. 4.2) is due to the enhanced oxidation in the presence of salt. This is attributed to the formation of polar oxygenated products by thermal oxidation of the PTFE surface resulting in a reduction of the hydrophobicity. These oxygenated products which were observed previously in XLPE cables are hydrophilic [27, 28 and 29].

Figs. 4.3 and 4.4 show the contact angle as a function of immersion time at 44 ± 2 and $73 \pm 2^\circ\text{C}$, respectively. θ was found to decrease with increasing time of immersion, increasing temperature and increasing salinity. Typically, Fig. 4.3 shows that θ decreased from 108° before immersion to 100, 88, 87 and 85° , respectively after immersion for 4,000 h in 0.005, 1, 10 and 100 mS/cm at $44 \pm 2^\circ\text{C}$ (Fig. 4.3). When the PTFE specimens were left in air at this temperature the contact angle remained in the range of $104 \pm 5^\circ\text{C}$ (Fig. 4.3).

Fig. 4.4 shows the contact angle as a function of immersion time at $73 \pm 2^\circ\text{C}$. It will be observed that θ decreased steadily at this temperature for all conductivity. Typically, after 3,000 h of immersion the contact angle decreased to 87° , 82° , 80° and 62° , respectively for 0.005, 1, 10 and 100 mS/cm (Fig. 4.4). The contact angle reached saturated values after immersion for 3,000 h and remained at these low levels up to 4,000h (Fig. 4.4). In air θ was $103.5 \pm 4.5^\circ$ during 3,000 h of heating at $73 \pm 2^\circ\text{C}$ (Fig. 4.4).

From Fig. 4.5, it will be observed that at 99°C the contact angle decreased relatively sharply than all the other temperature (0, 22, 44 and 73°C). θ decreased from 108 to 39° after 4000 h of immersion in 100 mS/cm saline solution. The effect of conductivity level at 99°C was very significant and the same observation is also reported in [7]. At 10, 1 and 0.005 mS/cm of conductivity the contact angle decreased to 65, 85 and 87.5° respectively.

Fig. 4.6 shows the saturated contact angle against temperature after immersion from 3,000 to 4,000 h at different salinity levels. It will be observed that, at a fixed conductivity level, θ decreased with increasing temperature of the solution, and this occurred for all conductivity levels (Fig. 4.6). This is due to the enhanced oxidation reaction with increasing temperature. At a fixed temperature θ also decreased with enhanced salinity (Fig. 4.6). This is also due to the oxidation reaction on the surface of PTFE becoming more effective with increasing the amount of salt in the solution.

4.3.2. Effect of humidity of air on the weight of PTFE at 22±4°C

A PTFE specimen was first cleaned and dried and then kept in air 22±4°C, for 4,000h. the humidity varied during this time in the range of 50±15%. The weight of the specimen remained constant within ±0.0025%.

4.3.3. Effect of R.F. discharges on the contact angle of PTFE

After subjecting the PTFE specimen to R.F. (radio frequency) discharges, for duration of 10, 15 and 20 min, the contact angle decreased from 108±5° to 87.25, 86.2 and 84.8°, respectively.

The reduction in the contact angle was larger; the longer was the duration of the R.F. discharge. After terminating the R.F. discharge, the PTFE specimens were left in air (at $24\pm4^{\circ}\text{C}$) in order to study the recovery of the contact angle. Fig. 4.7 shows the recovery of the contact angle in air at 22°C after subjecting the PTFE specimens to R.F. discharges for various duration. It took about 450 h for θ to recover its original value. This is consistent with a recovery time of 500 h reported for EPDM after the surface was subjected to RF discharges [19].

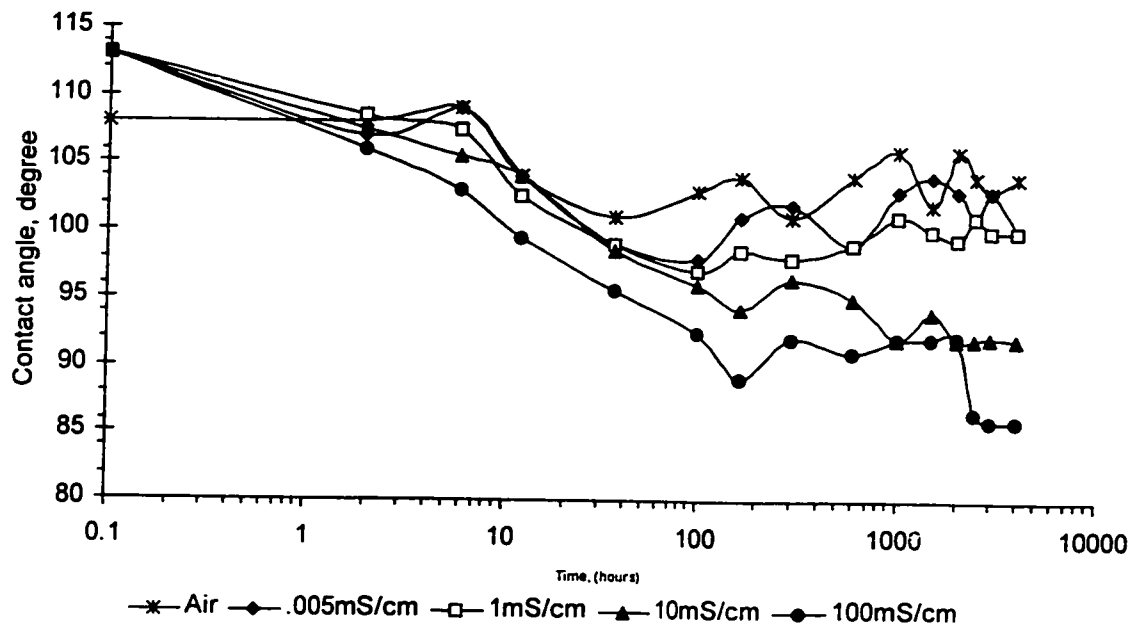


Fig.4.1. Dependence of the contact angle on the duration of immersion in saline solutions and in air at $0 \pm 1.5^\circ\text{C}$. (Material, PTFE).

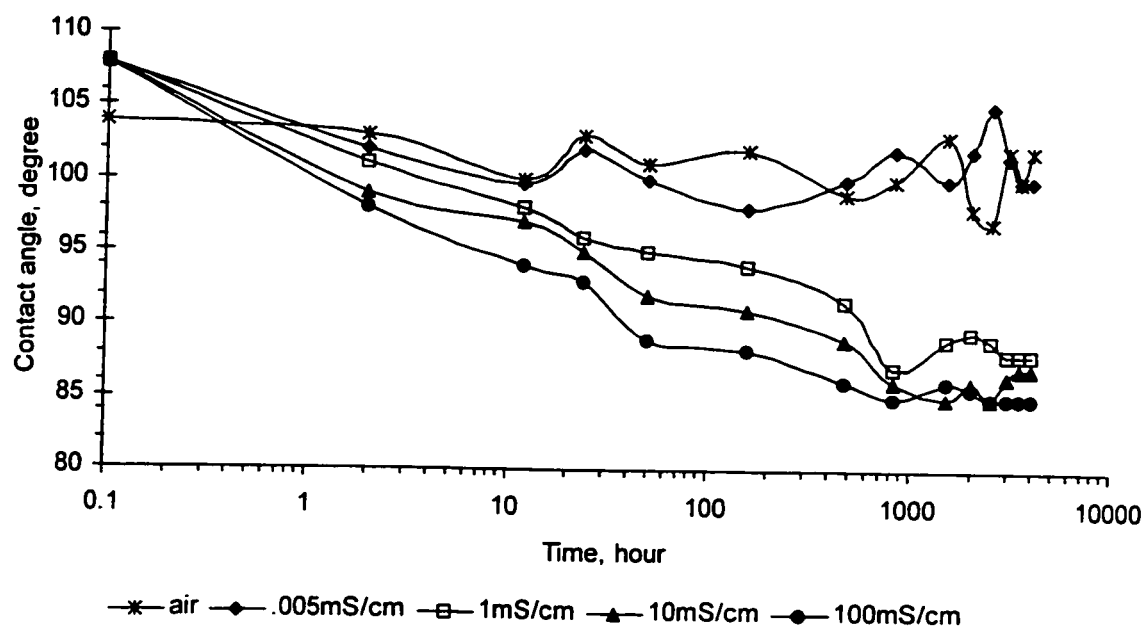


Fig.4.2. Dependence of the contact angle on the duration of immersion in saline solutions and in air at $22\pm4^{\circ}\text{C}$. (Material, PTFE).

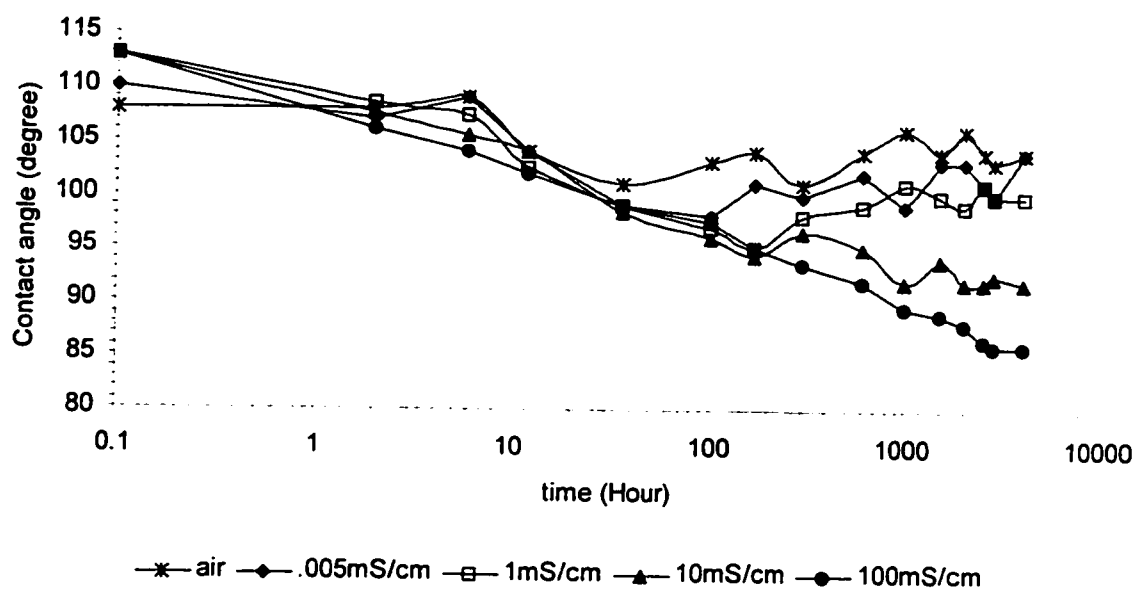


Fig.4.3. Dependence of the contact angle on the duration of immersion in saline solutions and in air at $44 \pm 2^\circ\text{C}$. (Material, PTFE)

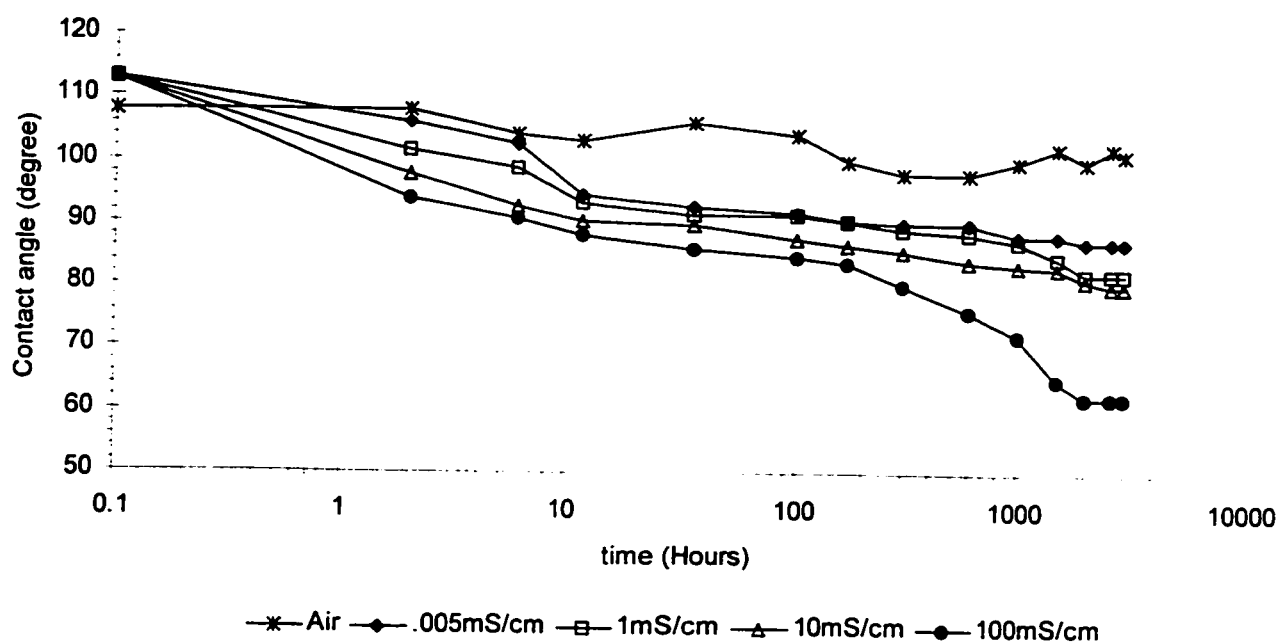


Fig. 4.4. Dependence of the contact angle on the duration of immersion in saline solutions and in air at $73\pm 2^{\circ}\text{C}$. (Material, PTFE).

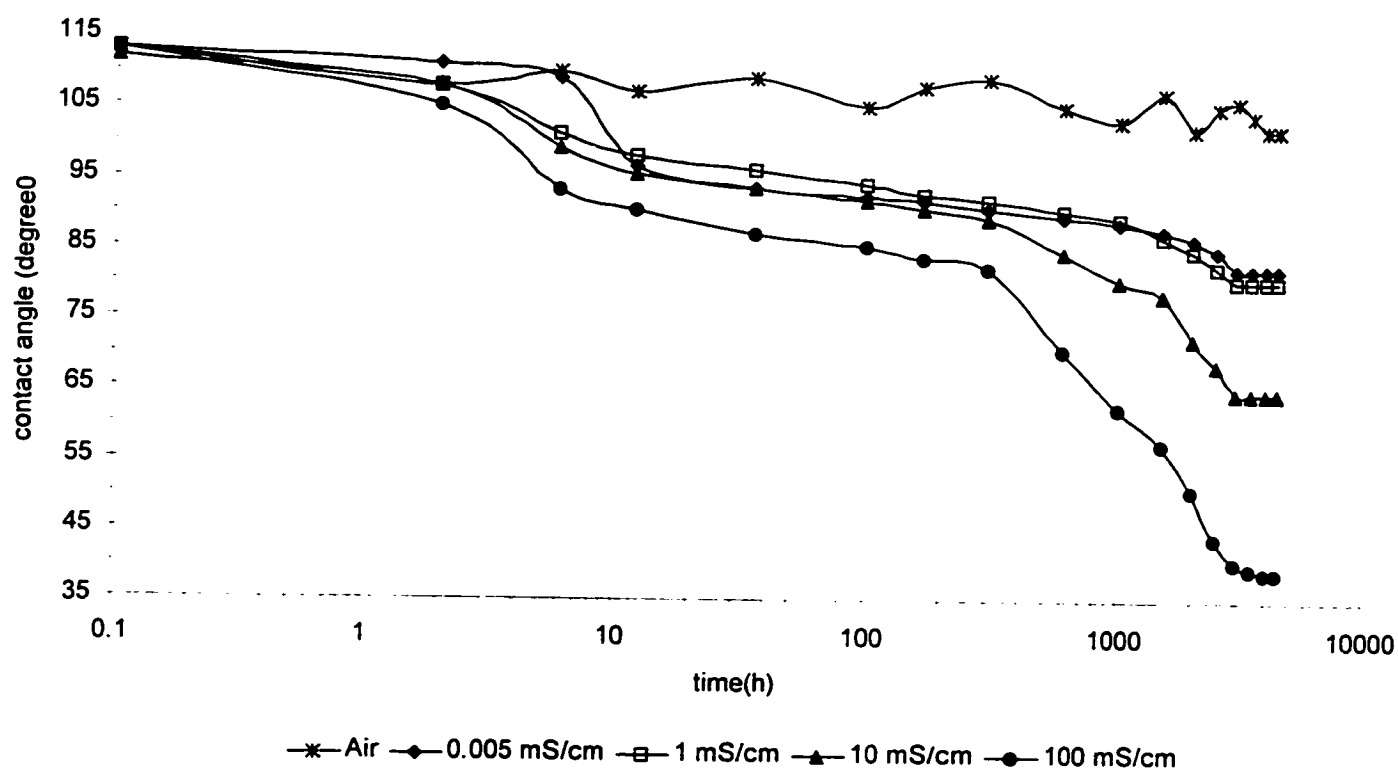


Fig. 4.5. Dependence of the contact angle on the duration of immersion in saline solutions and in air at $99\pm 2^\circ\text{C}$. (Material, PTFE).

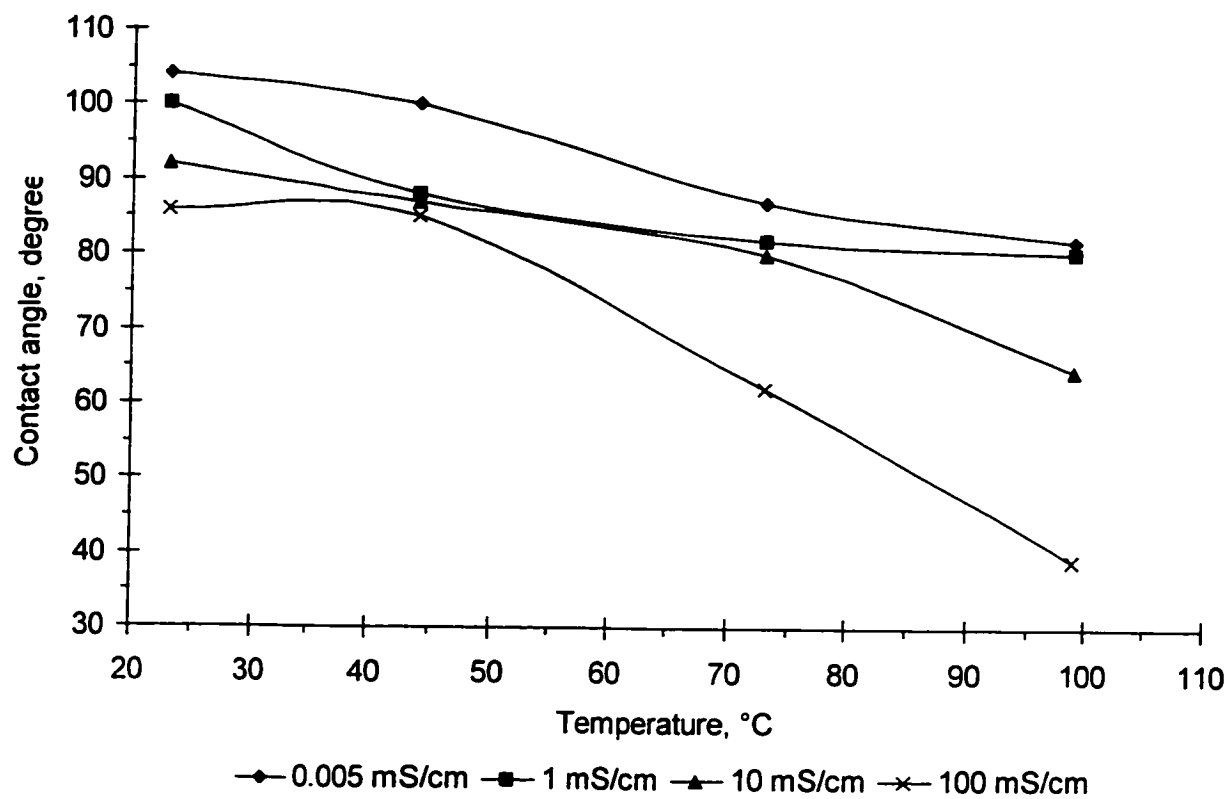


Fig. 4.6. Dependence of the contact angle on the temperature of the saline solution. Time of immersion 4,000h ($22\pm4^\circ\text{C}$ and $44\pm2^\circ\text{C}$), 3,500h ($73\pm2^\circ\text{C}$) and 3,500h ($99\pm2^\circ\text{C}$). (Material PTFE).

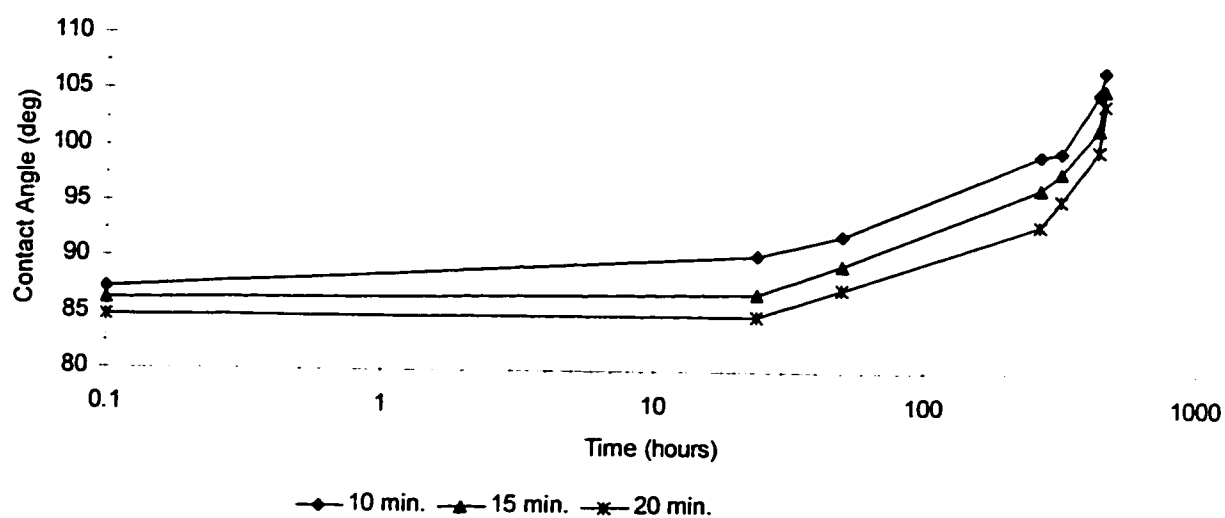


Fig. 4.7. Recovery of contact angle after application of RF discharges for duration from 10 to 20 min.

4.3.4 Effect of DC Stress on Contact Angle

Subjecting a PTFE specimen to 12.5 kV/cm, dc uniform field for 500h resulted in only small reduction in the contact angle θ from $108\pm5^\circ$ to $104\pm4^\circ$. For 7.5kV/cm θ was $106\pm5^\circ$. The lack of large change in θ with the application of a dc field is due to the negligible amount of current flowing over the surface of the PTFE rod at these field levels. The surface flashover field of a virgin specimen was also measured and found to be 26.50 ± 1.55 kV/cm at $22\pm4^\circ\text{C}$, with an electrode gap length of 9.9 ± 0.15 mm.

4.3.5. Average Surface Roughness

The average surface roughness (ASR) of the PTFE specimen was measured before and after immersion in the solutions as shown in Fig. 4.8.

Before immersion the ASR was 1.12 ± 0.32 μm . After immersion for 4,000 h, the ASR increased with increasing temperature and with increasing salinity. Typically, for 100 mS/cm, ASR roughness increased to 1.73, 1.82, 2.42 and 2.94 and 3.12 μm , respectively for 22, 44, 73, and 99°C . The increase in the surface roughness is due to the intake of water which caused a distortion of the surface of specimen.

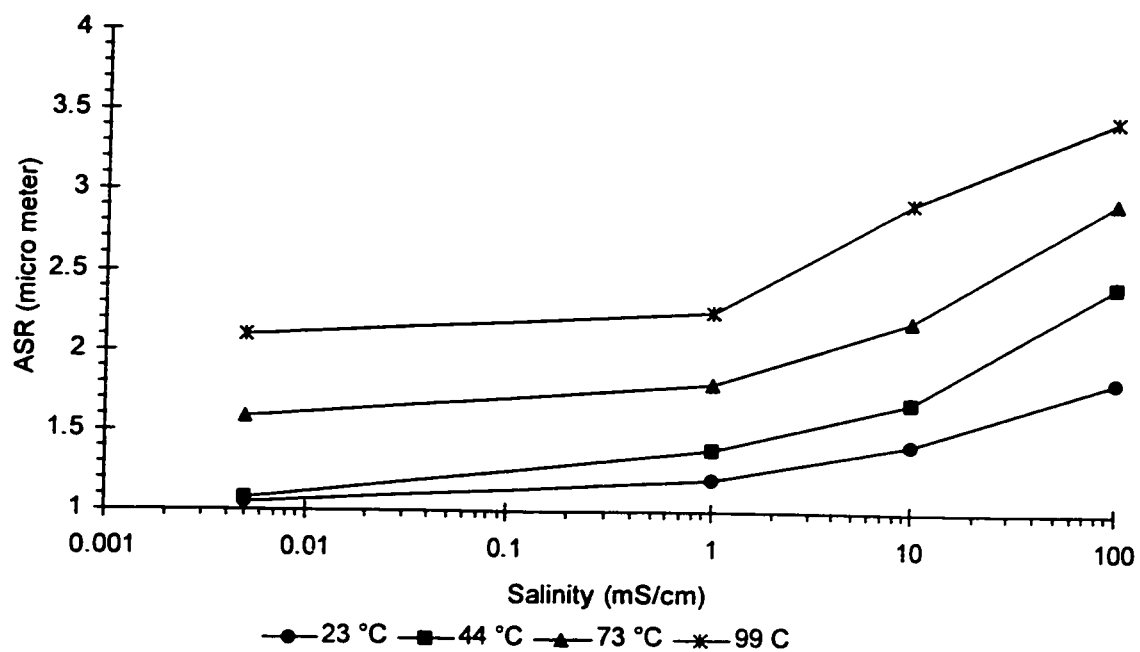


Fig. 4.8 Increase in average surface roughness against different salinities at 24 ± 4 °C, 44 ± 2 °C, 73 ± 2 °C and 99 ± 2 °C

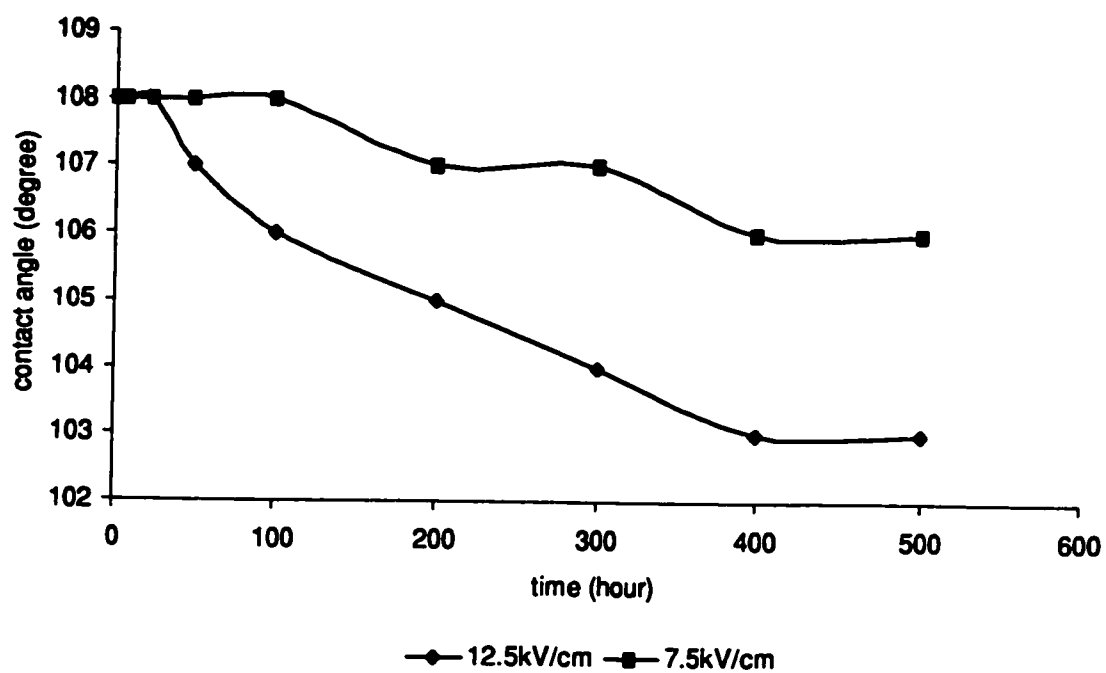


Fig.4.9 Time variation of contact angle of droplet of distilled water on PTFE after application of uniform dc stress up to 500 hours.

4.3.6 Calculation of Surface Tension (Surface Free Energy) of PTFE

Surface free energy γ_s for PTFE can be calculated by measuring the static contact angle of water (θ_w) and of methylene iodide (MI), (θ_M) on PTFE surface and solving the two equation (4.4) for water and methylene iodide simultaneously. For distilled water the literature values of γ_{LW} , γ_{LWD} and γ_{LWH} are 72.8×10^{-3} , 22.1×10^{-3} , 50.7×10^{-3} , respectively [1]. For methylene iodide (MI), γ_{LM} , γ_{LMD} and γ_{LMH} are 50.8×10^{-3} , 44.1×10^{-3} , 6.7×10^{-3} , respectively [1]. The contact angle with water (θ_w), was measured as 108° while the contact angle with MI, θ_M was measured as 77° on a virgin PTFE specimen. In this study the surface free energy and its components are calculated by using the computer program in "C" as described in appendix A. All three component γ_s , γ_{SH} and γ_{SD} were calculated. The calculated value is reported in section 4.2.1 and agrees with the literature value [1].

Fig 4.10, shows the change in the surface free energy and its components at 10 mS/cm at $0 \pm 1.5^\circ\text{C}$. It can be observed that surface free energy increased with the increasing time of immersion. Fig 4.11, 4.12, 4.13 and 4.14 shows the variation in the surface free energy and its components in 10 mS/cm at $22 \pm 4^\circ\text{C}$, $44 \pm 2^\circ\text{C}$, $73 \pm 2^\circ\text{C}$ and $99 \pm 2^\circ\text{C}$ respectively.

From Fig 4.14 it can be observed that γ_{SH} steadily increased with increasing time of immersion until saturation is reached while γ_{SD} changed only slightly during the aging process. The steady change in λ_{SH} was because it reflects the polarity of the surface while λ_{SD} reflects non-polar components of the surface tension and is independent of the polar forces [15]. From Fig 4.12 to 4.14 it can be observed that as the surface was wetted gradually the polarity of the surface increased due to the enhanced effect of the hydrogen

bonding forces and γ_{SH} also increased.

The surface free energy increased with increasing temperature and time of immersion. Table 4A, shows the effect of temperature on the surface free energy and its components after 4000 h of aging in 10mS/cm saline solution at $0\pm1.5^{\circ}\text{C}$, $22\pm4^{\circ}\text{C}$, $44\pm2^{\circ}\text{C}$, $73\pm2^{\circ}\text{C}$ and $99\pm2^{\circ}\text{C}$ respectively. It increased from 22.6×10^{-3} for the virgin sample to 44.3×10^{-3} for the 10 mS/cm saline solution at $99\pm2^{\circ}\text{C}$.

Fig 4.15, 4.16 and 4.17 shows the variation in the surface free energy with the time of immersion in 0.005, 1 and 100 mS/cm saline solution at $99\pm2^{\circ}\text{C}$. It is observed that the surface free energy increased with increasing time of immersion and increasing salinity. Table 4B shows the value of surface free energy and its components of a virgin sample and after 4000 hour of aging at $99\pm2^{\circ}\text{C}$ in 0.005, 1, 10 and 100mS/cm saline solution respectively.

Table 4A

Effect of temperature on the surface free energy of PTFE specimen in
10 mS/cm saline water solution.

Description of Specimen	$\gamma_{SD}, J/m^2$	$\gamma_{SH}, J/m^2$	$\gamma_S, J/m^2$
Virgin PTFE Specimen	20.538×10^{-3}	20.07×10^{-4}	22.545×10^{-3}
PTFE specimen in 10 mS/cm at $0 \pm 1.5^\circ C$, after 4000 hours	21.596×10^{-3}	7.644×10^{-3}	29.24×10^{-3}
PTFE specimen in 10 mS/cm at $22 \pm 4^\circ C$, after 4000 hours	21.056×10^{-3}	10.025×10^{-3}	31.081×10^{-3}
PTFE specimen in 10 mS/cm at $44 \pm 2^\circ C$, after 4000 hours	21.883×10^{-3}	13.029×10^{-3}	34.911×10^{-3}
PTFE specimen in 10 mS/cm at $73 \pm 2^\circ C$, after 4000 hours	21.883×10^{-3}	13.029×10^{-3}	34.911×10^{-3}
PTFE specimen in 10mS/cm at $99 \pm 2^\circ C$, after 4000 hours	23.836×10^{-3}	20.441×10^{-3}	44.277×10^{-3}

Table 4B

**Effect of salinity on the surface free energy of PTFE specimen at 99±2°C in
different saline solution of water.**

Description of Specimen	$\gamma_{SD}, J/m^2$	$\gamma_{SH}, J/m^2$	$\gamma_S, J/m^2$
PTFE Specimen, in air at 99±2°C	22.03×10^{-3}	36.25×10^{-4}	25.656×10^{-3}
PTFE specimen in 0.005 mS/cm at 99±2°C, after 4000 hours	21.212×10^{-3}	12.317×10^{-3}	33.529×10^{-3}
PTFE specimen in 1 mS/cm at 99±2°C, after 4000 hours	21.955×10^{-3}	12.754×10^{-3}	34.708×10^{-3}
PTFE specimen in 10 mS/cm at 99±2°C, after 4000 hours	23.836×10^{-3}	20.441×10^{-3}	44.277×10^{-3}
PTFE specimen in 100 mS/cm at 99±2°C, after 4000 hours	31.611×10^{-3}	30.736×10^{-3}	62.346×10^{-3}

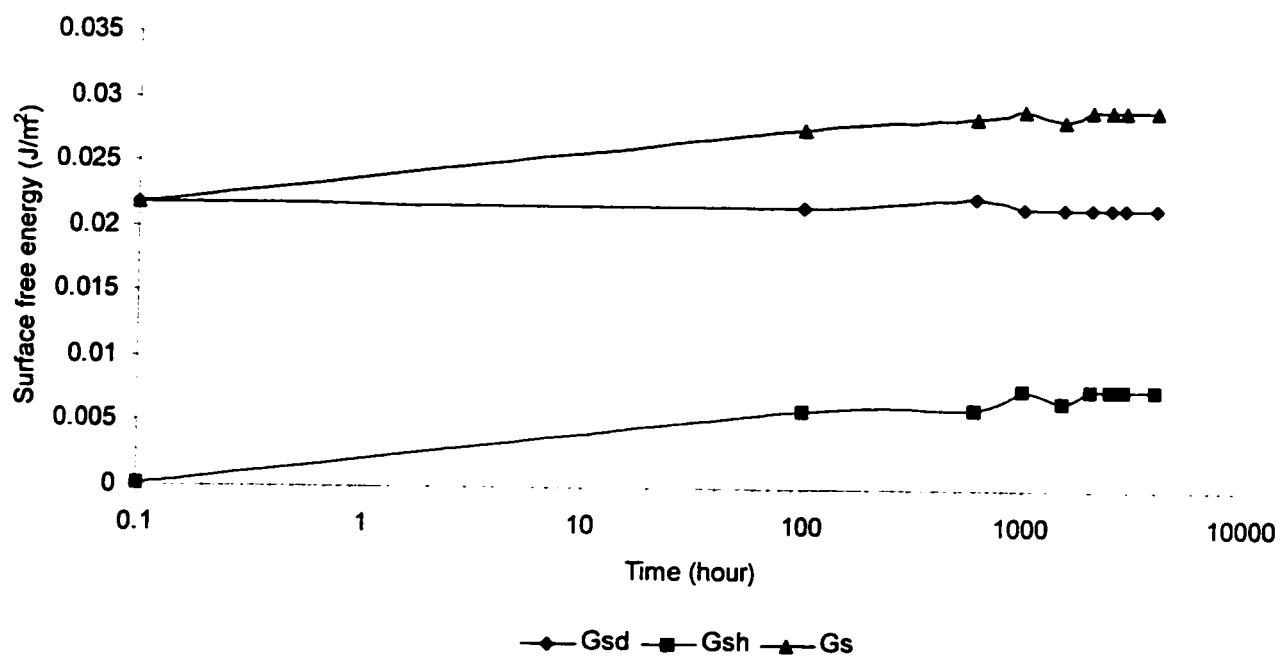


Fig 4.10. Time variation of surface free energy (and its components) of PTFE after immersion in 10 mS /cm saline solution of water at $0\pm1.5^{\circ}\text{C}$.

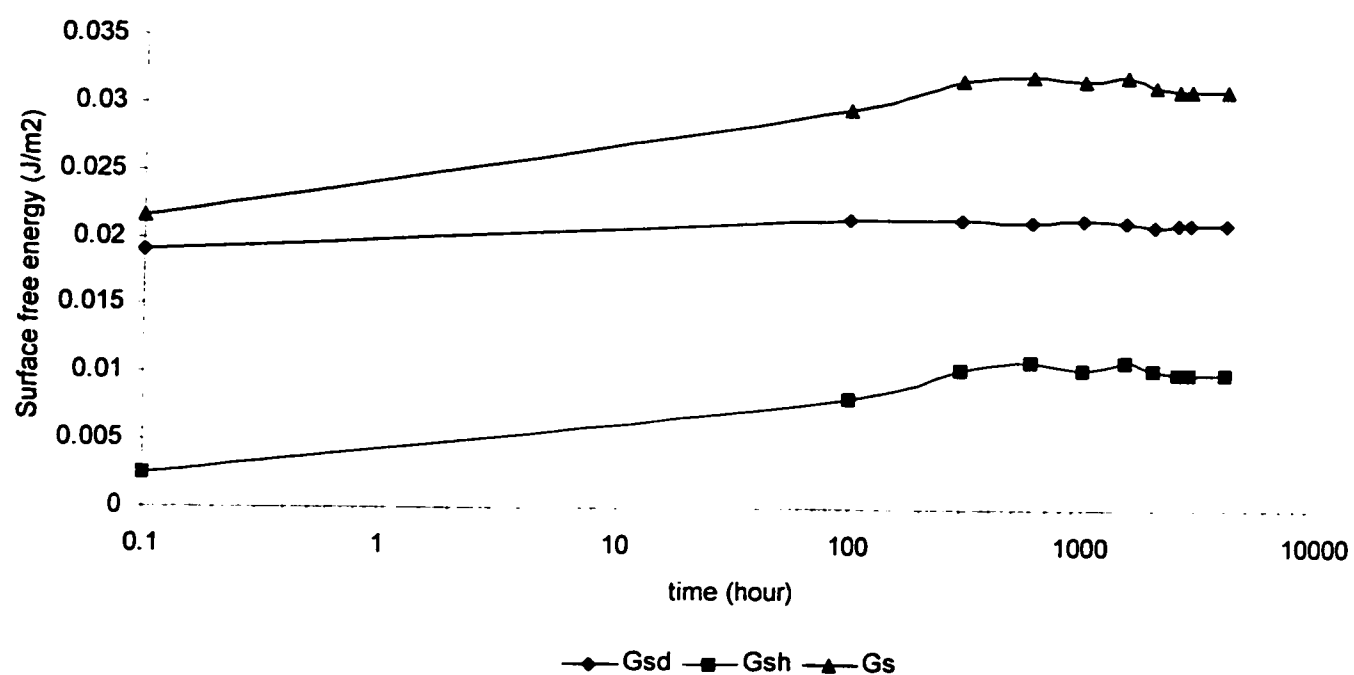


Fig. 4.11. Time variation of surface free energy (and its components) of PTFE after immersion in 10 mS /cm saline solution of water at 22 ± 4 °C.

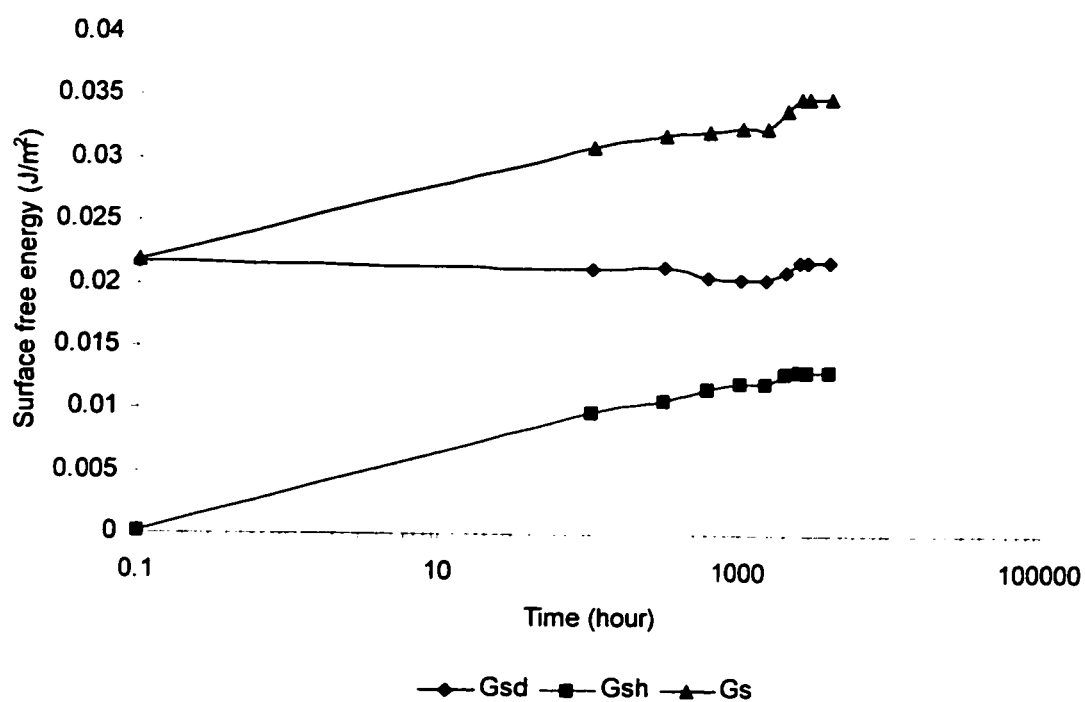


Fig. 4.12. Time variation of surface free energy (and its components) of PTFE after immersion in 10 mS /cm saline solution of water at 42 ± 2 °C.

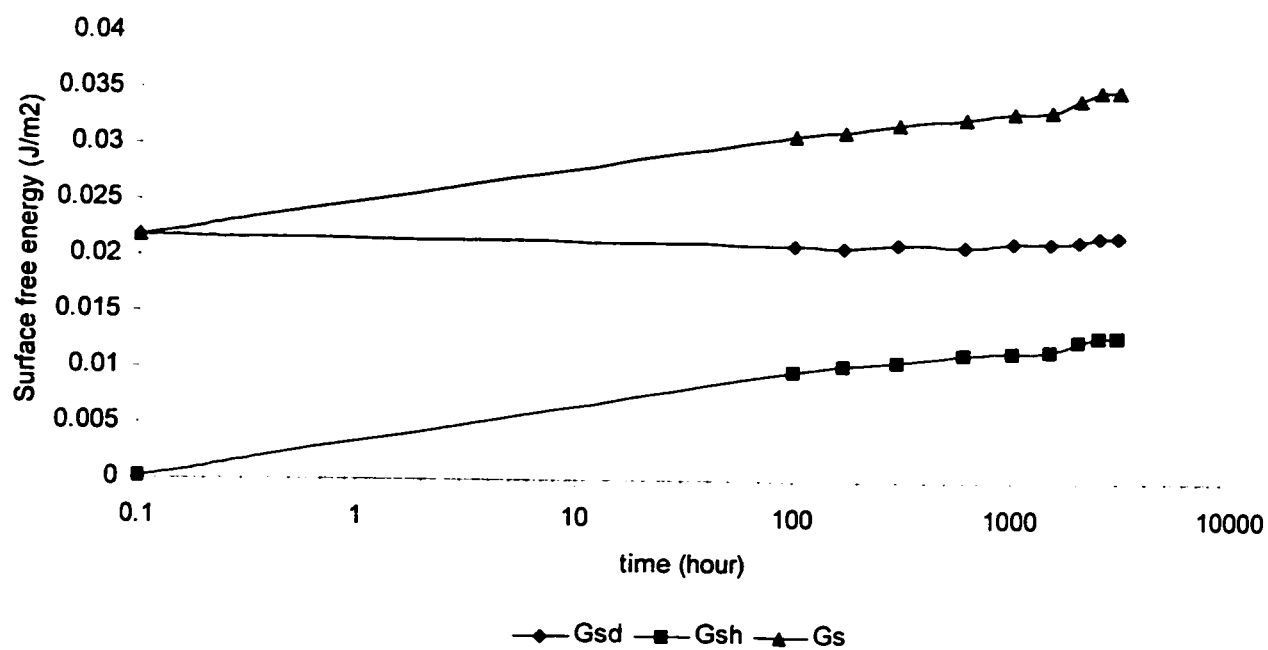


Fig. 4.13. Time variation of surface free energy (and its components) of PTFE after immersion in 10 mS /cm saline solution of water at 73 ± 2 °C.

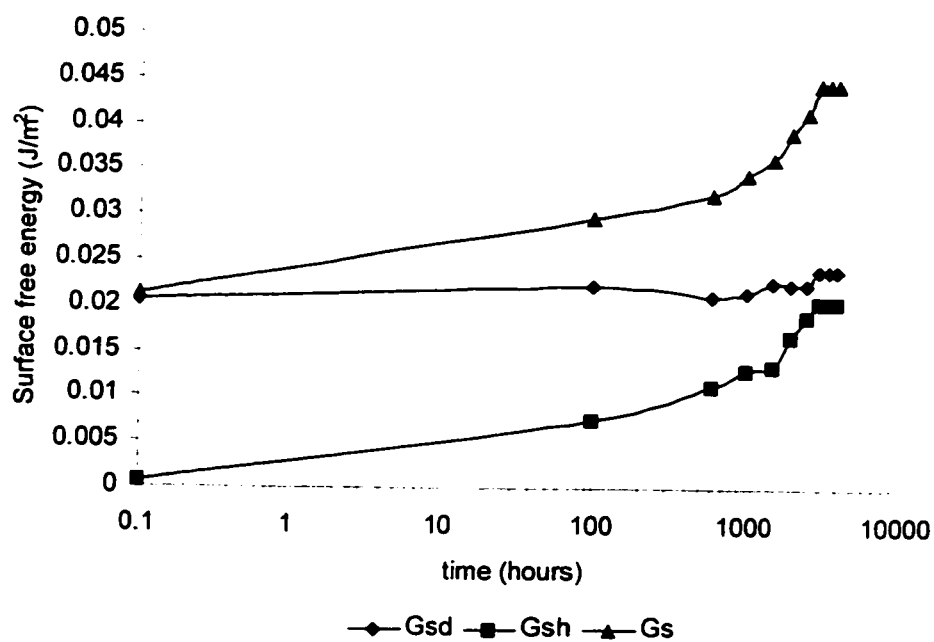


Fig. 4.14. Time variation of surface free energy (and its components) of PTFE after immersion in 10 mS /cm saline solution of water at 99 ± 2 °C.

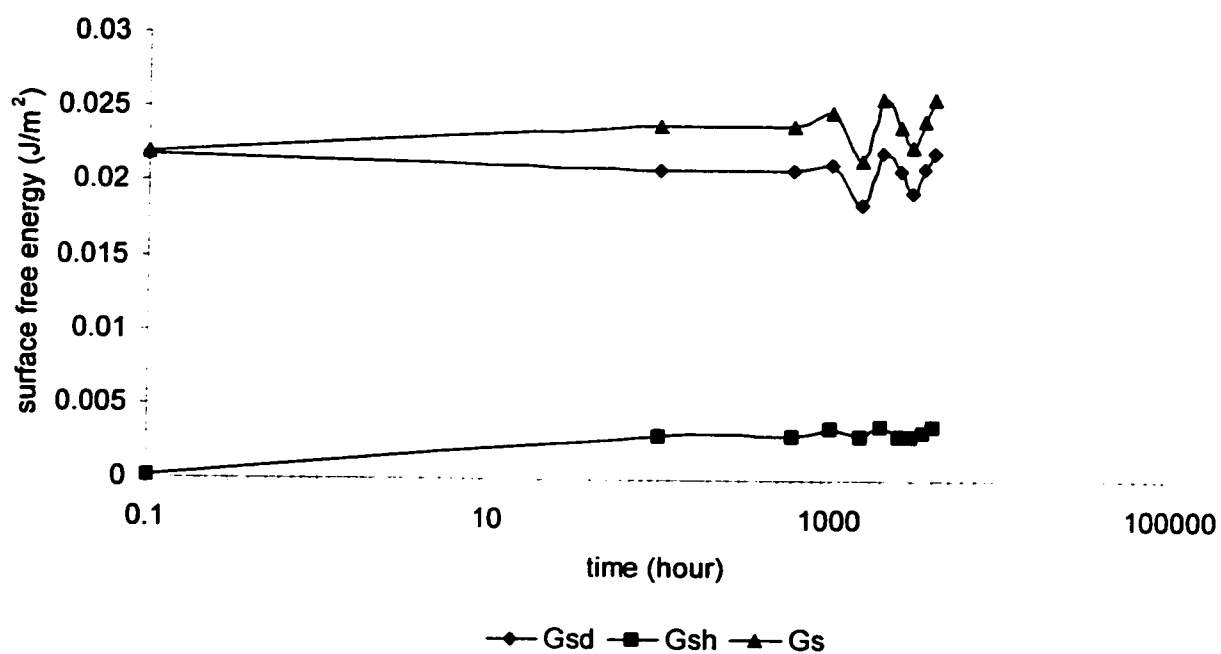


Fig. 4.15. Time variation of surface free energy (and its components) of PTFE in air at 22 ± 4 °C.

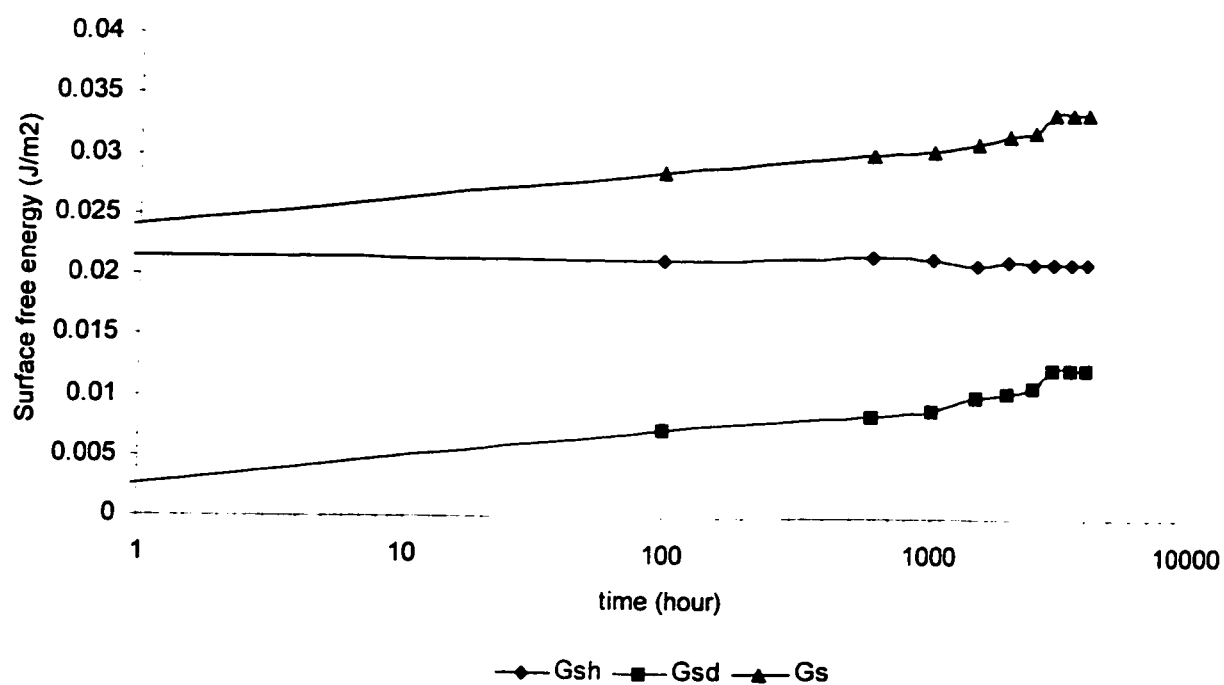


Fig.4.16. Time variation of surface free energy (and its components) of PTFE after immersion in 0.005mS /cm saline solution of water at 99±2 °C.

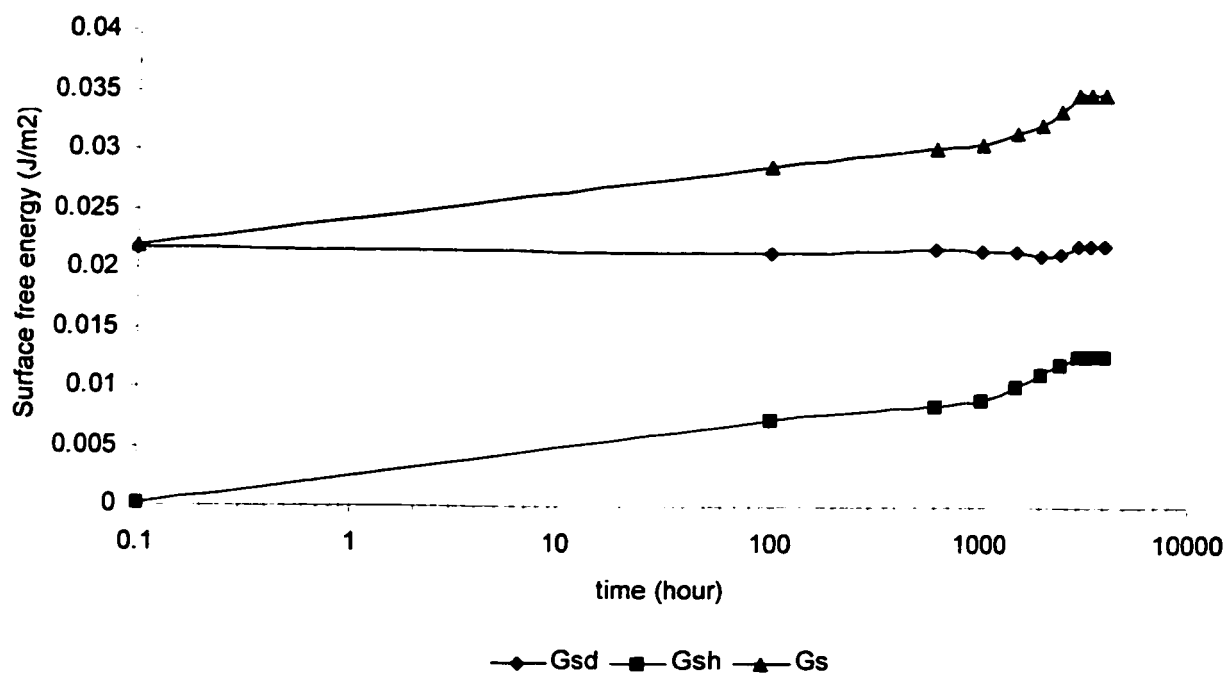


Fig. 4.17 Time variation of surface free energy (and its components) of PTFE after immersion in 1mS /cm saline solution of water at 99±2 °C.

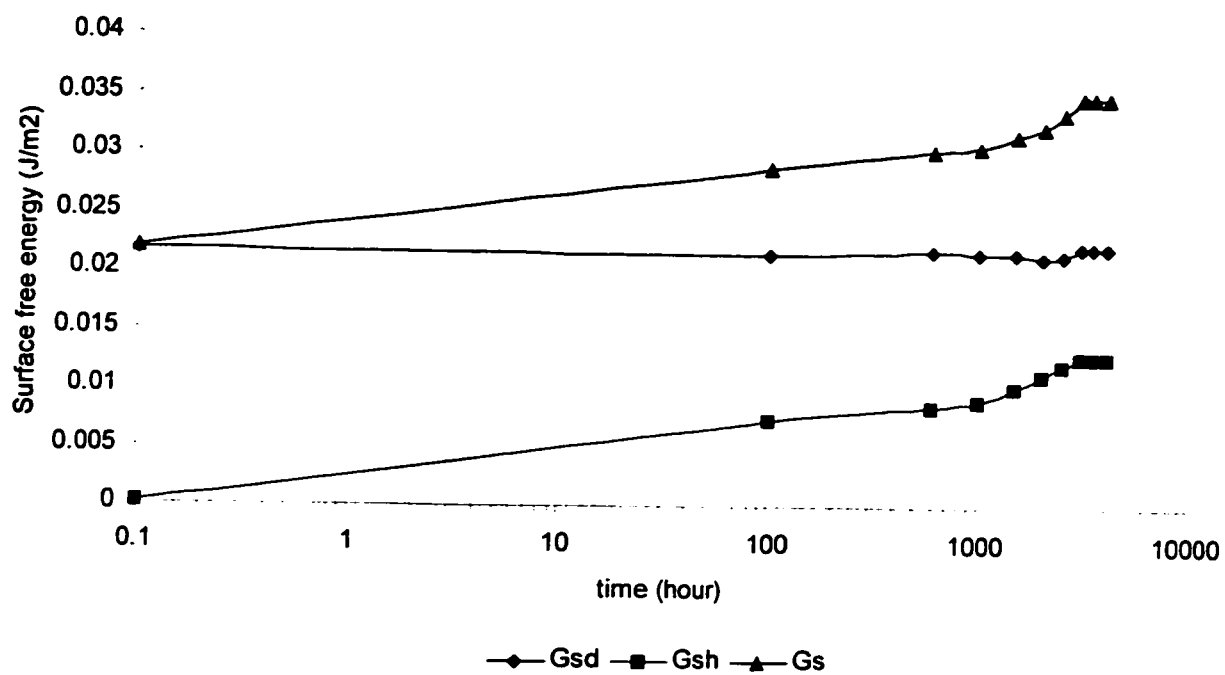


Fig. 4.18 Time variation of surface free energy (and its components) of PTFE after immersion in 100mS /cm saline solution of water at $99\pm 2^{\circ}\text{C}$.

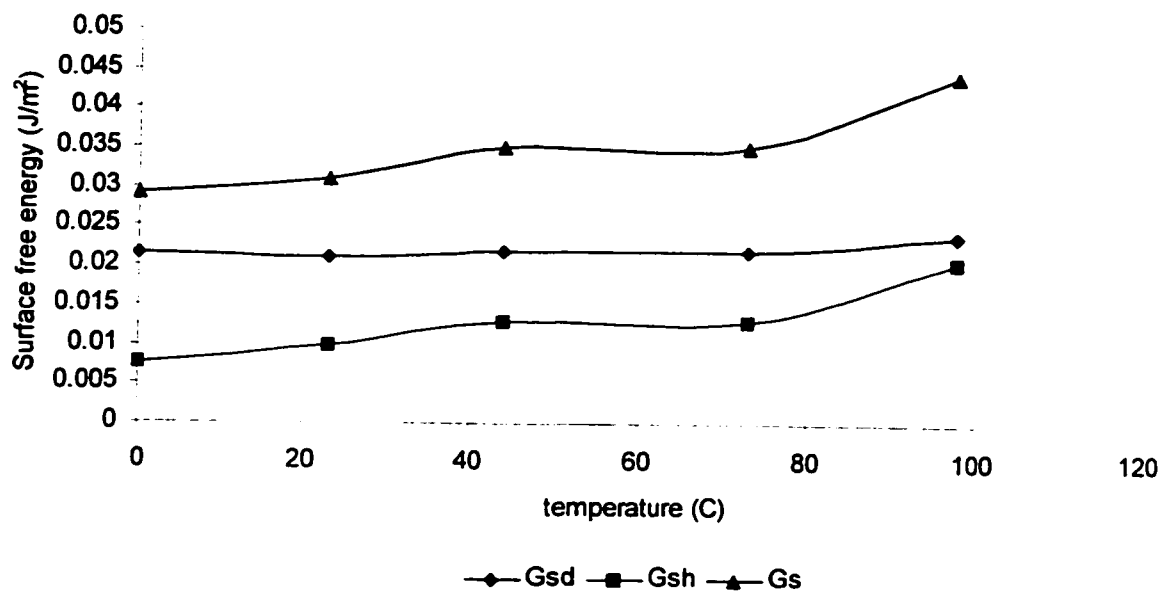


Fig. 4.19 Effect of temperature on surface free energy (and its components) of PTFE after 4000 h immersion in 10mS /cm saline solution of water.

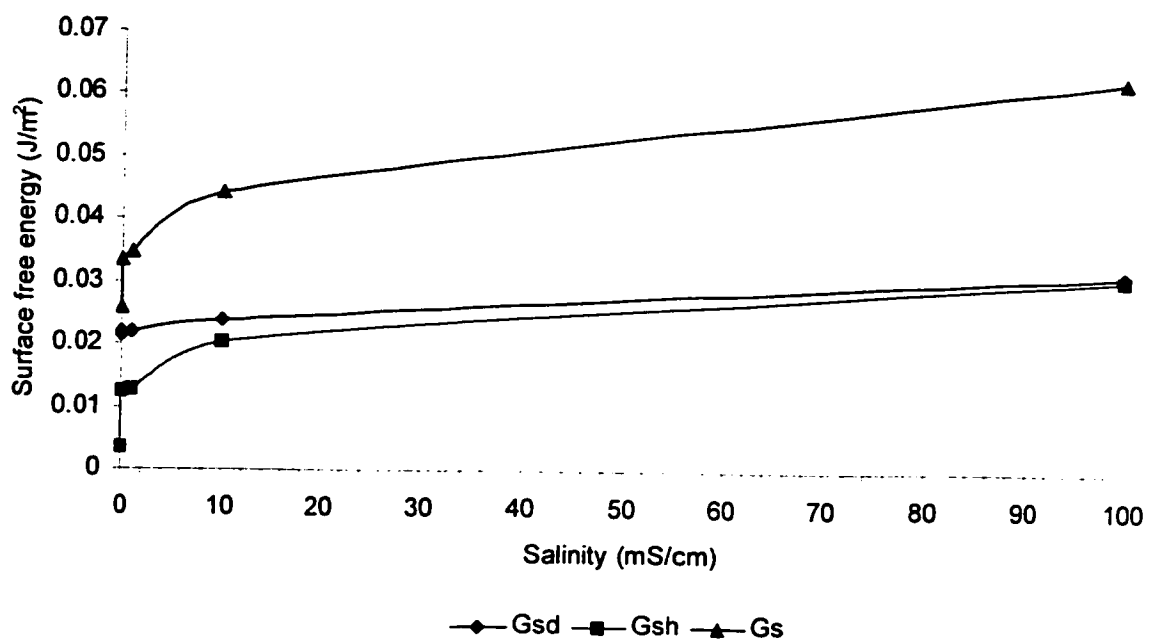


Fig. 4.20. Effect of salinity on the surface free energy (and its components) of PTFE after 4000 h immersion in different Saline solution of water at 99 ± 2 °C.

4.3.7. Water Uptake by PTFE During Immersion

Immersion of PTFE specimens in saline solutions, in the temperature range of 0 - 98°C resulted in an increase in the weight by 0.21%.

4.3.8 Calculation of Diffusion Coefficients

Fig. 4.21 shows the percentage change in the weight $M(t)$ of PTFE as function of time for different conductivity of saline solution at $0 \pm 1.5^\circ$. The maximum percentage increase in the weight was observed at 100 mS/cm and was 0.09% (Fig. 4.21). At the lower conductivity the increase in weight was less as compared to the value at 100 mS/cm. Fig 4.23, 4.25, 4.27 and 4.29 show the percentage change in the weight $M(t)$, at different temperature from 22° to 99° . It is observed that the change in weight was higher for the higher salinity and temperatures. The total percentage increase in the weight at $0 \pm 1.5^\circ\text{C}$ at 100 mS/cm was 0.0.9 % (Fig. 4.21) and increased to 0.21% at $99 \pm 2^\circ\text{C}$ (Fig 4.29).

Applying equation (4.8) the diffusion coefficient “D” can be determined at different salinities and temperature when $\Delta M(t) / \Delta M_\infty = 0.5$

Fig 4.22 shows the percentage variation of $M(t) / M_\infty$ as a function of $[t^{0.5}]$ for different conductivities of saline solution at 0°C . For the 10mS/cm solution $M(t) / M_\infty = 0.5$ occurs at $t^{0.5} = 63.2\text{h}^{0.5}$ and the thickness of PTFE specimen at this time was 0.86 mm. Using equation 4.3, the diffusion coefficients was calculated to be $D = 2.5\text{E-}15 \text{ m}^2/\text{s}$ at 0°C for 10mS/cm saline solution. Similarly the diffusion coefficients of 10mS/cm at $22 \pm 4^\circ\text{C}$,

44±2°C, 73±2°C and 99±2°C were calculated from Fig. 4.24, 4.26, 4.28 and 4.30 to be 2.60E-15, 3.97E-15, 4.53E-15 and 5.1E-15 m²/s respectively.

Table “4C” shows the diffusion coefficient at different temperatures in 10mS/cm saline solution.

The diffusion coefficients for the salinity of 0.005, 1.00, 10.00, 100.00 mS/cm were calculated at 99° from Fig 4.30 and found to be 2.9x 10⁻¹⁵ m²/s 3.1x 10⁻¹⁵ m²/s, 5.1x 10⁻¹⁵ m²/s and 7.5x 10⁻¹⁵ m²/s respectively. The diffusion coefficient increased with increasing salinity and the same observations are also reported by [7].

Table 4C

Diffusion coefficient of 10 mS/cm saline water for different temperature.

Temperature of the solution, C	$t_{0.5}$, hour	$L_{0.5}$, mm	D , m^2/s
0 °C	63.2	0.86	2.50E-15
23 °C	63.3	0.86	2.60E-15
44 °C	52.2	0.88	3.9E-15
73 °C	51.2	0.89	4.53E-15
98 °C	44.7	0.87	5.1E-15

Table 4D

Diffusion coefficient for different saline solution at 99 °C.

Salinity of the solution, C	$t_{0.5}$, hour	$L_{0.5}$, mm	D , m^2/s
0.005 mS/cm	63.2	0.92	2.88E-15
1 mS/cm	59.1	0.89	3.08E-15
10 mS/cm	44.7	0.87	5.1E-15
100 mS/cm	36.5	0.86	7.5E-15

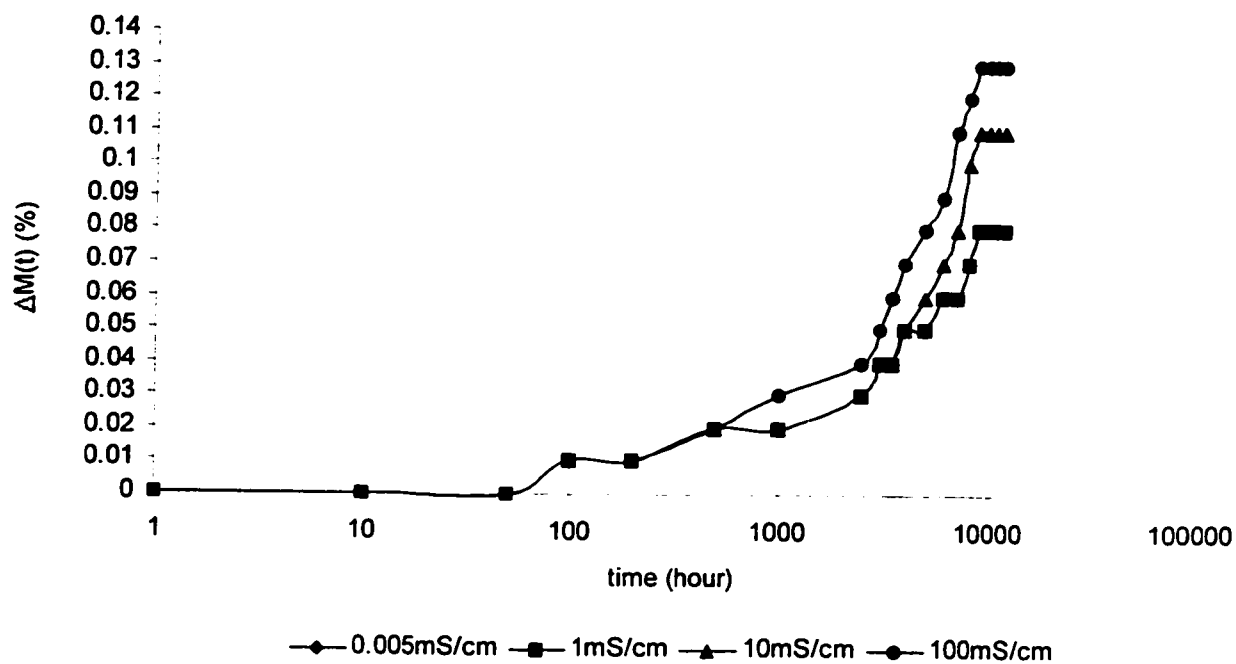


Fig 4.21: Variation of percentage increase in the weight of PTFE as a function of time for different conductivity of saline solution at $0 \pm 1.5^\circ \text{C}$.

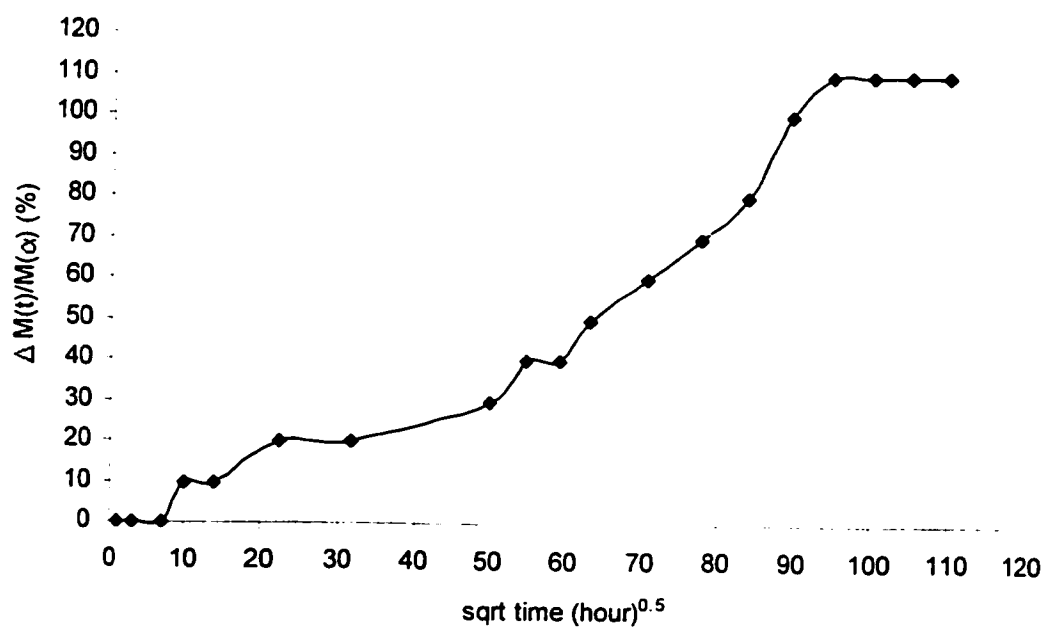


Fig 4.22: Variation of $M(t) / M_{\infty}$ (%) of PTFE as a function of square root of time in hours for different conductivity of saline solution at 0 ± 1.5 °C.

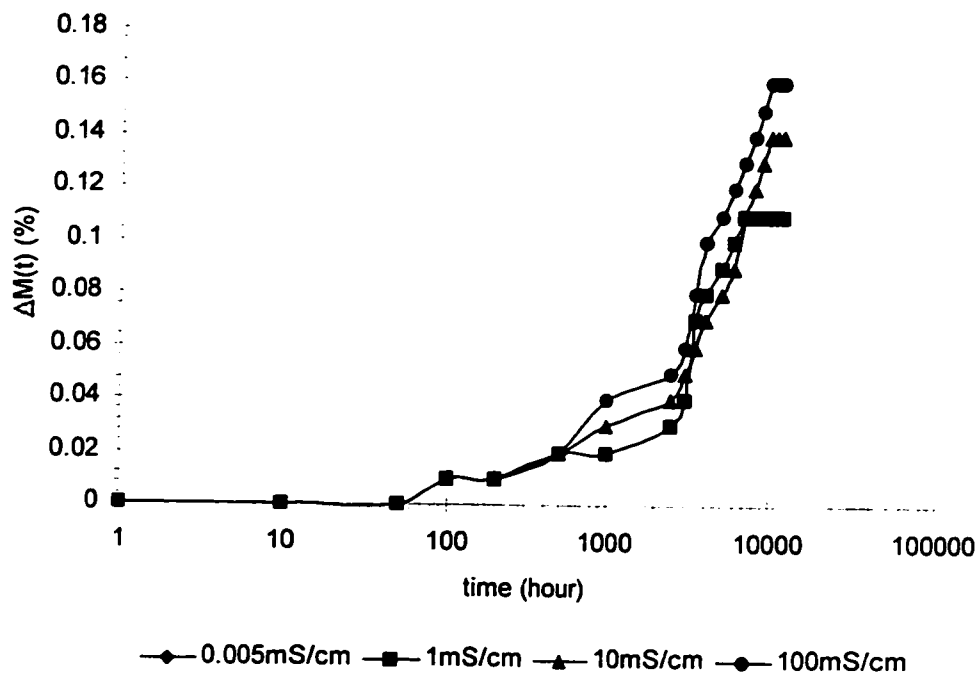


Fig 4.23: Variation of percentage increase in weight of PTFE as a function of time or different conductivity of saline solution at 22 ± 4 °C.

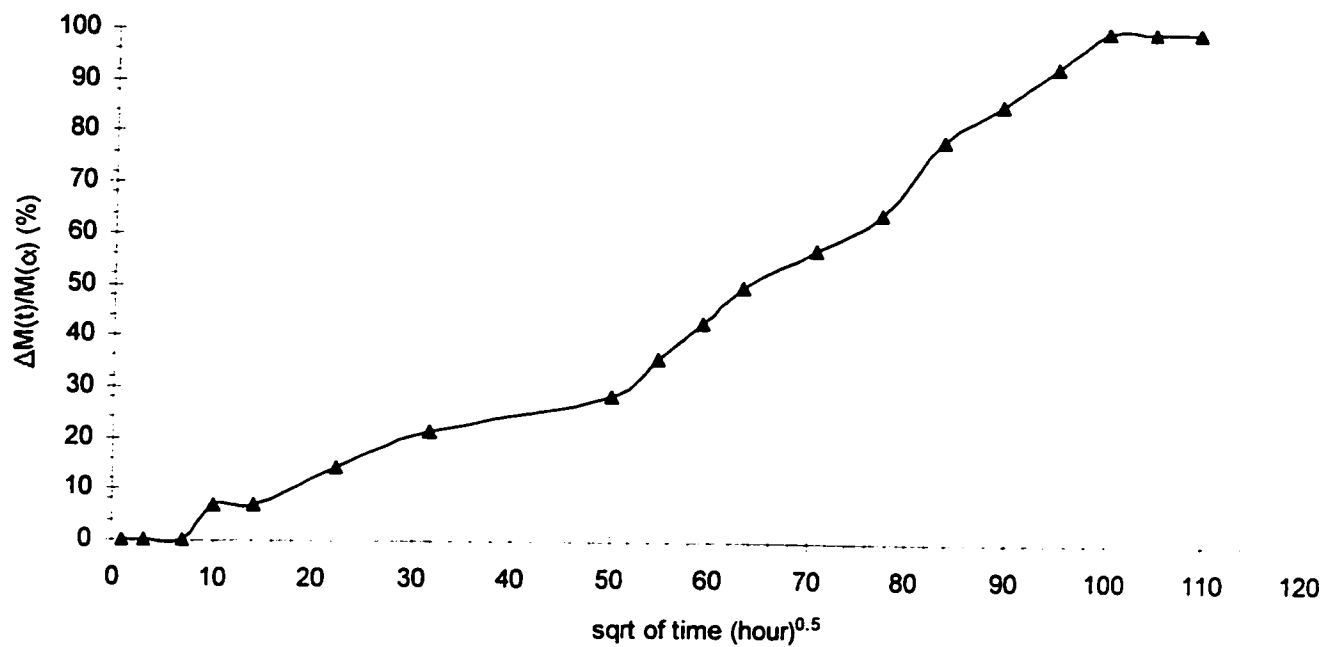


Fig 4.24: Variation of $M(t) / M_{\infty}$ (%) of PTFE as a function of square root of time in hours for different conductivity of saline solution at 22 ± 4 °C.

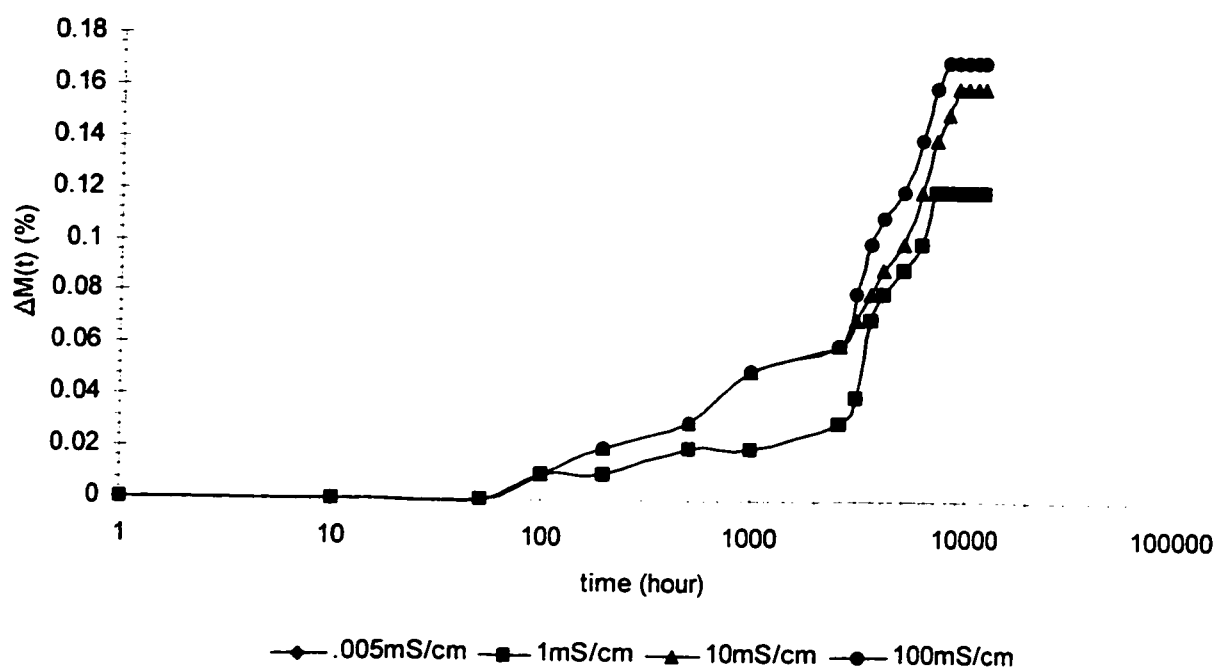


Fig 4.25: Variation of percentage increase in weight of PTFE as a function of time for different conductivity of saline solution at 44 ± 2 °C.

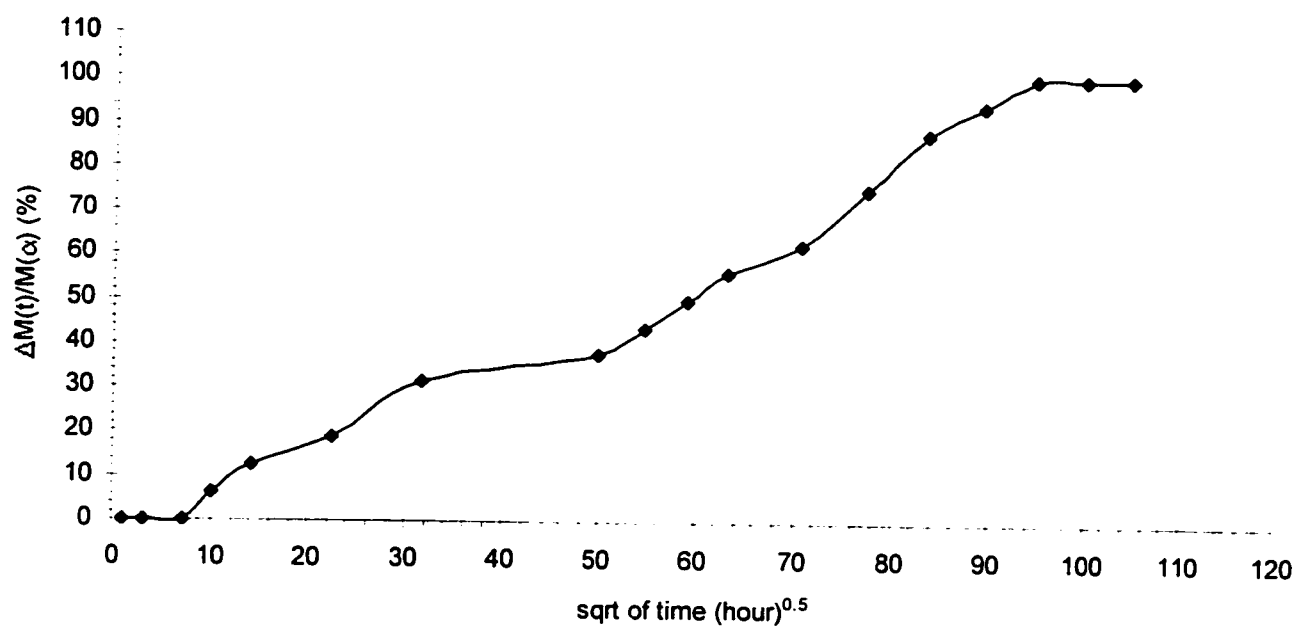


Fig. 4.26. Variation of $M(t) / M_{\infty} (\%)$ of PTFE as a function of square root of time in hours for different conductivity of saline solution at $44 \pm 2 ^\circ\text{C}$.

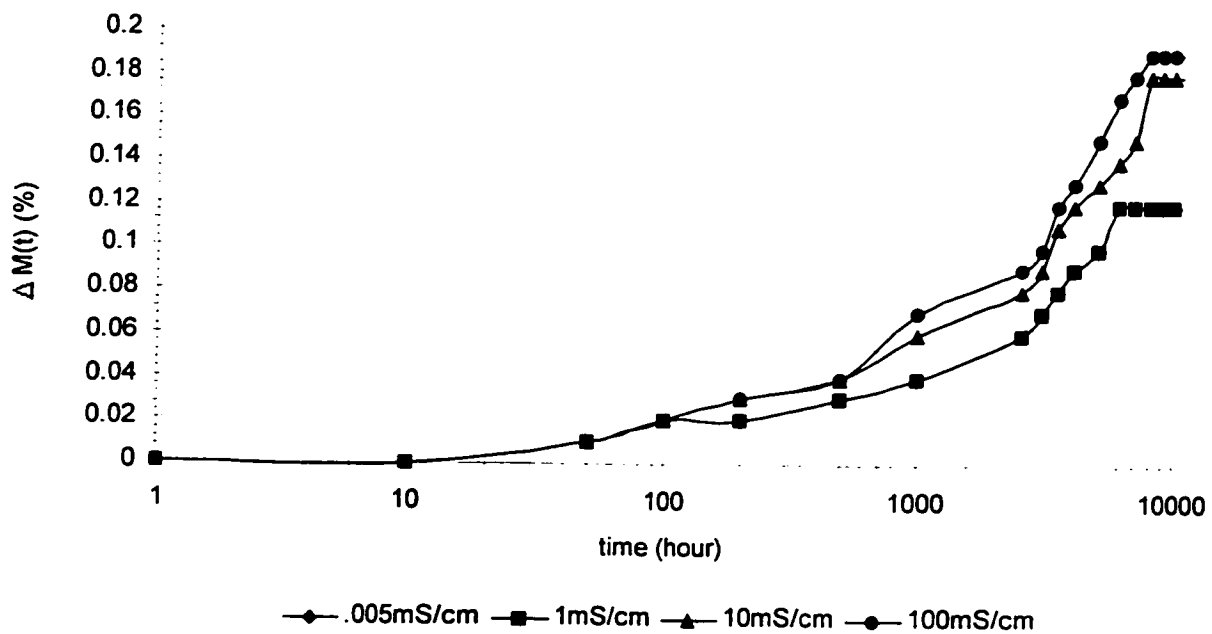


Fig. 4.27. Variation of percentage increase in weight of PTFE as a function of time for different conductivity of saline solution at 73 ± 2 °C.

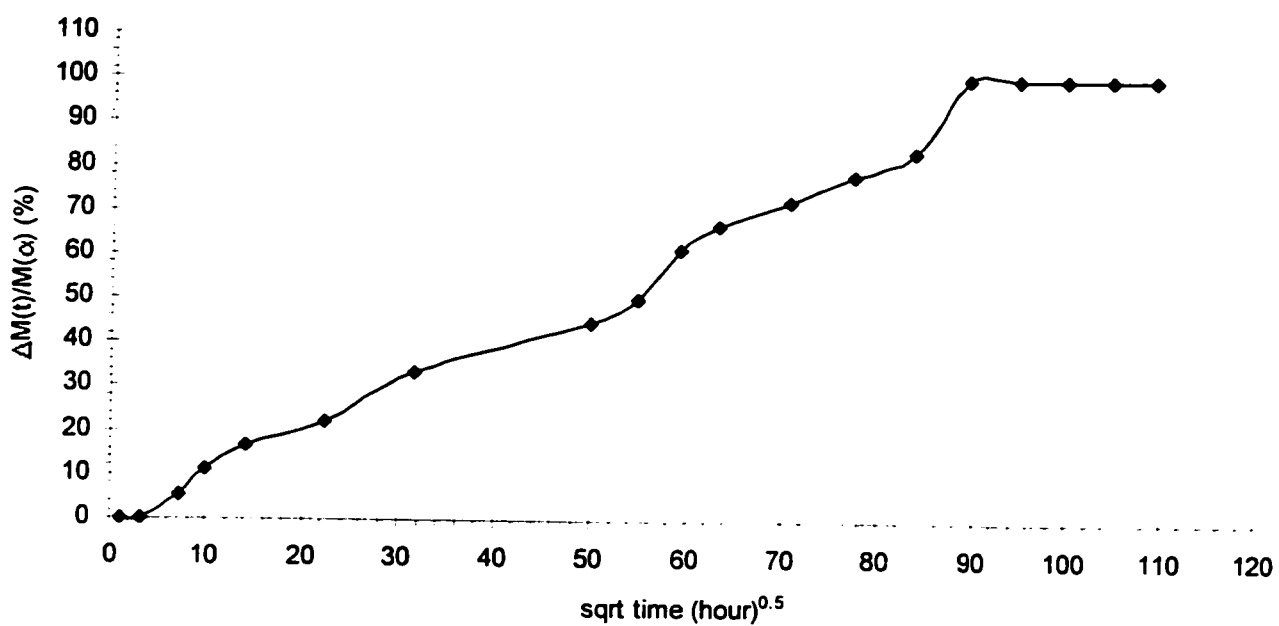


Fig. 4.28. Variation of $M(t) / M_{\infty} (\%)$ of PTFE as a function of square root of time in hours for different conductivity of saline solution at $73 \pm 2 ^\circ\text{C}$.

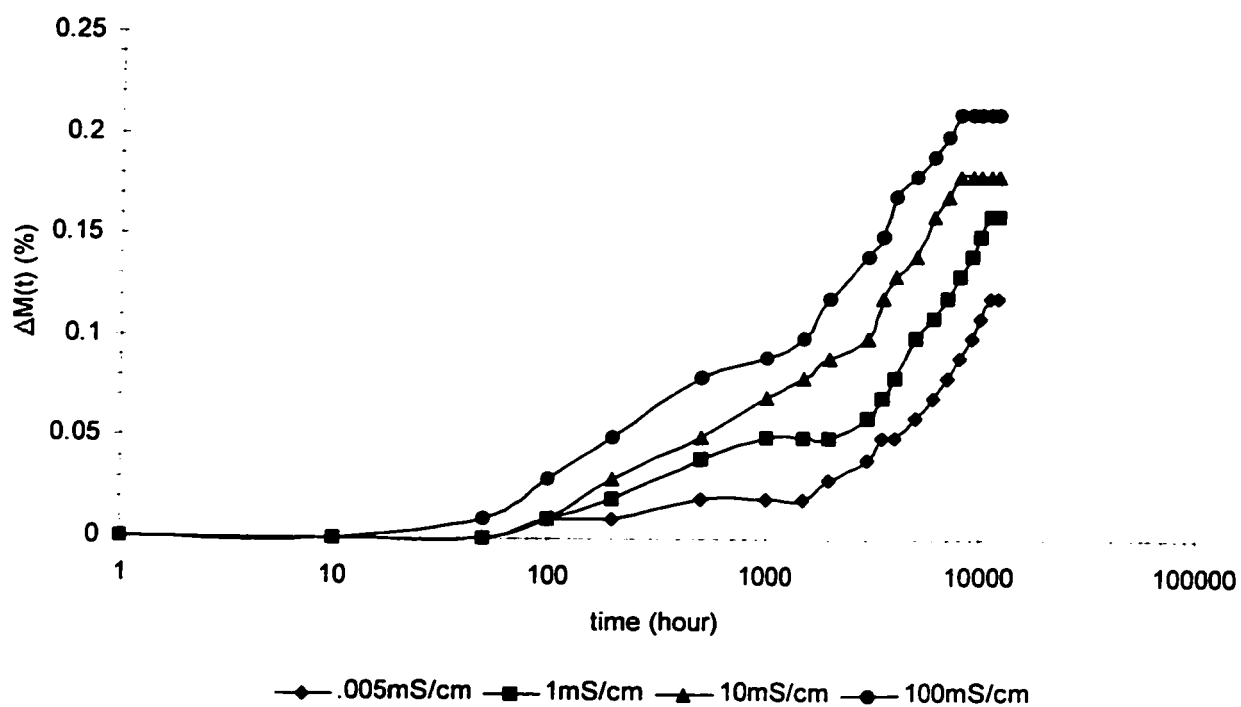


Fig 4.29. Variation of percentage increase in weight of PTFE as a function of time or different conductivity of saline solution at 99 ± 2 °C.

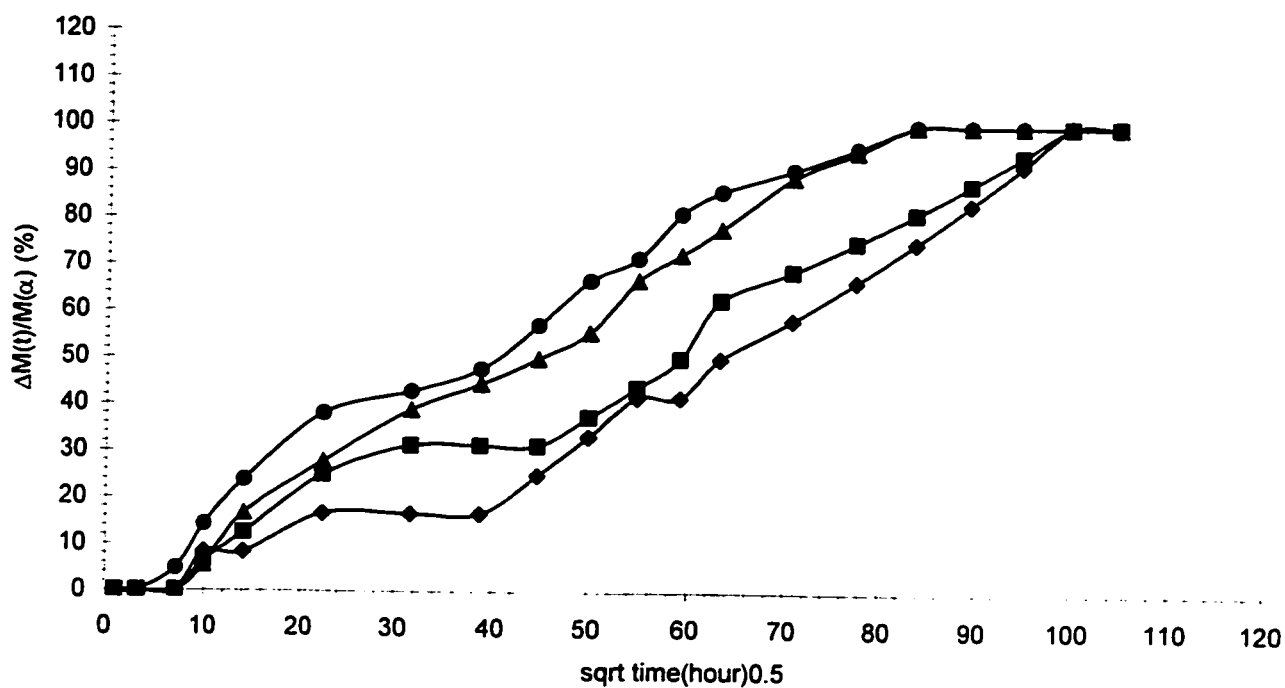


Fig 4.30 Variation of $M(t) / M_{\infty}$ (%) of PTFE as a function of square root of time in hours for different conductivity of saline solution at 99 ± 2 °C

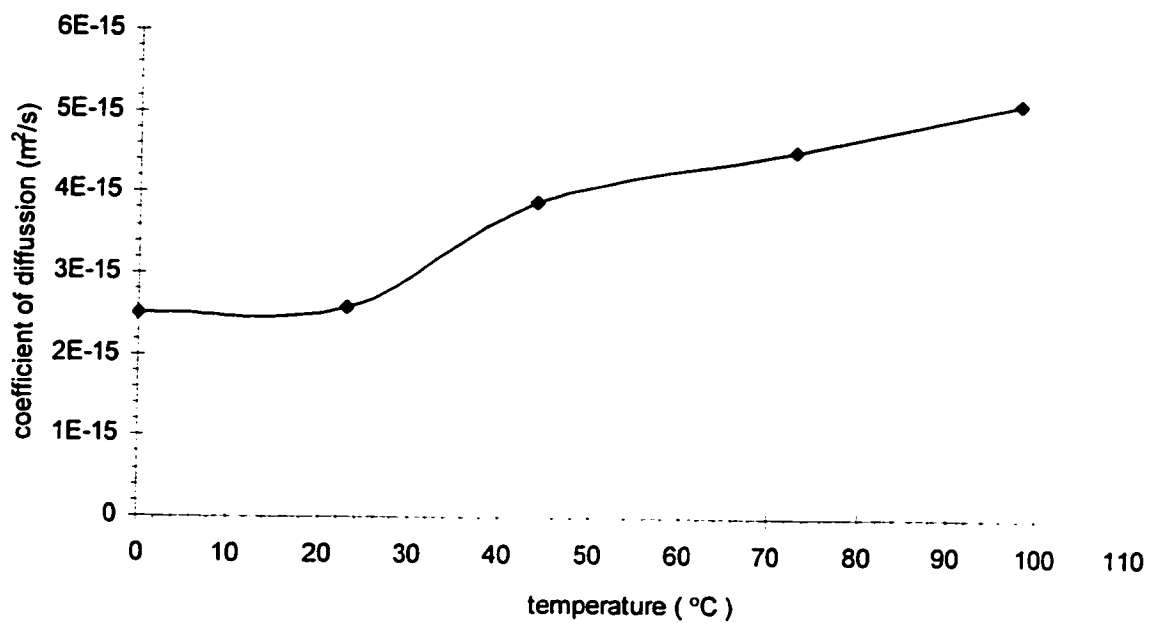


Fig 4.31 Variation of Diffusion coefficients with temperature in 10mS/cm saline solution.

4.3.9 Calculation of Activation Energy

The diffusion coefficient of water in PTFE increased with increase in the temperature and conductivity of the saline solution. The force that causes diffusion increases with increase in temperature of the system [7]. The activation energy can be calculated by Arrhenius equation as described below

$$D = D_0 \exp[-E_a / RT] \quad \text{----- (4.8)}$$

$$\text{or} \quad \ln D = \ln D_0 - E_a / RT \quad \text{----- (4.9)}$$

where $D \Rightarrow$ is the diffusion coefficient in m^2/s

D_0 is Constant

E_a is Activation Energy

R is Universal gas constant = $8314.3 \text{ J / kmole}^\circ\text{K}$ [A55]

T is Absolute temperature in $^\circ\text{K}$.

Using equation (4.9) the activation energy has been calculated and plotted as a function of conductivity as shown in Fig. 4.33.

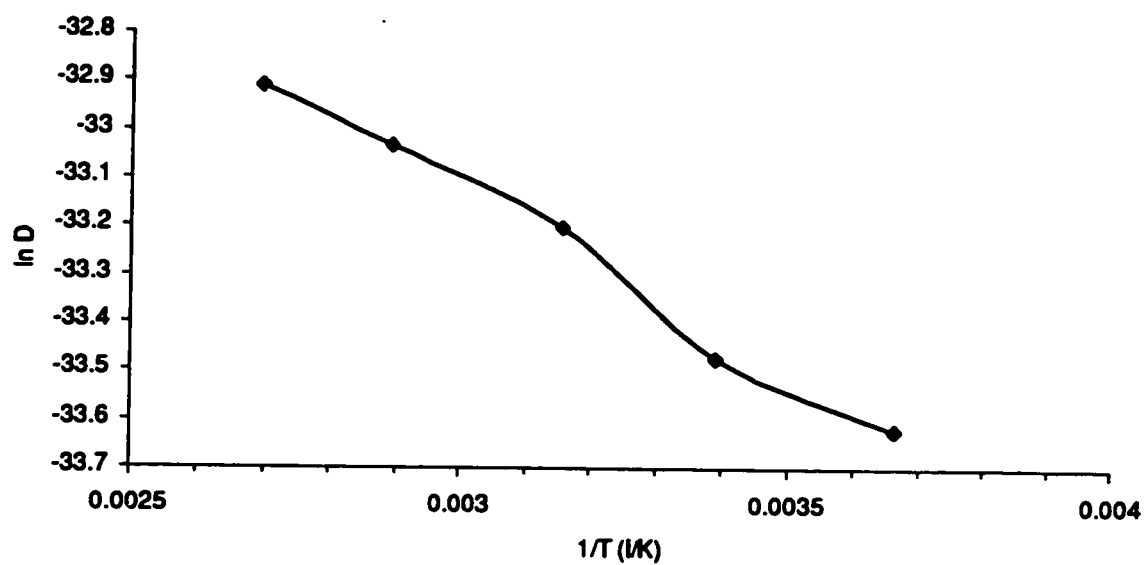


Fig. 4.32 Variation of $\ln D$ of PTFE as a function of $1/T$ for different conductivities of saline solution

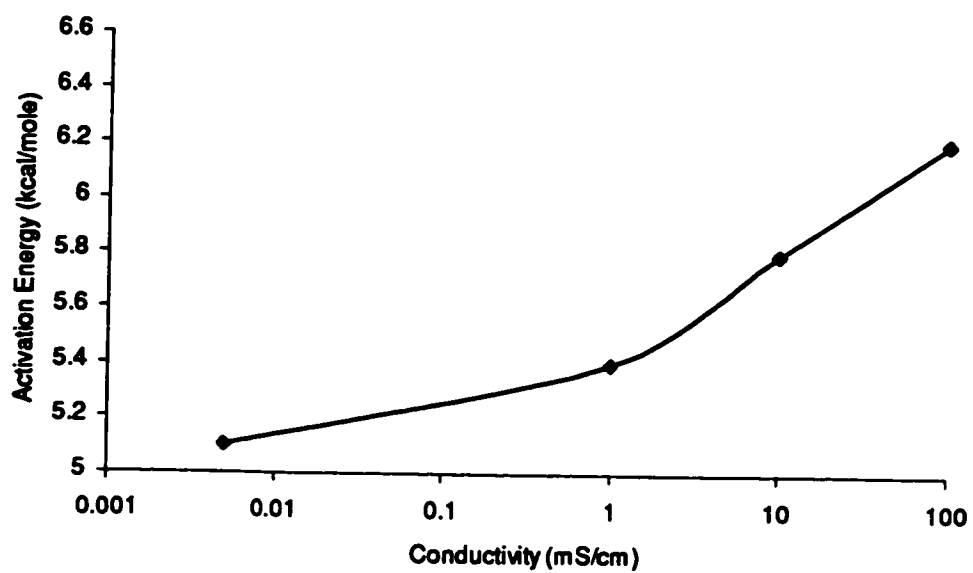


Fig.4.33 Variation of Activation energy of diffusion of saline solution in PTFE at different temperatures as a function of conductivity of saline solution.

4.3.10 Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) was applied to study the chemical changes in the polymer bulk and the surface due to the stress of heat and salinity over a long period of time.

In Infrared (IR) spectroscopy, the vibrational excitation is achieved by radiating the sample with a broadband source of radiation in the infrared region, which is generally 4000-200 cm^{-1} (2.5-50 μm). The wave number ν in cm^{-1} , is the number of waves per centimeter. The wave number is given by

$$\nu = 1/\lambda \text{ (cm}^{-1}\text{)} \text{ ----- (4.10)}$$

and is also equal to $\nu = \nu/c \text{ (cm sec}^{-1}\text{)} \text{ ----- (4.11)}$

In the IR region, the wavelength λ is given in micrometers (μm) or 10^{-6} m. When the sample is experiencing IR, its molecule is excited to a higher vibrational state by directly absorbing the infrared radiation. The transmittance at a given wavenumber can be calculated by following equation

$$T = I / I_0 \text{ ----- (4.12)}$$

where I is the intensity of the transmitted light (after sample absorption)

I_0 is the intensity of the transmitted light (before sample absorption)

Similarly, the absorbency at a given wavenumber can be obtained by using the Beer-Lambert equation as follows

$$A = \log (I / I_0) \text{ ----- (4.13)}$$

or $A = abc$,

where “a” is absorptivity, “b” is a cell thickness, and “c” is concentration.

FTIR is an improved method of infrared spectroscopy that reduces the signal to noise ratio of the instrument and provide added responses of intensity of chemical functional groups by accumulating the response of each group in the vicinity by using multiple scan method [40]. In this research work, FTIR has been used to study the aging effect on different PTFE specimens. FTIR provides information about the chemical changes in the polymer bulk and the surface due to different aging effects. Computerized FTIR set up 'Bruker Vector 22' which uses OPUS-NT software for analysis and data acquisition is used for FTIR. Twenty scans were carried in each case. PTFE was microtomed to the $15 \pm 10 \mu\text{m}$ to have maximum transmission.

4.3.11 FTIR Analysis of PTFE

Fig. 4.34 to 4.38 shows the FTIR spectrum of virgin and aged PTFE specimen in different saline solutions. It is clear from these spectra that there is no appreciable absorption of any chemical groups in case of 0.005 and 1 mS/cm salinity levels. The spectra of the aged specimens are not much different from that of virgin PTFE even after 3500 hours of immersion. However for higher temperatures and salinities, few peaks have started to develop in the different region. As described in [1], the strong bands at $1200 - 1350 \text{ cm}^{-1}$ are due to CF vibrations. The bands at 1600 and 1700 cm^{-1} are due to conjugated C=C bonds, at 2150 cm^{-1} are due to carbonyls, 2853 and 2925 cm^{-1} are due to CH_2 groups, at 2950 cm^{-1} are due to CH_3 groups, 3300 cm^{-1} are due to hydroxyl, and at 3400 cm^{-1} are due to NH groups.

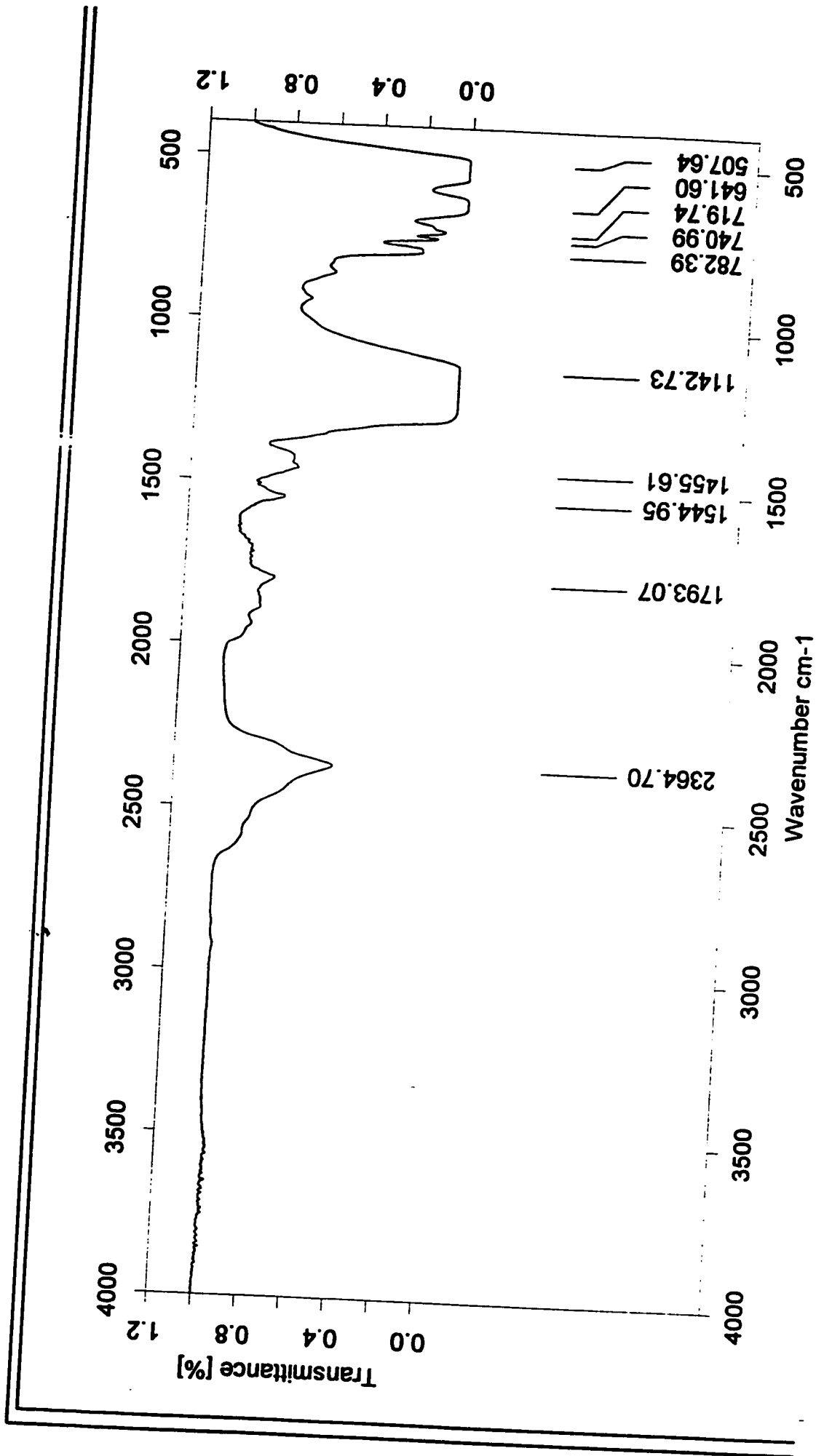
The CF_2 groups absorb between 1350 and 1200 cm^{-1} . The C-F absorption on these bonds is exceptionally very strong due to great electro-negativity of fluorine.

In Fig. 4.38, in the first harmonic region the wave number 1133.56 cm^{-1} shows the stretching vibrations of C-F band, the corresponding approximate position of the overtone will be 2365.08 cm^{-1} as observed in Fig. 4.38. Comparing Fig. 4.34 with Fig. 4.38 shows a broad change due to different aging environment. The intensity of the transmission reduces as the aging stress increases. This change in the intensity is the exact replica of the change in C-F band at wave number 1133.56 cm^{-1} . The reduction in the intensity of transmission can be associated as, either due to weakening of C-F bond or due to degradation of PTFE surface. However, it is more likely that both the factors contributed to the gradual reduction in the intensity of transmission.

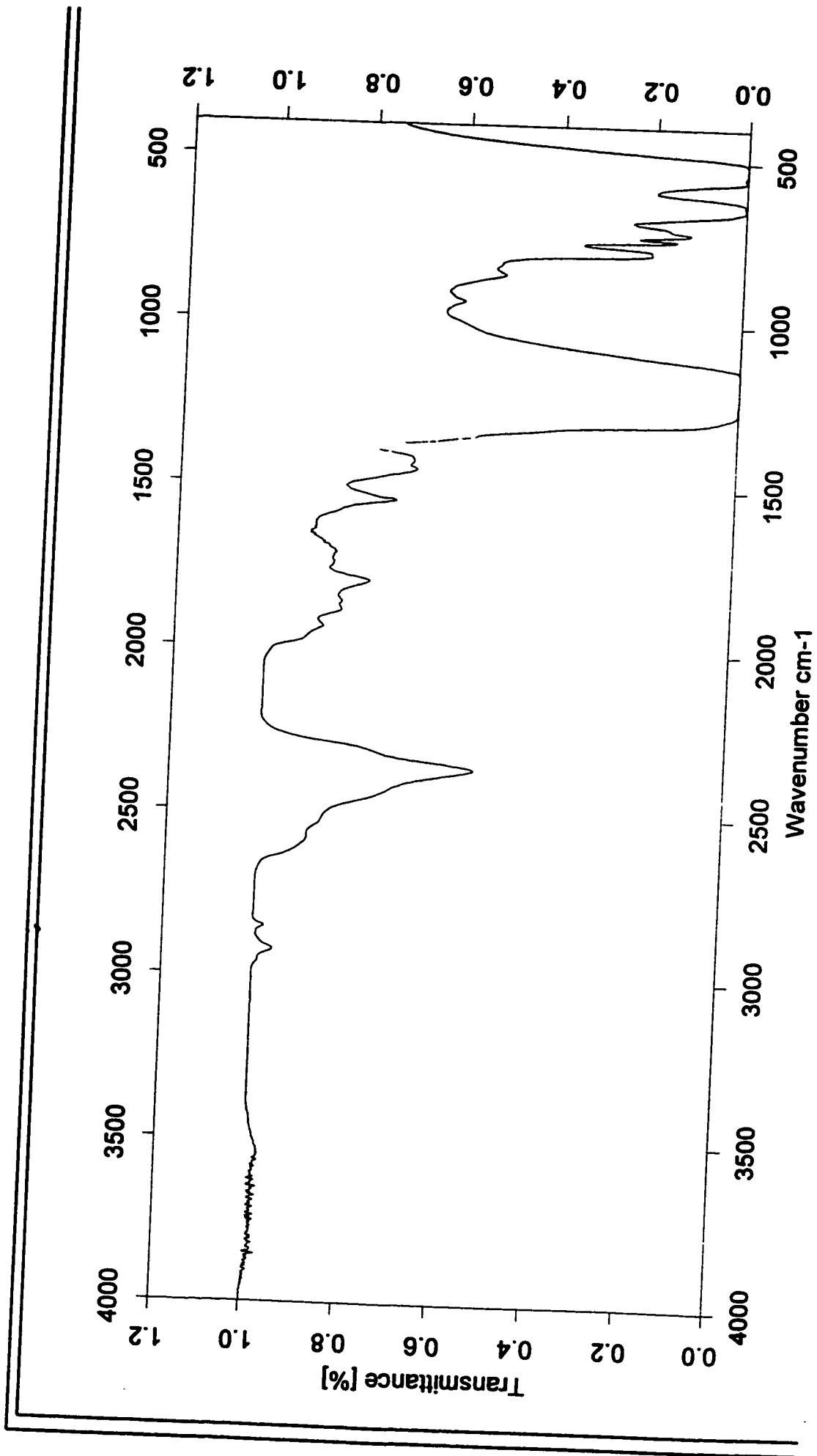
Development of a small peak was observed at 2853 and 2925 cm^{-1} and shows the presence of CH_2 groups. No significant difference in the FTIR spectra of virgin and aged PTFE specimens were observed for wave number 2150 cm^{-1} (carbonyls groups) 3300 cm^{-1} (hydroxyl groups). This shows that there is no indication of oxidation in the aged PTFE for all the salinity and temperature though the contact angles on these specimens showed a large reduction (Fig. 4.1 to Fig. 4.5).

It is concluded from the study of FTIR spectra (Fig. 4.34 to Fig. 4.38) that oxidation has probably been suppressed by the addition of an anti oxidant. The loss of hydrophobicity despite the lack of observable oxidation leads to the conclusion that the contact angle of an aged PTFE does not necessarily depends on the oxidation compounds on the surface or in the bulk of PTFE. The same was also observed in the study of XLPE by Khan [7].

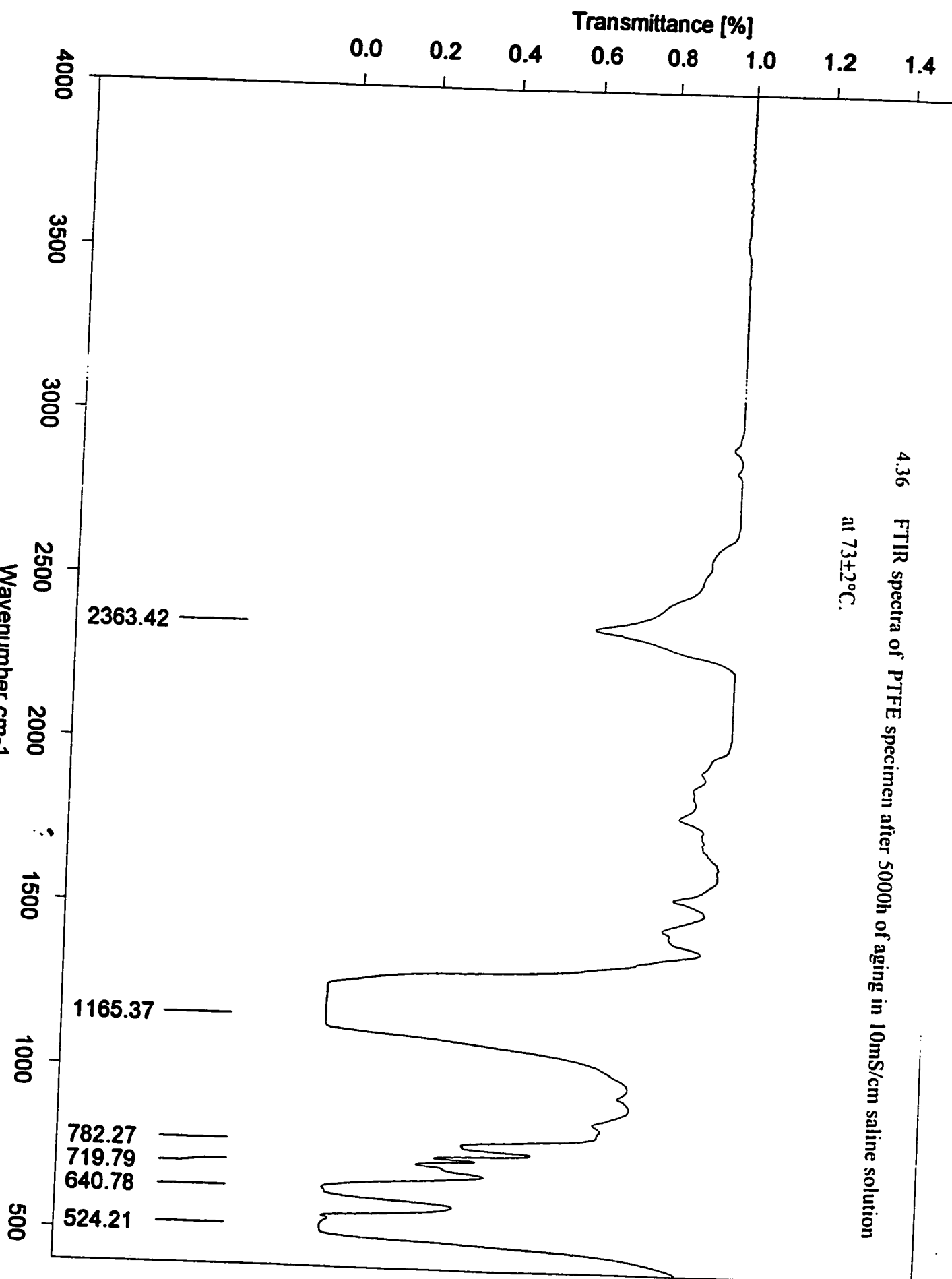
4.34 FTIR spectra of virgin PTFE specimens .



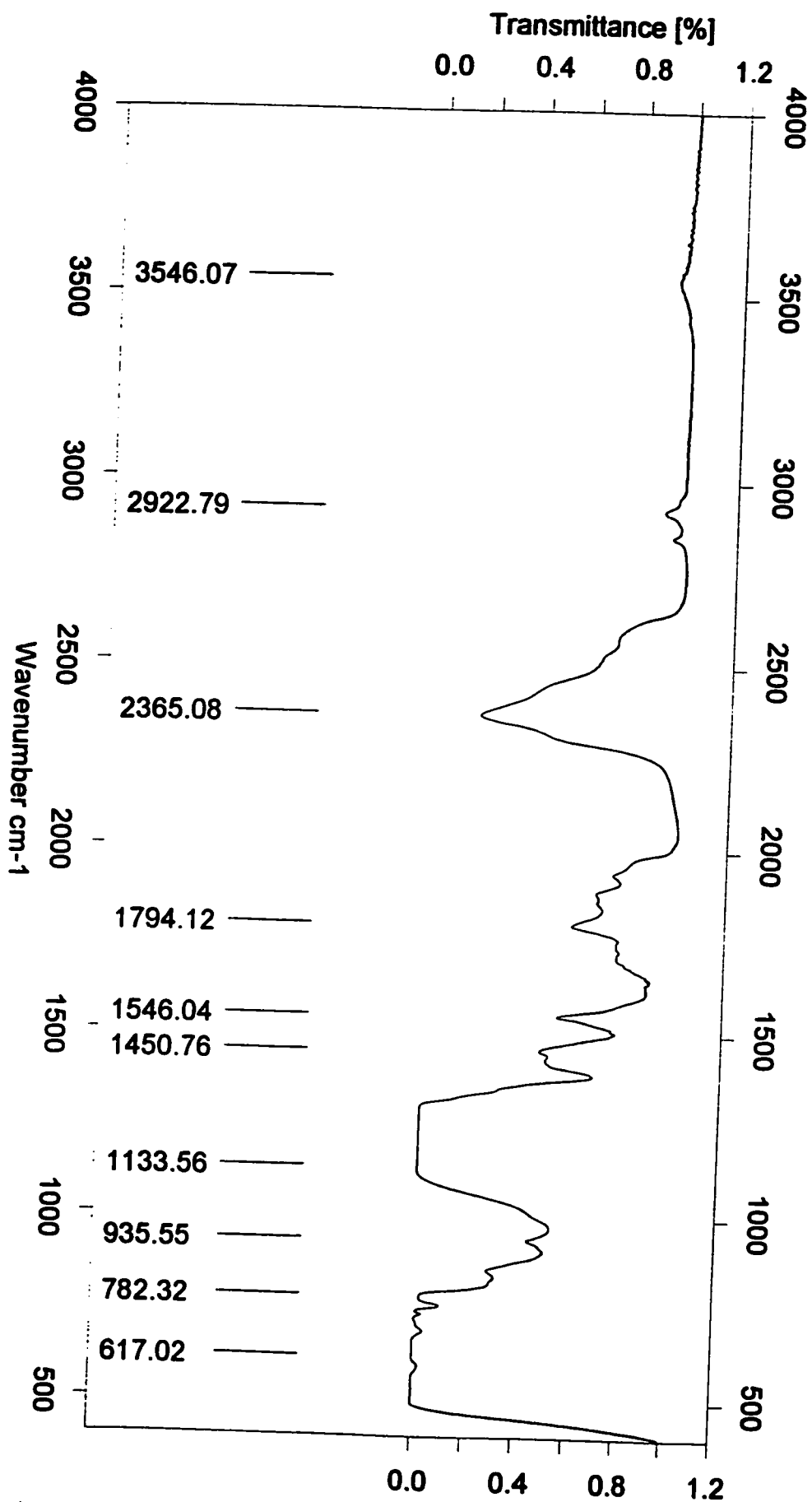
4.35 FTIR spectra of PTFE specimen after 5000h of aging in 100mS/cm saline solution at $44\pm 2^\circ\text{C}$.



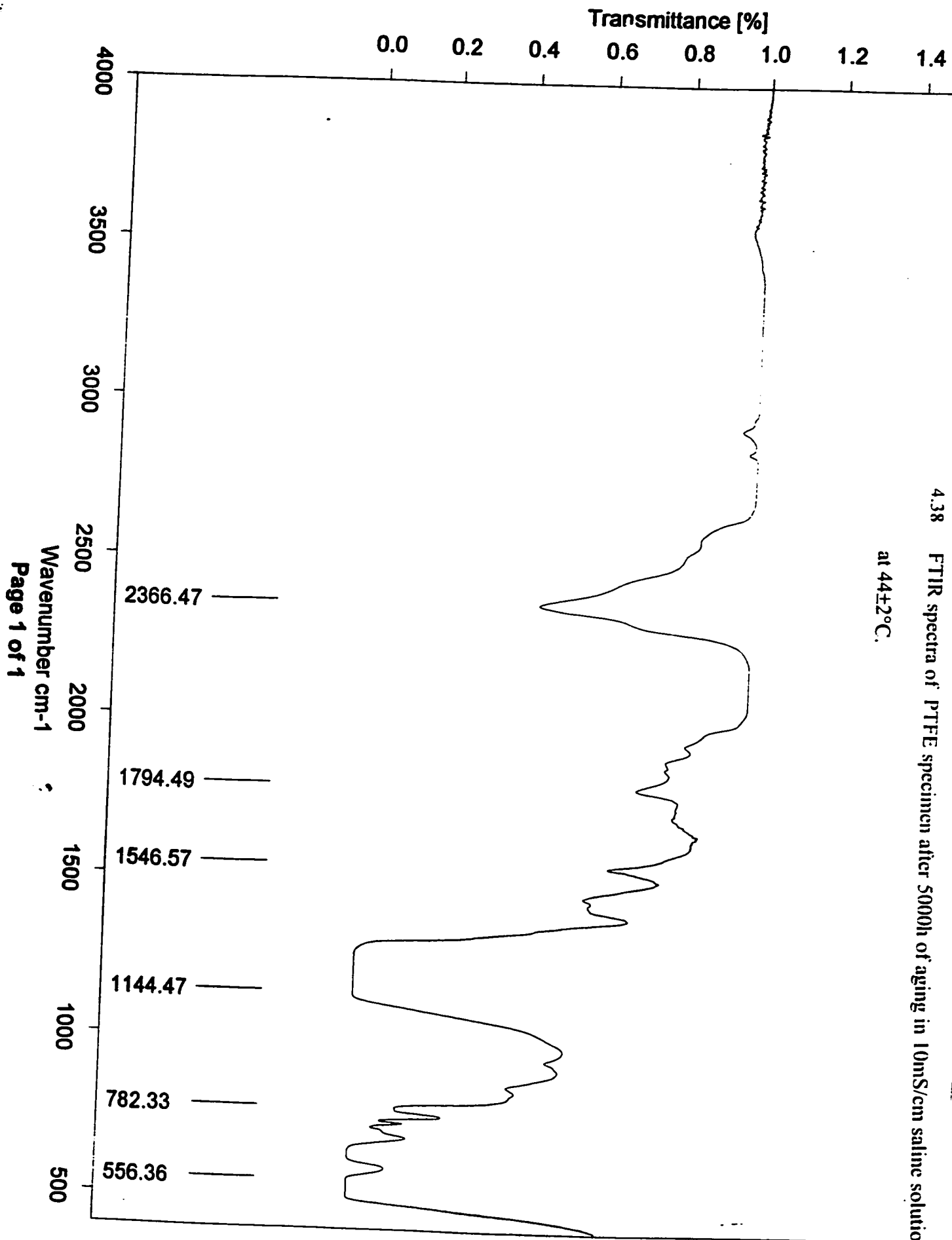
4.36 FTIR spectra of PTFE specimen after 5000h of aging in 10mS/cm saline solution
at 73±2°C.



4.37 FTIR spectra of PTFE specimen after 5000h of aging in 100mS/cm saline solution at 99±2°C.



4.38 FTIR spectra of PTFE specimen after 5000h of aging in 10mS/cm saline solution
at 44±2°C.



4.3.12 Scanning Electron Microscopy (SEM) of PTFE:

Scanning Electron Microscopy (SEM) was carried out in order to have an independent confirmation of the results of surface roughness measurements, taken by the surface detector Surftest-212 and to investigate into other surface changes of the PTFE. The specimens aged in different saline solutions at different temperatures and salinities were cooled down to room temperature and then washed in 0.005 mS/cm distilled water in an ultrasonic vibrator for a duration of approximately 5 minutes to remove the salt deposits and any other debris on the surface of the specimens.

Fig. 4.39 shows a SEM microphotograph of a virgin PTFE specimen. The magnification ratio is 500. The Fig. 4.39 shows the PTFE surface which is smooth clear and free of debris.

Fig. 4.40 shows changes on the surface of PTFE at $22\pm4^{\circ}\text{C}$ in air after 5000h of aging. It shows very minimum changes as compared to Fig. 4.39. The Fig. 4.40 shows a few white spots on the surface that may be some salts and other debris collected from atmosphere.

Fig. 4.41 and Fig. 4.42 shows the changes on the surface of PTFE specimen after 5000h of immersion in 0.005 mS/cm and 10 mS/cm saline solution at $0\pm1.5^{\circ}\text{C}$. These figures does not show any significant change in the surface roughness except the presence of some debris.

Fig. 4.43 and Fig. 4.44 shows the surface roughness of PTFE specimen after 5000h of immersion in 0.005 and 10 mS/cm saline solution at $22\pm4^{\circ}\text{C}$. It can be observed from Fig. 4.43 and Fig. 4.44 that, as the temperature and salinity increases, the degradation of PTFE surface was also increased. The same was observed from surface roughness measurements (Fig. 4.8).

From Fig. 4.45 to Fig. 4.48, it can easily be observed that as the temperature and salinity increases the surface roughness also increases.

Fig. 4.49 and Fig. 4.50 shows the increased in the surface roughness for 10mS/cm and 100mS/cm saline solution at $99\pm 2^{\circ}\text{C}$. These figures shows the presence of some white material on the surface of the specimen. It is thought to be calcium carbonate (CaCO_3) which may have come from the distilled water while heated at $99\pm 2^{\circ}\text{C}$. The surface roughness as detected by the surfest-212 is maximum in this case (Fig. 4.8), which agrees with the SEM microphotograph. It will also be noted from Fig. 4.50, that the contact angle for these two specimen after 5000h of immersion were also lower than the other temperatures and salinities, which proves that the loss of hydrophobicity increases with the increasing surface roughness [7].

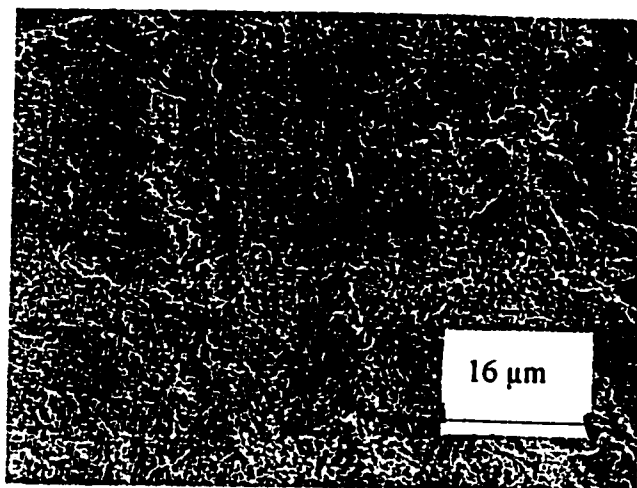


Fig. 4.39 Scanning electron microphotograph (SEM) of virgin PTFE specimen. (Magnification, 2,000).

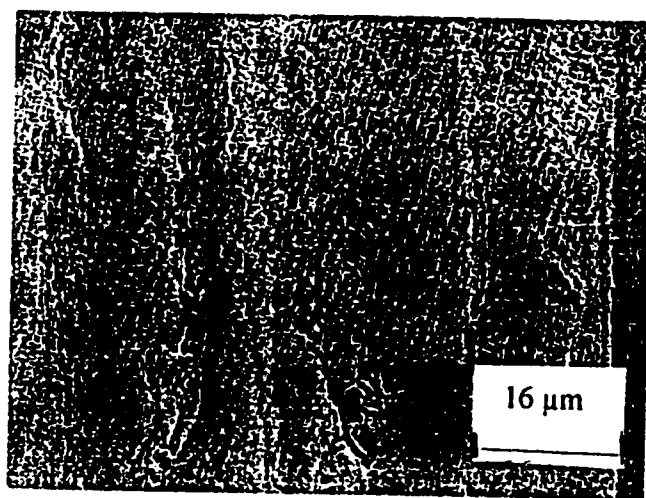


Fig. 4.40 Scanning electron microphotograph (SEM) of PTFE aged in air at $22 \pm 4^{\circ}\text{C}$, time of aging 5000 h. (Magnification, 2,000).

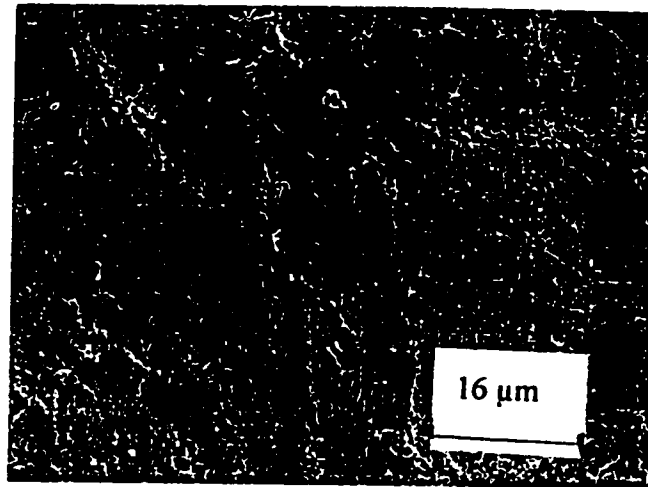


Fig. 4.41 Scanning electron microphotograph (SEM) of PTFE aged in 0.005mS/cm saline solution, at $0 \pm 1.5^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000)

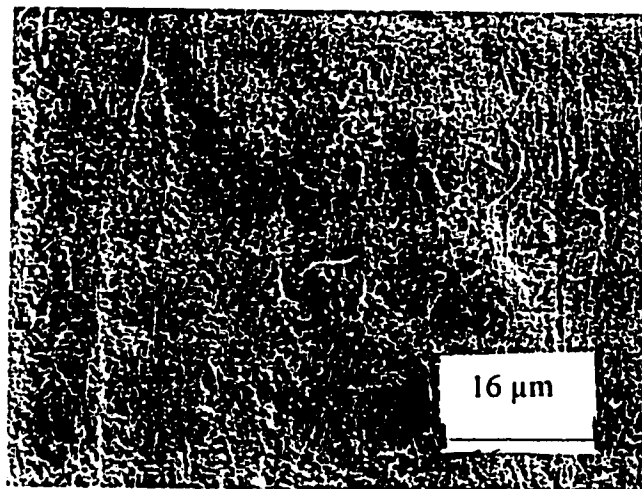


Fig. 4.42 Scanning electron microphotograph (SEM) of PTFE aged in 10 mS/cm saline solution, at $0 \pm 1.5^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000).

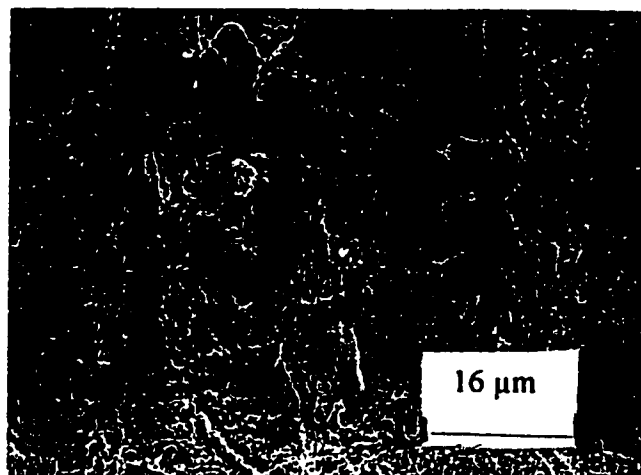


Fig. 4.43 Scanning electron microphotograph (SEM) of PTFE aged in 0.005mS/cm saline solution, at $22\pm4^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000).

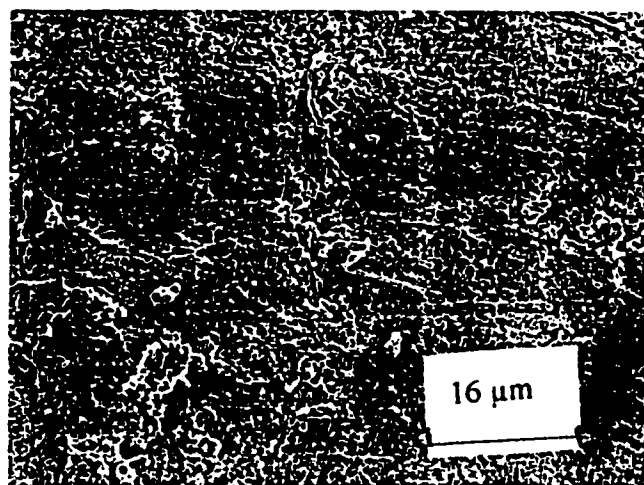


Fig. 4.44 Scanning electron microphotograph (SEM) of PTFE aged in 10 mS/cm saline solution, at $22\pm4^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000).

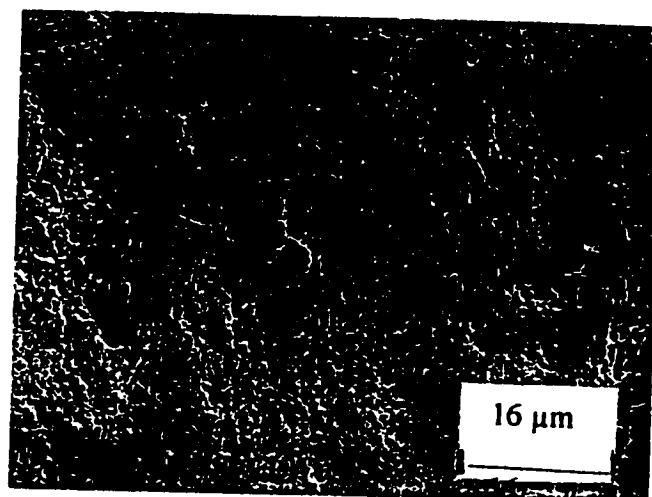


Fig. 4.45 Scanning electron microphotograph (SEM) of PTFE aged in 0.005 mS/cm saline solution, at $44\pm 2^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000).

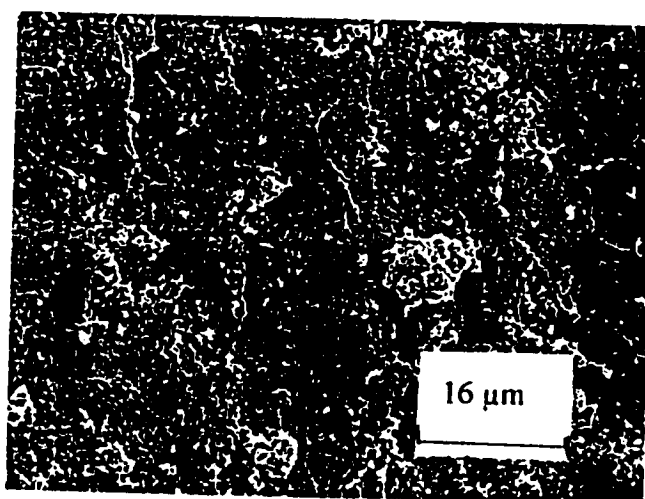


Fig. 4.46 Scanning electron microphotograph (SEM) of PTFE aged in 10 mS/cm saline solution, at $44\pm 2^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000).

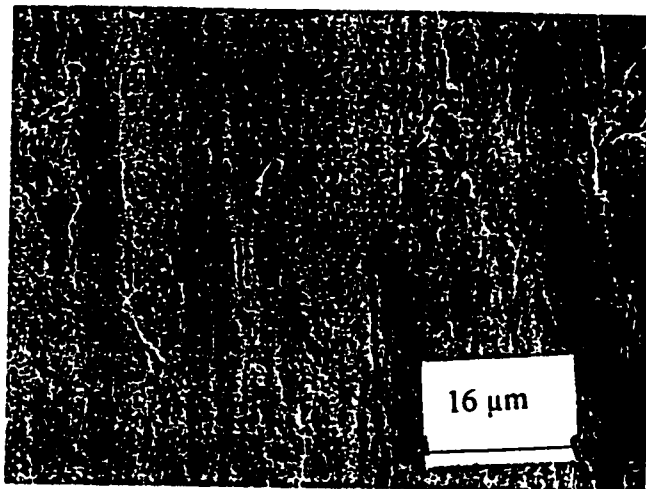


Fig. 4.47 Scanning electron microphotograph (SEM) of PTFE aged in air, at $73\pm 2^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000).

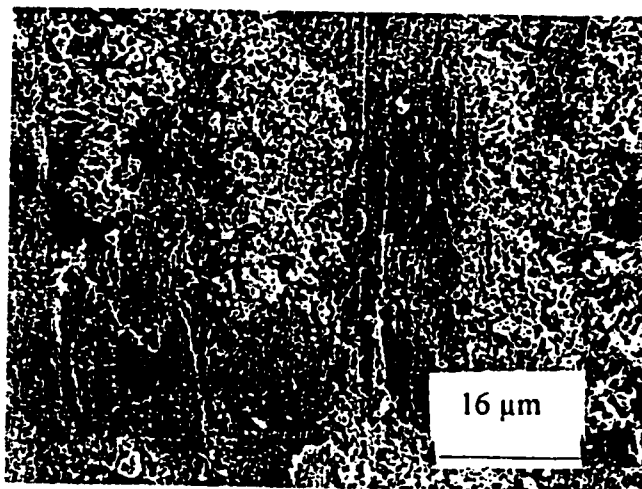


Fig. 4.48 Scanning electron microphotograph (SEM) of PTFE aged in 10 mS/cm saline solution, at $99\pm 2^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000).

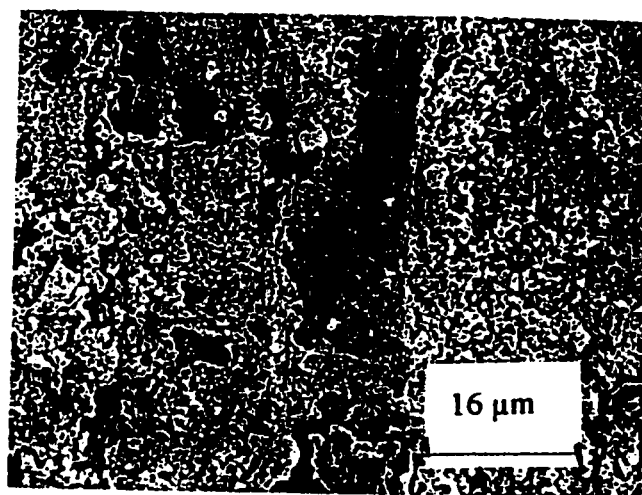


Fig. 4.49 Scanning electron microphotograph (SEM) of PTFE aged in 10 mS/cm saline solution, at $99\pm 2^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000).



Fig. 4.50 Scanning electron microphotograph (SEM) of PTFE aged in 100 mS/cm saline solution, at $99\pm 2^{\circ}\text{C}$, time of immersion 5000 h. (Magnification, 2,000).

4.4 Summary of the results:

1. The contact angle of PTFE remained within $104\pm 5^\circ$ in air for 0° , 22° , 44° , 73° and 99°C .
2. The contact angle of PTFE decreased with increasing time of immersion in saline water for the same conductivity and temperature.
3. The contact angle of PTFE decreased with immersion in saline water with the increasing temperature from 0°C to 99°C .
4. The contact angle of PTFE decreased with immersion in saline water with the increasing salinity from 0.005 mS/cm to 100mS/cm .
5. The surface roughness of PTFE increased with increasing time of immersion, increasing conductivity and increasing temperature of the saline solution.
6. The intake of water into PTFE increased with increasing time of immersion, temperature and salinity.
7. The contact angle of PTFE decreased with increasing surface roughness and weight of specimen due to intake of water.
8. The coefficient of diffusion of water into PTFE increased with increasing temperature from 0°C to 99°C and increasing salinity from 0.005 mS/cm to 100 mS/cm .
9. The activation energy of the diffusion of water into PTFE increased with increasing conductivity of the saline solution.
10. The surface free energy per unit area of PTFE increased with increasing time of immersion, conductivity and temperature, until saturation was reached.

11. Application of higher temperature up to 250 °C in air has no significant effect on the contact angle of PTFE.
12. Application of dc stress of up to 15 kV has no significant effect on the contact angle of PTFE.
13. The contact angle decreased after application of RF discharge and then recovered slowly within 450hrs.
14. From FTIR spectra it is concluded that the loss of hydrophobicity of PTFE specimen is also due to weakening of C-F bonds under stress of higher temperature and higher salinity.
15. The loss of hydrophobicity despite the lack of observable oxidation leads to the conclusion that the contact angle of an aged PTFE does not necessarily depend on the oxidation compounds on the surface or in the bulk of PTFE.
16. The microphotograph of Scanning Electron Microscopy proves that the surface degradation increases with the increasing temperatures and salinities and hence the loss of hydrophobicity.

Chapter 5

Effect of Water Salinity, Temperature and its duration on ac and dc Flashover Voltage of PTFE

5.1 Break Down Phenomena of Solid Insulator

5.1.1 Introduction

The failure of dielectrics under electrical stress, generally referred to as breakdown, is a study of practical importance. It is often found that the same material will exhibit a wide range of dielectric strength, depending upon its environment and mode of use [33]. However, even where the conditions of applications and field distribution are apparently the same, breakdown is still found to spread over a wide range of applied stresses [34,35]. Experimental evidence has made it clear that, in practice, the failure of a solid polymeric material is a complicated process, which often involves combinations of several of the breakdown mechanisms postulated in the literature [36].

Several types of breakdown are recognized as “**Intrinsic breakdown**”, which is electronic in nature, depending on the presence of electrons capable of migration through the lattice.

The second is “**thermal breakdown**” which arises from the dielectric conductivity, very generally, if a current is driven through the dielectric, by the application of a field. Thermal breakdown arises when local heat is generated by this current faster than it can be dissipated [33]. The third mechanism, “**discharge breakdown**”, is not perhaps strictly a basic one, as it depends upon the presence of voids in the dielectric material [37].

There are other mechanisms, which may be considered as basic, but are of a cumulative nature, gradually building up to breakdown. The most important of these are electromechanical and chemical effect; due to which the insulation gradually deteriorates to form with a higher conductivity. When this state is reached breakdown may occur by thermal effects, or by virtue of a lowered intrinsic strength, or by the formation of a discharge path (arc) between electrodes. Electrical conduction studies in solids are obscured by the fact that the transport phenomena besides electronic and ionic carriers include also currents due to the polarization processes such as slow moving dipoles (orientation polarization) and interfacial polarization [37].

5.1.2 Type of Breakdown

5.1.2.1 Intrinsic Breakdown

In the Intrinsic breakdown the material under test is pure and homogenous, the temperature and environmental conditions are carefully controlled, and the sample is so stressed that there are no external discharges [38]. As applied voltage is increased from zero a small current will begin to flow. As the voltage continues to rise this current remains constant until a certain critical voltage is reached. At this critical point the current suddenly rises rapidly to a high value; breakdown is said to have occurred and the critical voltage is the breakdown voltage. In case of intrinsic breakdown the current rises to the maximum value allowed by the supply in a time of the order of 10^{-8} seconds. It is therefore postulated that the breakdown is electronic in nature.

Frohlich [39] proposed a model, based on this assumption that the random motion of electrons is increased, which at the time of electrical breakdown gain energy from the electrical field faster than they lose it to the material. To date there has been no direct

experimental proof to show whether an observed breakdown is intrinsic or not, except for the plastic materials such as polyethylene and so conceptually it remains an ideal mechanism identified as the highest value obtainable after all secondary effects have been eliminated [36].

5.1.2.2 Thermal Breakdown

Thermal breakdown occurs when the heat input cannot be balanced by the heat loss from the insulation either macroscopically or, more usually, in a small area. As power is dissipated by the insulation, heating occurs which usually causes an exponential increase in the electrical conductivity as more carriers become available for conduction. Alternatively, the increased segmental motion may increase the mobility for intrinsic ionic conduction. If the electrical stress is maintained, the current density increases in the area of elevated temperature. This serves to further increase the local temperature through Joule heating and hence the conductivity, and so 'thermal runaway' may occur. In general this leads to a highly localized filamentary breakdown path; however under appropriate conditions, breakdown may occur on broad front [40].

5.1.2.3 Discharge Breakdown

It is difficult to eliminate voids in polymeric materials [37]. In partial discharge breakdown sparks occur within voids in the insulation causing degradation of the void walls and progressive deterioration of the dielectric.

5.1.2.4 Free Volume Breakdown

Free volume breakdown carriers are accelerated through spaces within low- density amorphous regions; the energy thereby gained is lost through collisions.

5.1.2.4 Electromechanical Breakdown

The effect of electrostatic forces in stressed dielectrics cannot be ignored. At fields approaching breakdown, such mechanical pressure can reach magnitudes of the order of several tens of kilograms per square centimeter and will cause deformation of compliant materials such as polymers. On this basis, Stark and Garton, have proposed that an electrically stressed material may fail by mechanical collapse. In the case of a plane slab of material with electrodes which will follow any motion allowed by the dielectric, for equilibrium, the electrostatic force is balanced by the elastic reaction so that for large strains

$$\frac{1}{2}[\epsilon'\epsilon_0(V/d)^2] = Y \ln (d_0/d) \quad \text{-----} \quad (5.1)$$

Where V is the voltage applied across a specimen of undistorted thickness, d_0 , and Young's modulus, Y . Differentiation of $d^2 \ln (d_0/d)$ readily shows that thickness, d , under stress conditions cannot have a stable value greater than $d_0 \exp (-1/2)$. Consequently, the critical value of stress will be given by

$$E_c = V_c/d \quad \text{-----} \quad (5.2)$$

$$E_c = (Y/\epsilon'\epsilon_0)^{1/2} \quad \text{-----} \quad (5.3)$$

which must be multiplied by $\exp(-1/2) \simeq 0.6$, if the undistorted thickness, d_0 , is used in computing the stress. This hypothesis assumes that breakdown has not occurred by

another process prior to the establishment of the critical stress and is also confined to substances which can deform sufficiently without fracture.

5.1.2.5 Flashover Voltage

Flashover is a breakdown of the air or gas in parallel with the solid or liquid insulation.

Flashover is a surface phenomena and can be controlled by

- 1) Adjusting the creepage distance
- 2) Insulator surface design.
- 3) Removing air or gas (other than electronegative types) from critical areas by varnish, impregnation, potting, oil filling, or embodiment of electrodes.
- 4) Changing the gas to electronegative type (such as SF₆) so those electrons may be trapped before they form a corona cloud.
- 5) Using semiconducting components or surface treatment materials to grade the stress below the corona inception voltage.
- 6) Using stress rings to alter the electric field in the vicinity of the gas-insulation-electrode area.
- 7) Avoiding the use of high-dielectric-constant materials adjacent to air.

Flashover voltage is affected by humidity, type of voltage, spacing, dielectric constant of solid insulator, contamination, and uniformity of field. Moisture as a surface contaminant promotes surface leakage, tracking, flashover, and dielectric breakdown by bridging weak spots

5.2 Effect of Water Salinity, Temperature and their Duration on AC and DC Flashover Voltage of PTFE

5.2.1 Abstract

The effects of aging of polytertraflouroethylene by immersion in a saline solution at different temperatures and for different duration's on the ac and dc flashover voltages in air at 1.01×10^5 Pa and $22 \pm 4^\circ\text{C}$ are reported. The PTFE specimens were immersed in 10mS/cm saline solutions, of water and were kept at $0 \pm 1.5^\circ\text{C}$, $22 \pm 4^\circ\text{C}$, $44 \pm 2^\circ\text{C}$ and $99 \pm 2^\circ\text{C}$ for duration of up to 125 days. The ac (60Hz) and dc flashover voltages were measured after 10, 23, 45, 100 and 125 days of immersion. The flashover voltage was found to decrease with increasing immersion time and increasing temperature of the solution. The contact angle, the average surface roughness and the change in the weight of specimens due to the uptake of water were also measured and correlated with the ac and dc flashover voltages.

5.2.2 Introduction

During exposure to a saline solution the surface of a polymeric insulating material incurs gradual changes, which impact on its withstand voltage. The surface of PTFE is usually hydrophobic due to its low surface energy [21,], but under continuous stress of salinity and higher temperatures, surface degradation is initiated due to oxidation and the surface gradually becomes hydrophilic. In this state the electric current readily flows across the surface. As the applied voltage is increased the current also increases until flashover occurs across the surface. In this paper the effect of immersion of PTFE in a saline

solution at different temperatures and for different duration's on the surface flashover voltage is reported.

5.2.3 Experimental Procedure

PTFE cylindrical rods of 10 mm in length and 19 mm in diameter were immersed in 10mS/cm saline solutions, at various temperatures and for up to 125 days. The specimens were removed from the solutions at different times and were placed between 152-mm diameter of stainless steel electrodes and the ac and dc flashover voltages were determined. A new specimen was used for each experimental reading.

The experimental procedure for the measurement of the contact angle is the same as described in [28]. The contact angle was determined at $22\pm4^{\circ}\text{C}$ and at a relative humidity of $55\pm20\%$. After the removal of the specimens from the saline solutions at $0\pm1.5^{\circ}\text{C}$, $22\pm4^{\circ}\text{C}$, $44\pm2^{\circ}\text{C}$ and $99\pm3^{\circ}\text{C}$, they were allowed to dry and reach room temperature before the contact angle, the surface roughness and the weight were measured. The contact angle was determined within 30s after placing a droplet of water on the specimen. The contact angle was measured at 8 different locations on each specimen. After these measurements, the flashover voltages were measured (within) two minute interval until a saturated value of the flashover voltage was reached.

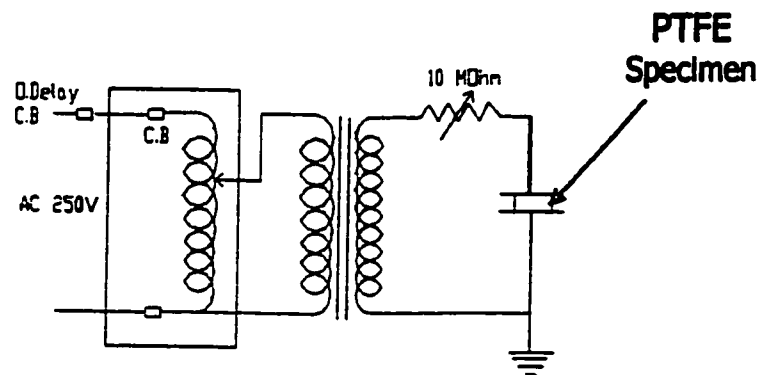


Fig. 5.1. Arrangement to measure the ac flashover voltage

5.3 Results and Discussion

5.4.1 AC Flashover Voltage

Fig. 5.2 shows the ac flashover voltage against number of shots. It can be observed that the flashover voltage rapidly increased until a saturated value was reached. The ac flashover voltage increased from 24.1 kV to 28.1 kV. The improvement in withstand voltage is attributed to the removal of asperities and debris from the surface.

Fig 5.3 shows the ac flashover voltage; after 125 days of aging in 10mS/cm solution at 0°C, 21°C, 44°C and 98°C. The flashover voltage decreased with increasing time of immersion. The saturated value of the ac flashover voltage of the virgin specimen was 28.1 kV, which decreased to 20.5, 19.2, 17.8 and 17.1 kV after 125 days of immersion in 10mS/cm saline solution of water at 0°C, 21.°C, 44°C and 98°C, respectively.

Fig 5.4 shows the effect of temperature on the ac flashover voltage of aged PTFE for 125 days in 10mS/cm solution. The flashover voltage decreased with the increasing temperature. Typically the flashover voltage decreased from 28.1 kV before immersion to 17.1 kV at 98°C after immersion. The increase in temperature reduces the ac breakdown voltage of PTFE [6].

The contact angle of the specimen was also measured. The contact angle decreased from 108° before immersion to 91.0° after 125 days of immersion at 0°C. At 21°C, 44°C and 98°C the contact angle decreased from 108° to 88.0°, 87.5° and 56.9° respectively, after 125 days of immersion in 10mS/cm saline solution. The contact angle decreased with the increasing temperature and salinity [3,7].

Fig 5.5. shows the effect of the hydrophobicity on the ac flashover voltage at 0°C. From Fig.5.5; it can be observed that as the contact angle decreases; the flashover voltage also

decreases. At 22°C the flashover voltage was 28.1 kV and the contact angle was 108° and decreased to 19.20 kV at 88°. This correlates the loss of hydrophobicity with the reduction in flashover voltage.

From Figs. 5.6, 5.7 and 5.8, it can be observed that the flashover voltage is affected by the loss of hydrophobicity. As the contact angle decreased the flashover voltage also decreased.

At 21°C, it decreased from 28.1 to 19.2 kV for contact angles of 105° and 88° respectively.

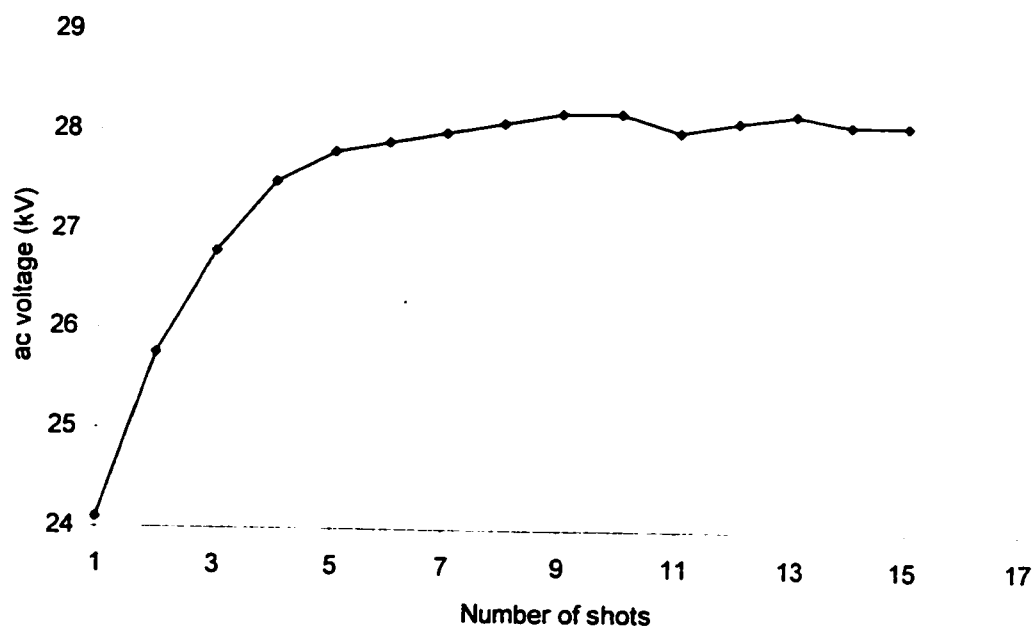


Fig. 5.2: AC flashover voltage (peak) versus number of shots at $22\pm 4^{\circ}\text{C}$. before immersion in a saline solution.

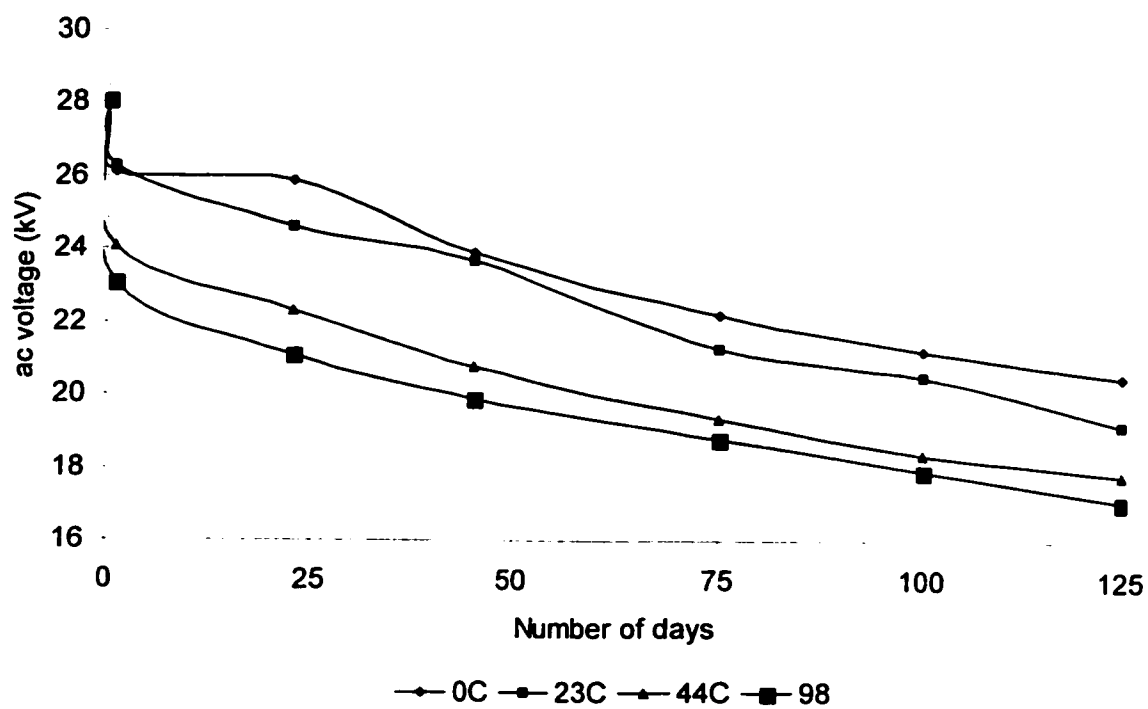


Fig. 5.3: Dependence of the AC flashover voltage (peak) on the duration of immersion in 10mS/cm saline solutions.

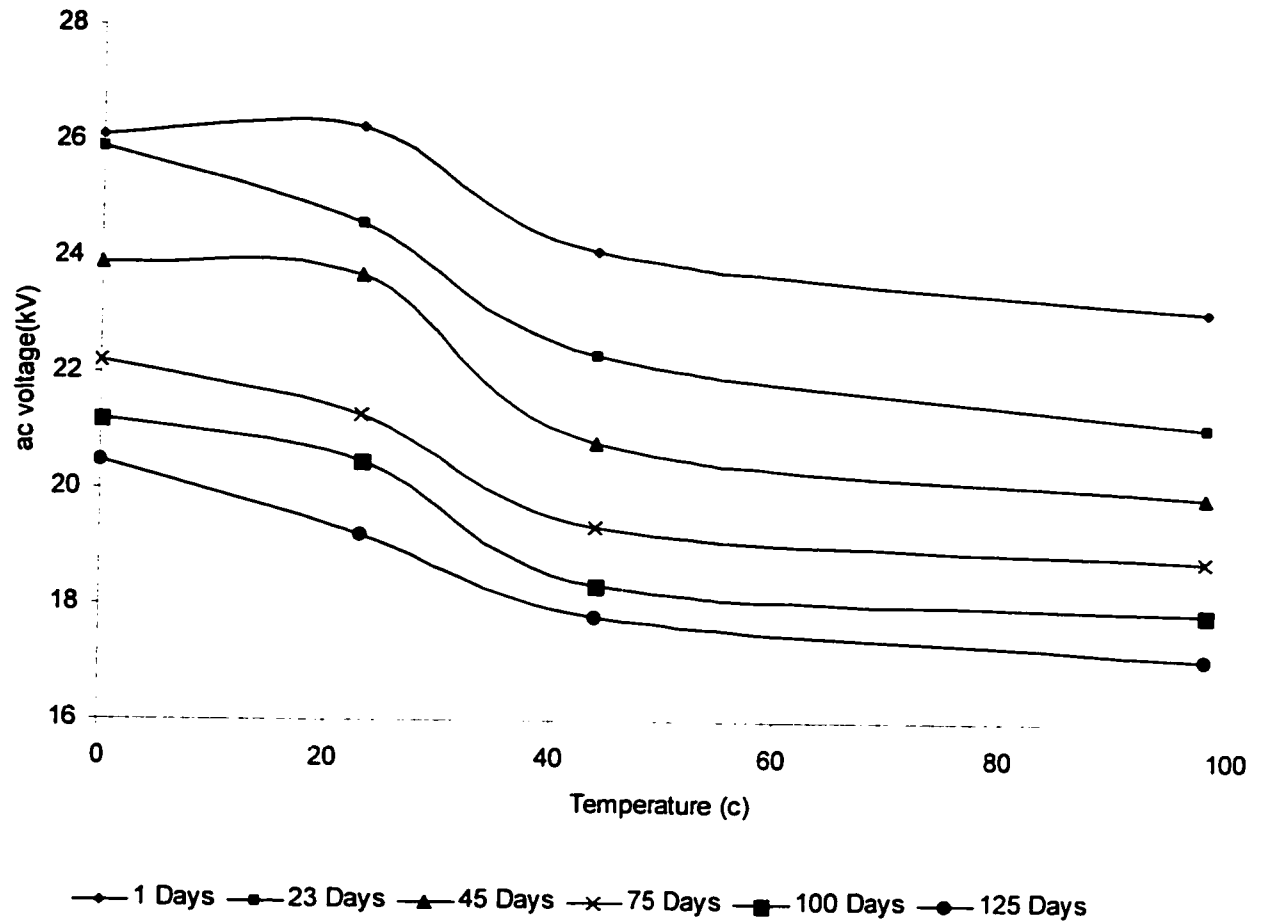


Fig 5.4: Dependence of AC flashover voltage (peak) on temperature; after immersion in 10mS/cm saline solutions.

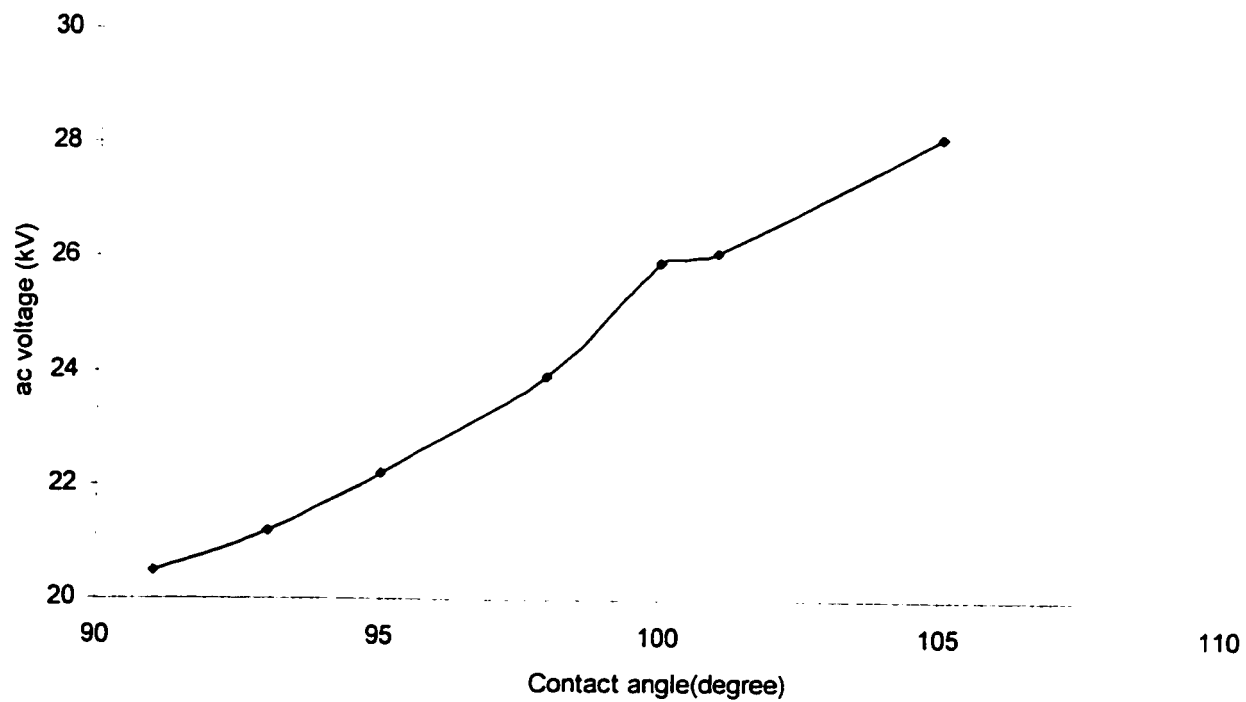


Fig. 5.5: Dependence of the ac flashover voltage (peak) on the contact angle, at $0\pm1.5^{\circ}\text{C}$.

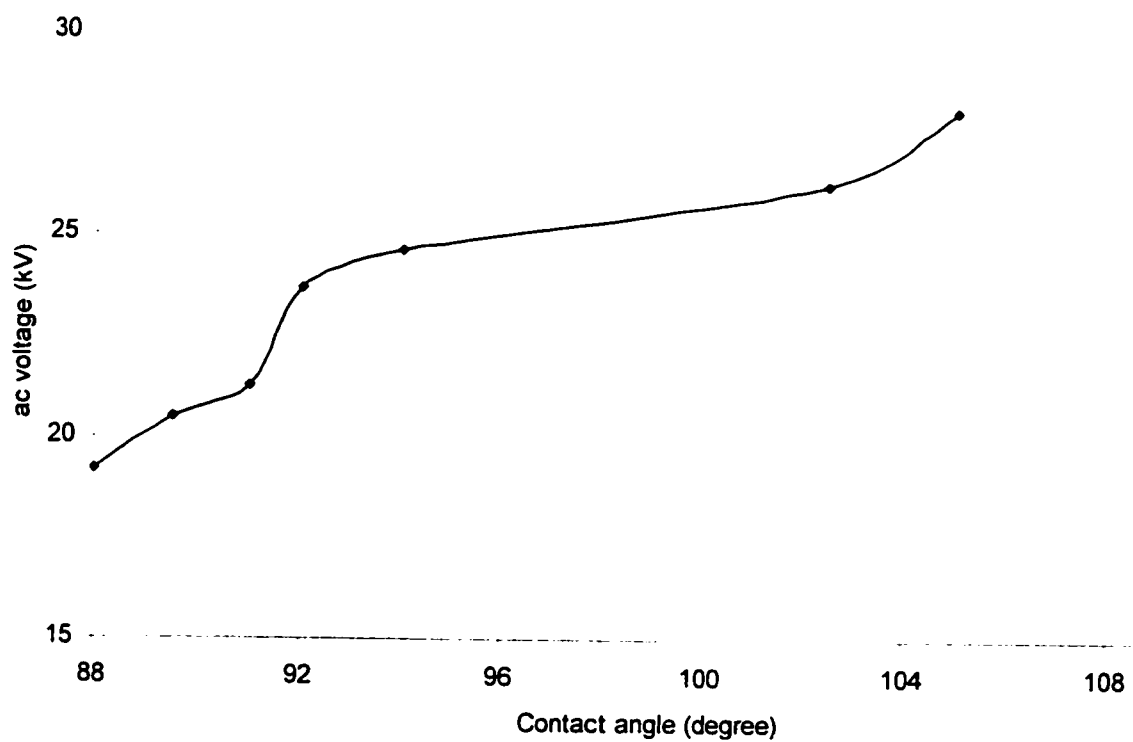


Fig. 5.6: Dependence of the ac flashover voltage (peak) on the contact angle, at $22. \pm 4^{\circ}\text{C}$.

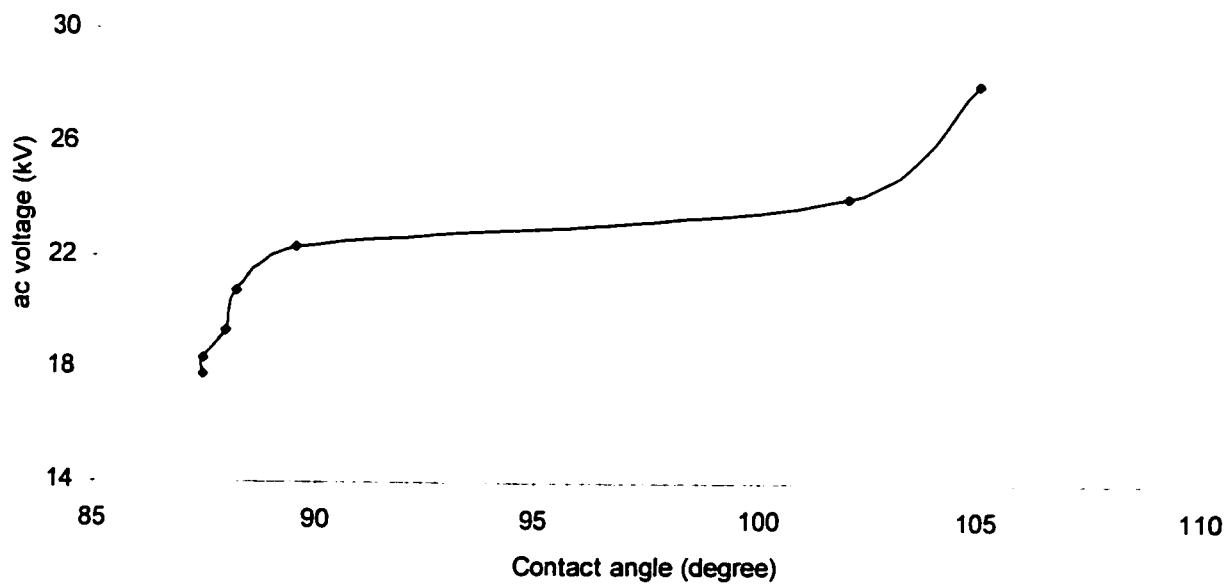


Fig. 5.7: Dependence of the ac flashover voltage (peak) on the contact angle, at $44\pm 2^{\circ}\text{C}$.

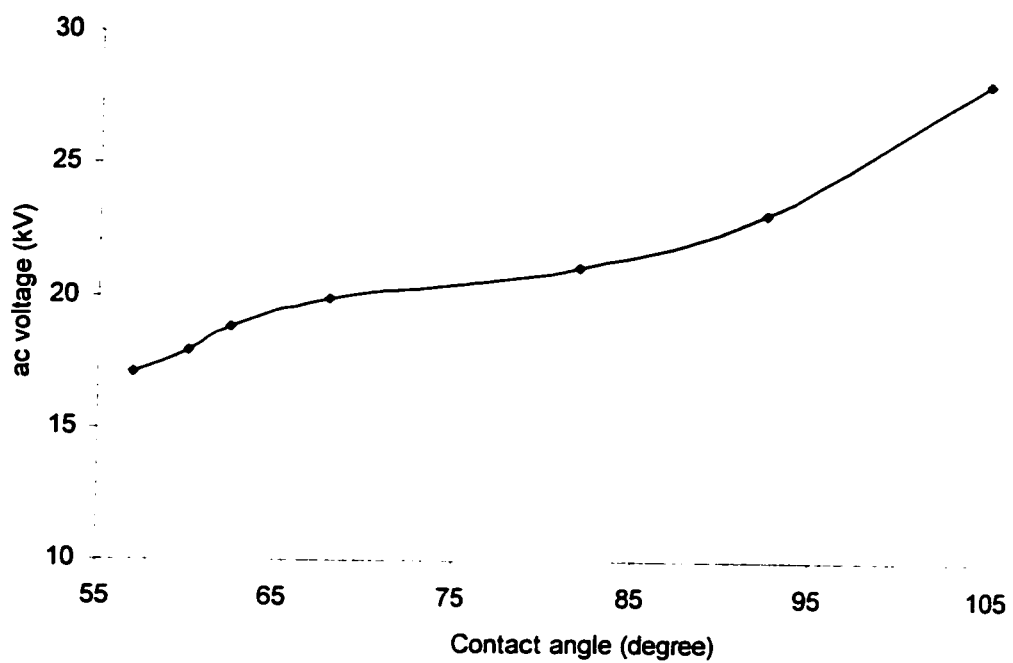


Fig. 5.8: Dependence of the ac flashover voltage (peak) on the contact angle, at $99\pm 2^{\circ}\text{C}$.

5.3.2 DC Flashover Voltage

The dc flashover voltage was also measured at 0°C, 22°C, 44°C and 99°C. The initial value of the dc flashover voltage before immersion in saline solution was 25.3 kV. The time between successive breakdowns was 2 minutes. It is observed from Fig. 5.10 that the flashover voltage increased rapidly to a saturated value after a few shots. The flashover voltage increased from 25.3 to 27.8 kV.

Fig. 5.11 shows that the dc flashover voltage decreased from 27.8 kV to 21.4 kV, after 125 days of immersion at 0°C. The contact angle decreased from 108° to 89°. The dc flashover voltage decreased to 20.9, 16.42 and 15.1 kV for 22°C, 44°C and 99°C, respectively. The dc flashover voltage decreased with the increasing temperature [6].

After one day of aging the flashover voltage at 22°C was 27.4 kV and 25.3 kV at 99°C. It can be observed from Fig. 5.12, that the flashover voltage decreased with increasing temperature and increasing time of immersion.

Fig. 5.13 to 5.16 shows the effects of the loss of hydrophobicity on the dc flashover voltage. It could be observed that the flashover voltage decreased with decreasing contact angle at 0°C from 27.4 at 105° to 21.4 kV at 99°C

At 21.5°C the flashover voltage at 108° was 27.45 kV and it decreased to 20.9 kV at 86°.

The dc flashover voltages at 44°C and 99°C were 16.42 and 15.1 kV respectively.

The absorption of water, which resulted in an increase in the weight of PTFE specimen, was also measured. After 125 days of aging at 99°C in 10mS/cm saline solution, the weight increased by 0.025%. At lower temperature the increase in the weight was much lower than at 99°C, in agreement with [3,7, 9].

The average surface roughness (ASR) of the PTFE specimens after immersion in 10mS/cm saline solution at 0°C, 22°C, 44°C and 99°C was also measured and found to be 1.67, 2.13, 2.38 and 2.89 μm respectively.

The loss of hydrophobicity of PTFE at higher temperature is mainly due to the absorption of water, weakening of C-F bond and increase in the surface roughness due to aging. Due to these influences the ac and dc flashover voltages also decreased.

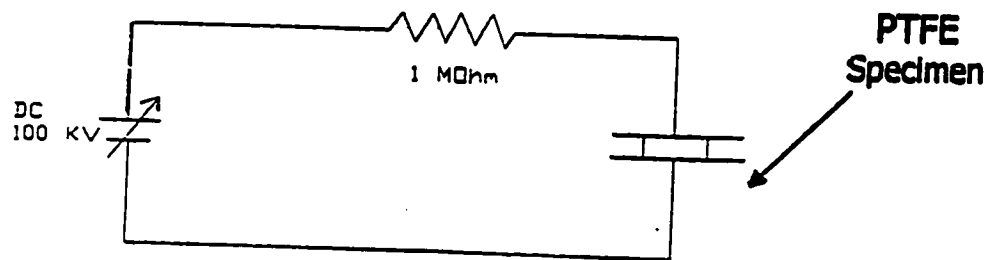


Fig. 5.9. Arrangement to measure the dc flashover voltage

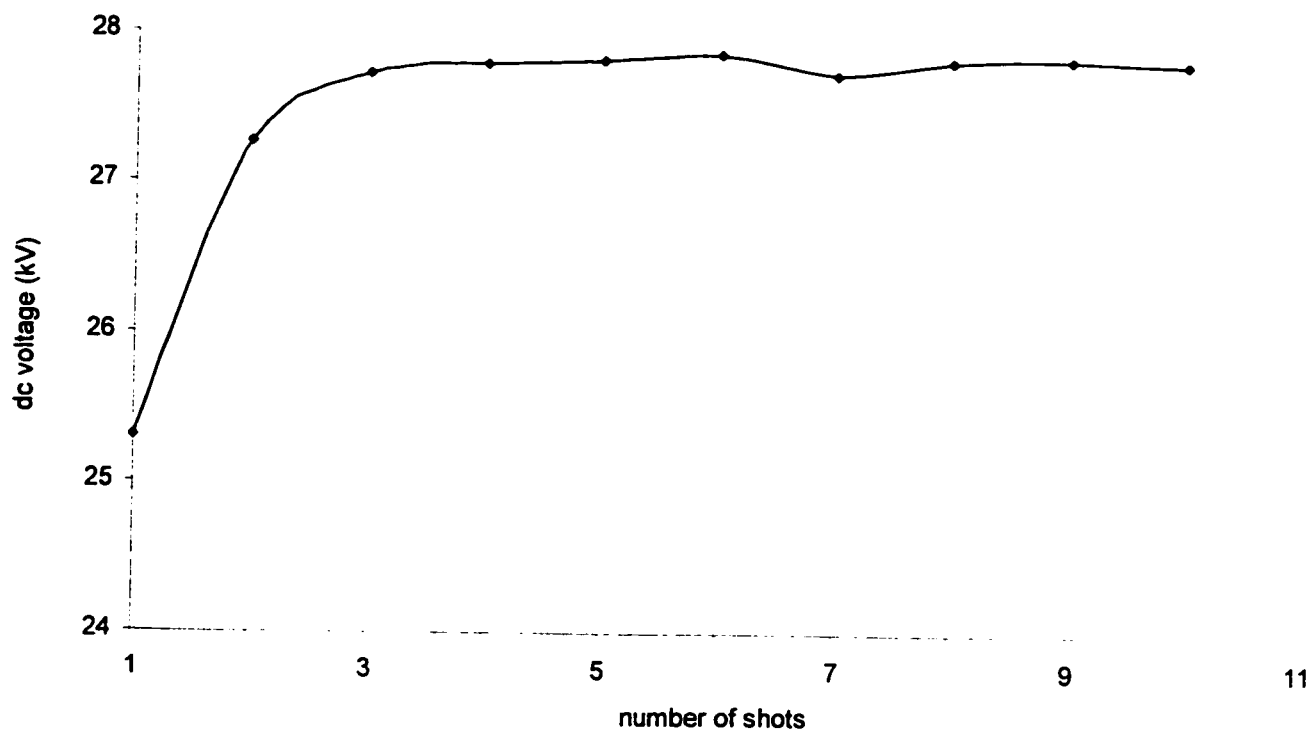


Fig. 5.10: DC flashover voltage versus number of shots at $22\pm 4^{\circ}\text{C}$.

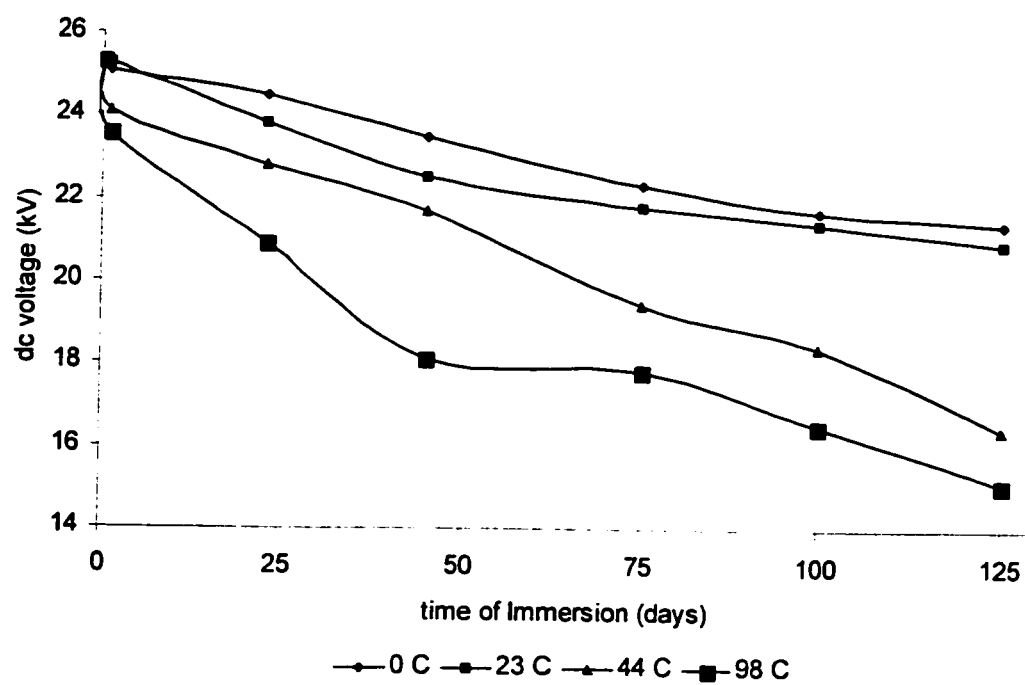


Fig 5.11: Dependence of DC flashover voltage on the duration of immersion in 10 mS/cm saline solutions.

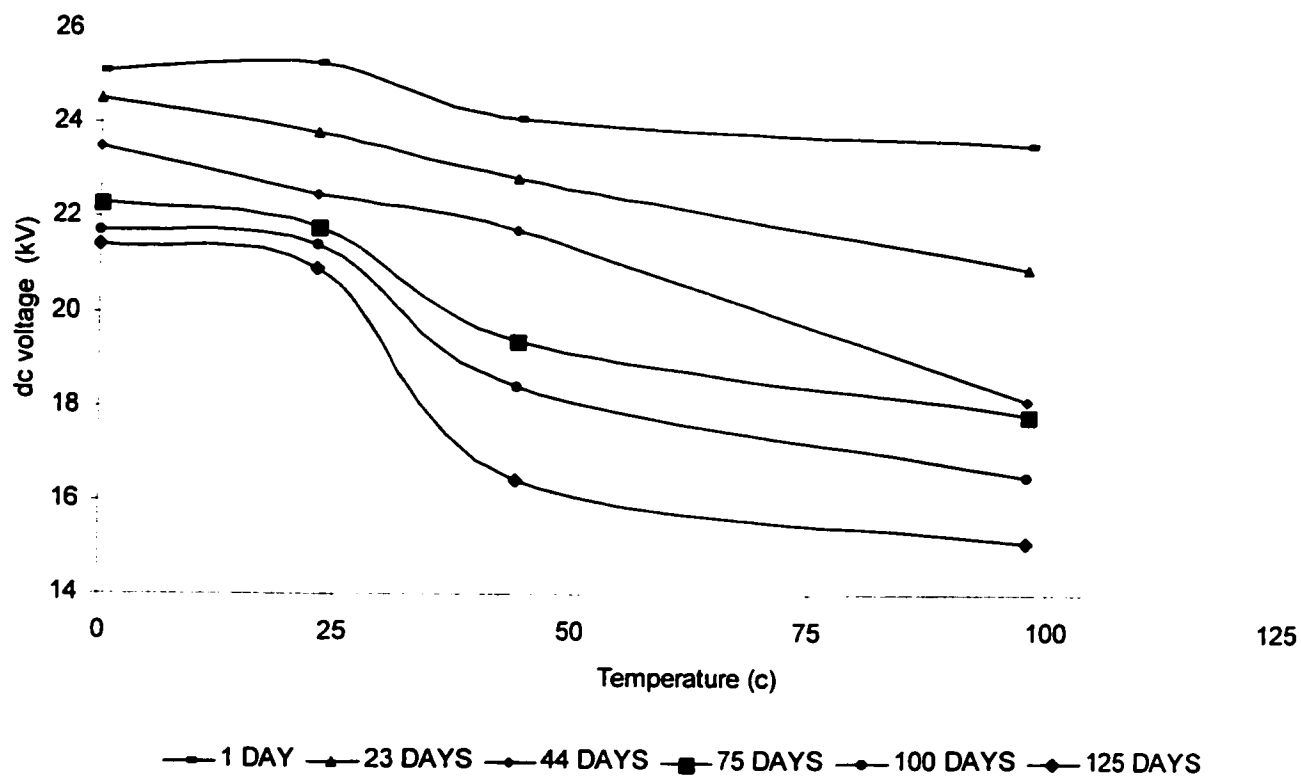


Fig. 5.12: Dependence of the DC flashover voltage on temperature, after immersion in 10mS/cm saline solutions

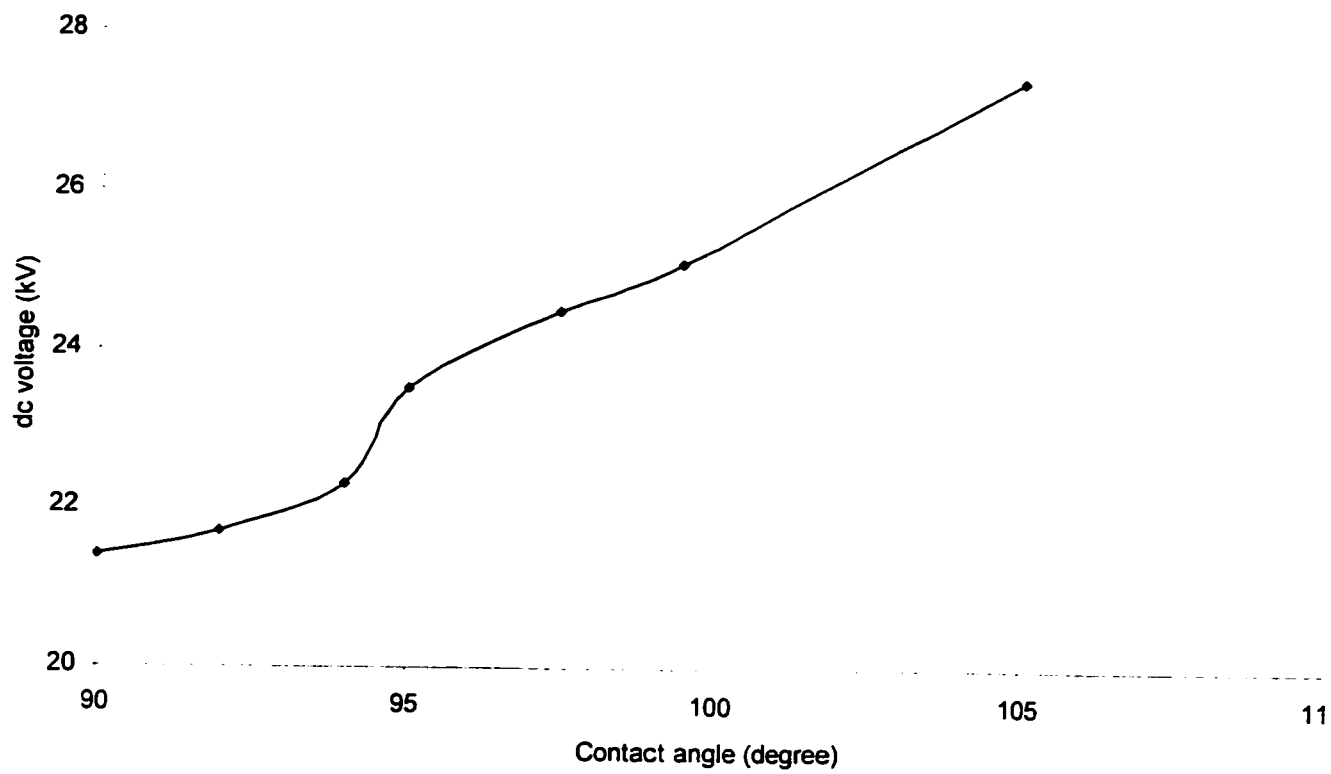


Fig. 5.13: Dependence of the DC flashover voltage on the contact angle, at $0 \pm 1.5^\circ\text{C}$.

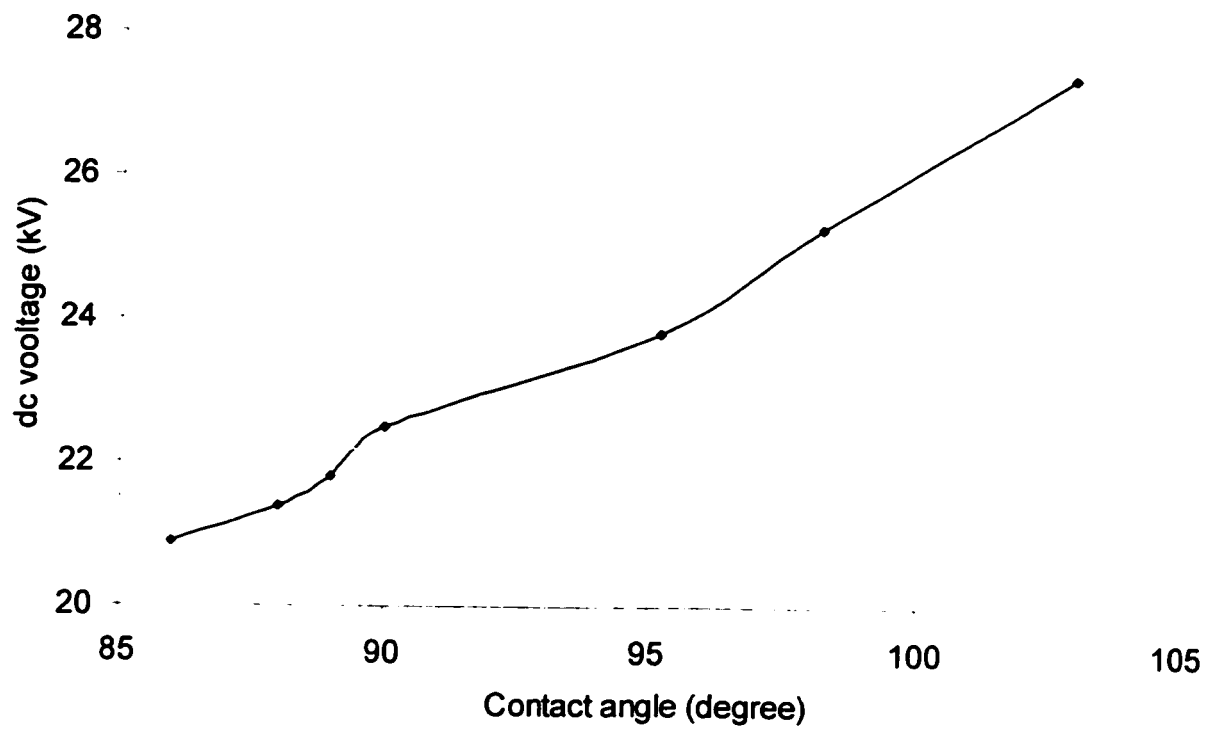


Fig. 5.14: Dependence of the DC flashover voltage on the contact angle, at $22\pm4^{\circ}\text{C}$.

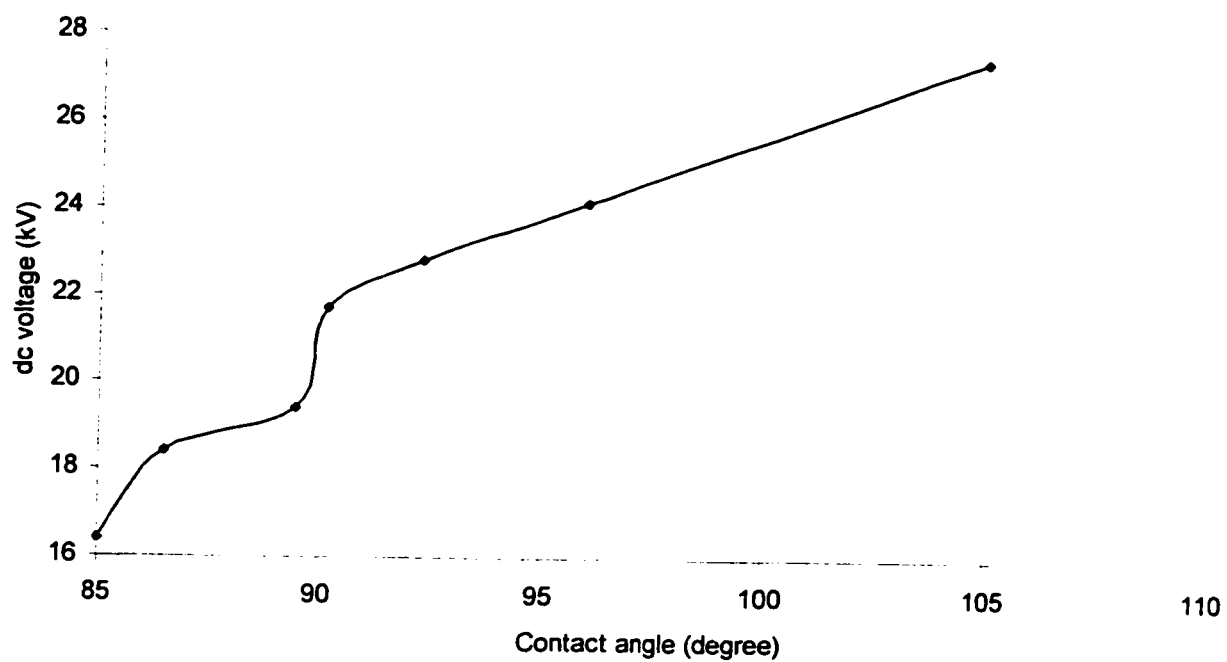


Fig. 5.15: Dependence of the DC flashover voltage on the contact angle, at $44\pm 2^{\circ}\text{C}$.

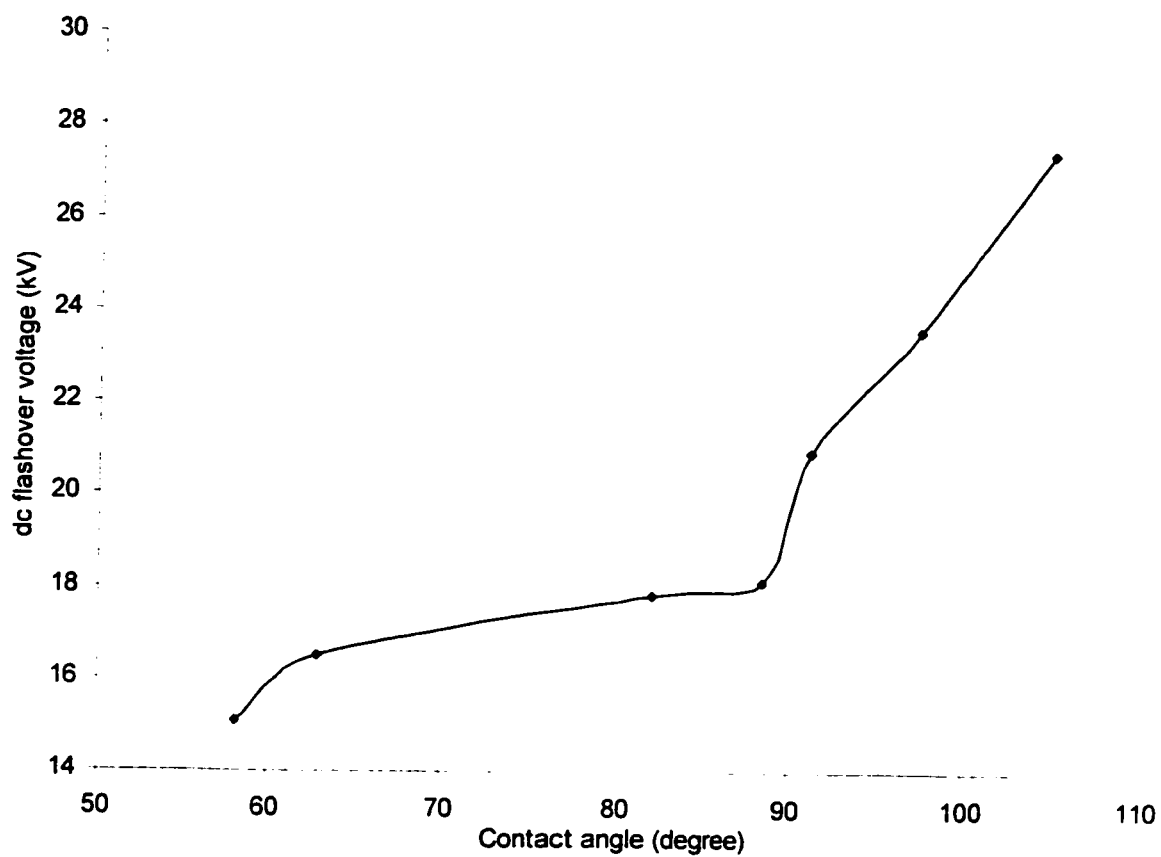


Fig. 5.16: Dependence of the DC flashover voltage on the contact angle, at $99\pm 2^{\circ}\text{C}$.

5.4 Summary of the Results

1. The contact angle decreased with increasing time of immersion.
2. The average surface roughness increased with the increasing time of immersion.
3. The peak surface roughness increased with the increasing time of immersion.
4. The ac and dc flashover voltage of PTFE decreased with increasing time of immersion.

Chapter 6

Recovery of Hydrophobicity in Polytetrafluoroethylene

6.1 Introduction

The increased usage of polymeric insulators has prompted much interest in the detailed investigations of the surface properties of insulating materials. The surface of polymer insulator is normally hydrophobic, but under stress of humidity and temperature it, gradually starts losing the hydrophobicity [3, 7]. This loss of hydrophobicity increases with the increasing temperature and time of immersion in the saline solution of water. The loss of hydrophobicity of polymer materials is attributed to uptake of water and oxidation of the polymer surface at higher temperature [3,7]. The result leads to deterioration of the insulator surface, loss of surface resistivity, increase in leakage current, followed by dry band arcing and tracking activity on the polymer surface and ultimately flashover leading to failure.

Polytetrafluoroethylene (PTFE) is widely used in cable insulation. The virgin PTFE is highly hydrophobic due to the strong carbon fluorine bond [21], but under wet or humid conditions and in the presence of pollution, a gradual loss of hydrophobicity occurs, which is accelerated at higher temperatures. A study on the loss of hydrophobicity due to aging of PTFE under different salinity of water and temperatures has been discussed in chapter 4. It was observed (chapter 4) that the hydrophobicity decreased with increasing salinity, temperature and time of immersion of saline solution. Recovery of

hydrophobicity normally starts after removal of these stresses. When the surface of an insulator recovers its hydrophobicity, the leakage current and the subsequent dry band arcing are suppressed. In this chapter the recovery of hydrophobicity of PTFE after aging by immersion in saline solutions having different conductivity and at different temperatures, is reported.

6.2 Experimental Procedure

PTFE cylindrical rods of 9.4 ± 0.6 mm in length and 19 mm in diameter were aged by immersion in different saline solutions, at various temperatures and for extended durations. Four different conductivities of the saline solutions of 0.005, 1, 10 and 100 mS/cm were used for the aging of PTFE. Five different temperatures of $0 \pm 1^\circ\text{C}$, $22 \pm 4^\circ\text{C}$, $44 \pm 2^\circ\text{C}$, $73 \pm 2^\circ\text{C}$ and $99 \pm 2^\circ\text{C}$ were applied for the aging of PTFE. The specimens of PTFE rods were also aged in air at these five temperatures. After immersion of about 5,000 h in saline solutions at different temperatures, saturation was reached in the values of the contact angle. The aged specimens were then removed from the saline solutions and kept in clean and dry glass containers in air at room temperature $22 \pm 4^\circ\text{C}$ and $55 \pm 15\%$ humidity for up to 3,500 h. The saturated value of contact angles are also used as initial reading for the recovery process. The weight, the surface roughness and the contact angle of the aged specimens were also recorded for the recovery process. Initially the contact angle, the surface roughness and change in the weight during the recovery of hydrophobicity were taken within a very small interval of time and then gradually increased up to 250 hrs.

6.3 Recovery of the Contact Angle

Figs. 6.1 to 6.5 show the recovery of the contact angles of PTFE as a function of time in air at 22 ± 4 °C, for specimens which were previously aged in different saline solutions, respectively at 0 ± 1.5 , 22 ± 4 , 44 ± 2 , 73 ± 2 and 99 ± 2 °C. The initial low values of the contact angle shown in Figs. 6.1 to 6.5 were measured immediately after removal of respective specimen from the saline solutions. The time of recovery of the contact angle was extended up to 3,500 h. The contact angle of the specimens which had been aged in air at temperatures up to 99 ± 2 °C was also measured during the recovery, as a function of time at 22 ± 4 °C. The variation in the contact angle was negligible at all the temperatures during the aging and subsequently during the recovery in air and remained at $104\pm 5^\circ$.

It may be observed from Fig. 6.1 that the contact angle recovered steadily from 92° and 88 to 104° in 2,000h for the PTFE specimens aged in 0.005 mS/cm and 1 mS/cm saline solutions respectively at 0 ± 1.5 °C. The time of the recovery process of the PTFE specimens aged in 10 and 100 mS/cm at this temperature were 2,500h. The final values for these specimens were 100 and 96° from 85 and 84° respectively. However, they did not achieve full recovery up to $108\pm 5^\circ$ even after 3,500 h. This may be attributed to the adsorption of highly saline solutions by the polymer surface and weakening of C-F bonds during the aging process thereby rendering its surface more hydrophilic [7, 21].

Figs. 6.2 to 6.4 shows that the contact angle increased gradually until saturation is reached. The saturated values of the contact angle were dependent on the level of the combined thermal and salinity stresses to which the specimens were subjected [7]. The

saturated value of contact angle for the higher salinities and temperatures were lower than the lower salinities and lower temperature. The same observations were reported in the studies on EPDM [3, 17], Nylon [9], XLPE and HDPE [7, 18]. The full recovery of the contact angle took place, only for the lower temperatures and lower salinities (Figs. 6.2 to 6.4).

Fig. 6.2 shows that for $22\pm4^{\circ}\text{C}$ the contact angle recovered to its original value of 104° only for 0.005 mS/cm after 2,500h and for 1 mS/cm after 3,000h. The saturated values of the contact angles for the higher salinities (10 and 100 mS/cm) were 102° and 99° respectively (Fig. 6.2).

Fig. 6.3, it can be observed that at 44°C , the contact angle reached its original value only for 0.005 mS/cm saline solution, after 3,000h, while for 1, 10 and 100 mS/cm salinities the saturated values were, 101° , 99° and 97° respectively. The saturated values of the contact angles were lower than the original value of the virgin specimens.

Fig. 6.4 shows the recovery of the contact angles in air at $22\pm4^{\circ}\text{C}$ after immersion in different saline solutions at $73\pm2^{\circ}\text{C}$. It took about 3000h to reach the saturated values of the contact angle. The contact angle recovered from 88° to 99° for 0.005 mS/cm, from 83° to 99° for 1 mS/cm and from 78° and 62° to 97° and 95° respectively for 10 and 100 mS/cm salinities.

Fig. 6.5 shows the recovery of the contact angles for the specimens of PTFE aged at $99\pm2^{\circ}\text{C}$ in different saline solutions. The recovery of the contact angles at $99\pm2^{\circ}\text{C}$ were relatively slower than the lower temperature. It took about 3,000h for 0.005 and 1.00 mS/cm salinities and 3,500h for 10 and 100mS/cm, to reach the saturated values of the contact angles. The slower recovery of contact angles may be partly due to the weakening

of C-F bonds and slower rates of desorption of water from these specimens [7, 21]. The contact angles recovered from 82°, 80°, 64.5° and 39° to 94°, 94°, 88° and 85°, after 3,000h for the specimens aged in 0.005, 1 mS/cm saline solution and 3,500h for the specimens aged in 10 and 100 mS/cm saline solutions at 99±2°C temperature.

Fig. 6.6 shows the saturated values of the contact angles of PTFE specimens after the recovery for 3,500h in air at 22±4°C, after aging in saline solutions of different conductivities and at different temperatures. It is observed from Fig. 6.6 that the saturated values of recovered contact angles decreased with increasing salinity for a fixed temperature. Similarly, for a fixed salinity level, the saturated values of contact angles decreased with increasing temperature. The lower saturated values of the contact angles at 99±2°C may be attributed to, weakening of the C-F bonds, increased water uptake and degradation of the surface during aging at higher temperature and salinity levels.

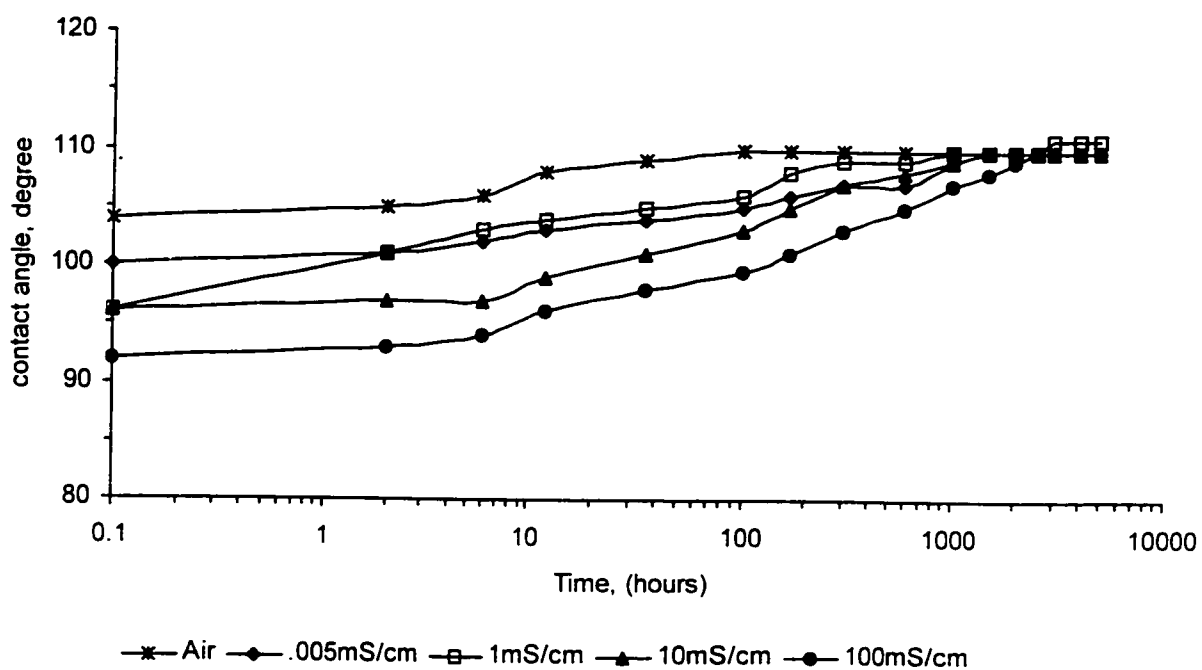


Fig.6.1. Recovery of contact angle on PTFE in air at $22\pm4^{\circ}\text{C}$ after immersion in different saline solutions for 3000 hours at $0\pm1.5^{\circ}\text{C}$.

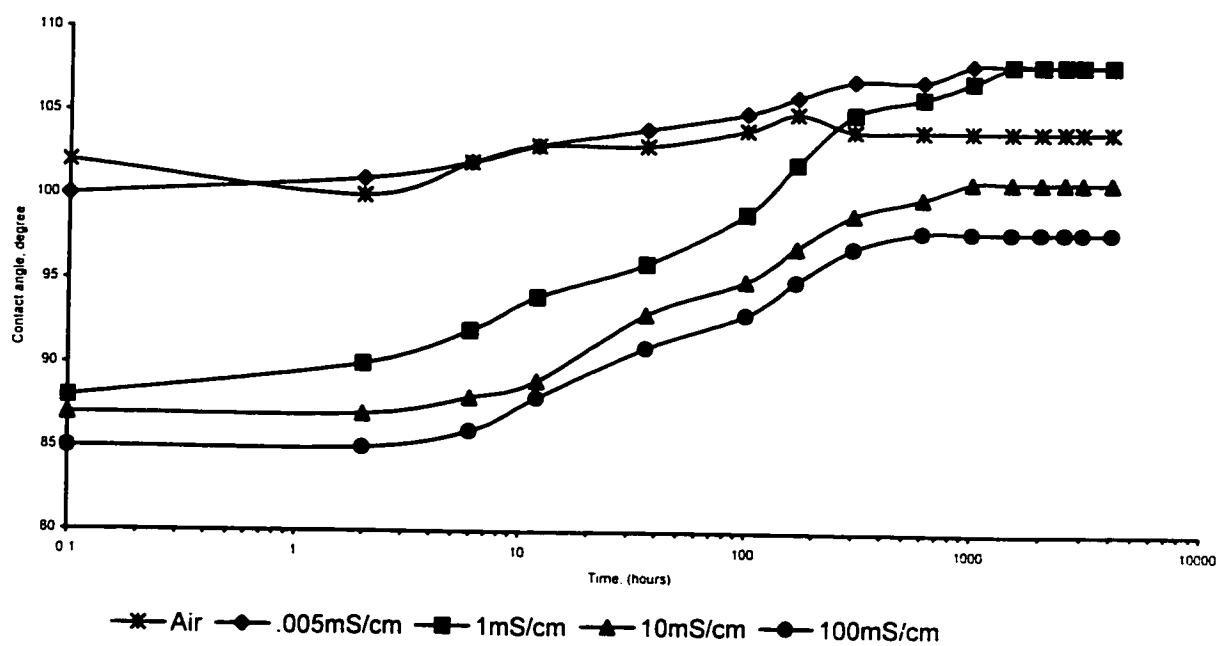


Fig. 6.2:Recovery of contact angle on PTFE in air at $22\pm4^{\circ}\text{C}$ after immersion in different saline solutions for 3000 h at $22\pm4^{\circ}\text{C}$.

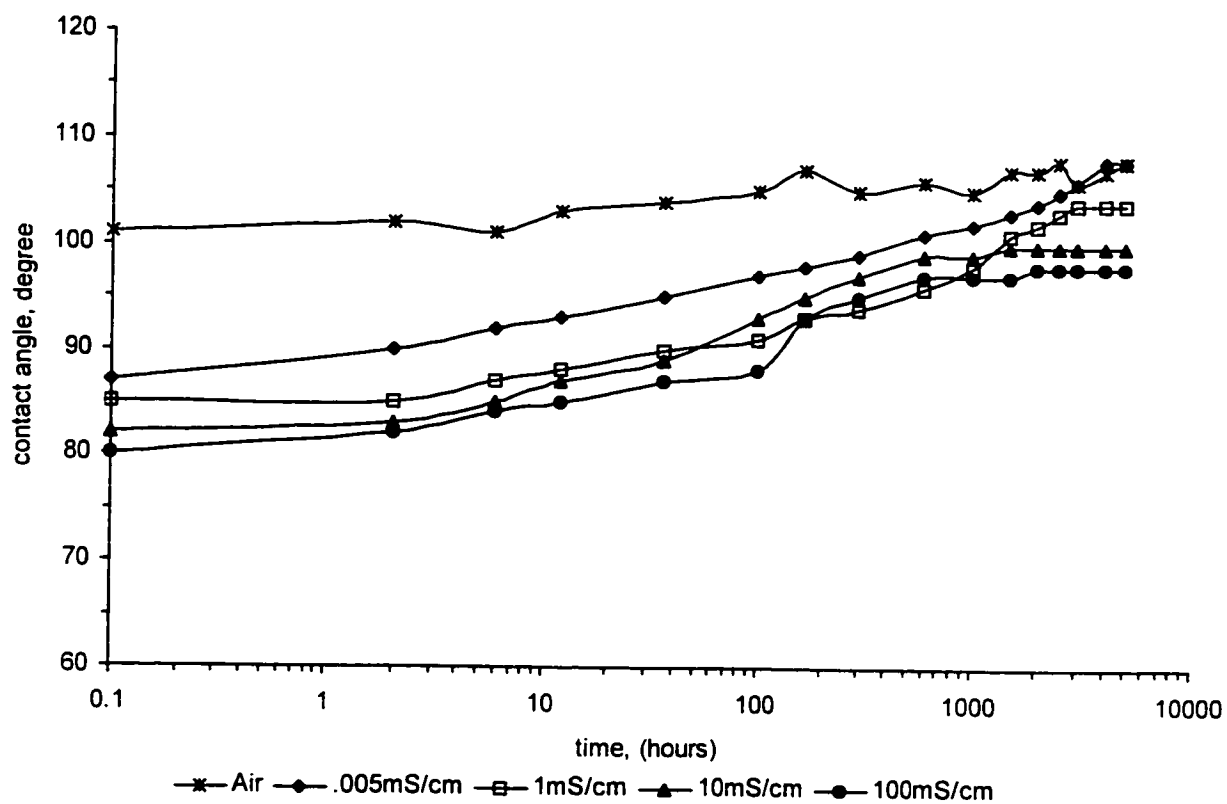


Fig. 6.3: Recovery of contact angle on PTFE in air at $22\pm4^{\circ}\text{C}$ after immersion in different saline solutions for 3000 hours at $44\pm2^{\circ}\text{C}$.

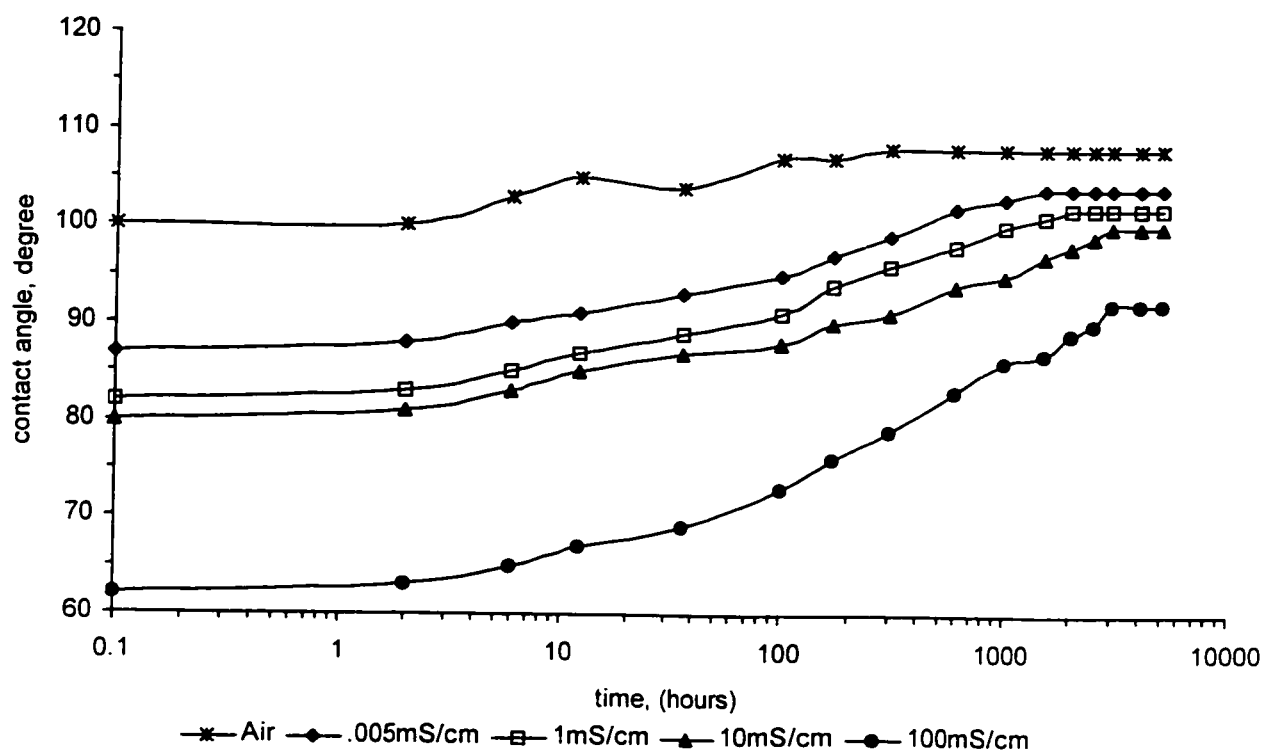


Fig. 6.4: Recovery of contact angle on PTFE in air at $22\pm4^{\circ}\text{C}$ after immersion in different saline solutions for 3000 hours at $73\pm2^{\circ}\text{C}$.

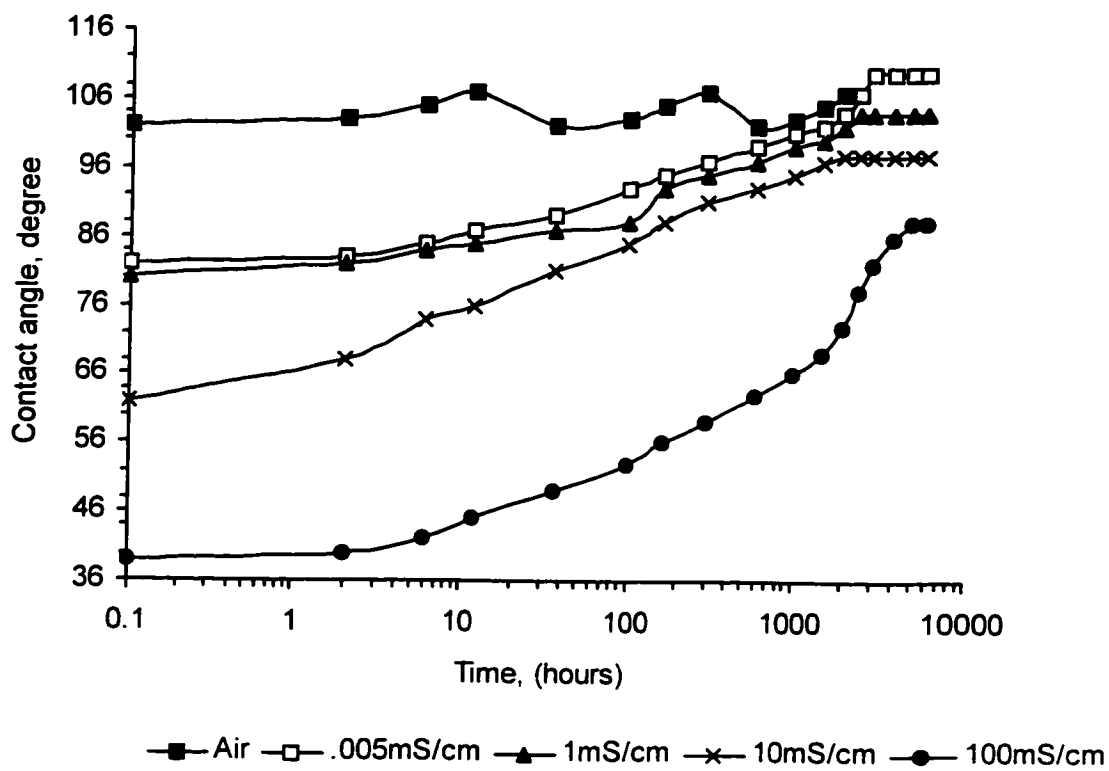


Fig. 6.5: Recovery of contact angle on PTFE in air at $22\pm4^{\circ}\text{C}$ after immersion in different saline solutions for 3500 h at $99\pm2^{\circ}\text{C}$.

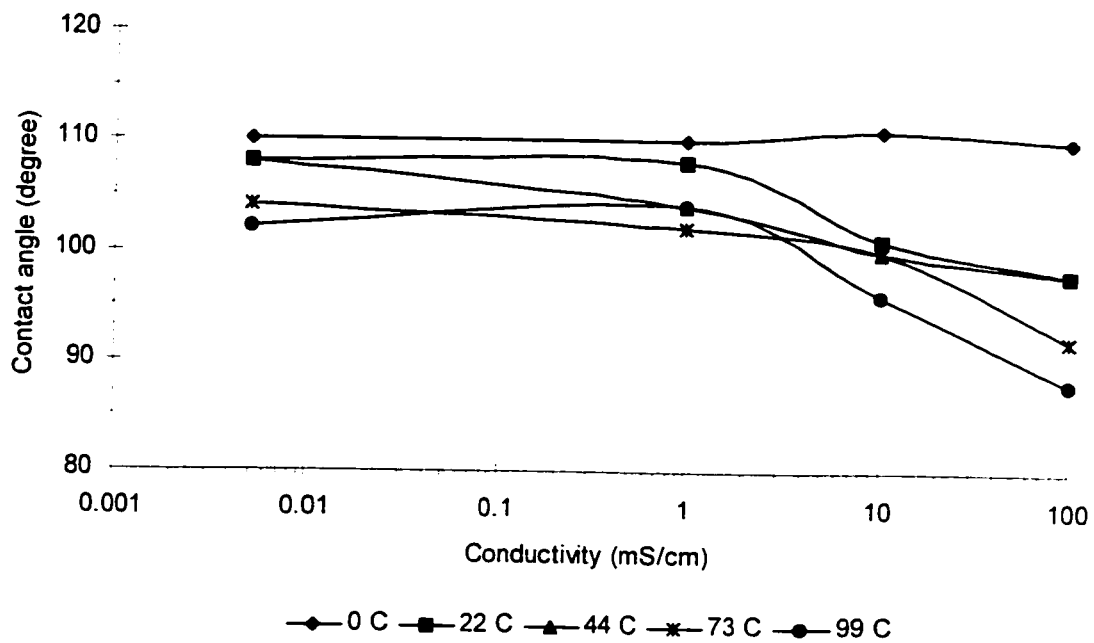


Fig. 6.6: Contact angle on PTFE after recovery for 3500 hours in air at $22\pm4^{\circ}\text{C}$ and subsequent to aging in saline solutions of different conductivity and at different temperatures.

6.4 Reduction in the Weight during Recovery

The percentage (%) decrease in the weight of PTFE specimens during the recovery after aging, in different saline solutions and temperatures were also measured.

Table 6A shows the percentage decrease in the weight of PTFE specimen aged in different salinities and temperatures after 3,500h of the recovery in air at $22\pm4^{\circ}\text{C}$ and $50\pm15\%$ humidity level. The percentage decrease in the weight of PTFE specimens was higher for higher salinities and temperatures. The maximum percentage reduction in the weight was 0.09 % and was observed for the specimen aged in 100mS/cm solution at 99°C . These specimens were previously immersed for 5,000h in solutions having different conductivities and at different temperatures. Table 6A shows that the percentage reduction in the weight at a fixed aging temperature increased with the increasing conductivity. At a fixed conductivity, the percentage reduction in the weight increases with the increasing temperature. It was observed in the previous chapter (Chapter 4) that the maximum increase in the weight after completion of aging was noted for 100 mS/cm salinity at 99°C and the maximum reduction in the weight is also observed for the same salinity and temperature. But the net increase in the weight after loss and recovery of hydrophobicity is also observed for the specimen aged in 100 mS/cm salinity and 99°C and recovered in air at 22°C . This is because at a fixed conductivity of the saline solution the intake of water was observed to be higher with higher temperatures and the loss of water during the recovery in air is also observed higher. It can be observed from Fig. 6.6 and Table 6A that the level of the recovered contact angle also depends on the amount

Table 6A

Percentage change in the weight of PTFE during recovery in air at 22±4°C for

3500h. Specimens were initially immersed at different conductivities

at 99±4°C for 5000 h.

S.No	Conductivity	% Change in weight from start of recovery	% change in weight after saturation of contact angle during recovery with virgin specimen, before aging
1	0.005	0.04	0.02
2	1	0.04	0.03
3	10	0.09	0.06
4	100	0.11	0.09

Table 6B

Percentage reduction in weight of PTFE after recovery in air at 22±4°C

for 3500h. Specimens were initially immersed at different

temperatures at 100mS/cm for 5000 h

S.No	Temperature	% Reduction in the weight	% change in weight after saturation of contact angle during recovery with virgin specimen, before aging
1	0±1.5°C	0.04	0.02
2	22±4°C	0.04	0.03
3	44±2°C 0	0.07	0.06
4	73±2°C 0	0.10	0.07
	99±2°C	0.11	0.09

of water lost from the specimens during the recovery in air [7]. To observe the effects of humidity at 22°C, in air, the measurements of a control specimen over the duration of these experiments were taken. The maximum variation in the weight of PTFE specimen during these experiments was $\pm 0.001\%$, which is negligibly small.

5.5 Surface Roughness

During the recovery of the contact angle in air at 22 ± 4 C°, the Average Surface Roughness (ASR) were also measured. The ASR, of the virgin specimens before immersion was 1.12 ± 0.32 μm . Fig. 4.8 (Section 4.3.5, Chapter 4) shows the variation of the ASR as a function of conductivity of the saline solution at different temperatures after immersion of PTFE specimens for 5,000 h. The ASR increased after immersion in the saline solution due to the intake of the saline solutions at higher conductivity levels and at higher temperatures. It increased from 1.12 ± 0.32 μm . to 1.73, 1.82, 2.42 and 2.94 and 3.12 μm , respectively for 23, 44, 73, and 99°C for 100 mS/cm.

Fig 6.8 shows the average surface roughness of PTFE specimen immersed in 100 mS/cm saline solution, as a function of temperature both immediately after immersion for 5,000h and subsequent to the recovery in air at $22 \pm 4^\circ\text{C}$ for a further 3,500h. For the PTFE specimen aged in 100 mS/cm, the surface roughness decreased to 1.37 μm , 1.42 μm , 1.58 μm , 1.78 μm , and 2.05 μm , for 0 ± 1.5 , 22 ± 4 , 44 ± 2 , 73 ± 2 and $99 \pm 2^\circ\text{C}$, respectively after 3,500h of recovery in air at $22 \pm 4^\circ\text{C}$. It will be observed from Fig. 6.8 that the surface

roughness decreased substantially after drying in air but the values were still above those of the virgin specimen ($1.12\text{ }\mu\text{m}$). The slightly higher surface roughness after 3,500h of recovery in air with the increasing temperatures (Fig.6.8), also contributed to the lower values of saturated contact angles at 100 mS/cm (Fig. 6.6).

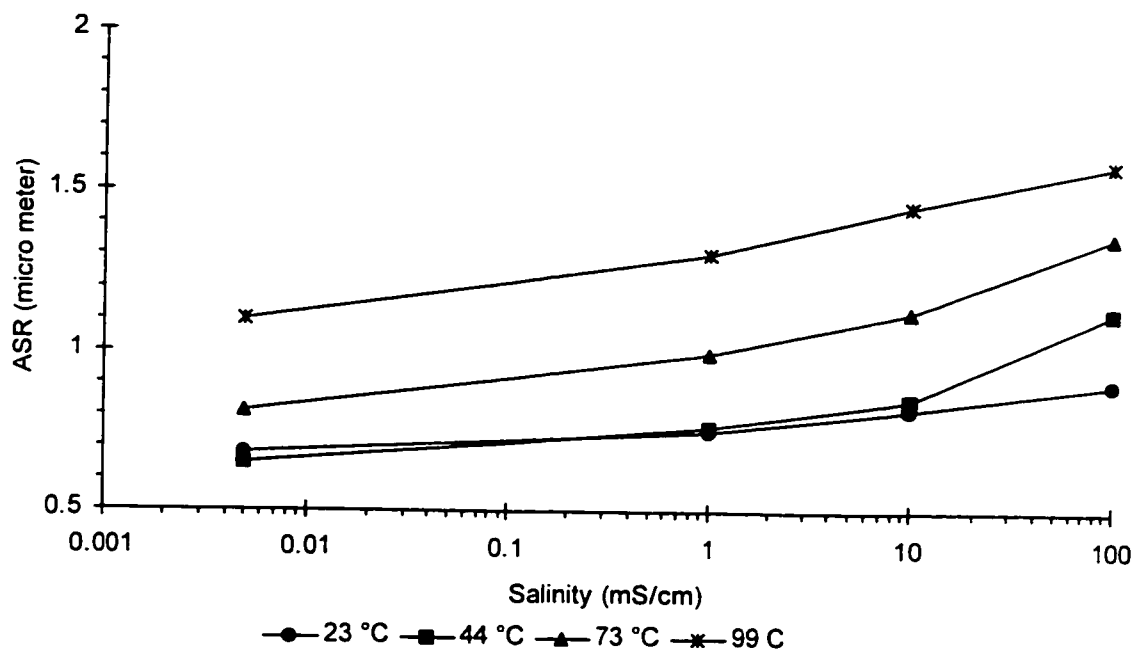
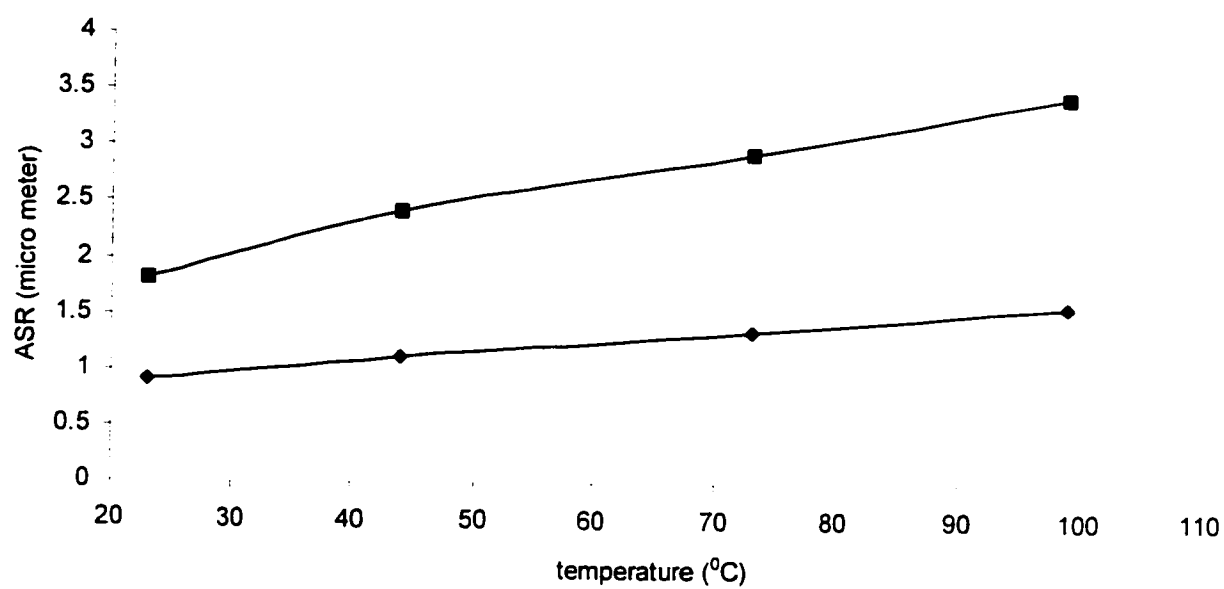


Fig. 6.7. Average surface roughness against different salinities at 22 ± 4 °C, 44 ± 2 °C, 73 ± 2 °C and 99 ± 2 °C, after recovery at 22 ± 4 °C.



—◆— ASR, after recovery of C.A. —■— ASR, after loss of hydrophobicity

Fig. 6.8. Average surface roughness against temperature at 100 mS/cm saline solution, after loss and recovery of hydrophobicity, at 22 ± 4 °C

6.6 Confidence Interval of the Contact Angle during Recovery:

Fig 6.9 shows the maximum variation and the confidence interval of the contact angle during recovery in air at $22\pm 2^{\circ}\text{C}$, after immersion in 100mS/cm at $99\pm 2^{\circ}$. The maximum variation of 95% confidence interval is $\pm 2.5^{\circ}$; where as the maximum variation of contact angle is $\pm 4^{\circ}$. This variation may be attributed to heterogeneity and method of measurement of contact angle. The standard normal distribution was used to calculate the 95% Confidence Interval (CI) by applying MS office 97 (XL).

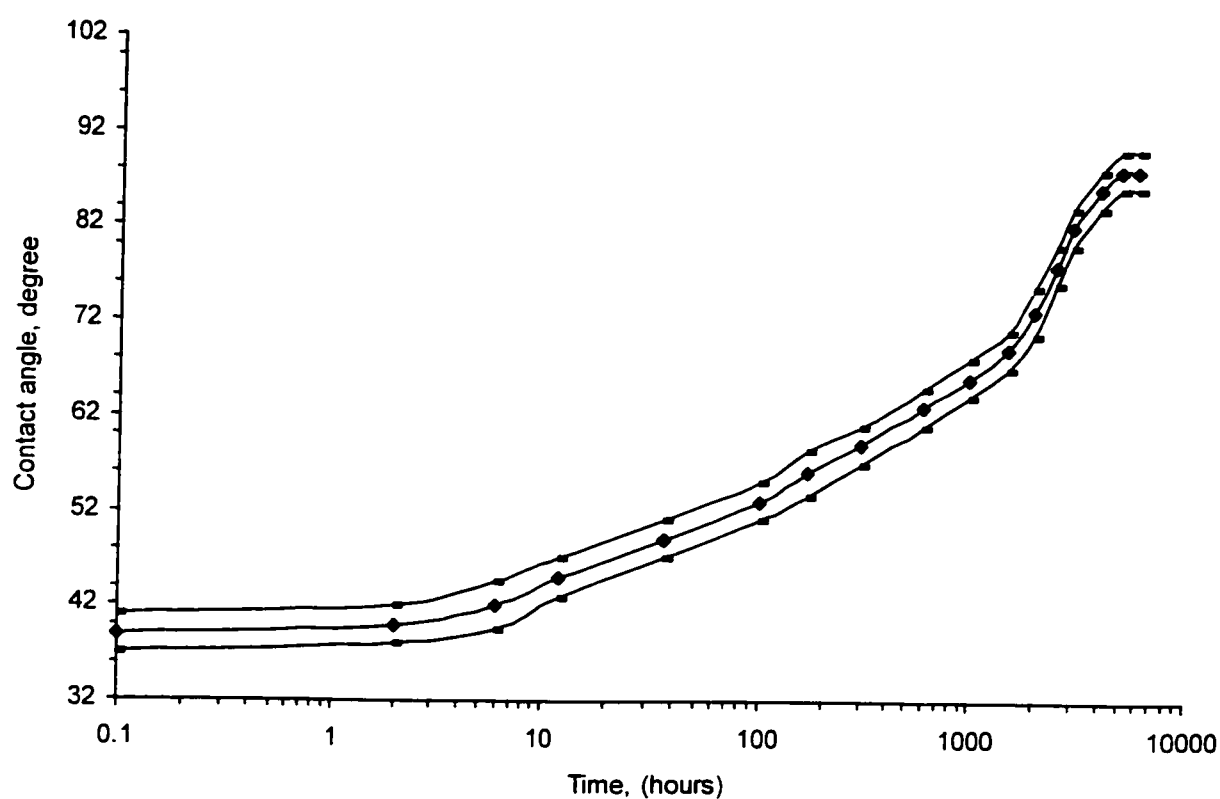
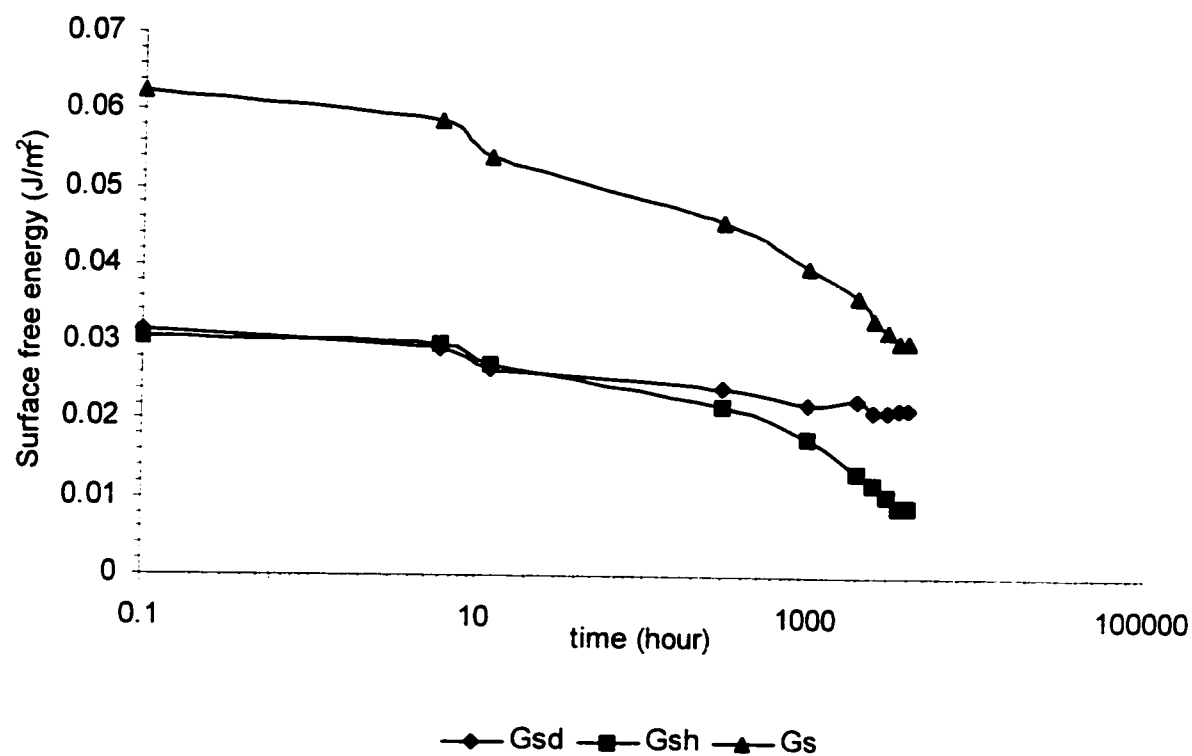


Fig. 6.9 95% confidence interval for standard normal distribution of contact angles on PTFE during recovery at $22\pm^{\circ}\text{C}$ after immersion in saline solution of 100 mS/cm at $99\pm 2^{\circ}\text{C}$. Continuous lines around the mean value curve represent 95% confidence interval.

6.7 Calculation of Surface Free Energies and its Components During the Recovery

The surface free energies were calculated previously during the loss of hydrophobicity (Chapter 4). In chapter 4, it was observed that the surface free energy increases with the decreasing contact angle. During the recovery, the surface free energies were also calculated by using the developed program, Surf Test, at $99\pm 2^\circ$ for 100mS/cm saline solution. It can be observed from Fig. 6.9. that the surface free energies decreased with increasing time of recovery till saturation was reached. It was expected according to equation 3.1, that the larger the contact angle the smaller will be the value of surface free energy and its components.

Fig. 6.10: Surface free energy and its components during recovery of contact angle on



PTFE in air at 22 ± 4 °C, after immersion in different saline solutions for 3500 hours at 99 ± 2 °C.

6.8 Summary of the results

1. The contact angle and hence the hydrophobicity increased in air at 22°C during the recovery.
2. The contact angle recovered fully for the lower salinity and lower temperature PTFE specimen.
3. The PTFE, which were previously immersed in higher saline solution with higher temperature, didn't recover the original contact angle.
4. The Average surface roughness decreased with the recovery of the contact angle. none of the specimen recovered its original value.
5. The weight of the PTFE specimen decreased with the recovery of the contact angle.

Chapter 7

Conclusion

7.1 Loss of Hydrophobicity:

7.1.1 Conclusion:

The loss of hydrophobicity is dependent on time of immersion in saline solution, salinity and temperature of the saline solution. With increasing temperature, salinity and time of immersion the hydrophobicity of PTFE decreased till saturation in the value of contact angle was reached. The coefficient of diffusion of water into PTFE is also increased with increasing salinity, temperature and time of immersion. The loss of hydrophobicity was due to increased surface roughness, uptake of water and weakening of C-F bonds. The loss of hydrophobicity due to oxidation of the PTFE surface could not be observed by FTIR spectra. The microphotograph by Scanning Electron Microscopy further proved the increasing degradation of the surface with increasing salinity and temperature. The contact angle decreased with increasing surface roughness and surface free energies of PTFE.

7.2 Effect of Water Salinity, Temperature and Time of Immersion on the ac and dc flashover voltage:

7.2.1 Conclusion:

This study shows that the ac and dc flashover voltages decreased after immersion in a saline solution and with increasing temperatures, [5, 6] The decrease in the flashover voltage correlates with the loss of hydrophobicity and increased surface roughness due to aging.

7.3 Recovery of Hydrophobicity:

7.3.1 Conclusion:

The hydrophobicity fully recovered for the PTFE specimen previously aged in lower salinity and lower temperature and recovered in air at 22°C. The contact didn't recovered fully for the higher salinity and temperature. This may be attributed partially to intake of water from saline solution and weakening of C-F bond due to aging of the surface at higher temperature and salinity.

The recovery of hydrophobicity is also correlated with the decrease in surface roughness and surface free energy.

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9. Publication in Support of the Thesis

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Presentation in Support of Thesis.

- [3] H.Z. Syed "Aging of PTFE insulation" Cage Club Student Conference 1998, University of Western Ontario, London, Ontario, Canada.

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