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Thermal Expansion Anomaly Near the Critical Consolute of Triethylamine-Water

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Dedication

To my parents, my husband and my son Abdelkrim with respect and love.

Acknowledgment

Thanks go to my Supervisor Prof. Issam Rashid Abdelraziq for his guidance and generous support and to my Co-Supervisor Dr. Mohammad Abu-Jafar for his efforts and help in the completion of this thesis.

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I would also like to thank my parents, my husband, my son, my sisters and my brothers for their patience and prayers for me.

الإقرار

أنا الموقعة أدناه مقدمة الرسالة التي تحمل العنوان:

Thermal Expansion Anomaly Near the Critical Consolute of Triethylamine-Water

أقر بأن ما اشتملت عليه هذه الرسالة إنما هو نتاج جهدي الخاص, باستثناء ما تمت الإشارة إليه حيثما ورد, وان هذه الرسالة ككل من أو جزء منها لم يقدم من قبل لنيل 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: اسم الطالبة: رنان منير بني فضل

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Date: التاريخ: 2017/1/17

Table of Contents

List of Tables

List of Figures

List of Abbreviations

TEA	Triethylamine
T_c	Critical temperate
X	Concentration by weight
X_c	Critical concentration by weight
T'_c	Pressure derivative of critical temperature along critical line
α_{pc}	Critical amplitudes of isobaric thermal expansion
α_{pb}	Background amplitudes of isobaric thermal expansion
η	Viscosity of the liquids
ρ	Mass density
ρ_c	Critical mass density
η_T	Dynamic viscosity at absolute temperature T
T	Absolute temperature
α	Critical exponent (~ 0.11)
τ	Reduced temperature $= \dfrac{T - T_c}{T_c}$
C_{pc}	Critical amplitude of specific heat at constant pressure
α_p	Isobaric thermal expansion
cP	Centipoise
UCST	Upper critical solution temperature
LCST	Lower critical solution temperature
ξ_o	Critical amplitude of correlation length
K_B	Boltzmann's constant

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Abstract

The dynamic shear viscosity of Triethylamine–water (or TEA–water) binary liquid mixture has been measured at temperature range 291.0-293.0K and concentration range 0.0%- 100.0% by weight of Triethylamine. The results show, as the temperature increases the dynamic viscosity values for each concentration decreases and the anomaly in the dynamic viscosity values was observed at the critical temperature T_c= 291.6K. The critical temperature T_c= 291.6K which has been found is in agreement with the value measured by Abdelraziq (Abdelraziq, 2003). On the other hand, the dynamic viscosity value increases as the concentration by weight of Triethylamine increases until the concentration becomes X_c = 72.3%. After that the dynamic viscosity value starts decreasing. The critical value of dynamic viscosity η_c was measured at the critical temperature T_c= 291.6K and the critical concentration X_c= 72.3% and was found to be 4.816cP. In addition, the mass density of the binary liquid mixture has been measured at the same concentration range and at the same temperature range. According to these results, the density decreases as temperature increases for each

concentration. Likewise, the density of the binary liquid mixture also decreases as the concentration of Triethylamine increases for each temperature. Moreover, the critical mass density ρ_c at the critical concentration X_C= 72.3 % and the critical temperatures T_c= 291.6K was measured and found to be $0.7752(\frac{gm}{cm^3})$.

The isobaric thermal expansion coefficient has been also studied near the critical temperatures T_c= 291.6K and at the critical concentration X_C = 72.3 %. Consequently, the critical and background isobaric thermal expansion coefficient were found to be α_{pc}= $4\times10^{-5}K^{-1}$ and α_{pb}= $4.304\times10^{-3}K^{-1}$, respectively.

The critical amplitude of specific heat at constant pressure (atmospheric pressure) C_{pc} for Triethylamine–water binary liquid mixture was also calculated and found to be $3.084\frac{J}{gm.K}$. This value was close to the value calculated by Abdelraziq which equal to $2.58 \frac{J}{gm.K}$ (Abdelraziq, 2003). In addition, the value of the pressure derivative of the critical temperature along the critical line T_c' was calculated and found to be 4.88 x $10^{-3}\frac{cm^3K}{J}$.

Chapter One

Introduction

1.1 Binary liquid mixtures

A binary liquid system is two liquids mixed together at the same pressure and temperature. The mixed components have extensive properties different from the unmixed components like volume, enthalpy, entropy, etc. (Saville, 2011).

Partially miscible mixtures usually form multiphasic systems. The phase separation occurs when two liquids combine in certain proportions and form partially miscible mixture. The two phases would be identical or would have different compositions. This difference is called a miscibility gap. The miscibility gap decreases as the temperature increases until at the upper critical solution temperature. At the critical temperature, the two liquid phases become identical, and the system becomes a single binary liquid mixture (Devoe, 2015).

The liquid mixtures have an upper critical solution temperature (UCST) if the phase separation occurs below the critical solution temperature. The two components of the liquid mixtures are completely soluble above the upper critical solution temperature, but they are partially soluble below this temperature. A mixture of two hydroxyl-compounds are a case in point. However, these mixtures have a lower critical solution temperature (LCST) if they components form two phases (partially soluble) above this

temperature and if they become completely soluble below this temperature. One example of this is Triethylamine- water mixtures. A few systems, like nicotine-water system, have both upper and lower critical temperatures which completely miscible above an upper critical solution temperature and below a lower critical solution temperature, separating into two phases between these temperatures.

There are two types of liquid mixtures with lower critical solution temperature. The first type contains liquids with a great difference in molecular size between the two components. The second type contains liquids where the interaction between the components occurs by means of a hydrogen bond like Triethylamine- water mixtures (Counsell *et al*., 2009).

The critical solution temperature is the temperature reached by increasing the temperature in some systems or lowering it in others. The two mixed liquids become completely miscible at this temperature (Barker and Fork, 1953).

The coexisting phase curves can be used to describe at which temperature range the binary mixtures are completely soluble and have two phases as shown in Figures 1.1, 1.2 and 1.3.
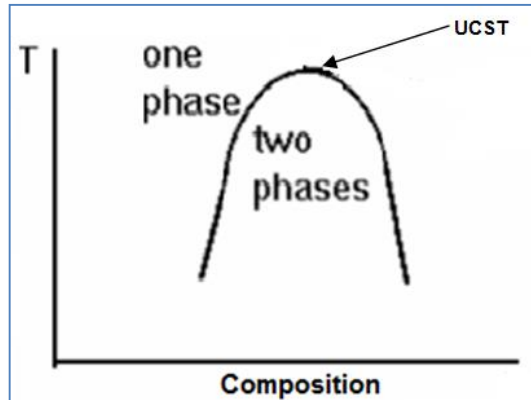
Fig. 1.1: Liquid mixtures with upper critical solution temperature, like Hexane-Nitrobenzene mixtures (Barker and Fork, 1953).
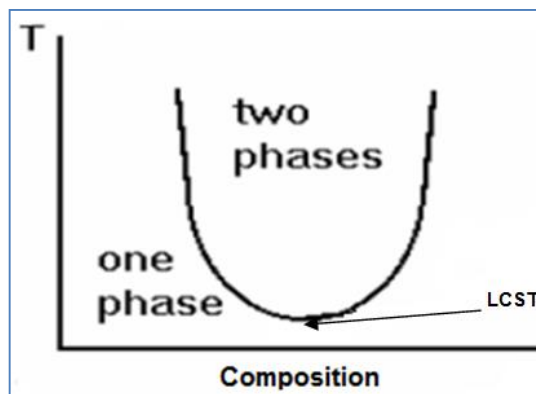
Fig. 1.2: Liquid mixtures with lower critical solution temperature, like 2-Methyloamine-water and 3-Ethyloamine-water (Barker and Fork, 1953).
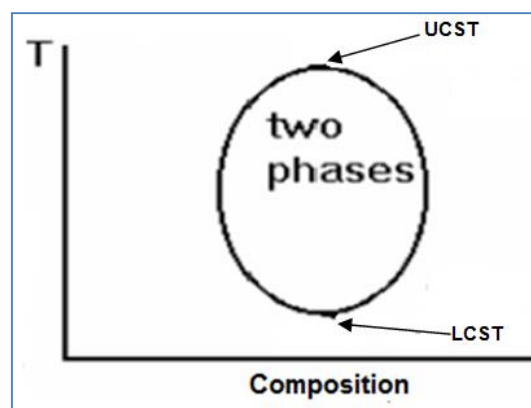
Fig. 1.3: Liquid mixtures with both upper and lower critical solution temperatures, like Nicotine-water, Glycerol-water (Barker and Fork, 1953).

The critical point is reached when coexisting phases become indistinguishable in terms of any physical property (Prausnitz *et al.*, 1999).

1.2 Previous studies

Scheibner and his group measured the volume as a function of temperature in the one-phase region of Methanol-Cyclohexane binary mixture near the critical solution point. They found an anomalous or a critical divergence in the thermal coefficient expansion near the critical point. This divergence was well described by a power law with critical exponent 0.11 ±0.07 in the range 11 mk $< T - T_c < 6.4$ K. The critical exponent value agreed with the predicted value of Ising model which equaled 0.125. They also observed unusual behavior over a small region of temperature centered at $T - T_c = 15$ mK. They found a mysterious hump at $T - T_c \approx 15$ mK similar to that seen by Morrison and Knobler at $T - T_c \approx 90$ mK and a stronger divergence for $T - T_c \leq 11$ mK (Scheibner *et al.*, 1978; Morrison and Knobler, 1976).

Greer and Jacobs measured the density as a function of temperature in the one-phase region above the critical solution temperature of two different composition samples of Polystyrene-Cyclohexane. Their results showed no critical anomaly in both samples. This means that the thermal expansion amplitude was very small (Greer and Jacobs, 1980).

Sandra and his group measured the density as a function of temperature in the one-phase region near the consolute point in Nitroethane-3-Methylpentane. They found a critical anomaly in the thermal expansion (Sandra *et al.*, 1975).

Graham and Knobler measured the volume as a function of temperature near the critical point of isobutyric acid-water. They found evidence of a critical anomaly in the thermal expansion and a small range of temperature in which it was negative. They found that the thermal expansion coefficient was in the range of 0.08-0.14 which could be compared to the Ising model (Graham and Knobler, 1976).

Miller, Clerke and Greer measured the mass density as a function of temperature at a near critical point in the one phase region above the upper critical solution point of Benzonitrile-Isooctane. They found that the density decreased near the critical point (Miller *et al.*, 1983).

Klein and Woermann calculated the critical part of specific heat and the coefficient of thermal expansion of binary liquid mixtures of critical composition from light-scattering data by using the two-scale-factor universality hypothesis of critical phenomena (Klein and Woermann, 1978).

D'Arrigo and his group measured the shear viscosity as a function of molar composition and temperature near the critical point of Aniline–Cyclohexane binary system. They analyzed the data using the mode coupling theory (D'Arrigo *et al*., 1977)**.**

Losada and his group studied the specific heat capacity anomaly near the upper critical point of the Nitrobenzene–Tetradecane system (Losada *et al*., 2010).

Sanchez and his group measured the heat capacity of the critical 3-Methylpentane - Nitroethane binary mixture. An analysis of these results

yielded universal parameters in good agreement with those expected for the d=3, n=1 (three-dimensional Ising Model) universality class (Sanchez *et al., 1983)*.

Chen and his group obtained the isothermal vapor liquid equilibria for benzene + nonane, toluene + o-xylene, m-xylene + sulfolane and o-xylene + sulfolane binary mixtures. They measured the Liquid and vapor densities, using two vibrating tube densitometers. They also determined the excess molar volumes of the liquid phase (Chen *et al*., 2010).

Wang and his group determined the density and tension of 1-ethyl-3-Methylimidazolium l-lactate ionic liquid. They also calculated the coefficients of thermal expansion from the experimental density results. They measured the density and surface tension for the binary mixture systems at a temperature of 298.15K and atmospheric pressure (Wang *et al*., 2011).

Akasaka and his group measured the vapor–liquid coexistence curves near the critical point of the binary mixture of 2, 3, 3, 3-Tetrafluoropropene (R-1234yf) + Difluoromethane (R-32). They identified the critical temperatures, critical densities, and critical molar volumes of the mixtures (Akasaka *et al*., 2013).

Zhang and his group measured the density over the whole concentration range of the binary system of polyethylene glycol and water (PEG 300-water) at five temperatures from 298.15 to 318.15 K. They calculated the excess molar volume and viscosity deviation. The values of the excess

molar volume for all mixtures were negative while the values of viscosity deviation were found to be positive (Zhang *et al*., 2012).

Chang and his group identified the order-parameter correlation function near the critical mixing point of 3-Methylpentane-Nitroethane. To this end, measured the intensity of scattered light as a function of temperature (Chang et al., 1979).

Abdelraziq and his group measured the ultrasonic velocity and absorption at the critical concentration as a function of temperature and frequency and the viscosity of the binary mixture of carbon tetrachloride and coconut oil (Abdelraziq *et al*., 1992).

Abdelraziq measured the shear viscosity coefficients of Nitroethane and 3-Methylpentane. The anomaly of shear viscosity was detected as a function of temperature and concentration (Abdelraziq, 2002).

Elayyat measured the shear viscosity of a binary liquid mixture of water and phenol at different temperatures and different concentrations and identified the critical temperature and critical concentration. The mutual force between binary mixture molecules was found to be a weak attractive force by calculating a large intermolecular force range L of water and phenol molecules in a binary mixture. The critical amplitude of specific heat at constant pressure was calculated (Elayyat, 2015).

Reehan measured the dynamic shear viscosity of the binary liquid mixture water – phenol. She detected a dynamic shear viscosity anomaly at the critical temperature 67 °C and critical concentration 33.9% by weight of phenol. The specific heat at constant pressure and critical point and the

background isobaric thermal expansion coefficients has been calculated (Reehan, 2015).

1.3 Objectives

The aim of this study is twofold: first, to study the anomalous and background terms of the thermal coefficient of expansion near the critical consolute of Triethylamine–water system and second, to verify the power law of isobaric thermal expansion.

The following physical properties will be determined:

- Critical temperate T_c.

- Critical concentration X_c.

- Critical amplitude of isobaric thermal expansion α_{pc} and background amplitude of isobaric thermal expansion α_{pb}.

- The Critical amplitude of specific heat at constant pressure C_{pc}.

- Pressure derivative of critical temperature along the critical line $T_c' = \frac{dT_c}{dP}$.

Chapter Two

Theory

2.1 Viscosity

Viscosity is the friction between surfaces that are completely separated due to the internal friction of the liquid, or due to the fluid's resistance to flow (Schmid *et al*., 2004).

Newton's law of viscosity defines viscosity as the ratio of shear stress to shear rate for a given temperature and pressure. The fluids which follow the Newton's law of viscosity are called Newtonian fluids where the viscosity is constant and independent of the shear rate. The fluids which do not obey the Newton's law of viscosity are called Non-Newtonian where the viscosity is not constant and dependent on the shear rate as shown in Figure 2.1. Dynamic viscosity, as defined in Newton's law of viscosity, is the coefficient of viscosity, while Kinematic viscosity is the dynamic viscosity divided by the density (George *et al.*, 2013; Bansal, 2005).
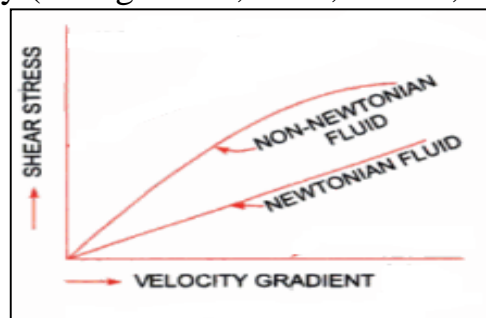
Fig. 2.1: Newtonian and Non-Newtonian Fluids (Bansal, 2005).

The viscosity of Newtonian fluids is affected by temperature, pressure, and composition (solutions and mixtures) (Themelis, 1995).

Newton's law of viscosity

$$\frac{F}{A} = \eta \frac{dv}{dz} \qquad (2.1)$$

Where $\frac{F}{A}$ is the shearing stress, $\frac{dv}{dz}$ is the velocity gradient in a fluid, v is the *viscosity* and dz is the *distance between layers*.

The kinematic viscosity (v) is a measure of resistive flow of a fluid under the influence of gravity and it's equal to

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

Where ρ is the density of the fluid (Elert, 1998-2015).

The fluid with high viscosity slide on surfaces hardly, tends to adhere and are unlikely to cause vorticity effects. These are due to its relatively strong molecular bindings. Therefore, it tends to oppose greater resistance to changing shape due to external forces, like the force of gravity (Coletto, 2003).

2.2 Viscosity and Concentration

Concentration is the relative amount of a given component within a mixture. There are many types of concentrations. One type is mass fraction where the mass fraction of B in the mixture X is

$$X = \frac{m_B}{m_A + m_B} \qquad (2.3)$$

When two liquids are mixed adiabatically, the mass is conserved, but the volume and temperature are not (Radermacher and Hwang, 2005).

The viscosity behavior of binary mixtures is very complex and depends on the mixing ratio as shown in Figure 2.2
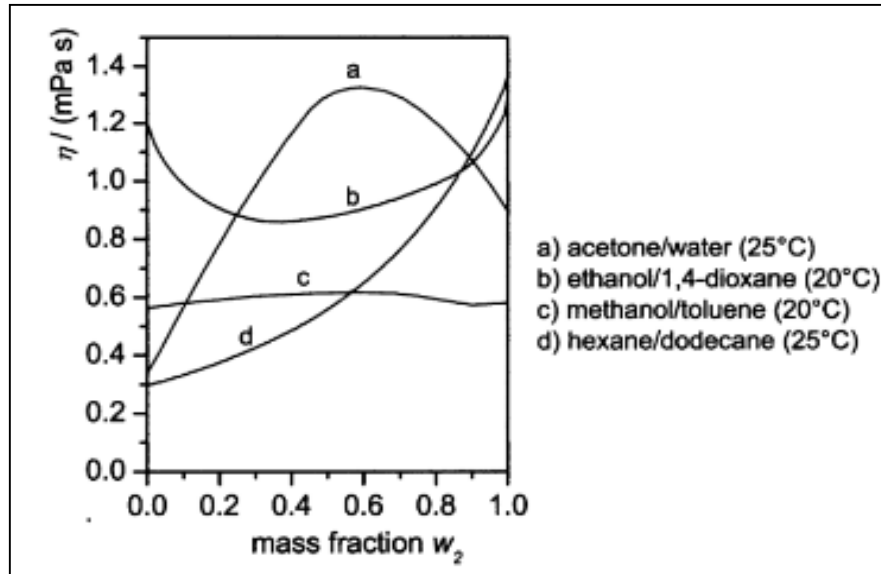
Fig. 2.2: Viscosity of mixtures of two solvents as a function of the mass fraction of the second component (Kulicke and Clasen, 2004).

2.3 Viscosity and Temperature

The viscosity of liquids decreases as temperature increases (White, 1992; Themelis, 1995). As temperature increases, the average speed of the molecules in a liquid increases and the amount of time they spend in contact with their nearest neighbors decreases. Then the average intermolecular forces decrease (Elert, 1998-2015). The viscous forces in a fluid are due to two causes: the intermolecular cohesive forces and the molecular momentum transfer. Liquid molecules are close together. Then the intermolecular cohesive forces dominate the molecular momentum transfer. However, these forces decrease as temperature increases. The viscosity decrease is as a result of the intermolecular cohesive forces decrease. The viscosity also decreases as the temperature increases as shown in the Figure 2.3.
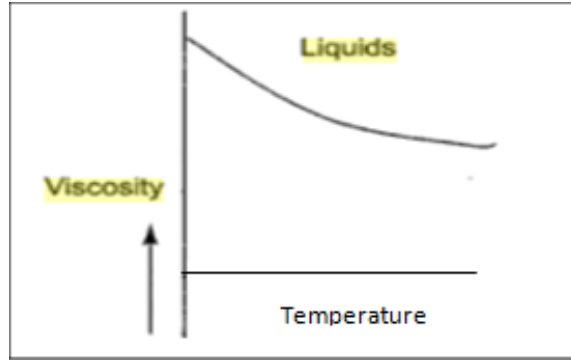
Fig. 2.3: Viscosity as function of temperature (Nag *et al*., 2008).

The relation between viscosity and temperature for liquids

$$\eta_T = A\,e^{B/T} \tag{2.4}$$

Where η_T is the dynamic viscosity at absolute temperature T, A and B are constants for a given liquid (Pandhare *et al.,* 2009).

2.4 Critical Points and Power Laws

The variety of systems that show critical phenomena can be divided into equivalence classes, also called universality classes. The critical universal quantities of all the systems within a given universality class are identical. Two factors are thought to distinguish the various universality classes: (1) the spatial dimensionality of the system (d) and (2) the number of components of the order parameter (n). Pure fluids (liquid-gas systems) and binary fluids belong to the universality class where d=3 and n=1 (Habdas, 1999).

Along various thermodynamic paths, several quantities exhibit simple power law dependence close to the critical point (Flewelling *et al*, 1996). Critical power laws represent the asymptotic behavior of a number of properties when the critical point approaches along specific thermodynamic paths (Sengers, 1980).

$$Y = Y_\circ \; \tau^{-\psi} \tag{2.5}$$

Where Y is a thermophysical property, ψ is the critical exponent which depends on thermophysical properties and τ is the reduced temperature $[\frac{T - T_c}{T_c}]$ (Zappoli *et al.*, 2015).

The critical exponents describing these relationships are universal and should depend only on a universality class determined by the order parameter (n) and spatial dimensionality of the system (d) (Flewelling *et al*, 1996).

The Renormalization Group approach provides a method for proving scaling relations, and for calculating exponents and universal amplitude ratios as expansions are in $\varepsilon = 4 - d$ or $1/n$, d the dimensionality and n the number of components of the order parameter (Hohenberg *et al.*, 1976).

The thermal expansion can be represented by a power law:

$$\alpha_p = \alpha_{pc}^+ \; \tau^{-\alpha} + \alpha_{pb}^+ \qquad \tau > 0, \; T > T_c \tag{2.6}$$

$$\alpha_p = \alpha_{pc}^- \; \tau^{-\alpha} + \alpha_{pb}^- \qquad \tau < 0, \; T < T_c \tag{2.7}$$

Where α_{pc} is the critical amplitude of the isobaric thermal expansion, α_{pb} is the background amplitude of the isobaric thermal expansion and α is the critical exponent and equal to 0.11 (Abedelraziq, 2003).

The two-scale factor universality is a relationship predicted by the Renormalization Group theory of critical. The universal relationship between the corresponding amplitudes implies the ratio

$$R_\xi = \xi_\circ \; \left(\frac{\alpha \, T_c \alpha_{pc}}{K_B T_c'}\right)^{1/d} = \xi_\circ \; \left(\frac{\alpha \, C_{pc}\rho_c}{K_B}\right)^{1/d} \tag{2.8}$$

Where C_{pc} is the Critical amplitude of specific heat at constant pressure, T_c' is the pressure derivative of the critical temperature along the

critical line, ξ_o is the correlation length of the binary mixture and K_B is the Boltzmann's constant. The theoretical estimates for the ratio are $R_\xi = 0.2547 \pm 0.007$ for high temperature series expansion and 0.2699 ± 0.0008 from the renormalization group theory and d=3 in the dimension of the space (Sengers, 1980: Abdelraziq, 2003).

Chapter Three

Methodology

3.1 Triethylamine

Triethylamine (or TEA) is a strong alkaline amine with high water solubility which equal to $15\frac{gm}{cm^3}$ and Chemical formula $(CH_3CH_2)_3N$, see Figure 3.1. It is a colorless flammable liquid with a fishy to strongly ammoniacal odor (Budavari, 1989).
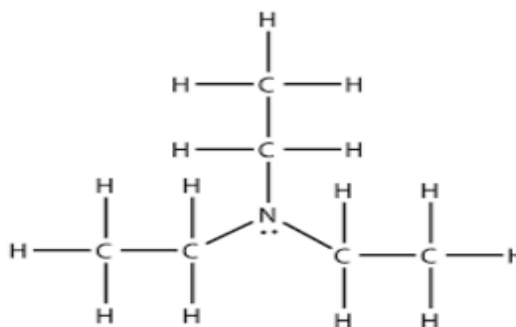
Fig. 3.1: Molecular structure of Triethylamine

Some physical and chemical properties of Triethylamine are given in Table 3.1.

Table 3.1: physical and chemical properties of Triethylamine

Property	Triethylamine
Molecular weight	$101.21\frac{gm}{mol.}$
Melting point	$-115^\circ C$
Boiling point	$89^\circ C$
Density	$0.729 \frac{gm}{cm^3}$ at $20^\circ C$

Triethylamine is present in a many processes in the chemical and pharmaceutical industries. It is used as a catalyst in polymer production in

foundries. In addition, it is used as a chemical intermediate in the production of pharmaceuticals, soaps and rubber products (Corn, 1993). Acute (short-term) exposure of humans to Triethylamine vapor causes eye irritation, corneal swelling, and halo vision. People have complained of seeing a blue haze or having smoky vision. Acute exposure can irritate the skin and mucous membranes. Chronic (long-term) exposure of workers to Triethylamine vapor has been observed to cause reversible corneal edema. Chronic inhalation exposure has resulted in respiratory and hematological effects and eye lesions in rats and rabbits. No information is available on the carcinogenic effects of Triethylamine in humans (Budavari, 1989). Occupational exposure may occur primarily via inhalation and dermal contact during its manufacture and use. The general population may be exposed to Triethylamine from ingesting contaminated food; Triethylamine has been identified in broiled beef.

3.2 Experimental Apparatus

3.2.1 Density apparatus

The density of the pure water and pure Triethylamine is calculated by using

- 10 ml Pycnometer is made of glass with a close fitting ground glass stopper, see Figure 3.1. The Pycnometer is used to measure the density of the pure water and pure Triethylamine with accuracy ±0.005%.

Fig. 3.2: 10 ml Pycnometer

- HX-Z electronic balance with accuracy ±0.005% to measure the mass of the sample which is shown in Figure 3.2

Fig. 3.3: Electronic Balance

The mass density of the pure water and pure Triethylamine was calculated in $\frac{gm}{cm^3}$ using

$$\text{Density} = \frac{mass\ of\ the\ substance}{10\ ml}$$

3.2.2 Julabo F25 Refrigerated and Heating Circulator

The temperature of the samples was controlled by using Julabo F25 Refrigerated and Heating Circulator with accuracy ±1% as Figure 3.3 shows.

Fig. 3.4: Julabo F25 Refrigerated and Heating Circulator

3.2.3 Capillary Viscometer

The viscosity of the samples was calculated by using Capillary Viscometer with viscometer constant K 0.28 ($\frac{mm^2}{s}$), see Figure 3.4.

Fig. 3.5: Capillary Viscometer

When two fluids of equal volume are placed in identical Capillary Viscometers and allowed to flow under the influence of gravity, a viscous fluid takes longer than a less viscous fluid to flow through the tube (Elert, 1998-2015).

3.2.4 Digital Thermometer

The temperature of the samples was measured by using the Digital Thermometer with ±0.5% accuracy Figure 3.5 shows.

Fig. 3.6: Digital Prima Long Thermometer

3.2.5 KK-5853 Quartz Timer

The KK-5853 Quartz Timer stopwatch is used to measure the time of flow from the mark points in the capillary with ±0.05% accuracy.

Fig. (3.7): KK-5853 Quartz Timer

3.3 Viscosity Measurements

The density of pure water and pure Triethylamine were measured by using the Pycnometer and the electronic balance.

Samples prepared for Triethylamine –water binary mixture were in the range of concentrations 0.00% to 100.00% by weight of Triethylamine.

The concentration of Triethylamine –water binary mixture by weight of Triethylamine is

$$X_{\text{Triethylamine}} = \frac{\rho_{\text{ triethylamine}} \times V_{\text{ triethylamine}}}{\rho_{triethylamine} \times V_{triethylamine} + \rho_{water} \times V_{water}} \tag{3.1}$$

Where $\rho_{\text{triethylamine}}$ is the density of the Triethylamine, $V_{\text{triethylamine}}$ is the volume of the Triethylamine, ρ_{water} is the density of the water and V_{water} is the volume of the water.

The viscosity of pure water was calculated using the equation

$$\text{Log}_{10}\,\eta = \frac{1301}{998.33 + 8.1855\,(T-20) + 0.00585\,(T-20)^2} \tag{3.2}$$

Where T is the temperature in the range of 0.0-20.0 °C (David, 1985).

The viscosity of the Triethylamine –water binary mixture is calculated by measuring the densities of the water and the Ttriethylamine –water binary mixture as well as the time of flow of each concentration. The relationship between viscosity and the time of flow is

$$\eta_1 = \eta_2 \frac{\rho_1 \times t_1}{\rho_2 \times t_2} \tag{3.3}$$

Where η_1 is the viscosity of the Triethylamine –water binary mixture, η_2 is the viscosity of the water, ρ_1 is the density of the Triethylamine –water binary mixture, ρ_2 is the density of the water, t_1 is the time flow between the mark points in the capillary of the Triethylamine –water binary mixture and t_2 is the time flow between the mark points in the capillary of the water (Generalic and Eni, 2014).

The critical temperature and critical concentration were found by plotting the viscosity as a function of temperature for each concentration.

The isobaric thermal expansion coefficient can be calculated by using the following equation:

$$\alpha_{\text{p}} = \frac{1}{V}\left(\frac{dV}{dT}\right)_{\text{p}} = \rho\left(\frac{d\rho^{-1}}{dT}\right) \tag{3.4}$$

(Zhou *et al.*, 2015).

The critical isobaric thermal expansion α_{pc} and the background isobaric thermal expansion coefficient α_{pb} were calculated by plotting the α_p as a function $\tau^{-\alpha}$ where τ is the reduced temperature $= \frac{T - T_c}{T_c}$ and α is the critical exponent 0.11(Abdelraziq, 2003).

The critical amplitude of specific heat at constant pressure C_{pc} and the pressure derivative of the critical temperature along the critical line T_c' were calculated by using equation (2.8).

Chapter Four

Results and Discussion

4.1 Critical Temperature and Critical Concentration

The dynamic viscosity is measured at different concentrations of Triethylamine–water binary liquid mixture samples in the range of 0.0%-100.0% by weight of Triethylamine and at different temperatures in the range of 291.0-293.0 K.   The result values are given in Table 4.1.

Table 4.1: Dynamic viscosity values of Triethylamine–water binary liquid mixture at temperature range 291.0-293.0 K for concentrations 0.0%-100.0% by weight of Triethylamine

T(K)	η(cP) 0.0%	η(cP) 6.8%	η(cP) 10.3%	η(cP) 17.9%	η(cP) 21.9%	η(cP) 31.7%	η(cP) 39.5%	η(cP) 49.5%	η(cP) 60.4%	η(cP) 72.3%	η(cP) 85.5%	η(cP) 92.3%	η(cP) 100.0%
291.0	1.053	2.150	2.107	2.269	2.356	2.385	2.767	3.566	3.773	4.355	1.944	1.023	0.600
291.2	1.048	2.119	2.060	2.232	2.371	2.257	2.727	3.534	3.641	4.260	1.843	1.021	0.611
291.3	1.045	2.169	2.162	2.269	2.411	2.313	2.638	3.477	3.589	4.209	1.837	1.027	0.666
291.4	1.043	2.150	2.150	2.222	2.383	2.304	2.616	3.446	3.477	4.211	1.823	1.049	0.639
291.5	1.040	2.162	2.179	2.139	2.464	2.342	2.509	3.429	3.445	4.340	1.803	1.095	0.669
291.6	1.037	2.450	2.465	2.477	2.750	2.892	2.944	3.955	4.149	**4.816**	1.958	1.287	0.679
291.7	1.035	2.045	2.047	2.008	2.257	2.165	2.390	3.350	3.420	4.238	1.764	1.022	0.577
291.8	1.032	2.098	2.043	2.087	2.362	2.344	2.316	3.282	3.308	4.095	1.806	1.103	0.606
291.9	1.030	2.105	2.088	2.046	2.357	2.286	2.304	3.253	3.308	4.023	1.769	1.047	0.611
292.1	1.025	2.072	2.103	2.038	2.339	2.272	2.225	3.196	3.288	3.977	1.768	1.081	0.634
292.5	1.014	2.045	2.109	2.054	2.429	2.266	2.208	3.140	3.221	3.714	1.594	1.057	0.588
292.8	1.007	2.039	2.126	2.024	2.521	2.294	2.156	3.082	3.040	3.515	1.568	1.081	0.662
293.0	1.002	2.010	2.065	1.988	2.474	2.264	2.116	3.060	2.986	3.353	1.529	1.046	0.600
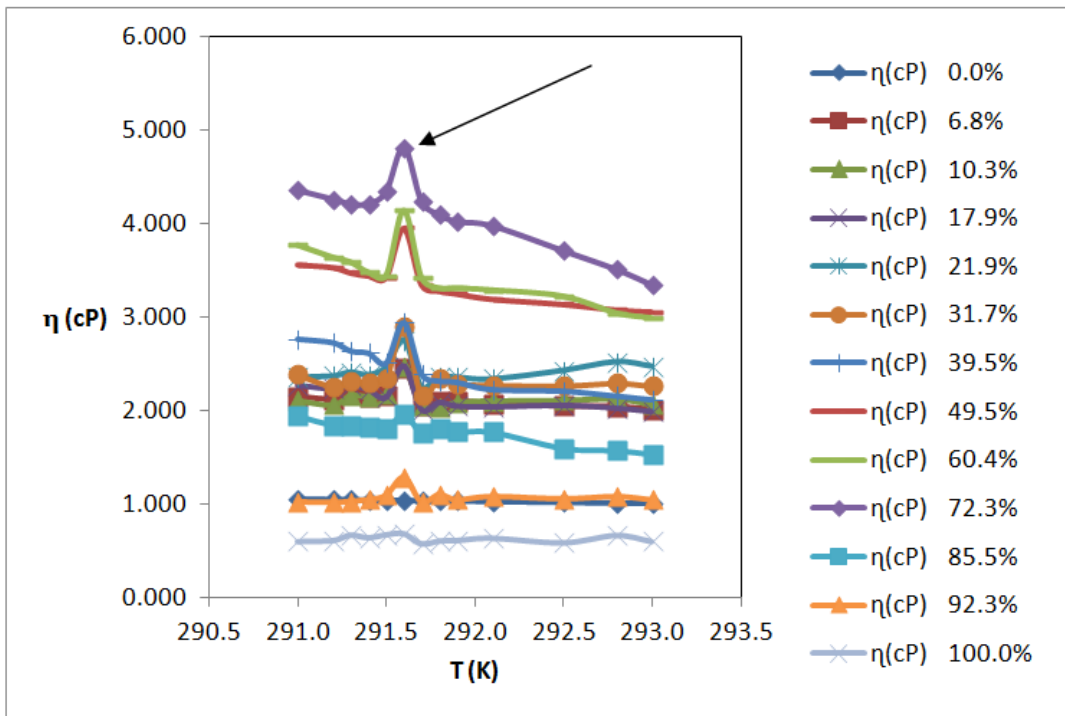
Fig. 4.1: Dynamic viscosity as a function of temperature of Triethylamine–water binary liquid mixture in concentrations ranges 0.0% - 100.0% by weight of Triethylamine

The measured dynamic viscosity values are plotted as a function of temperature for each concentration by weight of Triethylamine in Figure 4.1. The critical temperature T_c appears as anomaly in the dynamic viscosity values at 291.6K, as Figure 4.1 shows. Moreover, the dynamic viscosity values for each concentration decreases as the temperature increases, as shown in Figure 4.1. This is consistent with the fact that as the temperature increases, the intermolecular cohesive force decreases, and then does the viscosity.
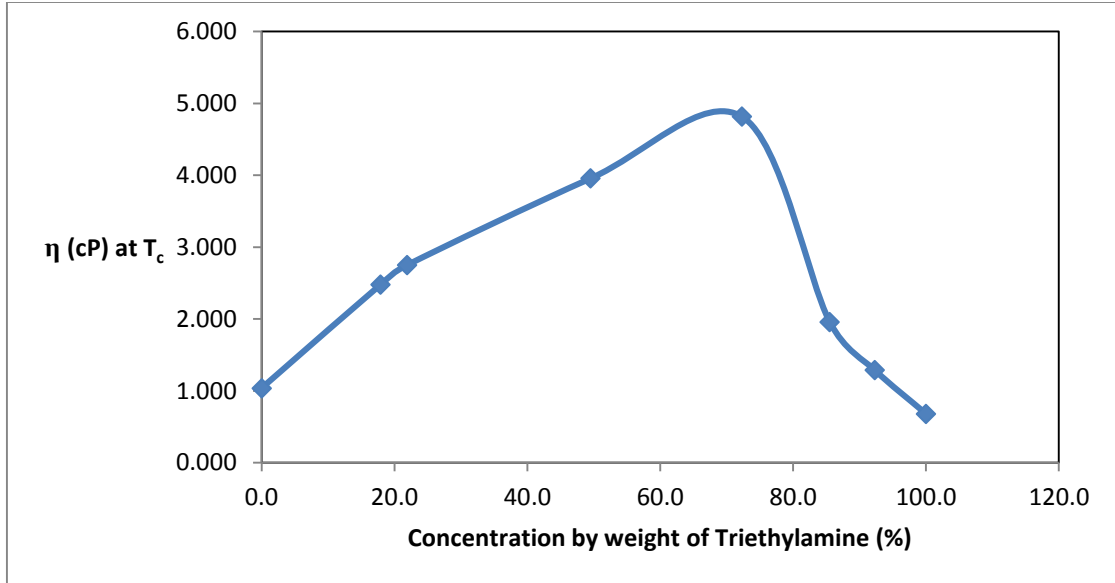
Fig. 4.2: Dynamic viscosity as a function of concentration by weight of Triethylamine for Triethylamine –water binary liquid mixture at critical temperature T_c 291.6 K

The dynamic viscosity as a function of concentration of Triethylamine–water binary liquid mixture at the critical temperature T_c= 291.6K is plotted in Figure 4.2. The result shows, the dynamic viscosity value increases as the concentration by weight of Triethylamine increases until the concentration becomes equal to X_c= 72.3%. After that the dynamic viscosity starts decreasing. At the critical point, the two phases of the binary liquid mixture become one phase. The dynamic viscosity increases at the critical point as a result of intermolecular cohesive force increases. For this reason, the largest value of the dynamic viscosity (the critical value η_c) was measured at the critical point and found to be 4.816cP.

4.2 Mass Density Measurements

The mass density of the Triethylamine–water binary liquid mixtures in the range of concentration 0.0%-100.0% by weight of Triethylamine and temperature range 291.0-293.0 K was measured. The measured values are given in Tables 4.2.

Table 4.2: Mass density values for Triethylamine–water binary liquid mixture at temperature range 291.0-293.0 K and concentration range 0.0%- 100.0% by weight of Triethylamine

| T (K)	$\rho(\frac{gm}{cm^3})$ 0.0%	$\rho(\frac{gm}{cm^3})$ 6.8%	$\rho(\frac{gm}{cm^3})$ 10.3%	$\rho(\frac{gm}{cm^3})$ 17.9%	$\rho(\frac{gm}{cm^3})$ 21.9%	$\rho(\frac{gm}{cm^3})$ 31.7%	$\rho(\frac{gm}{cm^3})$ 39.5%	$\rho(\frac{gm}{cm^3})$ 49.5%	$\rho(\frac{gm}{cm^3})$ 60.4%	$\rho(\frac{gm}{cm^3})$ 72.3%	$\rho(\frac{gm}{cm^3})$ 85.5%	$\rho(\frac{gm}{cm^3})$ 92.3%	$\rho(\frac{gm}{cm^3})$ 100.0%
291.0	0.9459	0.9258	0.9166	0.8994	0.8924	0.8609	0.8468	0.8191	0.7994	0.7774	0.7363	0.7125	0.6937
291.2	0.9444	0.9251	0.9165	0.8975	0.8905	0.8607	0.8448	0.8174	0.7989	0.7768	0.7358	0.7122	0.6934
291.3	0.9442	0.9250	0.9164	0.8967	0.8886	0.8605	0.8447	0.8173	0.7985	0.7767	0.7349	0.7121	0.6930
291.4	0.9437	0.9237	0.9162	0.8958	0.8879	0.8603	0.8445	0.8171	0.7983	0.7759	0.7366	0.7119	0.6926
291.5	0.9433	0.9226	0.9158	0.8957	0.8857	0.8586	0.8432	0.8165	0.7963	0.7756	0.7347	0.7118	0.6925
291.6	0.9432	0.9229	0.9156	0.8942	0.8835	0.8583	0.8430	0.8159	0.7960	**0.7752**	0.7366	0.7113	0.6922
291.7	0.9431	0.9226	0.9152	0.8938	0.8833	0.8580	0.8429	0.8149	0.7930	0.7748	0.7327	0.7111	0.6919
291.8	0.9428	0.9225	0.9151	0.8932	0.8815	0.8578	0.8424	0.8140	0.7890	0.7740	0.7324	0.7107	0.6917
291.9	0.9425	0.9215	0.9149	0.8928	0.8806	0.8571	0.8423	0.8131	0.7871	0.7736	0.7316	0.7106	0.6912
292.1	0.9423	0.9209	0.9145	0.8926	0.8806	0.8569	0.8419	0.8130	0.7845	0.7729	0.7314	0.7103	0.6907
292.5	0.9418	0.9201	0.9144	0.8925	0.8804	0.8561	0.8417	0.8128	0.7836	0.7721	0.7312	0.7101	0.6905
292.8	0.9416	0.9201	0.9143	0.8922	0.8803	0.8557	0.8415	0.8127	0.7834	0.7710	0.7311	0.7097	0.6903
293.0	0.9412	0.9199	0.9123	0.8920	0.8901	0.8552	0.8412	0.8124	0.7805	0.7699	0.7307	0.7095	0.6902
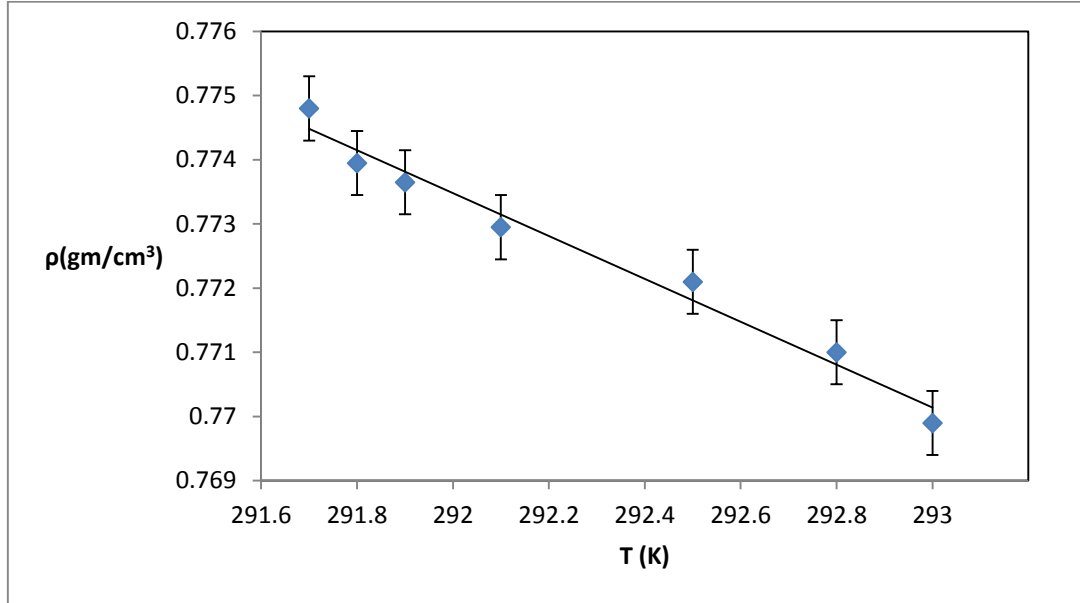
Fig. 4.3: The mass density as a function of temperature for Triethylamine–water binary liquid mixture at critical critical concentration X_c = 31.7% by weight of Triethylamine

The mass density values plotted as a function of temperature for Triethylamine–water binary liquid mixture at critical critical concentration X_c = 72.3% by weight of Triethylamine in Figure 4.3. As shown in the Figure, the density decreases as temperature increases at constant pressure (Atmospheric pressure). This result is expected, because when the temperature increases, the kinetic energy of the binary liquid mixture molecules increases, and the vibrations of the molecules tend to be increased. In addition, the density decreases as a result of vibrations of the molecules increases. Moreover, the critical mass density ρ_c at critical concentration X_C = 72.3 % and the critical temperatures T_c= 291.6K was measured and equal to $0.7752(\frac{gm}{cm^3})$.
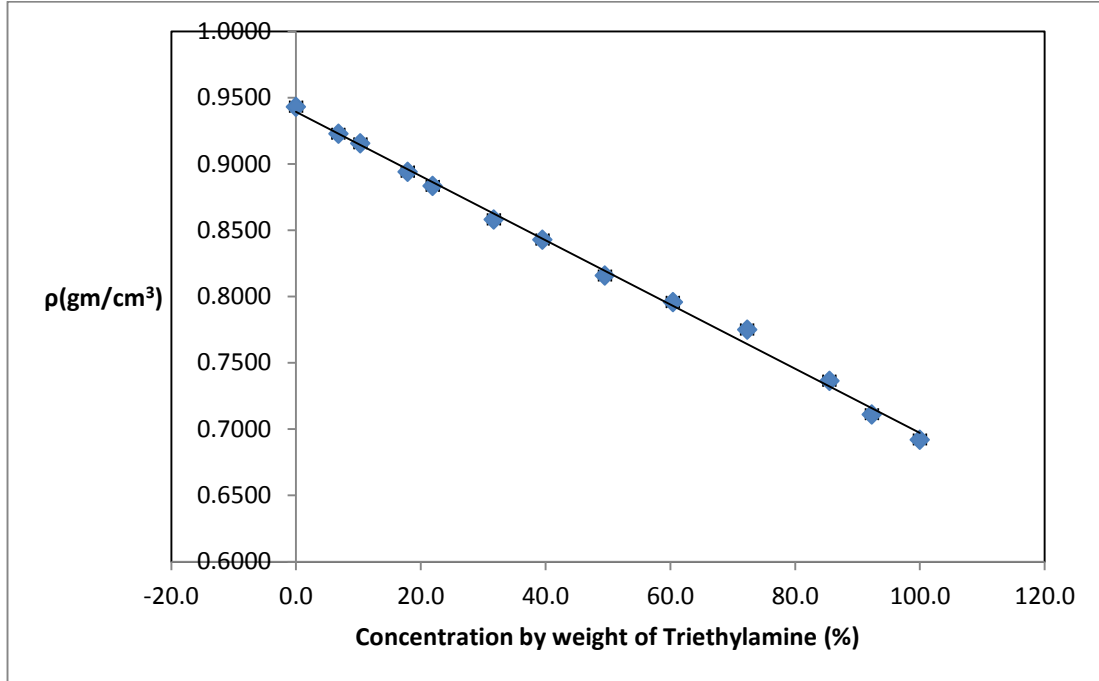
Fig. 4.4: Density as a function of concentration by weight of Triethylamine for Triethylamine –water binary liquid mixture at critical temperature T_c= 291.6 K

The density of Triethylamine–water binary liquid mixture is plotted as a function of concentration at the critical temperature T_c= 291.6K in Figure 4.4. The density of the binary liquid mixture decreases as the concentration of Triethylamine increases, as Figure 4.4 shows. This result is expected because the density of the pure Triethylamine $(0.6922\frac{gm}{cm^3})$ less than the density of the pure water $(0.9432\frac{gm}{cm^3})$.

4.3 Calculation of Isobaric Thermal Expansion Coefficient

The isobaric thermal expansion coefficient can be calculated by using the equation (3.4). The mass density reciprocal at different temperatures of the Triethylamine–water binary liquid mixture at the critical concentration X_c = 72.3% is calculated (See Table 4.3).

Table 4.3: Mass density reciprocal values for the Triethylamine–water binary liquid mixture at different temperatures range 291.7-293.0 K and at critical concentration X_c= 72.3% by weight of Triethylamine

T (K)	$\rho^{-1} (\frac{cm^3}{gm.K})$
291.7	1.2907
291.8	1.2921
291.9	1.2926
292.1	1.2937
292.5	1.2952
292.8	1.2970
293.0	1.2989

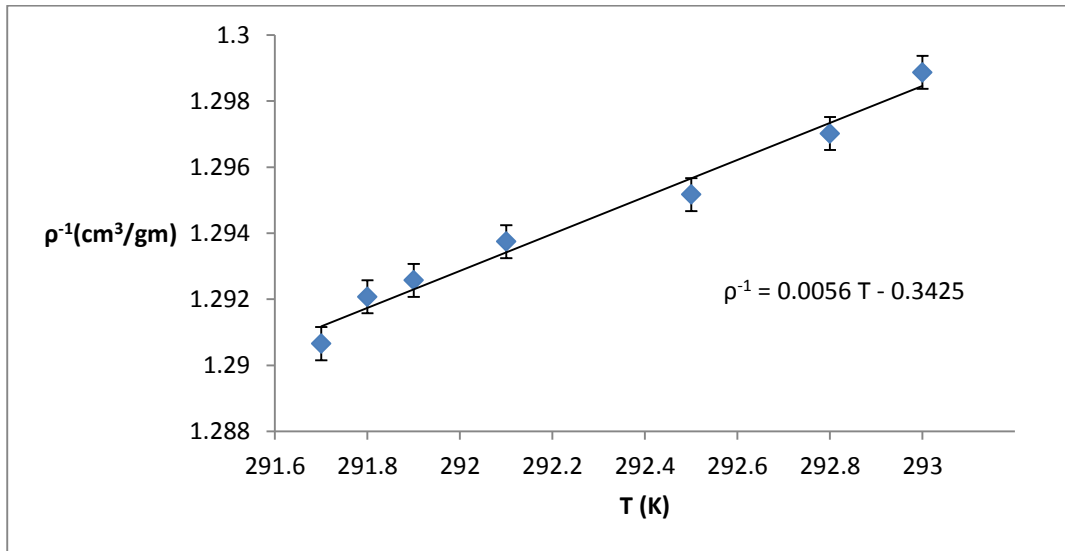**Fig. 4.5:** Mass density reciprocal as function of temperature for the Triethylamine–water binary liquid mixture at critical concentration X_c= 72.3% by weight of Triethylamine

The mass density reciprocal as a function of temperatures is plotted in Figure 4.5. The slope of the curves represented the value of $(\frac{d\rho^{-1}}{dT})$, which equal $0.0056\frac{cm^3}{gm.K}$.

The isobaric thermal expansion coefficient values α_p of Triethylamine–water binary liquid mixture at the critical concentration are calculated and are given in Table 4.4.

Table 4.4: Isobaric thermal expansion coefficient values for the Triethylamine–water binary liquid mixture at critical concentration X_c= 72.3% by weight of Triethylamine

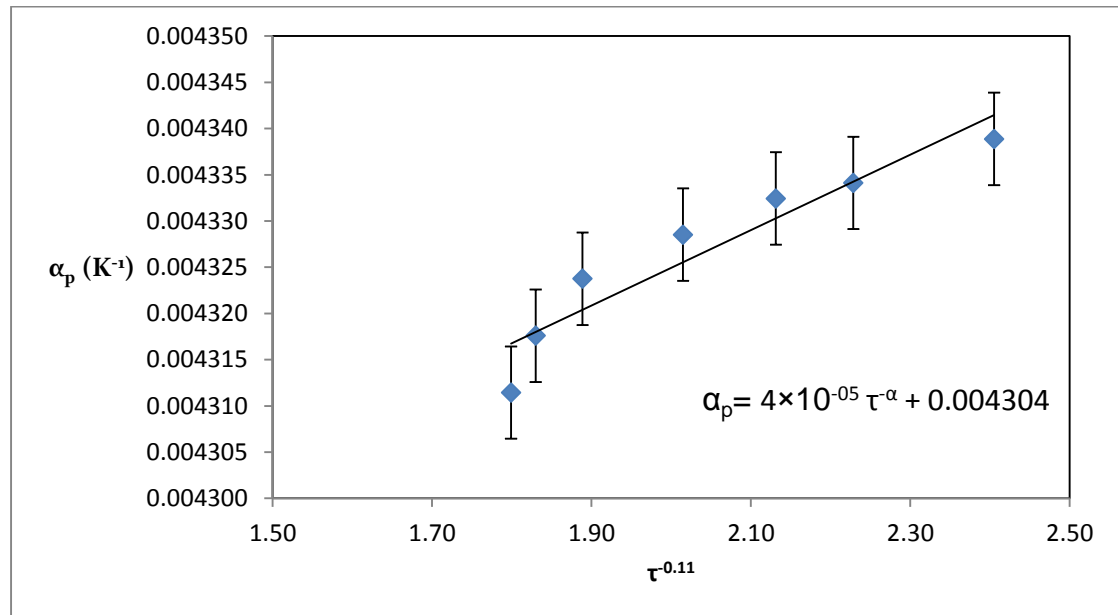| T (K)	$\tau^{-\alpha}$	ρ ($\frac{gm}{cm^3}$)	α_p (K^{-1})
291.7	2.41	0.7748	0.004339
291.8	2.23	0.7740	0.004334
291.9	2.13	0.7736	0.004332
292.1	2.01	0.7729	0.004329
292.5	1.89	0.7721	0.004324
292.8	1.83	0.7710	0.004318
293.0	1.80	0.7699	0.004311

Fig. 4.6: Isobaric thermal expansion coefficient as function of $\tau^{-\alpha}$ for the Triethylamine–water binary liquid mixture at critical concentration X_c 72.3% by weight of Triethylamine

The isobaric thermal expansion coefficient α_p is given by the relation (2.6), and plotted as a function of $\tau^{-\alpha}$, see Figure 4.6, where τ is the reduced temperature and equals to $\frac{T-T_C}{T_C}$. The critical isobaric thermal expansion coefficient α_{pc} is represented by the slope of the line and equal to $4 \times 10^{-5} \text{K}^{-1}$. The background isobaric thermal expansion coefficient α_{pb} is represented by the intercept of the line and equals $4.304 \times 10^{-3} \text{K}^{-1}$.

4.4 Calculation of Specific Heat

The critical amplitude of specific heat at constant pressure C_{pc} for Triethylamine–water binary liquid mixture can be calculated by using Two Scale Factor Universality relation (2.8), Where $R^+_\xi = 0.26711$, the correlation length for Triethylamine–water binary liquid mixture is $\xi_o = 1.0 \text{ Å}$, (Abdelraziq, 2003) the critical exponent $\alpha = 0.11$, Boltzmann's constant $K_B = 1.38 \times 10^{-23} \frac{J}{K}$, and the dimensionality $d = 3$.

The critical amplitude of specific heat at constant pressure C_{pc} for Triethylamine–water binary liquid mixture is calculated and equals $C_{pc} = 3.084 \frac{J}{gm.K}$.

The pressure derivative of the critical temperature along the critical line T'_c is calculated by using the Two-Scale Factor Universality relation (2.8), and equals $T'_c = 4.88 \times 10^{-3} \frac{cm^3 K}{J}$.

Chapter Five

Summary and Conclusion

The measured and calculated parameters for the Triethylamine–water binary liquid mixture are summarized in Table 5.1.

Table 5.1: Summary of the measured and calculated parameters for the Triethylamine–water binary liquid mixture

Parameter	Measured and calculated value
T_c	291.6 K
X_c	72.3%
η_c	4.816 cP
ρ_c	0.7752 $(\frac{gm}{cm^3})$
α_{pc}	4×10^{-5} K^{-1}
α_{pb}	4.304×10^{-3} K^{-1}
c_{pc}	3.084 $\frac{J}{gm.K}$
T_c'	4.88 x 10^{-3} $\frac{cm^3 K}{J}$

The dynamics shear viscosity coefficients were measured and plotted as a function of temperature for the Triethylamine–water binary liquid mixture in concentration range 0.0%-100.0% and temperature range 291.0-293.0K. The results show, the dynamic viscosity values for each concentration decreases as the temperature increases. This is consistent with the fact that as the temperature increases, the intermolecular cohesive force decreases, and then does the viscosity. The critical temperature T_c appears as anomaly in the dynamic viscosity values at 291.6K. The critical temperature T_c= 291.6K which has been found is in agreement with the value measured by Abdelraziq (Abdelraziq, 2003). Moreover, the dynamic viscosity value increases as the concentration

increases until the concentration becomes equal to $X_c = 72.3\%$. After that the dynamic viscosity value starts decreasing.

The two phases of the binary liquid mixture become one phase at the critical temperature T_c=291.6K and critical concentration X_c =72.3%, then the intermolecular cohesive force increases. Accordingly, the dynamic viscosity increases and has the largest value at the critical point. The critical dynamic viscosity η_c was found to be 4.816 cP.

The results of the density measurement for the Triethylamine–water binary liquid mixture shows, as the temperature increases at constant pressure (Atmospheric pressure) the density decreases. This result is expected, because when the temperature increases the kinetic energy of the binary liquid mixture molecules increases and the vibrations of the molecules tend to be increased. In addition, the density decreases as a result of vibrations of the molecules increases. Moreover, the critical mass density ρ_c at critical concentration X_C= 72.3% and the critical temperatures T_c= 291.6 K is found to be 0.7752($\frac{gm}{cm^3}$). The density of the binary liquid mixture decreases as the concentration of Triethylamine increases. This result is expected because the density of the pure Triethylamine (0.6922$\frac{gm}{cm^3}$) is less than the density of the pure water (0.9432$\frac{gm}{cm^3}$).

The isobaric thermal expansion coefficient has been studied near the critical temperatures T_c= 291.6 K and at the critical concentration X_C = 72.3 %. Consequently, the critical and background isobaric thermal

expansion coefficient were found to be equal to $\alpha_{pc} = 4 \times 10^{-5}\,\text{K}^{-1}$ and $\alpha_{pb} = 4.304 \times 10^{-3}\,\text{K}^{-1}$, respectively.

The critical amplitude of specific heat at constant pressure c_{pc} for Triethylamine–water binary liquid mixture is also calculated and was found to be $C_{pc} = 3.084\ \dfrac{J}{gm.K}$. This value was close to the value calculated by Abdelraziq which equal to $2.58\ \dfrac{J}{gm.K}$ (Abdelraziq, 2003). In addition, the value of the pressure derivative of the critical temperature along the critical line T_c' was found to be $4.88 \times 10^{-3}\ \dfrac{cm^3 K}{J}$.

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ب

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الملخص

في هذا البحث تم قياس معامل اللزوجة للمخلوط الثنائي ترايثايلمين والماء عند درجات حرارة ما بين 291.0 كلفن و293.0 كلفن وتركيز ما بين 0.0% و 100.0% من وزن التريثيلامين. أظهرت النتائج أن معامل اللزوجة يقل بزيادة درجة الحرارة عند نفس التركيز, بالاضافة الى ان درجة الحرارة الحرجة ظهرت على شكل شذوذ و زيادة مفاجئة في معامل اللزوجة عند درجة حرارة تساوي 291.6 كلفن . من ناحية اخرى, وجد انه بزيادة تركيز بالنسبة لوزن الترايثايلمين يزداد معامل اللزوجة حتى تركيز 72.3% ثم يبدأ بالنقصان. وتم ايضا قياس معامل اللزوجة الحرجة عند درجة الحرارة الحرجة 291.6 كلفن وعند التركيز الحرج 72.3% ووجد انه يساوي cP 4.816 . بالاضافة الى ذلك تم قياس كثافة المخلوط عند نفس درجات الحرارة ونفس التراكيز. أظهرت النتائج انه بزيادة درجة الحرارة تقل اللزوجة, وان زيادة التركيز تؤدي إلى نقصان الكثافة عند نفس درجة الحرارة. بالاضافة الى ذلك, تم قياس الكثافة الحرجة عند التركيز الحرج 72.3% و عند درجة الحرارة 291.6 كلفن ووجد انها تساوي 0.7752غم/سم^3.

تم حساب أيضا كل من معامل التمدد الحراري الحرج 4×10^{-5} كلفن$^{-1}$, بالإضافة إلى حساب الحرارة النوعية الحرجة للمخلوط الثنائي 3.084$\frac{جول}{كلفن.غم}$ ومشتقة درجة الحرارة الحرجة بالنسبة للضغط 4.88×10^{-3} سم^3 . كلفن/جول.