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Critical Behavior of the Electrical Conductivity for the Binary Mixture of Water and Phenol

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Dedication

To my mother who helped me in every single moment in my life, as well as, my husband who helped me to achieve this work.

This thesis is also dedicated to my brothers Amin, Ibrahim, Ahmed and Mohammed. To my dear sister Huda with love.

Acknowledgement

I would like to express my sincere gratitude to my advisor Prof. Issam Rashid Abdelraziq and to my co-supervisor Dr. Iyad Sasdeddin for the continuous support of my research, for their patience, motivation, enthusiasm, and immense knowledge. Their guidance helped me in all the time of research and writing of this thesis.

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الإقرار

أنا الموقع أدناه مقدم الرسالة التي تحمل العنوان

Critical Behavior of the Electrical Conductivity for the Binary Mixture of Water and Phenol

اقر بأن ما اشتملت عليه هذه الرسالة إنما هو نتاج جهدي الخاص، باستثناء ما تم الإشارة إليه حيثما ورد وأن هذه الرسالة ككل أو جزء منها لم يقدم من قبل لنيل أية درجة أو بحث علمي أو بحثي لدى أية مؤسسة تعليمية أو بحثية أخرى.

Declaration

The work provided in this thesis, unless otherwise referenced, is the researcher's own work, and has not been submitted elsewhere for any other degree or qualification

Student's name: إسم الطالبة : هبة زهرة

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Date: التاريخ : ٢١ /٥/ ٢٠١٥

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List of Symbols and Abbreviations

Symbol	Abbreviation
η	Dynamic Viscosity
τ	Shear stress
$\dfrac{\partial u}{\partial x}$	The rate of deformation or the velocity gradient
ν	The kinematic viscosity
ρ	Density of the fluid
σ	The electrical conductivity
σ_∞	The maximum electrical conductivity which is at infinite temperature
E_a	The activation energy for electrical conduction
K_B	Boltzman constant
VFT	Vogel-Fulcher-Tammann
T	The temperature in Kelvin
Λ_m	Molar electrical conductivity
c	The molar concentration
V_{SOL}	The volume of the solution
N	Number of moles
X_{phenol}	The concentration by weight of phenol
ρ_{phenol}	The experimental density of phenol
V_{phenol}	The volume of phenol
ρ_{water}	The experimental density of water
V_{water}	The volume of water
η_1	The viscosity of water – phenol mixture at temperature (T)
ρ_1	The experimental density of water – phenol mixture at temperature (T)
t_1	The time required mixture to flow from the upper mark to the lower mark in the viscometer at temperature (T)
η_2	The viscosity of water at temperature (T)
ρ_2	the experimental density of water at temperature (T)
t_2	The time required for water to flow from the upper mark to the lower mark in the viscometer at temperature (T)
α	The decoupling constant of the ions
K	Temperature – dependent constant

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Abstract

The dynamic viscosity was measured for water-phenol binary mixture at several concentrations and temperatures to determine the critical concentration and critical temperature. The critical concentration was determined at $X_C = 33.90\%$ by weight of phenol, the critical temperature was $T_C = 67.0°C$ determined from the viscosity measurements. The electrical conductivity measurements showed that $T_C = 67.8°C$ at $X_C = 33.90\%$ by weight of phenol. The electrical conductivity measurements for various concentrations (0.00%, 10.17%, 20.80% and 50.00%) by weight of phenol were fitted by using Arhenius equation. The activation energy and the electrical conductivity at infinite temperature were calculated for several concentrations. The electrical conductivity as a function of temperature was fitted by using power law above the critical temperature and critical concentration. The noncritical part of the measured electrical was found to be 30.26μS/cm and the critical exponent value was 0.032. The molar electrical conductivity was calculated and plotted by using Walden rule. The slope of Walden plot was found to be 0.9439. The water – phenol mixture is classified as a poor ionic mixture.

Chapter One

Introduction

Several properties for huge numbers of binary liquid mixtures have been studied near the critical point. Some of these properties are: viscosity, density, ultrasonic propagation, ultrasonic velocity, sound attenuation, electrical conductivity and other physical and thermodynamic properties of binary liquid mixtures (Fixman, 1962; Dikio *et al*, 2012; Weng-Zheng *et al*, 2005).

1.1 Binary Mixtures:

A mixture is a system contains two or more different materials. There are three types of mixtures depending on the physical state of the components: gas, liquid and solid mixtures.

A liquid mixture consists of two or more liquids. The mixture that contains two different liquids is called a binary liquid mixture such as water-ethanol, methanol-cyclohexane and water-phenol (Sadus, 1992; Sadus, 1999).

If a third component is added to the binary mixture, a ternary mixture will be formed for example (water-acetone-chlorobenzene) mixture.

The liquid binary mixture has two types depending on its phase: homogenous and heterogeneous. The phase of the binary liquid mixture become homogenous and completely miscible at a critical point where the coexisting phases become indistinguishable. Critical point is the term called when both temperature and concentration are stabilized at their critical values. The two liquids behave as one liquid with new physical characteristics. The

temperature and concentration at which the two liquids become completely miscible at all proportions are called critical temperature and critical concentration (Rice,1949; Sadus,1992; Sadus,1999).

The mixture is heterogeneous if its components does not mix with each other and completely or partially immiscible, for example mercury and water.

The solubility of the binary liquid mixture can be classified as:

1. Completely miscible, for example water and alcohol.

2. Partially miscible such as water and phenol.

3. Completely immiscible for example carbon disulphide and water.

1.2 Literature Review:

Many physicians are concerned about the behavior of the physical and thermodynamic properties of the binary mixture near the critical point. Experiments show that certain properties such as viscosity, density, ultrasonic velocity and electrical conductivity have anomalous behavior at the critical point (Kittany, 2014; Oleinikova and Bonetti, 2002; Abdelraziq, 2002; Abdelraziq, 1990).

Theories are available to study the ultrasonic absorption, velocity and viscosity. Some of these theories are dynamic scaling theory introduced by Ferrell and Bahattacharjee, renormalozation group theory by Kroll and Ruhland, mode-coupling theory by Fixman, Shiwa and Kawaski, Mistura and Chaban (Perl and Ferrell, 1972; D'Arrigo *et al*, 1977; Bhattacharjee *et al,* 1981; Abdelraziq, 2001).

Viscosity is a property for all liquids and it is a measure of the internal resistance of the liquid to flow which influenced by different factors, such as

catalyst, temperature, shear rate, molecular weight, pressure and concentration (Symon and Keith, 1971).

 The effect of temperature on dynamic viscosity for some vegetable oil was studied by Abramovic and Klofutar by using modified versions of the Andrade equation (Andrade, 1930; Abramovic and Klofutar, 1998).

The relation between the viscosity of liquid and its chemical nature was studied by Baranett team (Baranett *et al*, 1896). Vogel and De Guzman introduced a three-constant representation for the liquid viscosity as a function of temperature (De Guzman, 1913; Vogel, 1921).

 The anomaly of shear viscosity for the binary mixture was detected as a function of temperature and concentration (Senger, 1972; Abdelraziq, 1992; Abdelraziq, 2000; Abdelraziq, 2002). Ferrell observed that a finite shift in frequency and temperature brings the critical viscosity down from its diverging critical point (Ferrell and Bhattacharjee, 1983). Brown studied the ultrasonic velocity at various concentrations and temperatures for a mixture of aniline and cyclohexane (Brown and Richardson, 1959).

A method was developed by Bridgman to determine the viscosity of liquids over a wide range pressure at different temperatures and the method has been applied to 43 liquids in the pressure range between atmospheric and 12000 kg/cm at 30°C and 75°C (Bridgman, 1925).

Ferrell has proposed a closed-form for the crossover function which connects the asymptotic critical behavior of the viscosity away from the critical point (Bhattacharjee, 1981).

Abderaziq has measured the shear viscosity coefficient for a binary mixture of nitroethane and 3-methylpentane above the critical temperature (Abdelraziq, 2002).

Bell reported the acoustic velocity and the viscosity on mixtures of polydimethylsiloxane with toluene, covering a frequency range of 1 - 100MHZ and a concentration range from 0 to 1 volume fraction of polymer (Bell, 1979).

The ultrasonic velocity and the shear viscosity were studied for the liquid mixtures of polyvinylpyrrolidon - water at different temperature and concentration and a frequency value of 21 MHZ. An increase in the velocity was noticed when the temperature and concentration were increased (Spickler *et al*, 1989).

Electrical conductivity measures the ability of the mixture to conduct electricity. It has large applications in the industry, especially in detecting the existence of ions in the mixture. The solutions containing ions are called electrolytes. These ions are formed by dissociation of acids, bases or salts in a certain solvent for example water. The water is a good conductor because it consists of hydrogen ions (H^+) and hydroxide ion (OH^-) (Down and Lehr, 2005).

Binary mixtures of sulphuric acid with nitromethane, nitrobenzene and O-, M- and n-mitrotoluene were prepared in the laboratory. Experimental values for the densities, viscosities and electrical conductivity were measured (Liler and Kosanovic, 1959).

The electrical conductivity near the critical point for the binary mixtures of water-phenol, KCl - dopedphenol - water and isobatyric acid-water were measured. A singular contribution to the electrical conductivity was observed. The experimental data was fitted by using the least - square method (Ching – Hao and Walter, 1976).

Vitali has determined the liquid - solid equilibrium temperatures for the binary mixture strontium nitrate-acetanide. The supercoiling phenomenon was observed in limited form at specific mole fraction range. The measurements of viscosity and electrical conductivity were performed at a mole fraction $\chi = 0.8745$ (Vitali and Berchiesi, 1989).

The measurements of the density, viscosity and electrical conductivity were carried out for the mixture of N-butylureand sodium trifluoro acetate (Castellani *et al*, 1992).

Binary solutions of molten calcium fluoride (CaF_2), barium fluoride (BaF_2), strontium fluoride (SrF_2), beryllium fluoride (BeF_2) and magnesium fluoride (MgF_2) were prepared by Kim and Sadoway in order to measure their electrical conductivity by using electrochemical impedance spectroscopy. The highest value for the electrical conductivity of alkaline - earth fluorides was obtained for CaF_2. At the same conditions, BeF_2 has the lowest electrical conductivity between the alkaline - earth fluorides (Kim and Sadoway, 1992).

Electrical conductivity measurements were carried out at different temperatures and compositions by Rodrigues. An increase in the electrical

conductivity when the concentration of the binary mixtures increased was observed (Rodrigues *et al*, 2000).

The electrical conductivity at different temperatures and concentrations were measured for the binary liquid mixtures of $NdBr_3$ − alkali metal bromide. The data for the electrical conductivity was fitted by the classical Arrhenius equation (Gadzuric et al, 2004). In the next year, Ziolek and his group measured the electrical conductivity of the liquid binary $LaBr_3$ - alkali metal bromide, but the data for the electrical conductivity didn't obey the classical Arrhenius equation. The results were discussed in terms of complex formation in the melts (Ziolik et al, 2005).

The dependence of the electrical conductivity on temperature was studied for all $EuBr_2$ - alkali metal bromide liquid mixtures over a wide range of compositions. The activation energy was calculated for all liquid mixtures. It was found that Arrhenius equation does not fit the experimental data for any mixture (Rycerz *et al*, 2008).

Geng has fitted the dependence of electrical conductivity on temperature in the form of Arhenius equation and has examined the effect of concentration of ionic liquid on the electrical conductivity (Geng *et al*, 2008). The electrical conductivity for isobutric acid − water mixtures with added Zn^{2+} and SO_4^{2-} ions was measured at various temperatures and compositions ranges. The dependence of the electrical conductivity on the temperature was discussed by Arrhenius law over the whole range of compositions (Hadded and Bouanz, 2008).

The aqueous binary systems of [Bmim][BF$_4$] and [Bmim][PF$_4$] were prepared by Ya - Hung and his group to measure the molar heat capacity and electrical conductivity. The electrical conductivity data was measured from 293.2K to 353.2K with uncertainty of 0.001mS/cm (Ya – Hung *et al*, 2009). The electrical conductivity and dynamic viscosity for the binary mixture of 1-alkyl-3-methyl imidazolium tetrafluoroborata, [CnMIM] [BF$_4$] with water were measured at 288K, 298K, 308K and 318K and at different concentrations. There was an increase in the viscosity and electrical conductivity with increasing the concentration. A relation between the viscosity and the electrical conductivity was found by using Walden's rule. The molar conductivity and viscosity deviation were found and fitted with suitable equations. By comparing the data of this study with previous data, a good agreement was observed (Rilo *et al*, 2010).

Electrical conductivity and viscosity have been measured at 25 ^{0}C for binary mixtures of the tetrafluoroborate and the dicynamide salts by Stoppa (Stoppa *et al*, 2010).

Electrical conductivity as a function of temperature for four [Bmim(1-n-butyl-methyllimidazolium)] bases ionic liquids was described by using a modified version of VFT equation. A commercial conductivity meter with uncertainty ±1% was used to measure the electrical conductivity (Lin *et al*, 2010).

At the critical temperature and critical concentration, the electrical conductivity measurements for the binary liquid mixture of triethylamine + water were performed. The electrical conductivity as a function of temperature was explained by Vogel-Fulcher-Tammann relation. The

electrical conductivity for the pure components have a monotous deviation from VFT relation (Toumi *et al*, 2011).

The ternary mixture of 1-butyl-3-methylimidazolium hexafluorophosphate ([bmim][PF$_6$]) + water + ethanol and ([bmim][PF$_6$]) + water + acetone were prepared in the laboratory by Hezave and his group. The electrical conductivity at temperature ranges from 288.15 to 308.15 K was measured (Hezave *et al*, 2011).

A relation between the electrical conductivity and the static permittivity was detected for sixteen dipolar liquids and their binary mixtures with amides over the entire compositions range. An increase in the electrical conductivity was observed while increasing the permittivity. A linear variation between the conductivity and the concentration for the binary mixtures was investigated (Choudhary and Sengwa, 2012).

The electrical conductivity of the binary ionic liquid of caprolactum tetrabutylammonium halide (F, Cl and Br) with solvents (water or ethanol) was measured to study the structure of ionic liquids and their interaction with the solvents. An increase in the electrical conductivity of the ionic liquid with increasing temperature was noticed (Daun *et al*, 2012).

A binary mixture of ionic (RTIL) and molecular (acetonitrile) co-solvent was prepared. The ionic (RTIL) added to the binary mixture with different concentration ranges from 10% to 50% and the temperature varies from 278.15K to 328.15K. Electrical conductivity was measured and empirical correlations between ionic structure, concentration and temperature were calculated (Kalugin *et al*, 2013).

Seven binary systems of 1-ethyl-3-methyl imidazolium alkyl sulfate with water or ethanol were prepared and their electrical conductivity was measured at four different temperatures (288.15, 298.15, 308.15 and 318.15K) and for all ranges of concentrations. The molar conductivity was calculated and fitted with suitable equation (Rilo *et al*, 2013).

Vranes and his group have prepared binary liquid mixtures of 1-butyl-methyl imidazolium bis(trifluoromethylsolfony)imide + ɣ - butyrolactone. The density, electrical conductivity and viscosity were measured at different temperatures. The electrical conductivity as a function of temperature was found in the form of VFT equation (Vranes *et al*, 2014).

1.3 Objectives and Motivation:

In our thesis we are motivated to study some of the properties for the binary mixture of water and phenol for many reasons.

First, the binary liquid mixture of water and phenol play an important role in the chemical industry, cleaning products, medicine industry and cosmetics products.

Also, in 2001 it was found that 8 million tons of phenol from the chemical industry contaminate the water each year, so studying some of the properties of water-phenol binary mixture will give a good idea how to remove the phenol waste from the water. Finally, there are rarely published research articles about its physical and thermodynamics properties of this mixture.

The main goals of this thesis are to:

1. Determine the critical temperature (T_C) and critical concentration (X_C) for the binary liquid mixture of water and phenol.

2. Measure the electrical conductivity (σ) at different temperatures (T) and concentrations (X).

3. Find a relationship between the electrical conductivity and temperature σ (T).

4. Detect the anomaly behavior of the electrical conductivity near the critical temperature and concentration.

5- Calculate the molar electrical conductivity.

1.4 Thesis Layout:

In chapter one, an introduction about the liquid mixtures and some of the most important previous studies about its properties were discussed. Some equations that describe the relation between the electrical conductivity and the temperature will be presented in chapter two. In chapter three, the methodology including: the apparatus, properties of water-phenol mixture and the steps of the experiment will be investigated. The experimental data for electrical conductivity will be analyzed and the molar electrical conductivity will be calculated in chapter four. Finally, a discussion about the experimental and theoretical data of this study will be displayed in chapter five.

Chapter Two

Theoretical Approach

2.1 Viscosity of Liquids:-

Viscosity is a property for all liquids and it is a measure of the internal resistance of the liquid to flow. It corresponds to the informal notion of thickness (Symon and Keith ,1971).

There are two different forms of viscosity:

a- Dynamic Viscosity (η) in (Pa.s): it measures the internal friction of the fluid, and it can be written in the following form:

$$\eta = \frac{\tau}{\frac{\partial u}{\partial x}} \qquad (2.1)$$

Where τ is the shear stress in (N/m^2) and $\frac{\partial u}{\partial x}$ is the rate of deformation in (s^1) or the velocity gradient.

b- Kinematic viscosity (ν) in centistokes (cSt): The kinematic viscosity is the ratio of dynamic viscosity to the density of the fluid (ρ) and it can be written in the following form:-

$$\nu = \frac{\eta}{\rho} \qquad (2.2)$$

2.2 Electrical Conductivity Equations:-

Electrical conductivity is a property of the material and it measures its ability to conduct an electrical current **(Vila *et al*, 2007).**

The electrical current in solutions is carried by the cations and anions, where cations are the positive ions that lose electron and anions are the negative

one that gain electrons. Electrical conductivity depends on the temperature of the sample and the concentration of ions in it.

Many equations were used to explain the temperature behavior of the electrical conductivity.

a- One of these is Arhenius equation which is introduced by the Swedish chemist Svante Arrhenius who combined the concepts of activation energy and the Boltzmann distribution law into one of the most important relationships in physical chemistry and it can be written in the following formula:

$$\sigma = \sigma_\infty \exp\left[\frac{-E_a}{K_B\,T}\right] \tag{2.3}$$

Here σ is the electrical conductivity (S/m) where (S) means Siemens which equal (ohm^{-1}), σ_∞ is the maximum electrical conductivity which is at infinite temperature, E_a is the activation energy for electrical conduction which means the threshold energy that the reactants must acquire before reaching the transition state, K_B is Boltzman constant and T is the temperature in Kelvin. The term (K_BT) represents the average kinetic energy of the molecules and it is obvious that with higher temperature, the kinetic energy will be higher and thus speed up the reaction.

Arhenius equation failed to provide a good representation for all liquids.

b- Vogel-Fulcher-Tammann (VFT) equation was proposed to explain the dependence of the electrical conductivity on the temperature.

VTF equation can be written in different forms, but the popular form is:

$$\sigma = A \exp\left[\frac{-B}{T-T_g}\right] \tag{2.4}$$

Where A, B, and T_g are fitting parameters.

c- The modified version of the VTF- type equation would be written as:

$$\sigma = \sigma_\infty \exp\left[\frac{-E_a}{K_B\ (T-T_g)}\right]$$ (2.5)

Where σ_∞, E_a and T_g can be obtained from the best fit of the equation to experimental data (Vila *et al*, 2007).

2.3 Molar Electrical Conductivity:-

Molar electrical conductivity is a physical property of a solution and depends on the number of ions that exist in the solution (Castellan, 2004).

The molar conductivity (Λ_m) in (S.m^2/mole) can be expressed in the following formula (Vranes *et al*, 2013):-

$$\Lambda_m = \frac{\sigma}{c}$$ (2.6)

Where c is the molar concentration in (mole/L) which is also known as the molarity.

The molarity can be calculated from the following equation:-

$$c = \frac{n}{V_{SOL}}$$ (2.7)

Where n is the amount of the solute in (mole) and V_{SOL} is the volume of the solution in Liters.

Number of moles (n) is founded by using the equation:-

$$n = \frac{m}{M}$$ (2.8)

Where m is the mass of the substance and M is the molar mass of the substance.

The mass of the substance can be calculated from the following relation :-

$$m = \rho \times V \qquad (2.9)$$

Where ρ is the density of the substance and V is the volume of the substance.

2.4 Walden Rule:

An empirical rule suggested by P. Walden (1863–1957) concerning ions in solutions, stating that the product of the molar conductivity, Λ_m, and the viscosity, η, is approximately constant for the same ions in different solvents (Toumi *et al*, 2011; Sun *et al*, 2012).

Walden rule is a relation between the viscosity (η) and the molar conductivity (Λ_m) of the binary solution which can be useful in classifying the ionic liquid solutions and gives a good description about the dissociation of the solvent in the solute. It is summarized in the following formula (Toumi *et al*, 2011; Sun *et al*, 2012):

$$\Lambda_m \, \eta^{\alpha} = K \qquad (2.10)$$

Where α is the decoupling constant of the ions $0 < \alpha < 1$ and K (Pose.μS.cm^2/mole) is temperature - dependent constant.

The logarithm form of Eq. (2.10) is expressed in the following equation:

$$\log\Lambda_m = \alpha \, \log\eta^{-1} + \log K \qquad (2.11)$$

Here α equals the graph's slope of the logarithm form of Walden rule, while K can be calculated from the graph's intercept.

An ideal line was suggested by Walden which has a slope equal to 1 to classify the ionic liquids into "good ionic" or "poor ionic" as displayed in Fig (2.1)
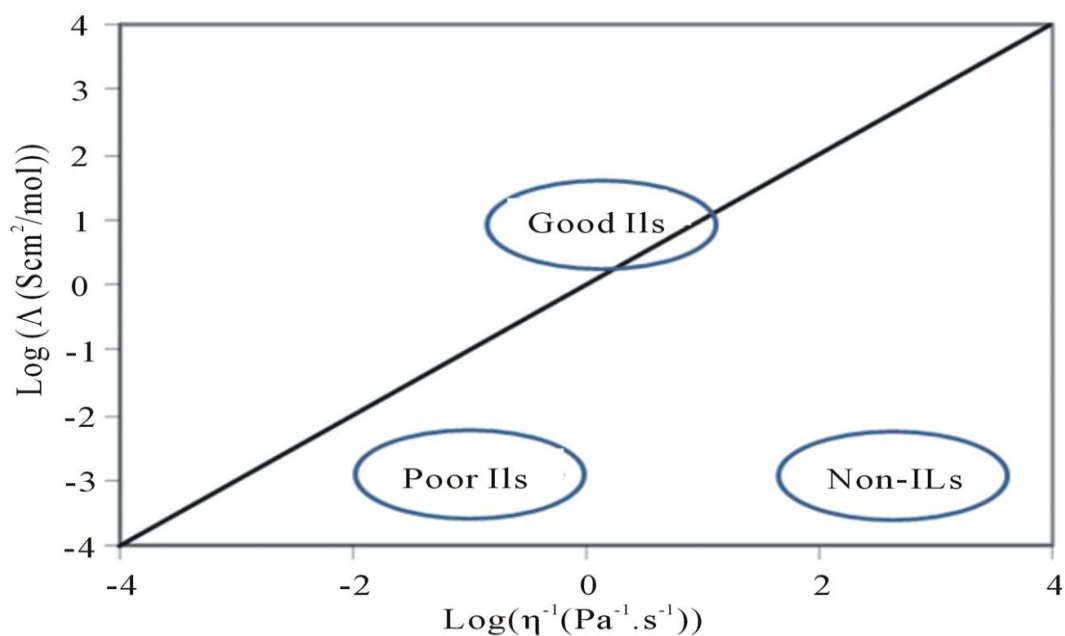
Fig.(2.1): Walden plot shows the classification of ionic liquid into good or poor ionic liquid

The figure above shows that if the measured points of the logarithm of the molar electrical conductivity versus the logarithm of the reciprocal of the dynamic viscosity lie below the ideal line then the ionic liquid will be classified as a poor ionic liquid (Angell *et al*, 2011).

Chapter Three

Methodology

3.1 Introduction:-

Samples of binary liquid mixtures of water and phenol at different concentrations from 0.00% to 100.00% by weight of phenol were prepared. Density, viscosity and electrical conductivity were measured at various temperatures from $32.0^{\circ}C$ up to $75.0^{\circ}C$. Each experimental data represents an average of two values.

The critical concentration and temperature were determined from plotting the viscosity as a function of temperature. The electrical conductivity as a function of temperature was plotted and fitted with Arhenius - like equation.

3.2 Properties of Binary Mixture Components:-

3.2.1 Water:-

Water is an amphoteric chemical compound with molecular formula H_2O. Each oxygen atom is bonded covalently with two hydrogen atoms. It has a density of 0.9982 g/cm^3 at T = 20.0 $^{\circ}C$ and 1atm. The density of water varies at different temperature (Campbell *et al*, 2006).

The water which was used in the experiment was distilled water with 99% of purity.

The pure sample of water has the following physical and chemical properties as shown in table (3.1) (Brown *et al*, 2008; Manahan, 1992).

Table (3.1):- Physical and chemical properties of water

Water	
Chemical formula	H_2O
Physical state (at 20.0 °C and 1atm)	Liquid
Freezing point	0 °C
Boiling point	100 °C
Dynamic Viscosity (at 20.0 °C and 1atm)	1.002 Cp
Density (at 20.0 °C and 1atm)	0.9982g/cm³
Color	Colorless
Purity	100%

3.1.2 Phenol:-

Phenol is an organic compound consists of a ring of benzene attached to (OH) group as shown in Fig. (3.1). It is partially soluble in water. Phenol is solid at room temperature (Weber *et al*, 2004).
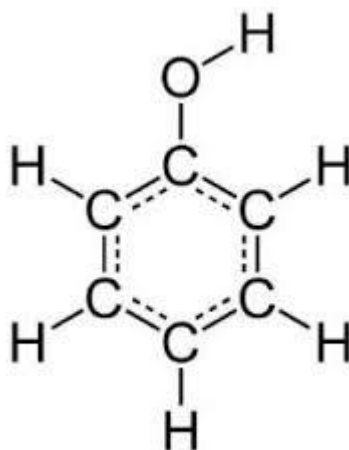
Fig. (3.1): The molecular structure of Phenol

The phenol which was used in the experiment has 99.0% purity.

The pure liquid of phenol has the following properties as listed in table (3.2) (Brown *et al*, 2008; Manahan, 1992):-

Table (3.2):- Physical and chemical properties of phenol

Phenol	
Chemical formula	C_6H_5OH
Physical state (at 20.0 ºC and 1atm)	Solid white crystalline
Freezing point	40.5 ºC
Boiling point	181.7 ºC
Dynamic Viscosity (at 20.0 ºC and 1atm)	12.74 cP
Density (at 20.0ºC and 1atm)	1.07g/cm³
Purity	99.0%
Color	White

3.2.3 Water-Phenol Solubility:-

Phenol is soluble in water to some extent. It is due to its ability to form hydrogen bonding with water molecules. However the large part of phenol molecule is phenyl group that is non polar and hence its solubility is limited in water.

The hydrogen bond between the water molecule and phenol can have many forms:-

1) The hydrogen bond between the phenol hydrogen in the hydroxyl group and the water oxygen.

2) The hydrogen bond between the phenol oxygen and the water hydrogen.

3) The hydrogen bond between the water molecule and the benzene ring (Dai, 1995; Gor *et al*, 2011; Kryachko and Nakatsuji, 2003).

3.3 Experimental Apparatus:-

3.3.1 Viscosity Apparatus:-

A glass capillary U-tube viscometer was used to measure the dynamic viscosity of the mixture. The viscometer has two bulbs and two marks, upper mark and lower mark as shown in Fig. (3.2). The instrument must first be calibrated with materials of known viscosity such as distilled water. Knowing the value of viscosity of one liquid, one can calculate the viscosity of other liquid.

Fig. (3.2): The Glass Capillary U-tube Viscometer

The apparatus is used to measure the viscosity of mixtures which is lower than 1cP.

3.3.2 Temperature Apparatus:-

The temperature of the sample was adjusted and controlled by using the Fried Electric model WB-23 as shown in the Fig. (3.3). The temperature of the sample was measured by using the Digital Prima Long Thermometer as

displayed in Fig. (3.4) which has an accuracy ± 1%. It measure temperatures ranges from -20 to 100ºC.

Fig. (3.3): The Fried Electric Model WB-23

Fig. (3.4): The Digital Prima Long Thermometer

3.3.3 Density Apparatus:

The density of pure components and the binary mixture was measured by using the Pycnometer Fig. (3.5).

Fig. (3.5): The Pycnometer

The mass of the samples was measured by the analytical balance HR-200 with accuracy 0.0005% as shown in Fig. (3.6).

Fig. (3.6): The Analytical Balance HR-200

3.3.4 Conductivity Apparatus:-

A Bante 520 Portable conductivity meter with an accuracy ±0.5% was used to measure the electrical conductivity of the binary mixture Fig. (3.7).

Fig. (3.7): A Bante 520 Portable Conductivity Meter

The meter with auto - ranging feature that can quickly measures the electrical conductivity of liquids. The meter has a conductivity range of 0-20 mS/cm. the meter is used in a temperature range of 0.0-100.0ºC.

3.4 Method of Experiment:-

3.4.1 Preparation of Samples:-

The density of water and phenol was measured at the room temperature by the pycnometer. It was found that the density of water has the value of 0.9987 g/cm³, and the phenol has the value of 1.437 g/cm³.

The values of density of the pure components were used to calculate the volume of water and phenol required to prepare the different concentrations of the binary mixture from (0.00% to 100.00%) by weight of phenol.

The concentration by weight of phenol was calculated by using the following relation:

$$X_{phenol} = \frac{\rho_{phenol}*V_{phenol}}{\rho_{phenol}*V_{phenol} + \rho_{water}*V_{water}}$$

(3.1)

Where X_{phenol} is the concentration by weight of phenol, ρ_{phenol} is the experimental density of phenol, V_{phenol} is the volume of phenol, ρ_{water} is the experimental density of water and V_{water} is the volume of water.

The volume of each sample was fixed to be 20 mL.

3.4.2: Viscosity and Density Measurements:

The Fried Electric model was turned on and adjusted at desired fixed temperature. The sample of each concentration of the mixture was put in the U-tube viscometer and then the viscometer was put into the water bath of the Fried Electric model to get the desired fixed temperature of the sample. The U-tube viscometer was cleaned with ethanol after each trial to remove the residue of the binary mixture.

The viscosity of water - phenol mixture at fixed temperature (T) was measured by using the following equation (Genaralic Eni., 2014):-

$$\eta_1 = \frac{\rho_1 t_1 \eta_2}{\rho_2 t_2} \qquad (3.2)$$

Where η_1 is the viscosity of water – phenol mixture at temperature (T), ρ_1 is the experimental density of water – phenol mixture at temperature (T), t_1 is the time required for the mixture to flow from the upper mark to the lower mark in the viscometer at temperature (T), η_2 is the viscosity of water at temperature (T) which is listed in Appendix A and the measurements were taken by IAPWS(The International Association for the Properties of Water and Steam) in 2008 (http://www.iapws.org/relguide/visc), ρ_2 is the experimental density of water at temperature (T) and t_2 is the time required for water to flow from the upper mark to the lower mark in the viscometer at temperature (T).

3.4.3 Conductivity Measurements:-

The water- phenol mixture was put into a graduated cylinder then into the water - bath to get its temperature. The cell of the conductivity meter was placed into the sample and the measurements were recorded from 32.0 to 75.0°C.

3.4.4 Data Analysis:-

The experimental data for viscosity of the water - phenol mixture will be plotted using the Excel program. The critical temperature and concentration will be determined from the figure of viscosity versus temperature.

The electrical conductivity measurements will be tabulated and analyzed using Excel program. Relation between the temperature and electrical conductivity of water – phenol mixture will be determined by fitting equations using the Excel program.

The strength of the correlation between the electrical conductivity and temperature will be determined by coefficient of determination R^2. It is a numerical value ranges from +1.0 to -1.0. It gives us an indication of the strength of relationship. Closer the coefficients are to +1.0 and -1.0, greater is the strength of the relationship between the variables.

Chapter Four

Results and Calculations

4.1 The Critical Point:-

Experimental data of dynamic viscosity were measured for water - phenol binary liquid mixture for the concentrations (0.00%, 10.17%, 20.80%, 30.20%, 31.80%, 32.90%, 33.90%, 35.00%, 37.00%, 40.00%, 50.00%, 60.00%, 70.00%, 80.00%, 90.00% and 100.00%) by weight of phenol and temperatures range (32.0°C -75.0°C) as given in Table (4.1) and (4.2).

There were many reasons to choose this range for temperature. First, the phenol below 40°C exists in a solid state, also the critical point appeared at T = 67°C so we weren't interested to go far away from the critical point.

In our experiment, first we increase the temperature 5 degrees in each step until the temperature reach 55.0°C, the layer which separates the phenol and water into two phases was observed to decrease with increasing temperature so we expect that the critical temperature will be in that range, so we began to increase the temperature one degree each step to record the critical temperature accurately.

Each measured data of the viscosity of water-phenol mixture samples represents the average of two measurements.

Table (4.1): Measured dynamic viscosity values at various concentrations (0.00%-35.00%) and at temperatures range (32.0-75.0ºC) of water-phenolmixture

T(ºC)	η(cP) 0.00%	η (cP) 10.17%	η (cP) 20.80%	η (cP) 30.20%	η (cP) 31.80%	η (cP) 32.90%	η (cP) 33.90%	η (cP) 35.00%
32.0	0.7650	0.8710	0.9950	1.8750	1.8750	2.6250	2.5040	3.3310
40.0	0.6530	0.8070	0.8400	2.1480	2.1480	2.0580	2.2860	2.7590
45.0	0.5960	1.0750	1.1390	3.5490	3.5490	2.8310	2.9500	3.6840
50.0	0.5470	0.6440	0.7160	1.8320	1.8320	1.6180	1.7500	2.0090
55.0	0.5040	0.6050	0.6390	1.6650	1.6640	1.4630	1.5380	1.9240
57.0	0.4890	0.5690	0.6050	1.4080	1.4080	1.3510	1.4680	1.7230
58.0	0.4810	1.0150	1.0720	2.4330	2.4330	2.3990	2.6090	3.1280
59.0	0.4740	0.5580	0.5920	1.3060	1.3060	1.2550	1.3190	1.6990
60.0	0.4670	0.5600	0.5740	1.3750	1.3750	1.2560	1.3930	1.7470
61.0	0.4600	0.5380	0.5570	1.2510	1.2510	1.2070	1.3300	1.6050
62.0	0.4530	0.5330	0.5410	1.1360	1.1360	1.1730	1.2360	1.5350
63.0	0.4470	0.5090	0.5380	0.9000	0.9000	1.1130	1.1150	1.4100
64.0	0.4400	0.4320	0.4600	0.9530	0.9520	0.9350	0.9550	1.1990
65.0	0.4340	0.4270	0.4670	0.8560	0.8550	0.8760	1.0710	1.0460
66.0	0.4280	0.4150	0.4690	0.7350	0.7350	0.8880	1.0910	0.9740
67.0	0.4220	0.4070	0.4520	0.6450	0.6440	0.9010	1.1100	0.9700
68.0	0.4160	0.4020	0.4450	0.6640	0.6440	0.8890	0.9910	0.8710
69.0	0.4100	0.4000	0.3770	0.5680	0.5680	0.9030	0.7320	0.8580
70.0	0.4040	0.3690	0.3830	0.5480	0.5480	0.7090	0.7570	0.8150
75.0	0.3780	0.3530	0.3760	0.5050	0.5050	0.5890	0.6810	0.7090

Table (4.2): Measured dynamic viscosity values at various concentrations (37.00%- 100.00%) and at temperatures range (32.0-75.0ºC) for   water-phenol mixture

T(ºC)	η (cP) 37.00%	η (cP) 40.00%	η (cP) 50.00%	η (cP) 60.00%	η (cP) 70.00%	η (cP) 80.00%	η (cP) 90.00%	η (cP) 100.00%
32.0	3.0950	2.2560	2.6480	3.3970	3.1580	3.3180	3.3350	Solid
40.0	2.4610	1.8800	2.0710	2.6000	2.5020	2.7360	2.6140	4.2810
45.0	3.3080	2.7940	2.8480	3.9950	3.3910	3.6710	3.5340	3.6780
50.0	1.9490	1.5860	1.7250	2.3590	2.0290	2.2110	2.0380	4.9380
55.0	1.7380	1.4190	1.5690	1.9790	1.8770	2.1050	1.9390	2.9340
57.0	1.7570	1.3400	1.4640	1.8960	1.8640	2.0250	1.8600	2.6810
58.0	3.0520	2.3440	2.7010	3.2920	3.1760	3.6370	3.3070	2.7050
59.0	1.5930	1.2500	1.4210	1.7760	1.7370	1.9100	1.8150	4.9690
60.0	1.5360	1.2840	1.3440	1.7040	1.7200	1.8580	1.8120	2.5380
61.0	1.4070	1.1500	1.2680	1.6410	1.7210	1.9090	1.8020	2.4600
62.0	1.5410	1.1560	1.2410	1.5890	1.6370	1.7810	1.7690	2.4210
63.0	1.3860	1.0380	1.1740	1.4990	1.6120	1.5960	1.6500	2.2880
64.0	1.2540	0.8750	0.9840	1.2940	1.2440	1.3810	1.3570	2.1700
65.0	1.1640	0.9480	0.9800	1.2440	1.1660	1.2130	1.3430	1.8500
66.0	1.2280	0.9090	0.9320	1.2160	1.1420	1.2030	1.1490	1.8220
67.0	1.1410	0.8510	0.9060	1.2010	1.1210	1.1720	1.1230	1.7910
68.0	1.0760	0.8350	0.8960	1.1600	1.1160	1.1640	1.0880	1.7200
69.0	1.1040	0.8440	0.8570	1.1780	1.1040	1.1680	1.0740	1.6930
70.0	1.0000	0.8300	0.8040	1.0900	1.0000	1.0530	1.0910	1.7800
75.0	0.8850	0.7510	0.7150	1.0120	0.8890	0.9260	0.9460	1.6050

The dynamic viscosity versus temperature is plotted at different concentrations for the binary liquid mixture of water-phenol in Fig. (4.1) and (4.2).
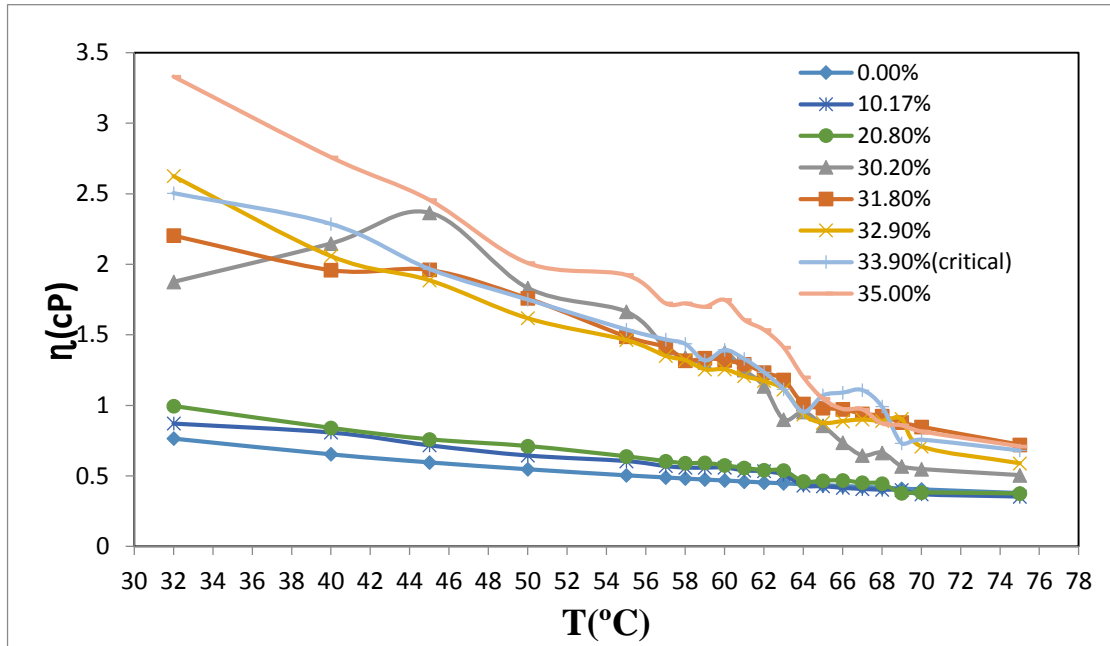
Fig. (4.1): The dynamic viscosity of water – phenol as a function of temperature for the concentrations (0.00% - 35.00%) by weight of phenol
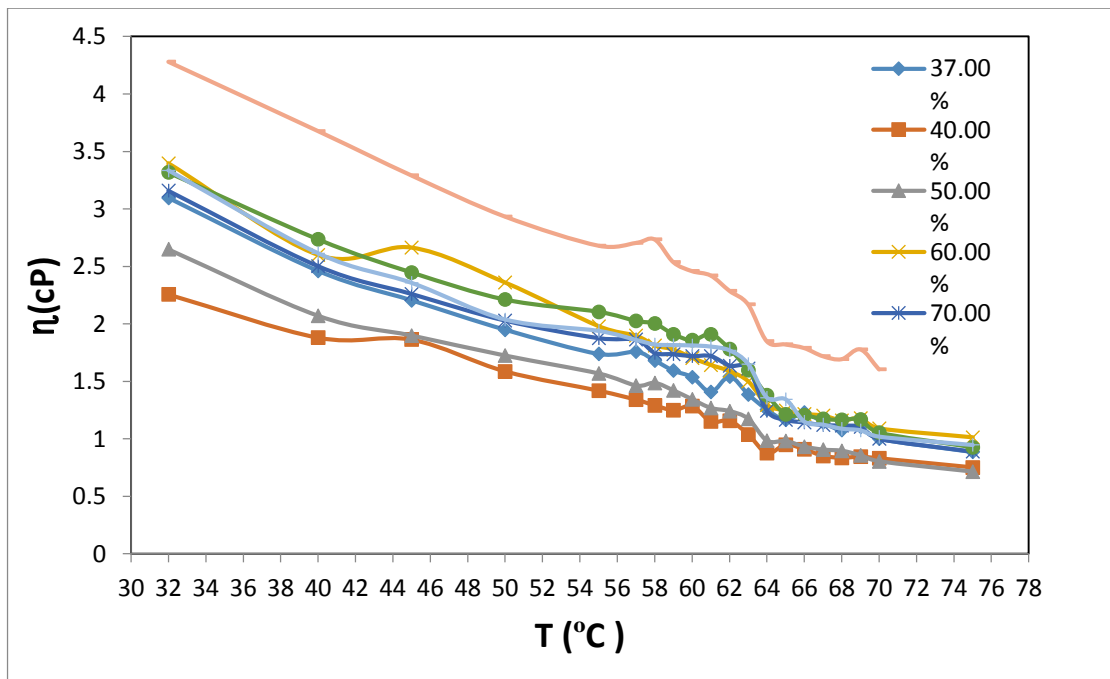
Fig. (4.2): The dynamic viscosity of water – phenol as a function of temperature for the concentrations (37.00% - 100.00%) by weight of phenol

The dynamic viscosity values are observed to decrease with increasing the temperature from Fig. (4.1) and (4.2). Anomaly in the behavior of the binary mixture is observed at T = 67.0 and 33.90% by weight of phenol. This point is the critical point for the mixture where the two liquids become in one phase and completely miscible which was observed by eye. Wilcox and Bailey have determined the critical point of the binary mixture water - phenol where the critical temperature was at 65.3°C and the critical concentration was at 36.50% by weight of phenol (Wilcox and Bailey, 1929). The difference between the values of their critical temperature and our critical temperature is because the difference in the purity of the samples of water and phenol which are used in the experiment. The binary mixture of water-phenol shows anomaly in its behavior at different points. The anomaly at these points was observed because there were some sources of error in the experiment such as the temperature was not adjusted very well or some bubbles of air were formed in the binary mixture which affected the value of the dynamic viscosity.

4.2 Electrical Conductivity Measurements:

The electrical conductivity of water - phenol mixture is studied for wide range of temperatures (32.0-75.0°C) and concentrations (0.00% - 100.00%) by weight of phenol as given in Table (4.3) and (4.4).

Table (4.3): Electrical conductivity measurements as a function of temperature and concentration for water-phenol binary liquid mixture

T(ºC)	σ(µS/cm) 0.00%	σ(µS/cm) 10.17%	σ(µS/cm) 20.80%	σ(µS/cm) 30.20%	σ(µS/cm) 31.80%	σ(µS/cm) 33.90%	σ(µS/cm) 35.00%
32.0	11.62	19.60	17.40	2.57	1.93	1.45	1.52
40.0	14.25	24.30	23.20	4.44	2.57	1.84	1.99
45.0	16.26	27.50	27.30	5.91	3.91	2.14	2.34
50.0	18.18	30.70	31.10	7.47	3.39	2.51	2.77
55.0	20.10	34.90	34.70	9.66	3.87	3.04	3.14
57.0	21.40	37.90	39.40	11.40	4.28	3.77	3.42
58.0	23.00	40.40	42.20	13.80	4.73	4.34	3.91
59.0	23.40	42.10	45.70	14.80	4.98	4.77	4.57
60.0	23.80	45.70	50.20	23.30	5.61	5.25	5.25
61.0	24.60	48.80	51.70	23.20	6.06	6.23	5.83
62.0	25.50	49.60	55.80	31.30	6.76	7.05	7.18
63.0	26.10	52.30	57.30	33.90	7.80	7.78	7.98
64.0	27.20	56.10	60.60	32.80	8.82	8.82	9.93
65.0	27.90	56.90	62.50	34.20	9.97	9.86	10.80
66.0	28.50	61.20	68.90	35.50	11.20	13.00	12.40
67.0	29.50	62.40	71.90	37.10	13.10	18.90	14.50
68.0	30.20	66.00	75.00	50.50	16.50	25.05	18.00
69.0	31.00	68.40	78.80	62.80	24.80	26.66	20.60
70.0	34.20	71.00	80.90	72.60	32.20	27.09	24.50
75.0	37.00	76.80	87.00	86.20	41.20	37.30	36.20

Table (4.4): Electrical conductivity measurements as a function of temperature (32.0-75.0ºC) and concentration (37.00%-100.00%) for the binary liquid mixture of water and phenol

T(ºC)	σ(μS/cm) 37.00%	σ(μS/cm) 40.00%	σ(μS/cm) 50.00%	σ(μS/cm) 60.00%	σ(μS/cm) 70.00%	σ(μS/cm) 80.00%	σ(μS/cm) 90.00%	σ(μS/cm) 100.00%
32.0	1.04	1.13	7.00	2.68	6.06	4.22	2.53	1.07
40.0	1.31	1.50	8.74	3.51	7.15	5.06	2.93	1.27
45.0	1.56	1.80	10.30	4.00	8.15	5.58	2.59	1.43
50.0	1.86	2.05	12.20	4.91	9.16	6.26	3.81	1.58
55.0	2.40	2.36	14.30	5.94	9.77	6.63	4.25	1.72
57.0	2.59	2.70	16.40	6.41	10.20	7.71	4.88	1.78
58.0	2.79	3.11	18.50	7.39	11.30	8.04	5.06	1.79
59.0	3.14	3.53	20.70	7.88	11.50	8.18	5.35	1.84
60.0	3.55	5.29	23.50	8.93	11.70	8.33	5.43	1.89
61.0	4.74	6.52	24.10	9.90	11.90	8.40	5.69	1.92
62.0	6.54	7.77	26.50	10.90	11.90	8.75	5.87	1.94
63.0	7.37	8.59	25.90	12.90	11.90	8.76	5.90	2.01
64.0	8.21	9.74	27.90	16.10	11.90	8.77	6.08	2.01
65.0	9.58	10.90	29.30	17.20	11.90	8.87	6.27	2.11
66.0	10.40	12.60	29.80	17.90	11.80	8.84	6.63	2.09
67.0	12.00	12.60	30.20	19.20	11.70	8.38	6.56	2.00
68.0	15.20	17.00	31.00	19.20	11.50	8.95	6.90	2.01
69.0	16.78	19.70	31.60	20.40	11.70	9.08	6.69	1.96
70.0	18.00	22.30	33.30	20.90	11.70	9.07	6.78	2.00
75.0	24.00	24.40	35.10	22.40	11.80	9.36	6.90	2.01

The electrical conductivity measurements as a function of temperature are plotted in Fig. (4.3) and (4.4).
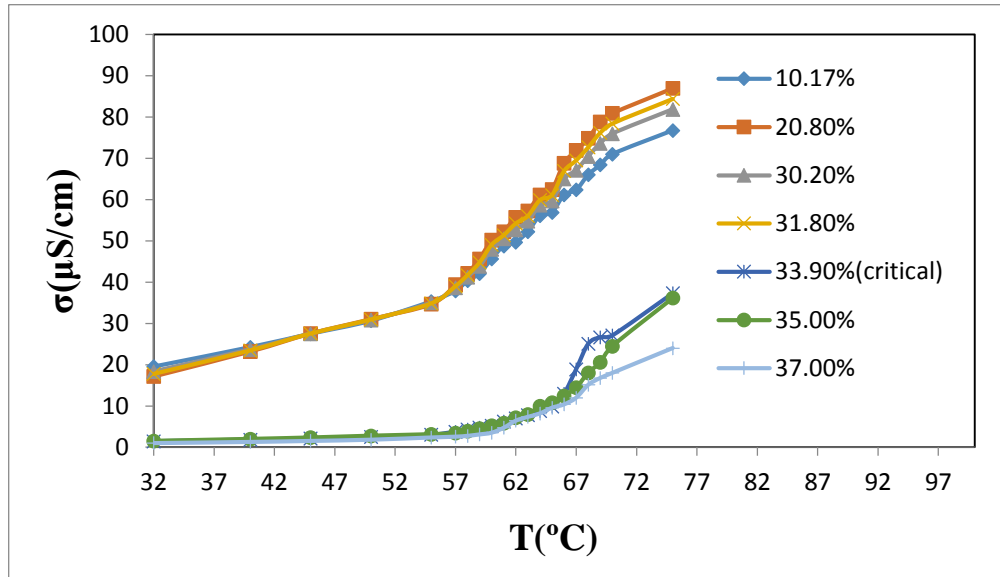
Fig. (4.3): The electrical conductivity versus the temperature for concentrations range (10.00% - 37.00%) by weight of phenol
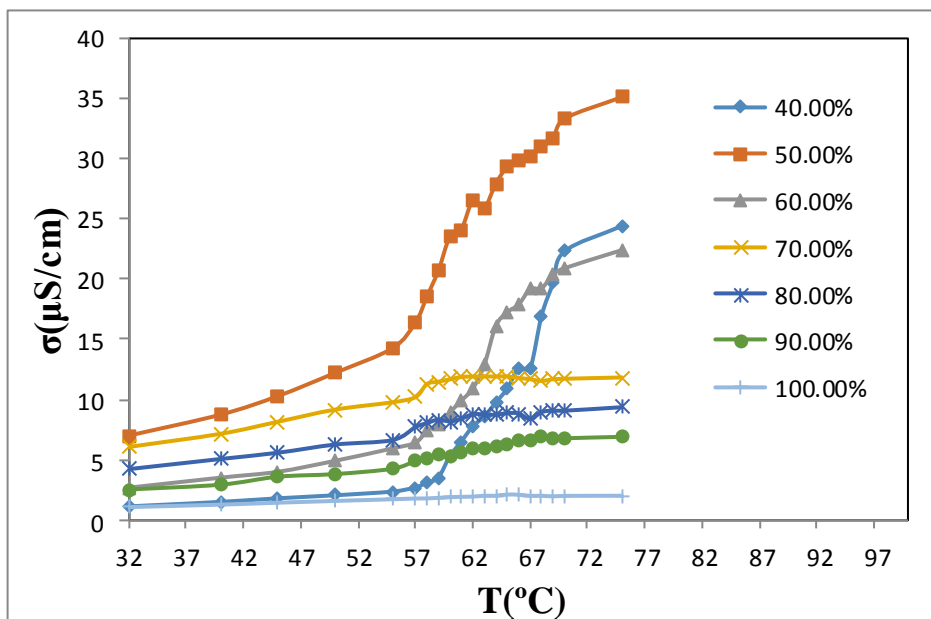
Fig. (4.4): The electrical conductivity versus the temperature for concentrations range (40.00% - 100.00%) by weight of phenol
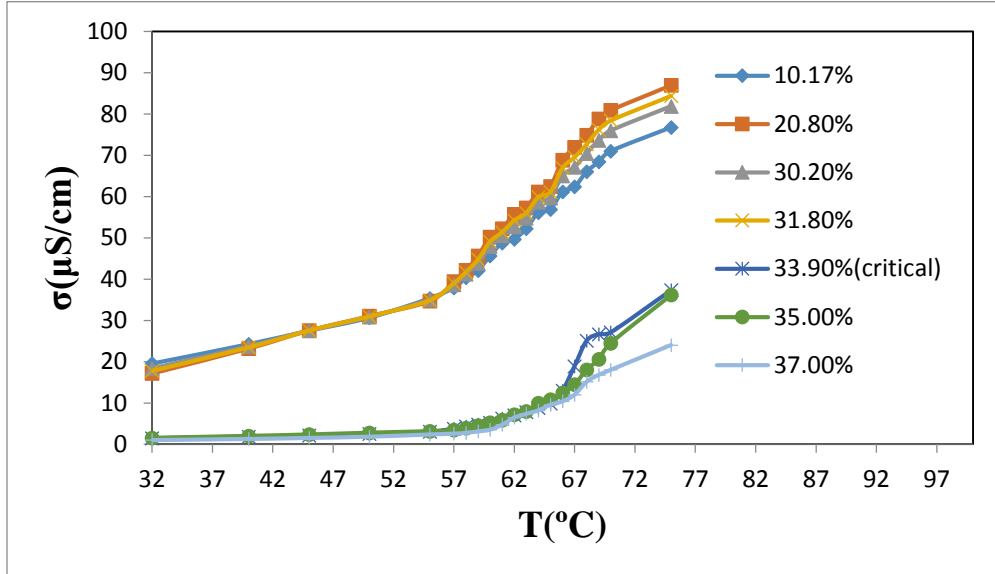
For each given concentration, the electrical conductivity was observed to increase with temperature. The increment in values of electrical conductivity is due to the increase in speed of ions of water-phenol binary mixture with increasing temperature.

Electrical conductivity values as a function of temperature obeys Arhenius equation which is given by the following formula:-

$$\sigma = \sigma_\infty \exp\left[\frac{-E_a}{K_B\,T}\right]$$

The natural logarithm of Arhenius equation can be written by the following equation:-

$$\ln\sigma = \ln\sigma_\infty - \frac{E_a}{K_B T}$$

The natural logarithm of the electrical conductivity for the concentrations (0.00%, 10.17%, 20.80% and 50.00%,) versus the reciprocal of the temperature are plotted in Fig. (4.5) to Fig. (4.9) to prove that Arhenius equation gives a good agreement with our experimental values. The other concentrations (20.80%, 30.20%, 31.80%, 35.00%, 37.00%, 40.00%,

60.00%, 70.00%, 80.00%, 90.00% and 100.00%) except the critical concentration $X_C = 33.90\%$ gives a good agreement with Arhenius equation but they aren't listed in the figures below to avoid the repetition.
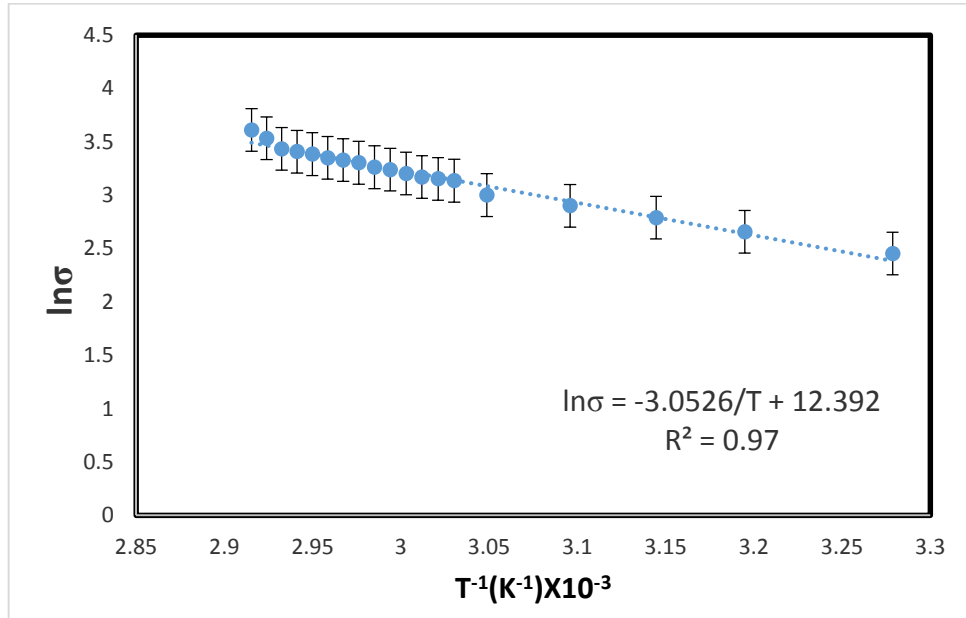
Fig. (4.5): The natural logarithm of the electrical conductivity for water only versus the reciprocal of temperature
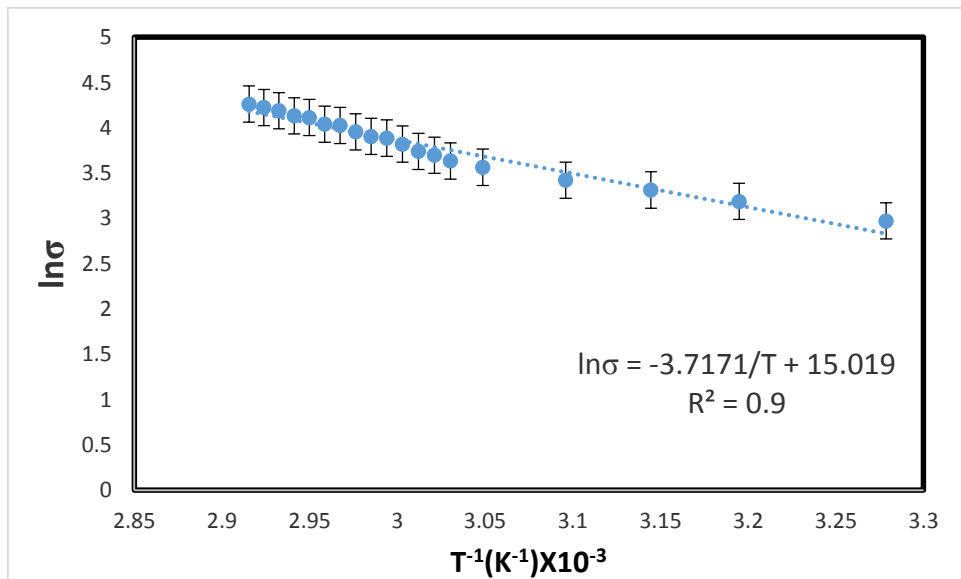
Fig. (4.6): The natural logarithm of the electrical conductivity versus the reciprocal of temperature at concentration 10.17% by weight of phenol
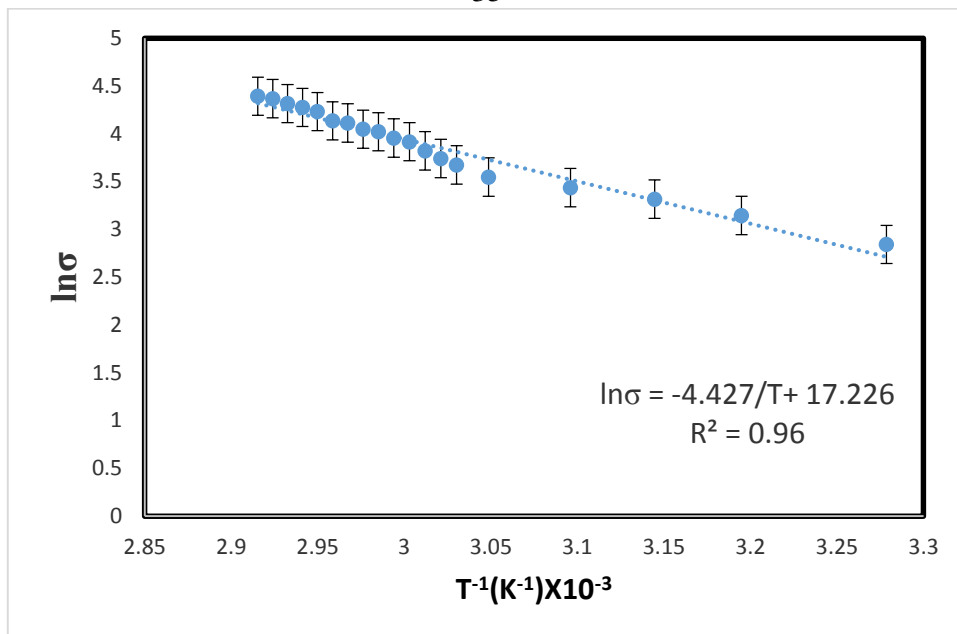
Fig. (4.7): The natural logarithm of the electrical conductivity versus the reciprocal of temperature at concentration 20.80% by weight of phenol
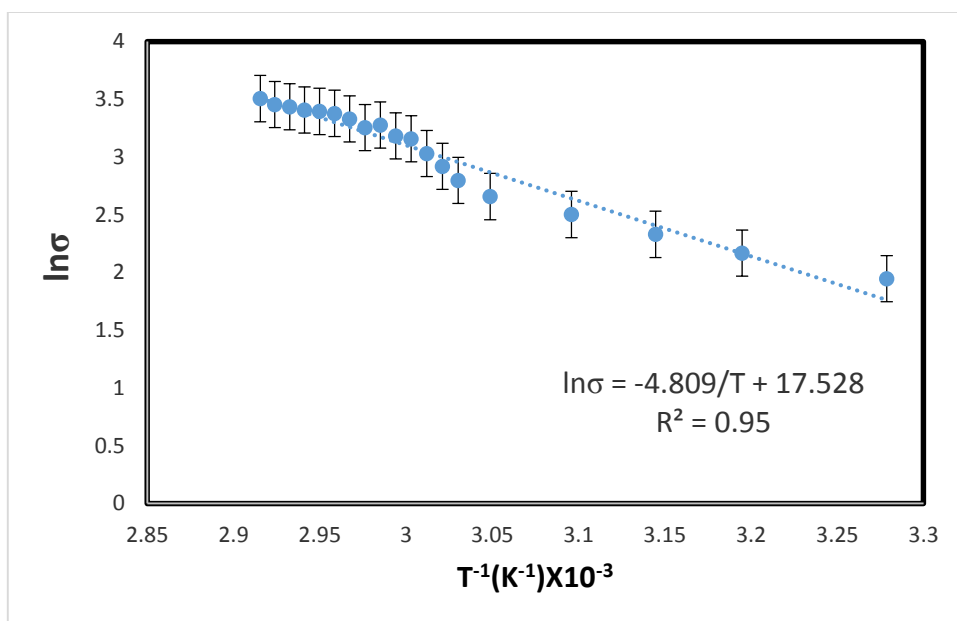
Fig. (4.8): The natural logarithm of the electrical conductivity versus the reciprocal of temperature at concentration 50.00% by weight of phenol

Arhenius equation gives a good agreement with the experimental values of electrical conductivity for the concentrations (0.00%, 10.17%, 20.80% and

50.00%) as observed in Fig. (4.5) to Fig. (4.9).An exponential relation between the electrical conductivity and the reciprocal of temperature is noticed from Fig. (4.5) to Fig. (4.9).

The slopes of Fig. (4.5) to Fig. (4.9) represent $(\frac{E_a}{K_B})$ value for each concentration, and the intercept with the y-axis represents the natural logarithm for the maximum value of the electrical conductivity $\ln\sigma_\infty$.

The activation energy and the maximum value of the electrical conductivity for each concentration were calculated and tabulated in Table (4.5).

Table (4.5): The activation energy and the maximum electrical conductivity for concentrations (0.00%, 10.17%, 20.80% and 50.00%) for water - phenol mixture

Concentration by Weight of Phenol	The Maximum Electrical Conductivity(μS/cm)	The Activation Energy ($\times 10^{-20}$J)
0.00%	151448.40	4.0128
10.17%	3355126.00	5.1325
20.80%	26429728.00	6.0481
50.00%	29151007.00	6.4842

4.3 Behavior of Electrical Conductivity above the Critical Point:-

The electrical conductivity above the critical temperature for the critical concentration doesn't obey Arhenius equation. A new equation was proposed above the critical point to fit our measurements.

Electrical conductivity values above the critical point ($T_C = 67.0^{\circ}$C and $X_C = 33.90\%$) was measured as a function of temperature. The measurements are listed in Table (4.6).

Table (4.6): The electrical conductivity measurements above the critical point ($T_C = 67.0$ and $X_C = 33.90\%$) of water - phenol binary mixture

Temperature (°C)	Electrical Conductivity (µS/cm)
67.0	18.90
67.2	19.96
67.6	23.40
67.8	30.20
68.0	25.05
68.8	26.49
69.0	26.66
69.5	26.90
70.0	27.09
71.0	30.10
72.0	32.90
73.0	35.70
74.0	36.30
75.0	37.30

The electrical conductivity as a function of temperature for the critical concentration was plotted as shown in Fig (4.10).
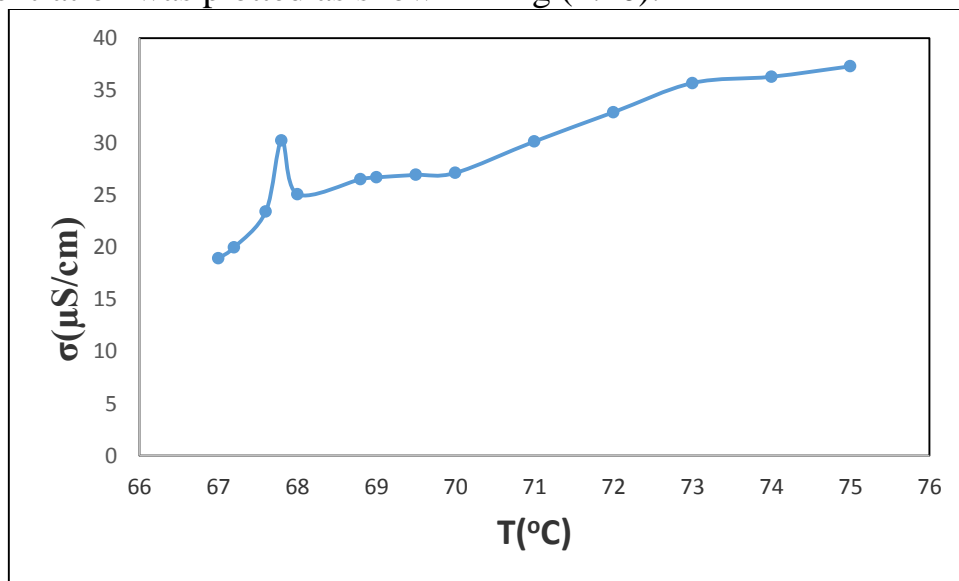
Fig.(4.9): The electrical conductivity above the critical point ($X_c = 33.90\%$ and $T_c = 67.0$°C) as a function of temperature for water-phenol mixture

The electrical conductivity at the critical temperature ($T_C = 67.8^\circ C$) shows anomaly in the behavior of water-phenol binary mixture in Fig. (4.9).

The critical temperature obtained from the electrical conductivity measurements ($T_C = 67.8^\circ C$) is different from the critical temperature obtained from the dynamic viscosity measurements $T_C = 67.0^\circ C$. The difference in the values of the critical temperature is due to some sources of error in the experiment such as the temperature of the water path was not adjusted very well.

The electrical conductivity is temperature dependent at the critical concentration which is given by the power law.

The electrical conductivity above the critical temperature is fitted by using power law:-

$$\sigma = \sigma_o\, \tau^{\chi_\sigma \upsilon} \qquad (4.1)$$

Which can be written in the following formula:-

$$\ln\sigma = \ln\sigma_o + \chi_\sigma\upsilon\ln\tau \qquad (4.2)$$

Where τ is the reduced temperature, which is given by :-

$$\tau = \frac{T - T_c}{T_c} \qquad (4.3)$$

υ is critical exponent = 0.64 (D'Arrigo $et\ al$, 1977; Klein and Woermann, 1978), $\chi_\sigma\upsilon$ is critical exponent = 0.037 and σ_o is the noncritical part of the measured electrical conductivity which represents the electrical conductivity at inifinite temperature.

The electrical conductivity measurements for the critical concentration (X_C = 33.90%) and above the critical temperature ($T_C = 67.80°C$) are listed in Table(4.7).

Table (4.7): The electrical conductivity measurements for the critical concentration (X_C =33.90%) for water - phenol binary mixture above the critical temperature

T(°C)	σ(μS/cm)	T-T$_c$	$\tau = \dfrac{T - T_c}{T_c}$	lnσ	lnτ
68.0	25.05	0.2	0.0030	3.2209	-5.8260
68.2	25.66	0.4	0.0059	3.2449	-5.1329
68.4	26.03	0.6	0.0089	3.2591	-4.7274
68.6	26.29	0.8	0.0118	3.2691	-4.4397
68.8	26.49	1.0	0.0148	3.2769	-4.2166
69.0	26.66	1.2	0.0177	3.2833	-4.0342
69.2	26.67	1.4	0.0207	3.2835	-3.8801
69.4	26.79	1.6	0.0236	3.2881	-3.7466
69.6	26.90	1.8	0.0266	3.2922	-3.6288
69.8	27.00	2.0	0.0295	3.2959	-3.5234
70.0	27.09	2.2	0.0325	3.2992	-3.4281

lnσ versus lnτ is plotted and fitted by using power law as shown in Fig. (4.11).
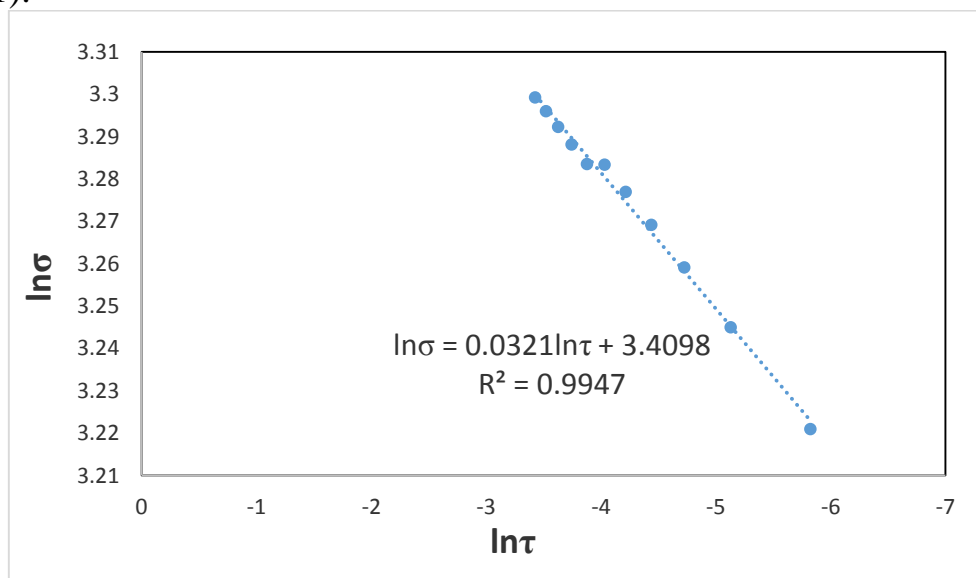
Fig. (4.10): lnσ versus lnτ for critical concentration and above the critical temperature

The electrical conductivity above the critical point behaves as power law as Fig. (4.11) shows.

By comparing Eq. (4.1) with the result in Fig. (4.10), the value of the critical exponents constant is presented by slope of the curve which equal to 0.032. The critical exponent $\chi_\sigma\upsilon$ has a good agreement with D'Arrigo and Klein values which equal 0.037 (D'Arrigo *et al*, 1977; Klein *et al*, 1978).

The intercept of the curve represented the natural logarithm of the noncritical part of the measured electrical conductivity $\ln\sigma_o = 3.4098$, so the value of the noncritical part equal to $30.26\mu S/cm$. The power law of the electrical conductivity in Eq. (4.1) and the theoritical value of the critical exponent are used to calculate the noncritical part of the measured electrical conductivity by plotting σ Vs. $\tau^{0.037}$ as shown in Fig. (4.11)

$$\sigma = 26.849\tau^{0.037} + 3.4552$$
$$R^2 = 0.9941$$
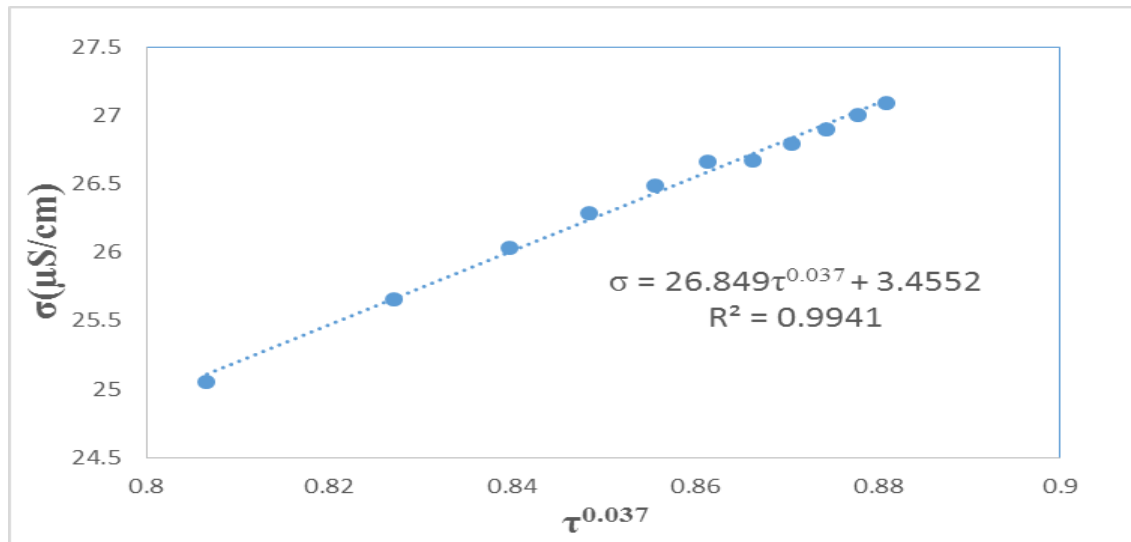
Fig. (4.11): The measured electrical conductivity versus $\tau^{0.037}$

The slope of Fig. (4.11) represents the noncritical part of the electrical conductivity and equals $26.85\mu S/cm$ which is close to the value obtained from Fig. (4.10).

4.4 Calculations of Molar Electrical Conductivity:-

The concentration of ions in the binary mixture are studied by calculating the molar electrical conductivity and applying Walden's rule.

The molar electrical conductivity were calculated for the concentrations (10.17%, 20.80%, 30.20%, 31.80%, 33.90%, 35.00%, 37.00%, 40.00%, 50.00% and 60.00%) by the weight of phenol by using Eq. (2.6):-

$$\Lambda_m = \frac{\sigma}{c}$$

The mass of phenol at each temperature and concentration was calculated from Eq. (2.9), and then from Eq. (2.8) the number of moles (n) of phenol were found and tabulated in Table (4.8) and (4.9).

$$n = \frac{m}{M}$$

$$m = \rho \times V$$

The density of phenol at each temperature and for each concentration is listed in appendix B.

Table (4.8): The number of moles of phenol for concentrations (10.17%, 20.80%, 30.20%, 31.80% and 33.90%) by weigh phenol at different temperatures

T(^{o}C)	n(moles) 10.17%	n (moles) 20.80%	n(moles) 30.20%	n(moles) 31.80%	n(moles) 33.90%
67.0	0.0199	0.0342	0.0607	0.0640	0.0695
68.0	0.0199	0.0342	0.0607	0.0640	0.0695
69.0	0.0198	0.0342	0.0606	0.0639	0.0694
70.0	0.0201	0.0346	0.0614	0.0647	0.0703
75.0	0.0201	0.0346	0.0613	0.0647	0.0703

Table (4.9): The number of moles of phenol for concentrations (35.00%, 37.00%, 40.00%, 50.00% and 60.00%) by weigh phenol at different temperatures

T(°C)	n(moles) 35.00%	n (moles) 37.00%	n(moles) 40.00%	n(moles) 50.00%	n(moles) 60.00%
67.0	0.0706	0.0750	0.0817	0.1026	0.1247
68.0	0.0706	0.0750	0.0816	0.1026	0.1246
69.0	0.0705	0.0749	0.0815	0.1025	0.1245
70.0	0.0714	0.0759	0.0826	0.1038	0.1261
75.0	0.0714	0.0758	0.0825	0.1037	0.1260

The molarity (c) of the solution at each temperature and concentration was calculated and listed in Table (4.10) and (4.11) by using Eq. (2.7):-

$$c = \frac{n}{V}$$

Table (4.10): The molarity of water-phenol solution for concentrations 10.17%, 20.80%, 30.20%, 31.80% and 33.90%) by weight of phenol at different temperatures

T(°C)	c(mole/mL) 10.17%	c(mole/mL) 20.80%	c(mole/mL) 30.20%	c(mole/mL) 31.80%	c(mole/mL) 33.90%
67.0	0.0010	0.0017	0.0030	0.0032	0.0035
68.0	0.0010	0.0017	0.0030	0.0032	0.0035
69.0	0.0010	0.0017	0.0030	0.0032	0.0035
70.0	0.0010	0.0017	0.0031	0.0032	0.0035
75.0	0.0010	0.0017	0.0031	0.0032	0.0035

Table (4.11): The molarity of water-phenol solution for concentrations (35.00%, 37.00%, 40.00%, 50.00% and 60.00%) by weight of phenol at different temperatures

T(°C)	c(mole/mL) 35.00%	c(mole/mL) 37.00%	c(mole/mL) 40.00%	c(mole/mL) 50.00%	c(mole/mL) 60.00%
67.0	0.0035	0.0038	0.0041	0.0051	0.0062
68.0	0.0035	0.0037	0.0041	0.0051	0.0062
69.0	0.0035	0.0037	0.0041	0.0051	0.0062
70.0	0.0036	0.0038	0.0041	0.0052	0.0063
75.0	0.0036	0.0038	0.0041	0.0052	0.0063

The molar electrical conductivity is calculated by using Eq. (2.6) and listed in Table (4.12) and (4.13)

$$\Lambda_m = \frac{\sigma}{c}$$

Table (4.12): The molar electrical conductivity of water-phenol solution for concentrations (10.17%, 20.80%, 30.00%, 32.00% and 33.90%) by of phenol at different temperatures

T(°C)	Λ_m (S.cm^2/mole) 10.17%	Λ_m (S.cm^2/mole) 20.80%	Λ_m (S.cm^2/mole) 30.20%	Λ_m (S.cm^2/mole) 31.80%	Λ_m (S.cm^2/mole) 33.90%
67.0	0.0627	0.042032	0.012224	0.0041	0.0086
68.0	0.0664	0.043815	0.016651	0.0051	0.0072
69.0	0.0689	0.046141	0.020726	0.0077	0.0113
70.0	0.0706	0.046757	0.023650	0.0099	0.0125
75.0	0.0764	0.050297	0.028104	0.0127	0.0163

Table (4.13): The molar electrical conductivity of water-phenol solution for concentrations (35.00%, 37.00%, 40.00%, 50.00% and 60.00%) byweight of phenol at different temperatures

T(°C)	Λ_m (S.cm^2/mole) 35.00%	Λ_m (S.cm^2/mole) 37.00%	Λ_m (S.cm^2/mole) 40.00%	Λ_m (S.cm^2/mole) 50.00%	Λ_m (S.cm^2/mole) 60.00%
67.0	0.0040	0.0031	0.0030	0.0058	0.0030
68.0	0.0051	0.0040	0.0041	0.0060	0.0030
69.0	0.0058	0.0044	0.0048	0.0061	0.0032
70.0	0.0068	0.0047	0.0053	0.0064	0.0033
75.0	0.0101	0.0063	0.0059	0.0067	0.0035

The concentrations of the ions in the binary mixture was studied by applying Walden rule as mentioned in Eq. (2.10) and Eq. (2.11)

$$\Lambda_m \eta^\alpha = K$$

$$\log \Lambda_m = \alpha \log\eta^{-1} + \log K$$

The logarithm of molar electrical conductivity and the logarithm of the reciprocal of dynamic viscosity at several concentrations at $T = 68.0^\circ C$ is listed in Table (4.14) and drawn as shown in Fig. (4.13). The Walden plot which is drawn in Fig. (4.13) is independent of temperature and the value of (α) will be the same with different temperatures.

Table (4.14): The logarithm of reciprocal of the dynamic viscosity and the logarithm of molar electrical conductivity

X_{phenol}	Λ_m (S.cm^2/mole)	η(Pose)	η^{-1}(Pose^{-1})	$\log\eta^{-1}$	$\log \Lambda_m$
10.17%	0.0664	0.0040	248.7562	2.3957	-2.1772
20.80%	0.0438	0.0045	224.7191	2.3516	-1.3583
30.20%	0.0166	0.0066	150.5152	2.1178	-1.7785
31.80%	0.0051	0.0064	155.2795	2.1911	-2.2865
33.90%	0.0072	0.0099	100.9082	2.0039	-2.1420
35.00%	0.0051	0.0087	114.8106	2.0599	-2.2914
37.00%	0.0040	0.0108	92.5926	1.9665	-2.3944
40.00%	0.0041	0.0083	120.4819	2.0809	-1.3810
50.00%	0.0060	0.0090	111.1111	2.0457	-2.2186
60.00%	0.0030	0.0116	86.2069	1.9355	-2.5106

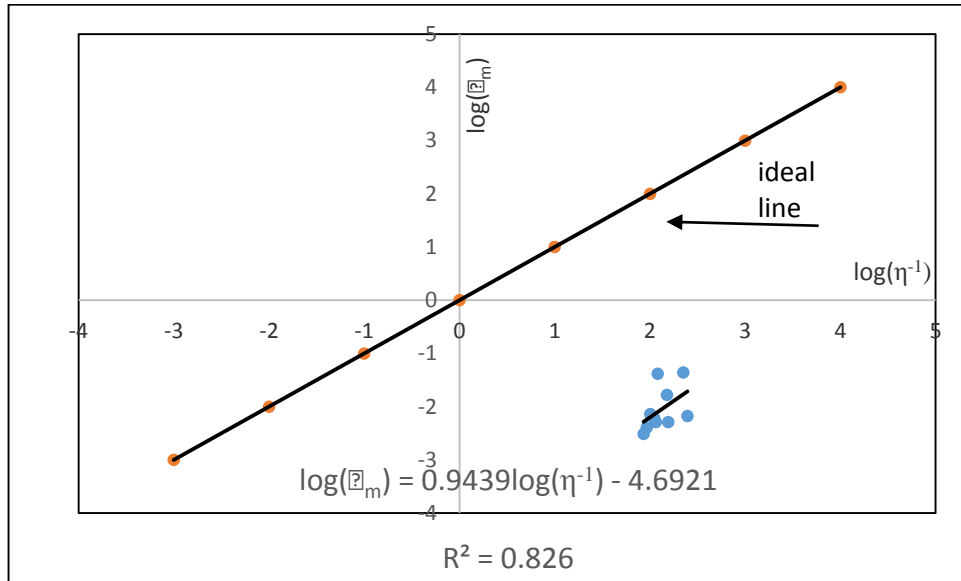**Fig. (4.12)**: The logarithm of molar electrical conductivity versus the logarithm of reciprocal of the dynamic viscosity at T = 68.0ºC

It can be noticed from Fig. (4.12) that the slope of the figure $\alpha = 09439$ which is lower than 1 and the calculated points for the water-phenol binary mixture lie below the ideal line. This means that water-phenol mixture can be considered as poor ionic solution.

Chapter Five

Conclusion

The dynamic viscosity was measured for water-phenol binary mixture at several concentrations and temperatures to determine the critical concentrations and critical temperatures. The critical concentration is determined at 33.90% by weight of phenol and the critical temperature was 67.0^{o}C from the dynamic viscosity measurements and 67.8^{o}C from the electrical conductivity measurements.

Wilcox and bailey found the critical concentration and critical temperature and it was (36.50% of phenol and 65.3^{o}C) (Wilcox and Bailey, 1929). The difference between their results and our results is due to the difference in the purity of the water and phenol which are used in the experiment.

The electrical conductivity measurements is recorded for water-phenol binary mixture for concentration ranges (0.00% - 100.00%) by weight of phenol and temperature range from 32.0^{o}C to 75.0^{o}C. As the temperature values increase, the electrical conductivity values are observed to increase because the speed of collisions among the species of the mixture increase.

The electrical conductivity measurements at different concentrations are fitted by Arhenius equation.

The activation energy and the electrical conductivity at infinite temperature are calculated for various concentrations.

The electrical conductivity is fitted by using power law above the critical temperature and for the critical concentration.

The noncritical part of the electrical conductivity is found to be 30.35µS/cm and the critical exponent was 0.032.

The ionic properties of the binary mixture is studied by calculating the molar electrical conductivity and then Walden rule is applied to give a relation between the molar electrical conductivity and the shear viscosity of the water-phenol mixture. It is observed from Walden rule, that the slope of the line is 0.9439 which is lower than one. These results mean that the water-phenol mixture has low concentrations of ions which carry the current and can be considered as a poor ionic liquid.

The main results of this work and other works are summarized in Table (4.15).

Table (4.15): The main results of this work and other works

Parameters	This work	Previous works
T_C	67.0°C (from dynamic viscosity measurements) 67.8°C (from electrical conductivity measurements)	65.3°C [a]
X_C	33.9%	36.50% [a]
E_a	4.0128($J \times 10^{-20}$) at (0.00%) 5.1325($J \times 10^{-20}$) at (10.17%) 6.0481($J \times 10^{-20}$) at (20.18%) 6.4842($J \times 10^{-20}$) at (50.00%)	-
σ_∞	151448.40μS/cm at (0.00%) 3355126.00μS/cm at (10.17%) 26429728.00μS/cm at (20.18%) 29151007.00μS/cm at (50.00%)	-
$\chi_\sigma \upsilon$	0.032	0.037 [b] 0.037 [c]
σ_o	30.26μS/cm 26.85 μS/cm	-
α	0.9439	-

[a]: (Wilcox and Bailey, 1929), [b]: (D'Arrigo *et al*, 1977), [c]: (Klein *et al*, 1978)

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Appendix A

Viscosity of Water at Different Temperature

Table A.1: The viscosity of water as a function of temperature

T(^{o}C)	η(cP)
32.0	0.765
40.0	0.653
45.0	0.596
50.0	0.547
55.0	0.504
57.0	0.489
58.0	0.481
59.0	0.474
60.0	0.467
61.0	0.460
62.0	0.453
63.0	0.447
64.0	0.440
65.0	0.434
66.0	0.428
67.0	0.422
68.0	0.416
69.0	0.410
70.0	0.404
75.0	0.378

Appendix B

Density of Phenol

The density of phenol which was used in the experiment was measured at different temperature and listed in table A.1 as given below:-

Table A.1: The density of phenol as a function of temperature

T(^{o}C)	ρ(gm/cm^3)
40.0	1.0440
45.0	1.0530
50.0	1.0530
55.0	1.0530
57.0	1.0520
58.0	1.0510
59.0	1.0550
60.0	1.0240
61.0	1.0230
62.0	1.0320
63.0	1.0310
64.0	1.0300
65.0	1.0290
66.0	1.0290
67.0	1.0370
68.0	1.0370
69.0	1.0360
70.0	1.0490
75.0	1.0480

السلوك الحرج للموصلية الكهربائية للخليط الثنائي

المكون من الماء والفينول

إعداد

هبة زهرة

إشراف

أ. د. عصام راشد عبد الرازق

د. إياد سعد الدين

ب

السلوك الحرج للموصلية الكهربائية للخليط الثنائي المكون من الماء والفينول

إعداد

هبة زهرة

إشراف

أ. د. عصام راشد عبد الرازق

د. إياد سعد الدين

الملخص

تم قياس معامل اللزوجة للخليط الثنائي للماء والفينول عند درجات حرارة وتراكيز مختلفة لتحديد الدرجة الحرجة والتركيز الحرج. الدرجة الحرجة كانت تساوي 67.0^oC من خلال قياسات اللزوجة و $67.8^o C$ من خلال قياسات الموصلية الكهربائيثة أما التركيز الحرج كان يساوي 33.90%. تم قياس الموصلية الكهربائية عند درجات حرارة وتراكيز مختلفة. تم تحليل نتائج الموصلية الكهربائيةلتراكيز مختلفة (%50.50 , 20.80% , 10.17%, 0.00%) باستخدام معادلة أرهينيوس. تم حساب طاقة التفعيل والموصلية الكهربائية عند درجات الحرارة العالية لعدة تراكيز مختلفة. تم تحليل نتائج الموصلية الكهربائية فوق الدرجة الحرجة باستخدام قانون القوة. وتم حساب الجزء غير الحرج من الموصلية الكهربائية وكان يساوي$\mu S/cm$30.26 وتم حساب الأس الحرج وكان يساوي 0.032. تم حساب الموصلية الكهربائية المولارية ورسمها باستخدام قاعدة ولدن وكان ميل الرسمة يساوي 0.9439 ومن خلالها تم تصنيف الخليط الثنائي انه محلول أيوني ضعيف.