

Physical Properties Analysis of Amine Based Solvent (STONVENT)

by

NURSYIEFFA BINTI MOHD NOOR

Dissertation submitted to the
Chemical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(CHEMICAL ENGINEERING)

JUNE 2010

Supervisor:

AP Dr Azmi Shariff

Co-Supervisor:

AP Dr. Mohamad Azmi Bustam

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

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Approved by,

(AP Dr Azmi M Shariff)
FYP Supervisor

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

NURSYIEFFA BINTI MOHD NOOR

ABSTRACT

This project concerns the solvent development of STONVENT, an unknown solvent that has the potential to be used in capturing high carbon dioxide, CO₂ content in natural gas which has become a crucial problem in oil and gas industry nowadays. The objectives of this work are: (1) to perform physical analysis of the STONVENT; (2) to study the effect of temperature change on the properties of STONVENT; (3) to study the effect of concentration change on the properties of STONVENT, (4) compare the result with conventional solvent; Diethanolamine, (DEA) and (5) to study the characteristic of STONVENT before and after absorbing CO₂ through Fourier Transform InfraRed (FTIR) Analysis.. This project is an experimental based project and for the time period given, the experimental work will cover physical analysis which consists of the determination of density, refractive index and surface tension and the effect of temperature change and concentration change on the STONVENT. For FYP II, the equipment has been identified and the experimental procedure has been concluded. Thus from the experimental data obtained, it been further analyzed and appropriate discussion been made.

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CHAPTER 1

PROJECT BACKGROUND

1.1 BACKGROUND OF STUDY

It is now scientifically evident that human activities have caused concentrations of greenhouse gases (GHGs) to rise significantly over the last 200 years, contributing to the global warming problem. Therefore, the desire to alleviate this problem has resulted in serious environmental concerns deriving from the need to reduce GHG emissions from industrial sources. Global and national emission reduction targets were set, signed, and ratified or acceded by 124 countries under the 1997 Kyoto Protocol (Rubin, 2001).

Davis and Cornwell (1991) stated that the major greenhouse gases that contribute to the problem are methane (CH_4), carbon dioxide (CO_2), nitrous oxide (N_2O) and halogens such as chlorofluorocarbons (CFCs) and hydrofluorocarbons (HFCs). Among these, CO_2 is the primary contributor to the problem due to its abundance, and is thus a major target for reduction.

1.2 PROBLEM STATEMENT

Carbon dioxide, which falls into the category of acid gases (as does hydrogen sulfide, for example) is commonly found in natural gas streams at levels as high as 80% (Dortmundt and Doshi, 1999). In combination with water, it is highly corrosive and rapidly destroys pipelines and equipment unless it is partially removed or exotic and expensive construction materials are used. Carbon dioxide also reduces the heating value of a natural gas stream and wastes pipeline capacity (Dortmundt and Doshi, 1999).

Based on current technologies, the gas absorption with a chemical reaction process using amine is considered to be the most cost effective and has the best proven operability record. However, the cost of absorption using conventional solvents such as aqueous solutions of monoethanolamine (MEA) is still relatively high. A major reason for the high cost is that the conventional solvents introduce a variety of practical problems including:

- (i) a high energy consumption for the solvent regeneration
- (ii) a high rate of corrosion of the process equipment
- (iii) a fast evaporation rate causing high solvent losses
- (iv) a high rate of degradation in the presence of oxygen.

all these problems translate into high capital and operating costs (Veawab et al, University of Regina).

In addition to that, most of the conventional absorption technologies have been designed to be carried out onshore, at a very low pressure condition (1 atm) and the amine based solvent used only suitable for low pressure operating condition (Veawab et al, University of Regina).

One way to reduce the process cost is to use better solvents in the CO₂ separation process. These solvents should have higher CO₂ absorption capacity as well as faster CO₂ absorption rates. They should also have high degradation resistance, and low corrosivity (Veawab et al, University of Regina).

STONVENT is an unknown solvent that has the potential of having a better performance as compared to the conventional amine based solvent is going to be analyzed physically (density, surface tension and refractive index) in this research. All the properties stated are important to be obtained from the experiment work will be used to design the absorption unit. Plus, the effect of temperature and concentration change with each property also will be studied.

1.3 OBJECTIVES

The objectives of the research project are:

1. To perform physical properties analysis (density, surface tension and refractive index) of the STONVENT.
2. To study the effect of temperature change on the properties of STONVENT.
3. To study the effect of concentration change on the properties of STONVENT.
4. To compare the results obtained with conventional solvent, Diethanolamine (DEA).
5. To study the characteristic of STONVENT before and after absorbing CO₂ through Fourier Transform InfraRed (FTIR) Analysis.

1.4 SCOPE OF STUDY

Basically this study is an experiment based project. Since the time given is quite short, thus this project will be focusing on the solvent development which is the analysis of physical properties of STONVENT. The effect of temperature and concentration change on the properties of STONVENT also will be examined. In addition to that, the properties obtained will be compared to the conventional alkanolamines-- diethanolamine (DEA). Besides, the ability of STONVENT will be studied by characterization analysis which is FTIR Analysis.

1.5 RELEVANCY OF PROJECT

Carbon Capture and Storage (CCS) has become a key technology in climate change mitigation programs worldwide. CCS is well-studied in terms of greenhouse gas emission reduction potential and cost of implementation. Impacts on human health and the environment have, however, received considerably less attention (Veltman et al., 2009). The results which will be presented in this paper can expose great potential of STONVENT that might lead to a better approach for development of advanced technologies in CO₂ capture with minimum environmental effects and operational cost-saving.

1.6 FEASIBILITY OF THE PROJECT WITHIN THE SCOPE AND TIME FRAME

Abide by the suggested milestone, the project scope has been narrowed down to make it feasible and accomplished within 14 weeks.

CHAPTER 2

LITERATURE REVIEW

Increasing energy costs and growing demand for natural gas have driven the development of sour gas fields around the world. About forty percent or 2600 Tcf of the world's natural gas reserves are in the form of sour gas where H_2S and CO_2 compositions exceed 10% volumetric of the raw produced acid gas (Rameshni, WorleyParsons). In some cases the acid gas composition in these reserves is very high and economics of producing pipe line quality gas are marginal. Natural gas almost always contains contaminants or other unacceptable components, including heavy hydrocarbons, mercaptans, mercury, water and the acid gases H_2S and CO_2 .

2.1 TECHNOLOGY OPTIONS FOR CO_2 CAPTURE

A wide range of technologies currently exist for separation and capture of CO_2 from gas streams (Refer to Figure 1 and Table 1), although they have not been designed for power-plant-scale operations. They are based on different physical and chemical processes including absorption, adsorption, membranes, and cryogenics. The choice of a suitable technology depends on the characteristics of the flue gas stream (Rao & Rubin, 2002).

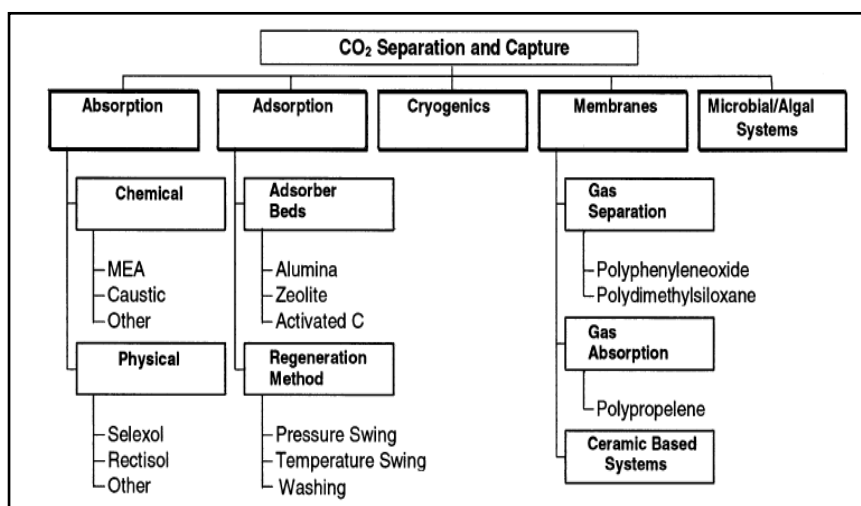


Figure 2.1: Technology options for CO_2 separation and capture. (Source: Rao & Rubin, 2002)

Chemical Solvent	Physical Solvent	Direct Conversion
MEA	Selexol [®]	Iron Sponge
DEA	Rectisol	Stretford
TEA	Purisol	Unisulf
MDEA	Spasolv	Takahax
DIPA/ Shell ADIP [®]	Propylene Carbonate	LO-CAT [®]
DGA/ Fluor Econamine [®]	Estasolven	Lacy-Keller
Proprietary Amine	Alkazid	Townsend
Benfield (Hot Carbonate)		Sulfint
Catacarb (Hot Carbonate)		
Giammarco-Vetrocoke (Hot Carbonate)		
Diamox		
Dravo/Still		
Specialty Solvent	Distillation	Gas Permeation
Sulfinol [®]	Ryan Holmes	Membrane
Amisol	Cryofrac	Molecular Sieve
Flexsorb PS		
Ucarsol LE 711		
Optisol		
Zinc Oxide		
Sulfa Check		
Slurrisweet		
Chemsweet		
Merox		

Table 2.1: Type of process used for CO₂ capture

(Source: Dr Maurice Stewart (2005), Acid Gas Sweetening, Stewart Training Corporation)

2.2 GENERAL OVERVIEW OF ALKANOAMINES

In 2004, Aroonwilas & Veawab's study shows that the removal of CO₂ from gas streams can be achieved by a number of separation techniques including absorption into a liquid solvent, adsorption onto a solid, cryogenic separation, permeation through membranes, and chemical conversion. Referring to Kohl, 1997, among these techniques, absorption into a liquid solvent is the most suitable process for removing CO₂ from high-volume flue gas streams.

Maddox, 1984 stated that the commonly used solvents are aqueous solutions of alkanolamines, such as monoethanolamine (MEA), diethanolamine (DEA), diisopropanolamine (DIPA), and methyldiethanolamine (MDEA). These alkanolamines have been competing with another class of acid gas treating solvents, the sterically hindered amines (Sartori et al, 1994).

Aqueous solutions of alkanolamines are the most widely used solvents for removing CO₂. The most commonly used alkanolamines are the primary amine monoethanolamine (MEA), the secondary amine diethanolamine (DEA), and the tertiary amine methyldiethanolamine (MDEA). Primary and secondary amines react rapidly with CO₂ to form carbamates. By the addition of a primary or secondary amine to a purely physical solvent such as water, the CO₂ absorption capacity and rate is enhanced manifold (Rinker, Ashour and Sandall, 2000).

However, because there is a relatively high heat of absorption associated with the formation of carbamate ions, the cost of regenerating primary and secondary amines is high. Primary and secondary amines also have the disadvantage of requiring 2 mol of amine to react with 1 mol of CO₂; thus, their loadings are limited to 0.5 mol of CO₂/mol of amine. Tertiary amines lack the N-H bond required to form the carbamate ion and therefore do not react directly with CO₂. However, in aqueous

solutions, tertiary amines promote the hydrolysis of CO_2 to form bicarbonate and the protonated amine (Rinker, Ashour and Sandall, 2000).

However, the heat of reaction associated with the formation of bicarbonate ions is much lower than that associated with carbamate formation, and thus, the regeneration costs are lower for tertiary amines than for primary and secondary amines. Another advantage with tertiary amines is that the stoichiometry is 1:1, which allows for very high equilibrium CO_2 loadings (Rinker, Ashour and Sandall, 2000).

At the present time, AMP and MDEA are receiving a great deal of attention because they require relatively low energy consumption for solvent regeneration, leading to significant savings in process costs (Aroonwilas & Veawab, 2004).

2.3 SOLVENT DEVELOPMENT

Hong (1980) stated that it is well known to contact carbon dioxide containing acidic gas mixtures with a liquid solvent to remove these acidic gases. Two general classes of solvents are used in such scrubbing processes; “physical” solvents which physically absorb the acidic gas, and “chemical” solvents which chemically react with the acidic gas for removal of same. Chemical solvents invariably are provided in aqueous form and effectively operate on gaseous mixtures containing low concentrations of acidic gas, while physical solvents invariably are provided in anhydrous form and operate very effectively on gaseous mixtures containing high concentration of acidic gas. Physical solvents are known to provide low circulation rates and low regeneration energies for their recovery, but have very low selectivity for preferentially absorbing the acidic gas over the remainder of the gaseous mixture.

2.3.1 Definition

Solvent can be defined as substances that can physically dissolve other substances; specifically they are inorganic and organic liquids able to dissolve other gaseous, liquid or solid substances (Scheithauer, 2000). A qualifier for the suitability as a solvent is that, during the solution, neither the solvent nor the dissolved substance undergoes chemical change, i.e., the components of the solution may be recovered in their original form by physical separation processes, such as distillation, crystallization, sublimation, evaporation, and adsorption (Scheithauer, 2000).

2.3.2 Physical analysis

The physical properties of solvents are essential for the design and development of gas absorption units. The physical properties can be used to derive other properties (Rinker et al, 1994). Physical properties of process solvents greatly affect the liquid-film mass transfer coefficient and are important for mass transfer rate modelling of absorbers and regenerators (Al-Ghawas et.al, 1989). Physical properties that will be analyzed are as follows:

(a) Density

The density (ρ) is elementary physical property of matter. For a homogeneous object it is defined as the ratio of its mass, m to its volume

$$\rho = m/V \quad [1]$$

Numerically it represents the mass per unit volume of matter. As it follows from equation [1], the SI unit of density is kg/m^3 . However, g/cm^3 is another unit commonly used in a laboratory. The volume of an object increases with increasing temperature, because of the matter's volumetric thermal expansion. Therefore, according to equation [1], the density of an object depends on its temperature, with higher temperature resulting in lower density (N. Kučerka).

(b) Surface tension

Poling, Prausnitz and O'Connell, 2000 stated that the boundary between a liquid phase and a gas phase can be considered a third phase with properties distinct from those of the liquid and gas. A qualitative picture of the macroscopic surface layer shows that there are unequal forces acting upon the molecules; i.e. at low gas densities, the surface molecules are attracted sidewise and toward the bulk liquid but experience little attraction in the direction of the bulk gas. Thus, the surface layer is in tension and tends to contract to the smallest area compatible with the mass of material, container restraints, and external forces, e.g. gravity. Molecules on the surface of a liquid experience an imbalance of forces as indicated in Figure 2 below

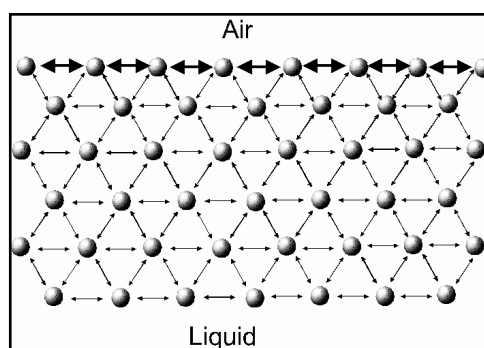


Figure 2.2: Molecules behaviour on the surface of a liquid
(Source: KSV Instruments Ltd.)

This tension can be presented as surface tension, σ , defined as the force exerted in the plane of the surface per unit length. The common units for surface tension are dynes/cm or mN/m. analogously, the boundary between two liquid phases may also be considered a third phase which is characterized by the interfacial tension. Surface tension decreases with temperature as cohesive forces decrease with an increase of molecular thermal activity.

(c) Refractive index (RI)

Refractometry is a technique that measures how light is refracted when it passes through a given substance, in this case, an unknown compound. The amount by which the light is refracted determines the refractive index (RI). Refractive index can be used to identify an unknown liquid compound, or it can be used as a means of measuring the purity of a liquid compound by comparing it to literature values (Chem 211: Refractrometry). The closer the refractive index is to the literature values, the purer the sample. Refractive index is very important since it is related to such fundamental thermodynamic properties as solvent density, phase composition, solute concentration, and interfacial tension. Refractive index is defined as the ratio of the velocity of light in air to the velocity of light in the medium being measured: $n_D = [V_{\text{air}}] / (V_{\text{liquid}})$. The RI of a substance is strongly influenced by temperature and the wavelength of light used to measure it, therefore, care must be taken to control or compensate for temperature differences and wavelength.

CHAPTER 3

METHODOLOGY/PROJECT WORK

3.1 Research Methodology

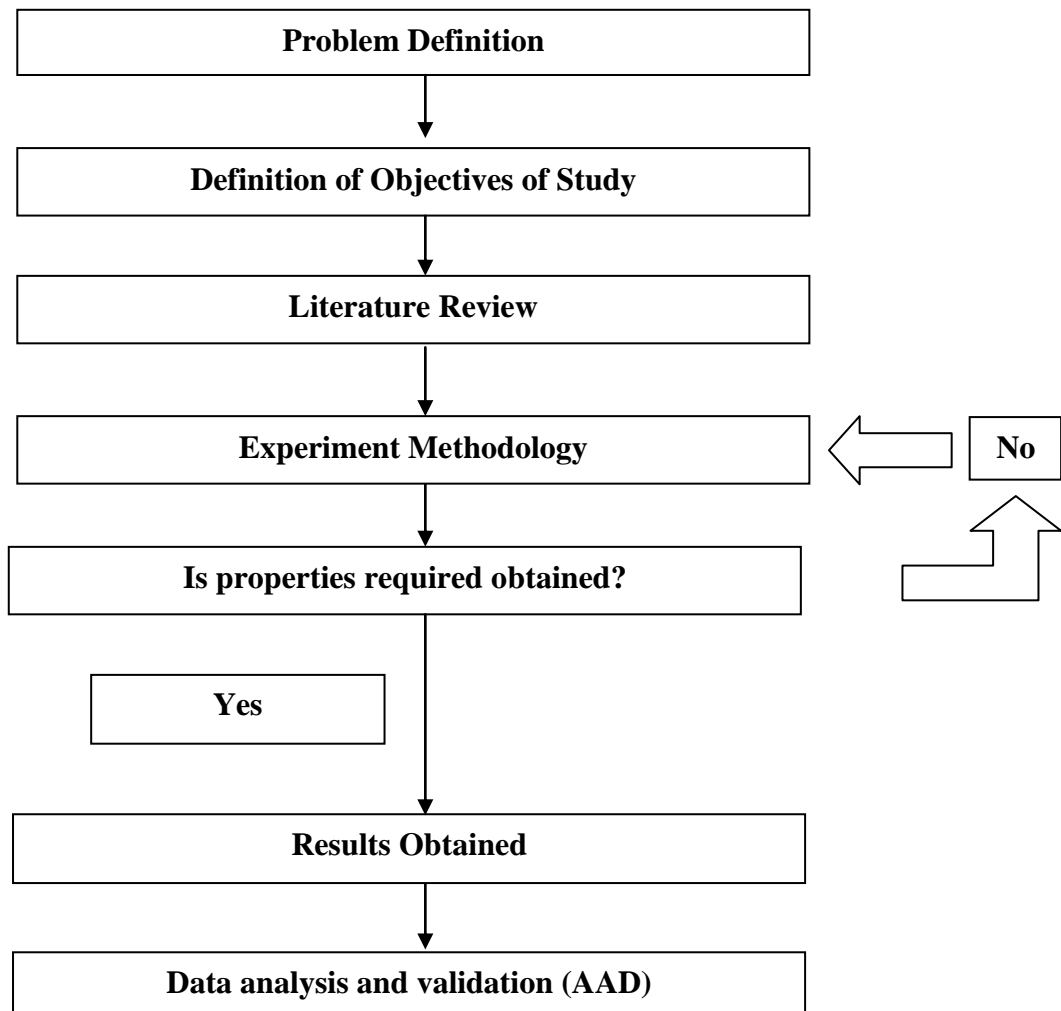


Figure 3.1: Research Methodology

3.2 Project Activities

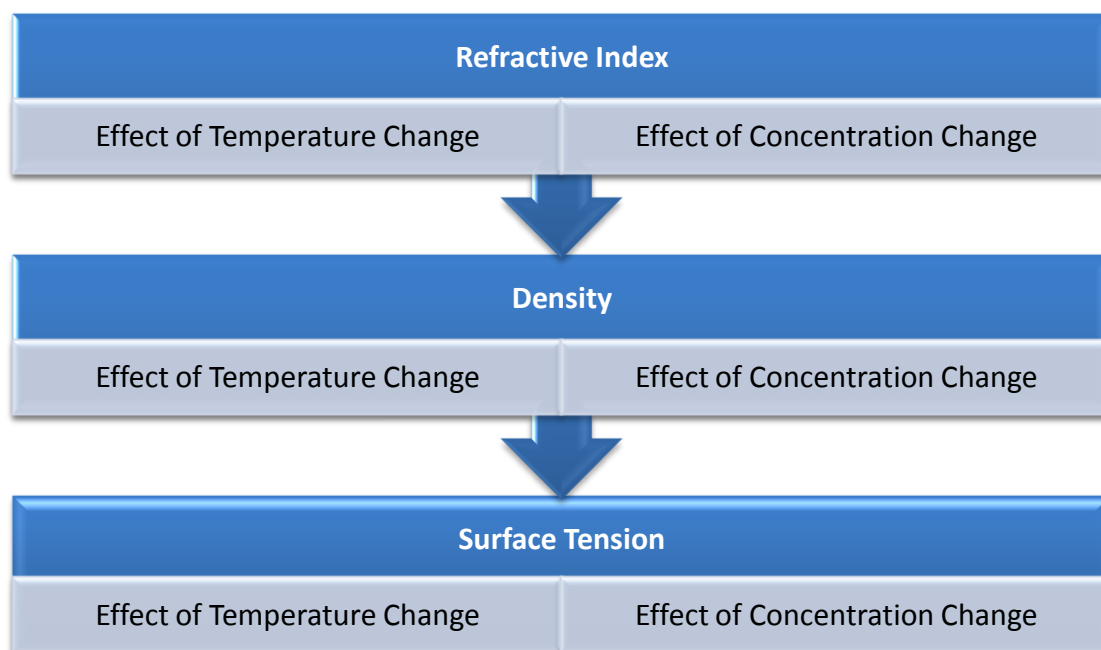


Figure 3.2: Project Activities

3.3 Key Milestone of FYP II

No	Action Item	Action By	Date	Note
1.	Briefing & update on students progress	Coordinator / Students / Supervisors	27 January 2010	WEEK 1
2.	Project work commences	Students	-	WEEK 2 -4
3.	Submission of Progress Report 1	Students	25 February 2010	WEEK 5
4.	Submission of Progress Report 1 Marks	Supervisors / FYP Committee	5 March 2010	WEEK 6
5.	Submission of Progress Report 2 (Draft of Final Report)	Students	09 April 2010	WEEK 11
6.	Poster Exhibition / Pre-EDX / Progress Reporting	Students / Coordinator	08 & 09 April 2010	WEEK 11
7.	EDX	Selected Students/ Coordinator		WEEK 12
8.	Submission of Final Report (CD Softcopy & Softbound)	Students / Supervisors	30 April 2010	WEEK 14
9.	Delivery of Final Report to External Examiner / Marking by External Examiner	FYP Committee / Coordinator	07 May 2010	WEEK 15
10.	Final Oral Presentation	Students/ Supervisors / Internal & External Examiners/ FYP Committee	31 May - 11 June 2010	WEEK 18 - 19
11.	Submission of hardbound copies	Students	18 June 2010	WEEK 20

Figure 3.3: FYP II (Jan 2010) Timeline

3.4 Gantt Chart of FYP II

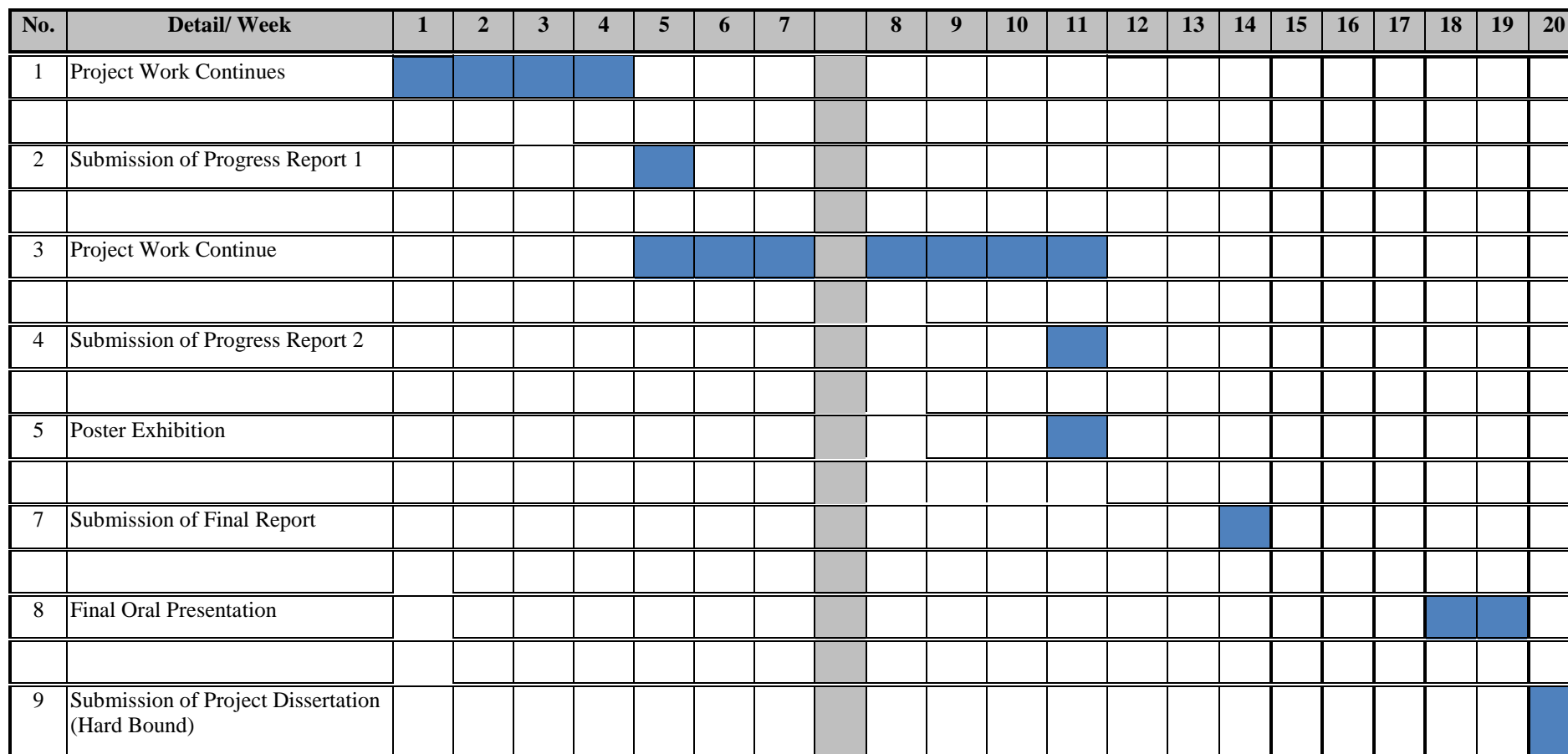




Figure 3.4: FYP II (Jan 2010) Gantt chart

3.5 Equipments for Experimental Work

No	Parameter	Equipment & Principle	Location
1	Density Temperature range : (20-40°C)	<p>Density Meter (DMA 35N, Anton Paar)</p>  <p>Figure 3.5: DMA 35N</p> <p>The battery-operated portable density meter, DMA 35N is ideal for use in the chemical industry and for concentration measurement. Just fill the measuring cell via the pipette pump and get the result seconds later.</p>	Block 15, Level 2
2	Surface tension Temperature range : (25-60°C)	<p>Interfacial Tension Meter (IFT 700, Vinci Technologies)</p>  <p>Figure 3.6: IFT 700</p> <ul style="list-style-type: none"> Designed to determine interfacial tension and contact angle, but also to observe heat and mass transfer phenomena Basically, a pendant drop or standing bubble/drop may be generated in a second immiscible fluid. The drop shape image is computed, and then the interfacial tension is computed from solving algorithm of the Laplace equation. 	Block 15, Level 2

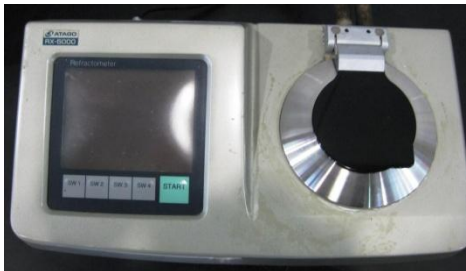
3	Refractive Index Temperature range : (20-80°C)	<p>Digital Refractometer (RX-5000, alpha, Atago)</p>  <p>Figure 3.7: RX-5000(alpha)</p> <ul style="list-style-type: none"> - an automatic digital refractometer which can set measurement temperature internally - has a thermo-module to control temperature, a constant temperature water bath is not necessary. - The measurement starts automatically after the sample reaches your targeted temperature. - The refractive index at your targeted temperature is speedily displayed. - High refractive index ± 0.00004 accuracy can be obtained. 	Block 4, Level 2
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Table 3.1: Equipments for Experimental Work

CHAPTER 4

RESULT AND DISCUSSION

In determining this final year project II, there are three parts of experimental work that had been completed within the time frame given;

- (a) Measuring density
- (b) Measuring refractive index
- (c) Measuring surface tension

Sample preparation:

The aqueous (STONVENT + water) and (DEA + water) solutions were prepared gravimetrically using an **analytical balance (Mettler Toledo model AS120S)**.

4.1 DENSITY

For density, the experimental data obtained been further analyzed where thermal expansion coefficients are calculated from the experimental densities, using an empirical correlation. In addition to that, the experimental data was compared to the literature review data and the average absolute deviation (AAD) was calculated to ensure the accuracy of the equipment used for density measurement and the data itself to be further used in the design and development of gas absorption unit.

4.1.1 Density measurement

Experimental Procedure:

The density of varying concentration of **STONVENT (100%, 40%, 30%, 20% and 10%)** and **DEA (10%, 20%, 30%, and 100%)** were determined using **digital density meter (DMA 35N, Anton Paar)**. All the measurements were performed at **temperatures (298.15 to 313.15) K** with a temperature control accuracy of $\pm 0.05^{\circ}\text{C}$ by using a simple water bath.

Result:

The results obtained are as follows:

T		STONVENT + H ₂ O (g/cm ³)				
C	K	w _{stonvent} =1.0	w _{stonvent} =0.4	w _{stonvent} =0.3	w _{stonvent} =0.2	w _{stonvent} =0.1
25	298.15	1.1528	1.0561	1.0414	1.0277	1.0131
30	303.15	1.1475	1.0526	1.0386	1.0256	1.0117
35	308.15	1.1405	1.0496	1.0350	1.0231	1.0099
40	313.15	1.1306	1.0452	1.0305	1.0204	1.0083

Table 4.1: Data of densities for STONVENT + H₂O

T		DEA + H ₂ O (g/cm ³)			
C	K	w _{DEA} =1.0	w _{DEA} =0.3	w _{DEA} =0.2	w _{DEA} =0.1
25	298.15	1.0942	1.0333	1.0213	1.0096
30	303.15	1.0936	1.0310	1.0191	1.0079
35	308.15	1.0894	1.0274	1.0146	1.0054
40	313.15	1.0862	1.0235	1.0124	1.0034

Table 4.2: Data of densities for DEA + H₂O

The densities of aqueous solutions of STONVENT and DEA are plotted versus temperature in Figures 11 and 12 respectively. The graph plotted shows that with an increase in temperature, the densities values decrease. However, an increase in densities values was observed with an increase of STONVENT and DEA concentration.

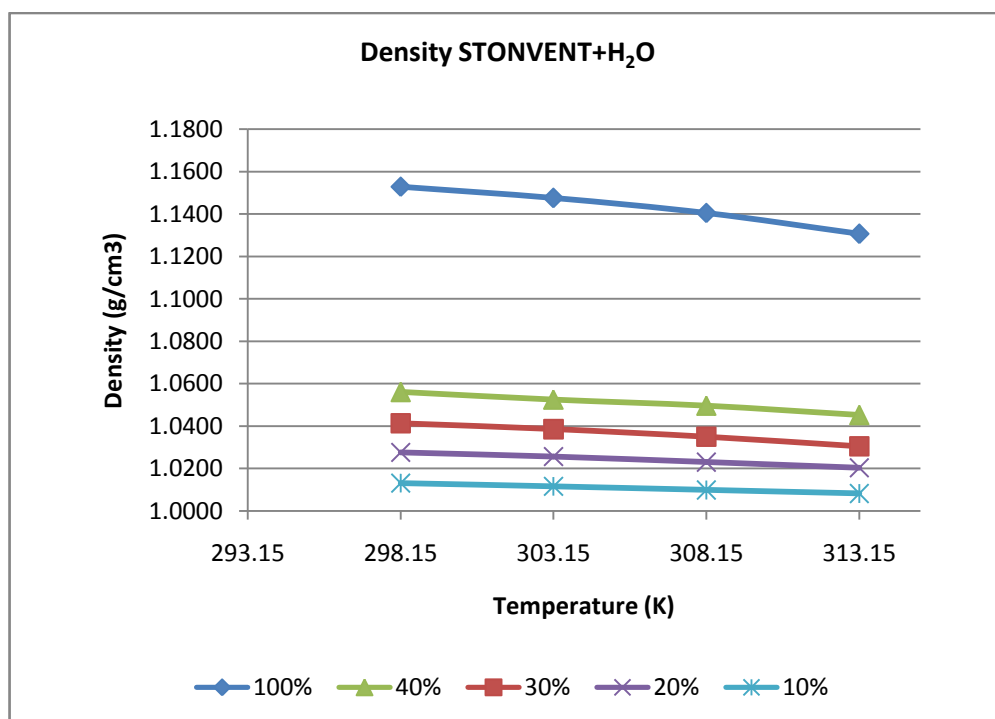


Figure 4.1: Graph of Densities of STONVENT + H₂O

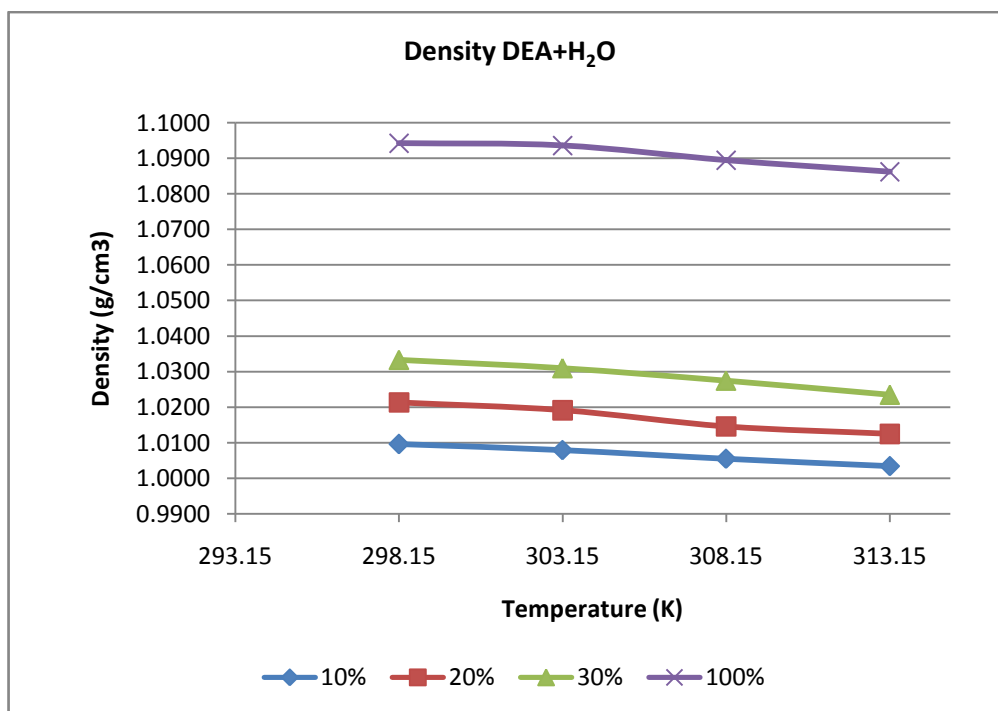


Figure 4.2: Graph of Densities of DEA + H₂O

4.1.1 Comparison with literature data

To establish the accuracy of density meter used, the experimental data obtained for DEA has been compared with the reported value from literature review done in the first part of the project.

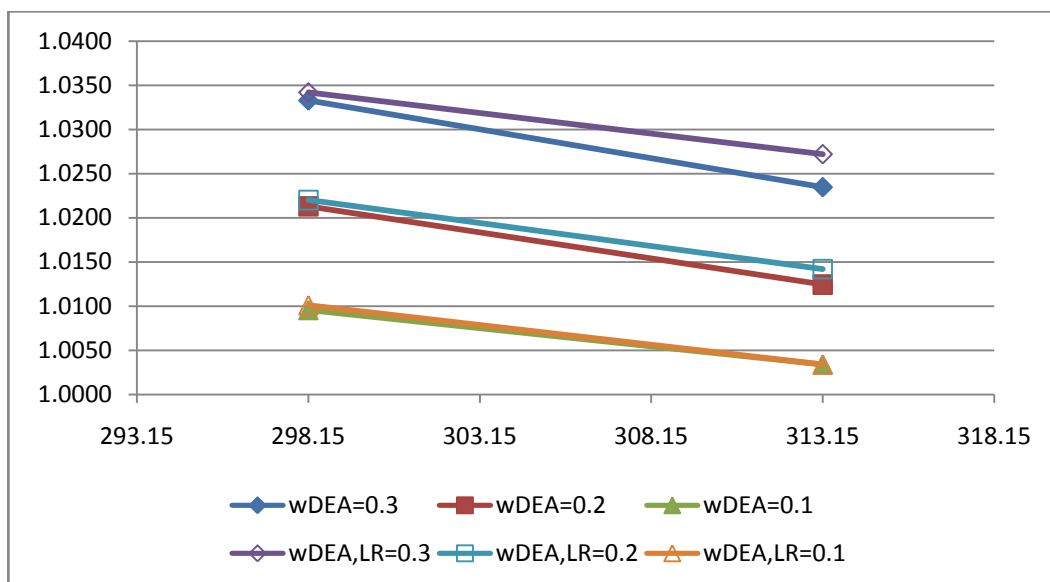


Figure 4.3: Density of aqueous solutions of DEA: all-filled marker represents experimental data obtained and non-filled marker; Rinker et al, 1994.

Average Absolute Deviation (AAD) calculated: **0.13%**

4.1.2 Calculated thermal expansion coefficient

The influenced of temperature on the density of all the compositions of binary mixtures of DEA+ H₂O was found to be linear and based from Ayyaz et al, 2008, it can be correlated using

$$\rho = A_0 + A_1 T \quad (1)$$

where A_0 and A_1 are the fitting parameters which were estimated using a method of least-squares and presented in Table 5 below. The overall AAD also been calculated which is **0.10%**.

Conc	Fitting Parameters	
wDEA	A ₀	A ₁
1	1.1092	-0.0006
0.3	1.0502	-0.0007
0.2	1.0371	-0.0006
0.1	1.0202	-0.0004

Table 4.3: Fitting Parameters of DEA+ H₂O obtained using Equation (1)

The coefficients of thermal expansion values for the system studied in this project are calculated from their experimental density data using

$$\alpha_P = -1/\rho(\delta\rho/\delta T)_P = -A_1 / (A_0 + A_1T) \quad (2)$$

where α_P is the coefficient of thermal expansion; ρ is the density; T is the temperature; and A_0 and A_1 are the correlation coefficients taken from (1) by fitting experimental density data. It can be observed from Table 6 that the change in coefficients of thermal expansion values is not significant and the volume expansion of the systems studied in the present work could be considered as independent of temperature. However, the values of α_P varied from $(3.96 \text{ to } 6.85) \times 10^{-4} \text{ K}^{-1}$, with the increase in composition of aqueous DEA solutions (0.1 to 1.0).

T		$\alpha_P \times 10^{-4} \text{ K}^{-1}$			
C	K	WDEA=1.0	WDEA=0.3	WDEA=0.2	WDEA=0.1
25	298.15	5.4835	6.7783	5.8703	3.9596
30	303.15	5.4985	6.8014	5.8875	3.9675
35	308.15	5.5137	6.8246	5.9049	3.9754
40	313.15	5.5289	6.8480	5.9224	3.9833

Table 4.4: Coefficients of Thermal Expansion of DEA+ H₂O using Equation (2)

Since STONVENT has quite similar CO₂ loading with DEA, thus from STONVENT data also we can determine its fitting parameter and calculate the thermal expansion coefficient.

Conc	Fitting Parameters	
Wstonvent	A ₀	A ₁
1	1.1908	-0.0015
0.4	1.074	-0.0007
0.3	1.06	-0.0007
0.2	1.04	-0.0005
0.1	1.0214	-0.0003

Table 4.5: Fitting Parameters of STONVENT obtained using Equation (1)

T		STONVENT + water				
C	K	Wstonvent=1.0	Wstonvent=0.4	Wstonvent=0.3	Wstonvent=0.2	Wstonvent=0.1
25	298.15	13.0062	6.6257	6.7146	4.8662	2.9589
30	303.15	13.0913	6.6477	6.7372	4.8780	2.9633
35	308.15	13.1775	6.6698	6.7600	4.8900	2.9677
40	313.15	13.2649	6.6922	6.7829	4.9020	2.9721

Table 4.6: Coefficients of Thermal Expansion of STONVENT using Equation (2)

4.1.3 Discussion

From Figure 13, we can see that the difference between the experimental data and the literature data (taken from Rinker et al, 1994) of DEA+H₂O is more or less the same. Plus, from the AAD calculated, it showed that the percentage error by using the respective density meter is small which 0.13% is only. This proved that the method and equipment used in measuring densities for STONVENT in this project is valid and the data obtained can be used in the absorption unit design.

As for the thermal expansion coefficients, from the calculations done, since the AAD is also small which is 0.10%, this also shows the validity of correlations formulated to be used in determining the thermal expansion coefficients from the experimental densities data obtained.

4.2 REFRACTIVE INDEX

For refractive index data, it also been compared to the literature review data in ensuring the accuracy of equipment used and the validity of data obtained.

4.2.1 Refractive Index Measurement

Experimental Procedure:

The refractive indices of varying concentration of **STONVENT (100%, 40%, 30%, 20% and 10%)** and **DEA (10%, 20%, 30%, and 100%)** were determined using **programmable digital refractometer (RX-5000 alpha, Atago)**. All the measurements were performed at **temperatures (293.15 to 353.15) K** with a temperature control accuracy of $\pm 0.05^{\circ}\text{C}$

Result:

The results obtained are as follows:

T		STONVENT + H ₂ O				
C	K	wstonvent=1.0	wstonvent=0.4	wstonvent=0.3	wstonvent=0.2	wstonvent=0.1
20	293.15	1.40467	1.35296	1.35066	1.34511	1.34026
30	303.15	1.40305	1.35083	1.34847	1.34386	1.33914
40	313.15	1.40125	1.34874	1.34591	1.34259	1.33788
50	323.15	1.39927	1.34658	1.34362	1.34109	1.33637
60	333.15	1.39679	1.34508	1.34142	1.33940	1.33466
70	343.15	1.39335	1.34319	1.34093	1.33778	1.33280
80	353.15	1.38881	1.34091	1.34044	1.33695	1.33172

Table 4.7: Data of refractive indices for STONVENT + H₂O

T		DEA + H ₂ O			
C	K	wDEA=1.0	wDEA=0.3	wDEA=0.2	wDEA=0.1
25	298.15	1.47776	1.37621	1.3624	1.34853
30	303.15	1.47397	1.37533	1.36095	1.34808
40	313.15	1.47325	1.37418	1.35995	1.34675
50	323.15	1.47179	1.37189	1.35845	1.34524
60	333.15	1.46881	1.36969	1.35676	1.34353
70	343.15	1.46537	1.3692	1.35514	1.34167

Table 4.8: Data of refractive indices for DEA+ H₂O

The refractive indices of aqueous solutions of STONVENT and DEA are plotted versus temperature in Figures 14 and 15 respectively. The graph plotted shows that with an increase in temperature, the refractive indices values decrease. However, an increase in refractive indices values was observed with an increase of STONVENT and DEA concentration.

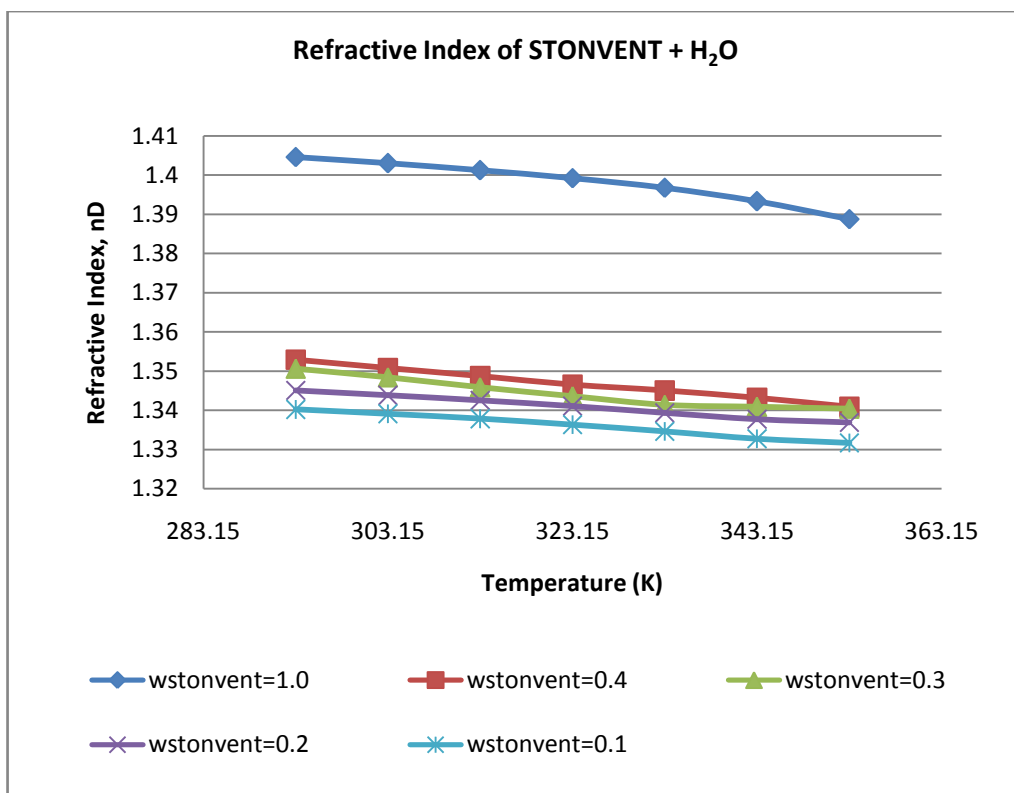


Figure 4.4: Graph of Refractive Index of STONVENT + H₂O

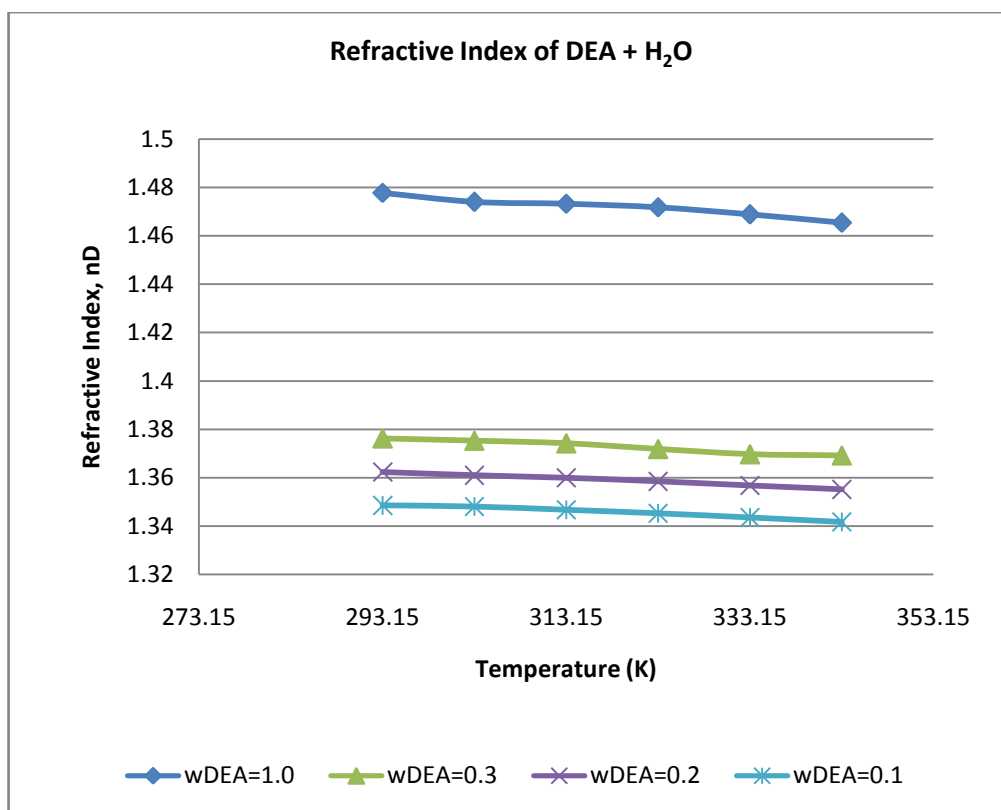


Figure 4.5: Graph of Refractive Index of DEA + H₂O

4.2.2 Comparison with literature data

To establish the accuracy of refractometer used, the experimental data obtained for DEA has been compared with the reported value from literature review done in the first part of the project.

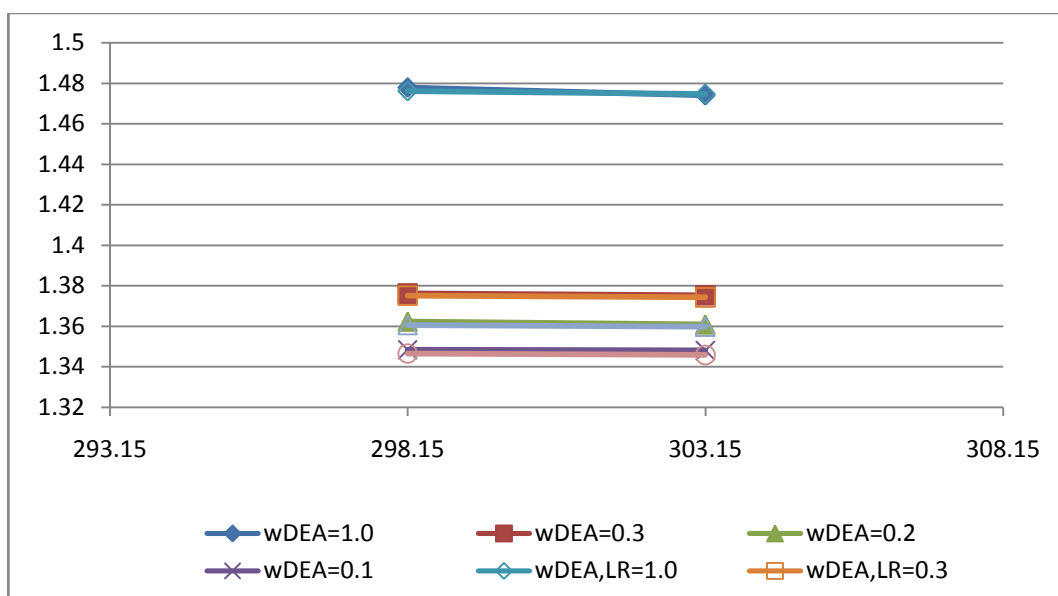


Figure 4.6: Refractive Indices of aqueous solutions of DEA: all-filled market represents experimental data obtained and non-filled marker; Tseng and Thompson, 1964.

Average Absolute Deviation (AAD) calculated: **0.09%**

4.2.3 Discussion

From Figure 16, we can see that the difference between the experimental data and the literature data of DEA+H₂O is very less. Plus, from the AAD calculated, it showed that the percentage error by using the respective refractrometer is very small which 0.09% is only. This proved that the method and equipment used in measuring refractive indices for STONVENT in this project is valid and the data obtained can be used in the absorption unit design.

4.3 SURFACE TENSION

For surface tension data, it also been compared to the literature review data in ensuring the accuracy of equipment used and the validity of data obtained.

4.3.1 Surface tension measurement

Experimental Procedure:

The surface tension of varying concentration of **STONVENT (100%, 40%, 30%, 20% and 10%)** and **DEA (10%, 20%, 30%, and 100%)** were determined using **Interfacial Tension Meter (IFT 700, Vinci Technologies)**. All the measurements were performed at **temperatures (293.15 to 333.15) K** with a temperature control accuracy of $\pm 0.05^{\circ}\text{C}$.

For this experiment, we need to form one drop of the liquid with suitable needle size (1.0-1.5mm). The camera will capture the image of the droplet which allows for surface tension calculations. The data were obtained by using WDROP for Windows software. The settings of the software are as follows:

1. Calibrate the tension meter each time forming a new drop; we need to adjust the Focus, Histogram, Vertical Setup, and Optical Calibration with tube.



Figure 4.7: Adjusting the Focus



Figure 4.8: Adjusting the Histogram

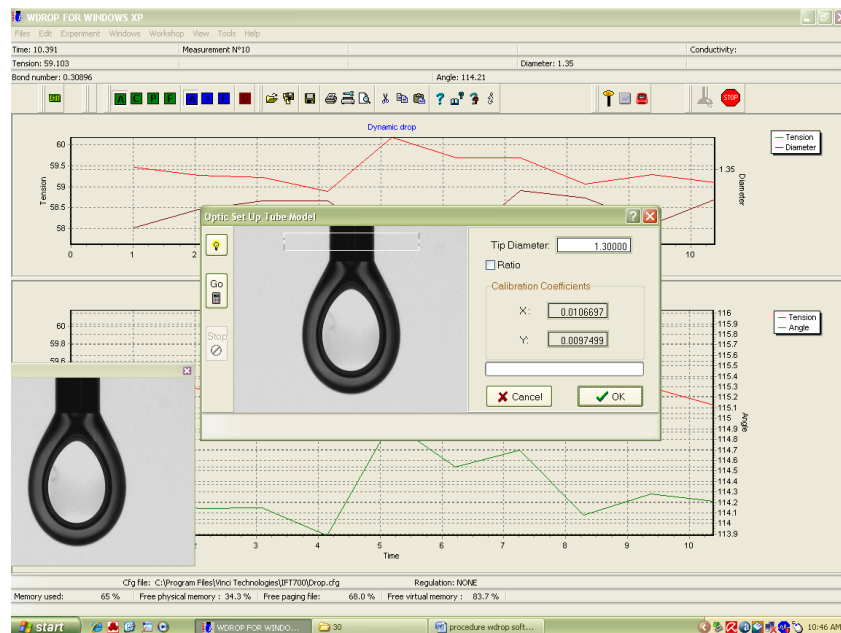


Figure 4.9: Adjusting the Optical Calibration

- Set the setup measurement before running the experiment and obtained the result after 60 seconds of calculation. Data been obtained.

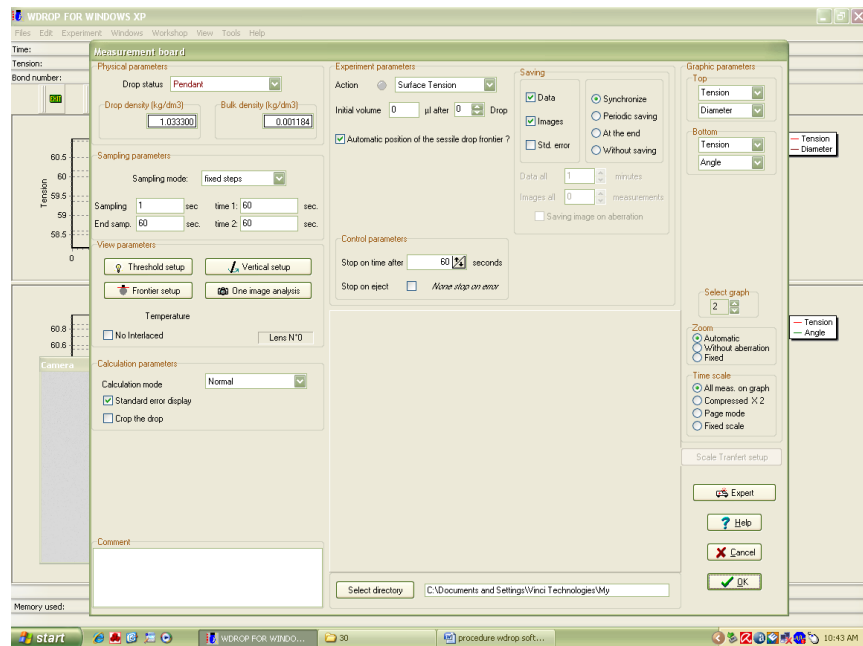


Figure 4.10: Adjusting the Setup Measurement

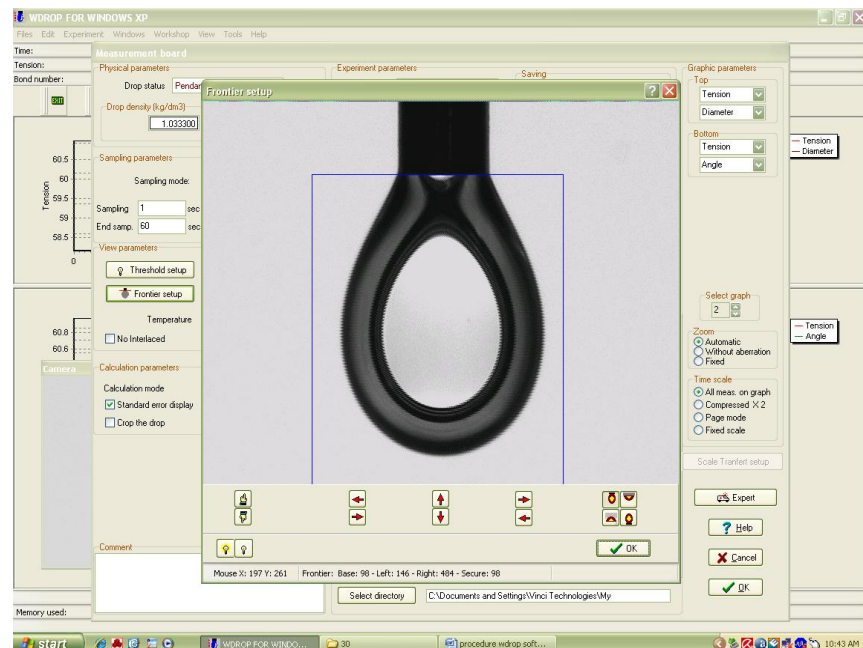


Figure 4.11: Adjusting the Frontier Setup

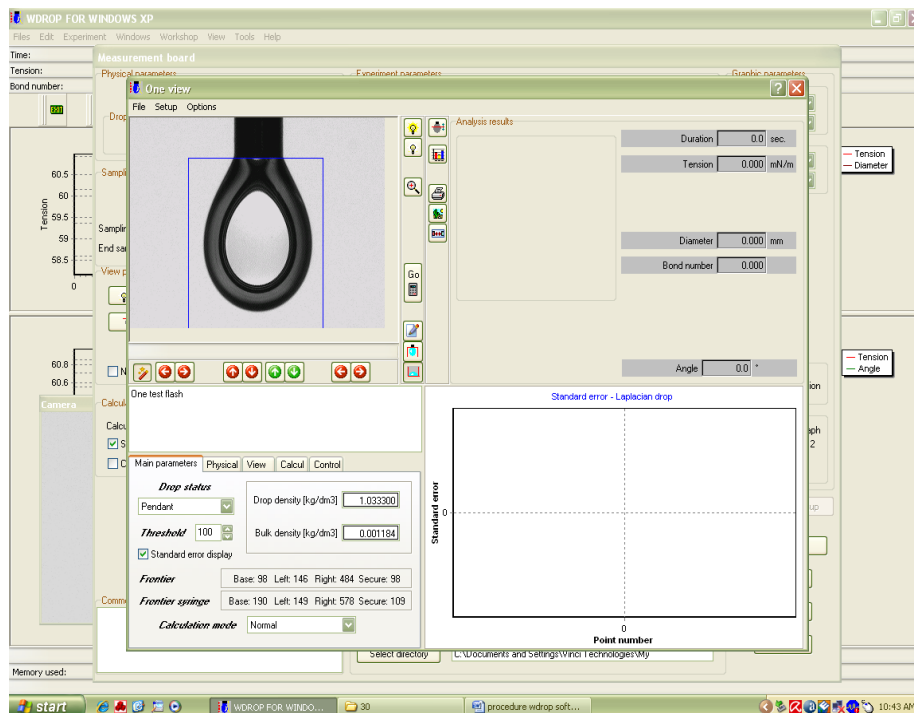


Figure 4.12: Adjusting the One View

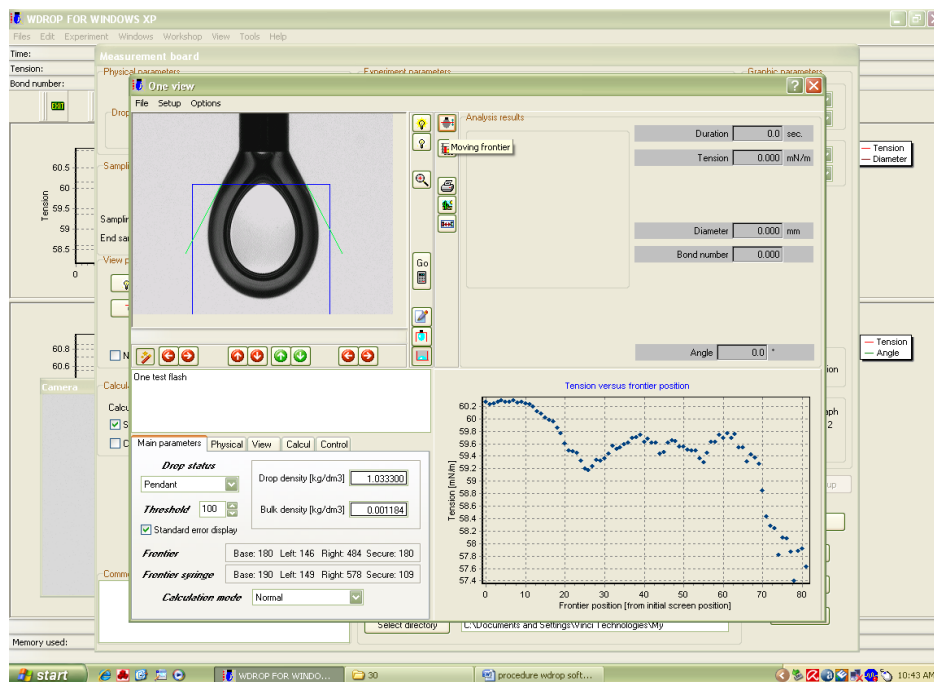


Figure 4.13: Testing Moving Frontier

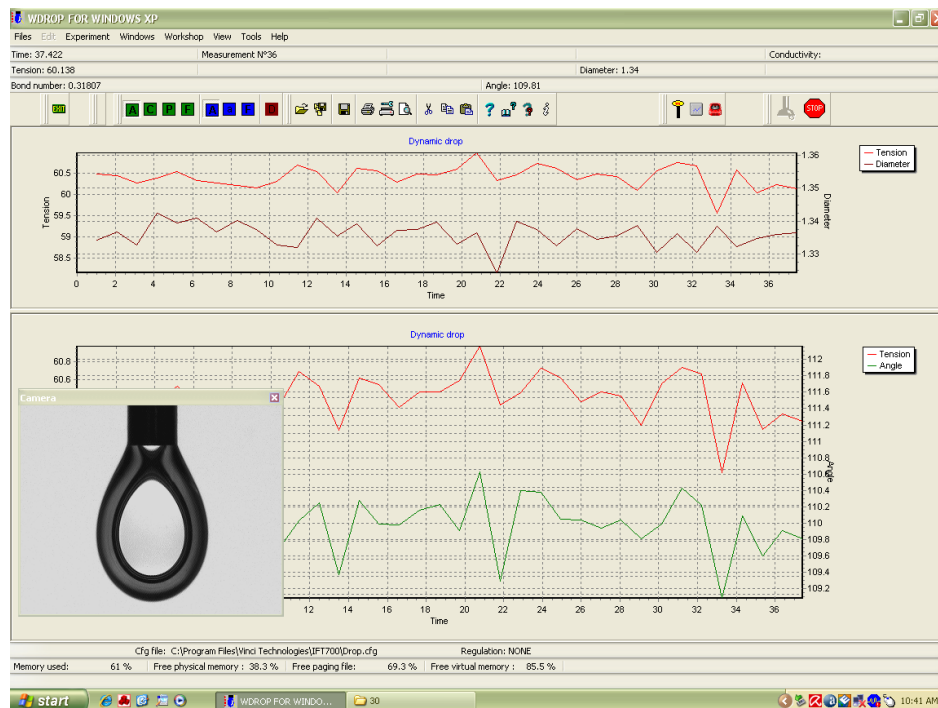


Figure 4.14: Running Measurement

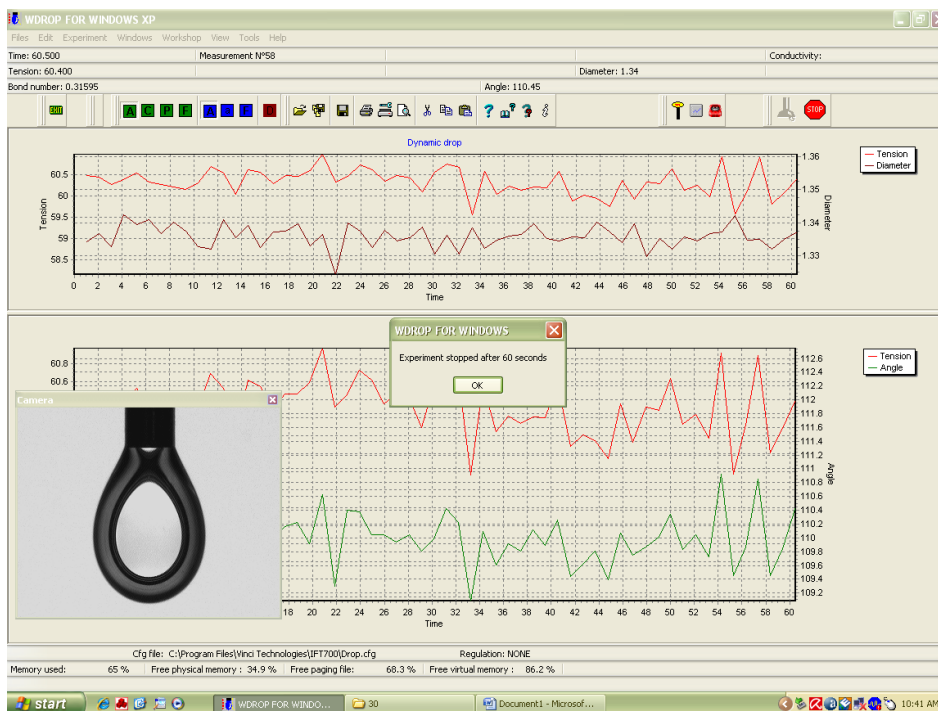


Figure 4.15: The calculation stops after 60 seconds



Figure 4.16: Results been produced

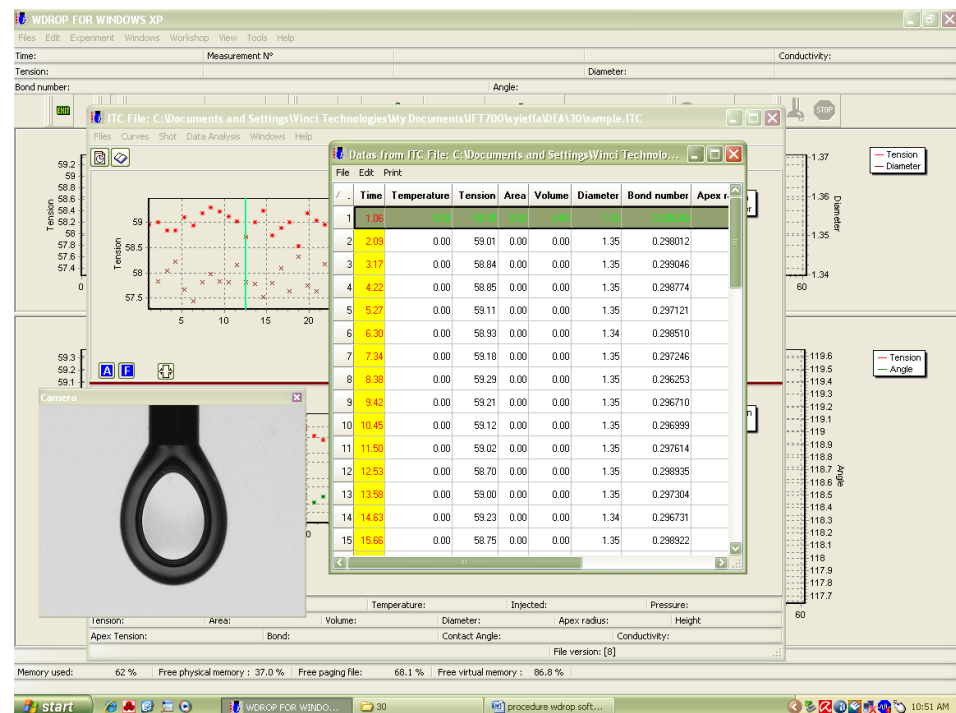


Figure 4.17: Results obtained

Result:

The results obtained are as follows:

T		STONVENT (mN/m)
C	K	w _{stonvent} =1.0
25	298.15	64.1433
30	303.15	63.3067
35	308.15	62.5233
40	313.15	61.6100
50	323.15	59.5200
60	333.15	57.3767

Table 4.9: Data of surface tension for STONVENT

T		DEA + water (mN/m)		
C	K	w _{DEA} =0.3	w _{DEA} =0.2	w _{DEA} =0.1
25	298.15	62.4067	60.3267	58.0733
30	303.15	61.3533	58.4000	56.2633
35	308.15	60.6200	57.0600	53.9200
40	313.15	59.4933	55.6767	51.7867
50	323.15	56.9633	52.4867	48.6500
60	333.15	54.7900	49.8133	45.1000

Table 4.10: Data of surface tension for DEA +H₂O

The surface tension of aqueous solutions of STONVENT and DEA are plotted versus temperature in Figures 25 and 26 respectively. The graph plotted above showed that with an increase in temperature, the surface tension values decrease. However, an increase in surface tension values was observed with an increase of STONVENT and DEA concentration.

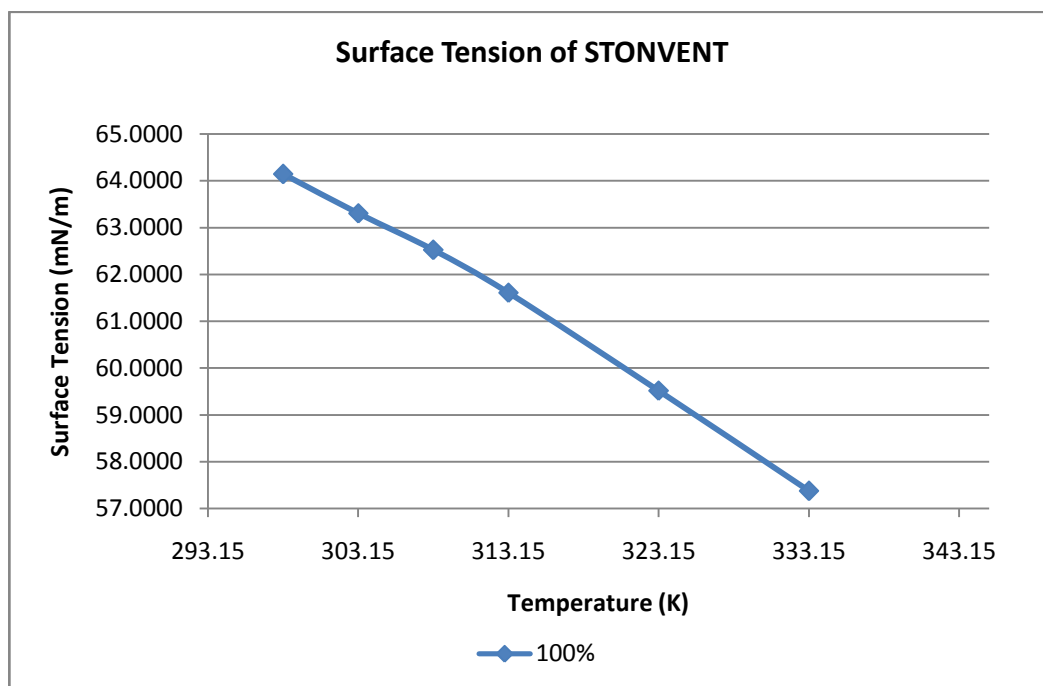


Figure 4.18: Graph of Surface tension of STONVENT

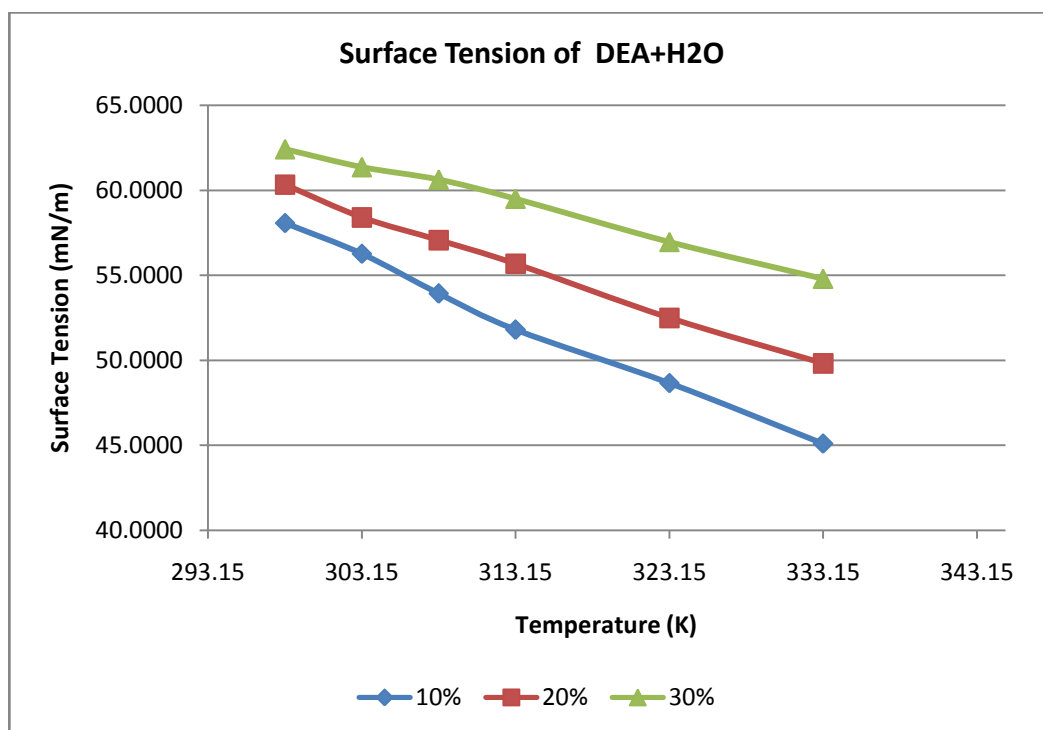


Figure 4.19: Graph of Surface tension of DEA +H₂O

4.3.2 Comparison with literature data

To establish the accuracy of tension meter used, the experimental data obtained for DEA has been compared with the reported value from literature review done in the first part of the project.

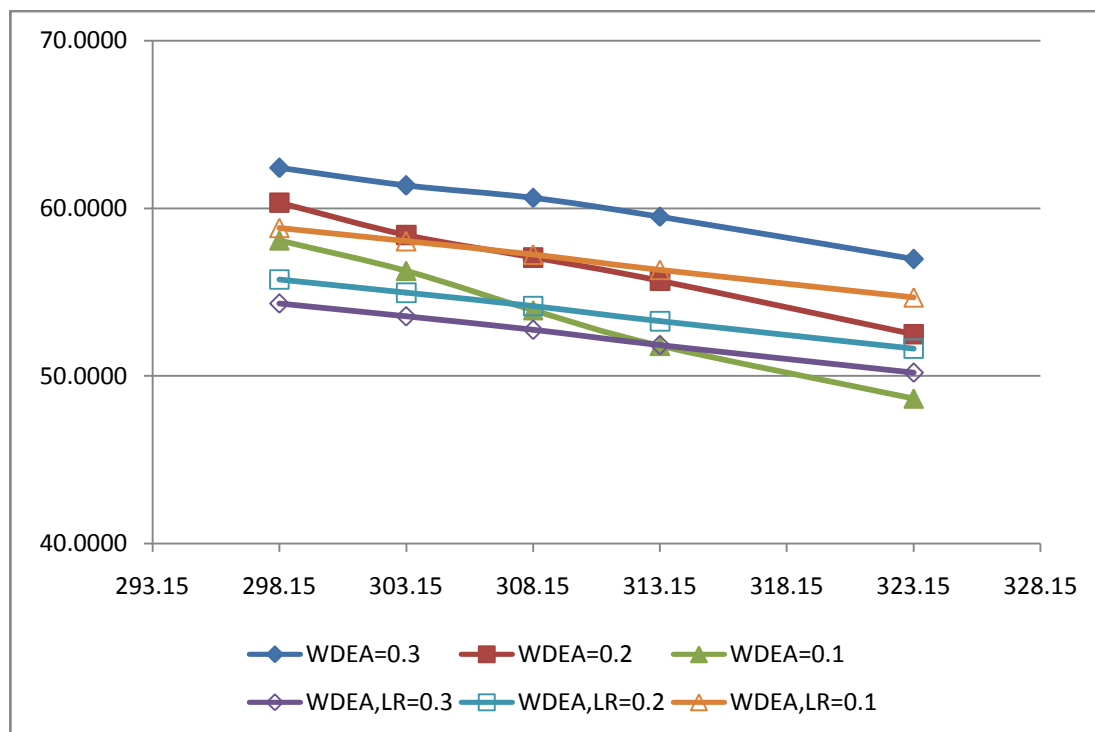


Figure 4.20: Refractive Indices of aqueous solutions of DEA: all-filled market represents experimental data obtained and non-filled marker; Vazquez et al, 1996.

Average Absolute Deviation (AAD) calculated: **3.75%**

4.3.3 Discussion

From Figure 30, we can see that the difference between the experimental data and the literature data of DEA+H₂O is quite differ, plus, from the AAD calculated, it showed that the percentage error by using the respective tension meter is very quite large which about 3.75%. The difference showed by the calculations done reflects maybe the inappropriate of the equipment and method used in measuring refractive indices for STONVENT in this project. The experimental work should be repeated with alternative equipment and method in ensuring the accuracy of the data obtained which will be used later in the design of absorption unit.

4.4 FTIR ANALYSIS OF STONVENT

To study the characterization of STONVENT before and after absorbing CO₂ gas, both pure STONVENT (before reaction) and reacted STONVENT (after reaction) have been analyzed its characteristic by Fourier Transform Infra Red (FTIR) technique. The FTIR analysis result was taken from the analysis done by my colleague who is working on the same solvent, STONVENT (Nur Akissha Mohd Ruba'ai, Biological, Corrosion and Characterization Analysis of STONVENT for CO₂ removal). The pure STONVENT was analyzed at room temperature and ambient pressure while reacted STONVENT was analyzed at 30°C and 30 kPa. This temperature and pressure condition which performed during CO₂ absorption was selected due to its highest loading CO₂ / amine, represents the best operating condition.

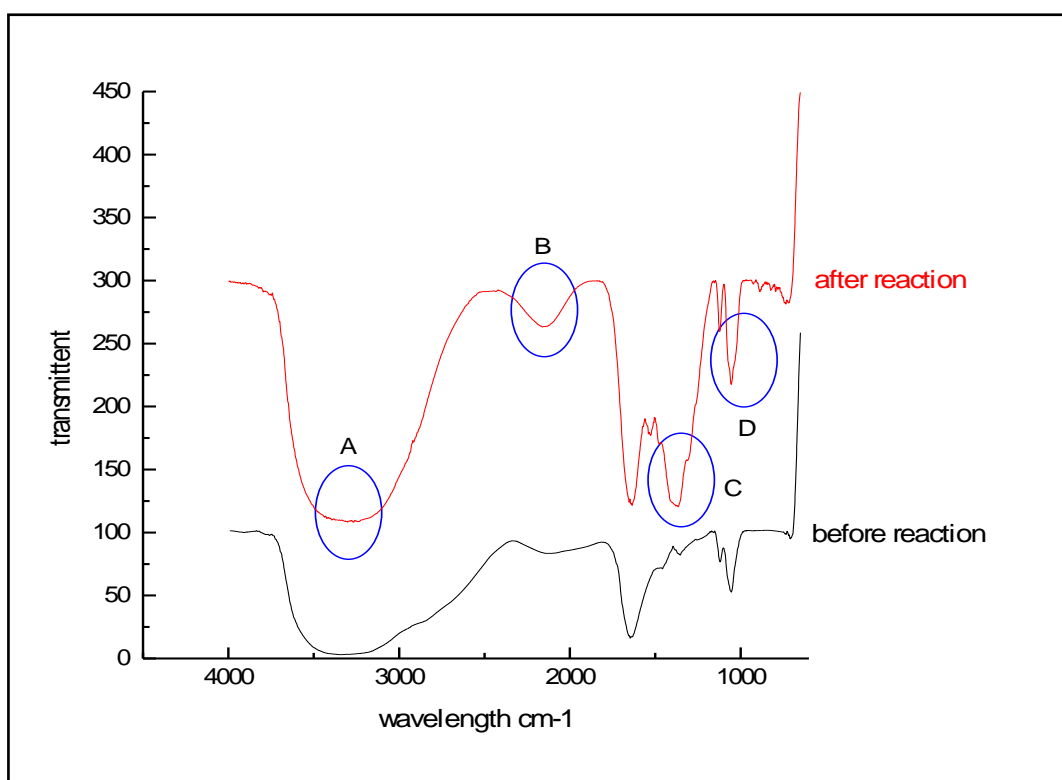


Figure 4.21: FTIR transmission spectra for STONVENT at before and after reaction of CO₂ absorption

Before reaction, from 3200cm^{-1} to 3500cm^{-1} , the line was largely stretched and alcohol functional group with O-H bond or amine functional group with N-H bond might be present. A small bend at the peak from 2200cm^{-1} to 2100cm^{-1} might exhibit the alkynes functional group, $\text{C}\equiv\text{C}$ meanwhile, a repetitive small peak found from 1050cm^{-1} to 1500cm^{-1} which probably comes from the carboxylic acid functional group, C-O bond and ketones functional group with C-C bond. From 1025cm^{-1} to 1200cm^{-1} , a slight sharp peak might represent the amine functional group with C-N alkyl bond or alkyl halide group with C-F bond stretch.

As shown in Figure 24, at location A, the transmittent peak ranged from 3200cm^{-1} to 3400cm^{-1} are attributed to the O-H functional groups and we can see after reaction, O-H stretching was slightly narrowed compared to before reaction. It can simply describe that, after undergone CO_2 absorption; STONVENT might experience certain loss of identity without presence of high concentration O-H bond. At location B, the transmittent peak ranged from 2100cm^{-1} to 2140cm^{-1} may exhibited the presence of $\text{C}\equiv\text{C}$ group with the weak terminal alkynes. The $\text{C}\equiv\text{C}$ bond appears to be more bending after the reaction shows that, formation of byproduct solid might associate to the $\text{C}\equiv\text{C}$ group. In addition, at location C, the new sharp transmittent peak was detected at a range of 1250cm^{-1} to 1300cm^{-1} . The atomic bonds and functional group that belongs to this transmittent peak is carboxylic acid, C-O. Therefore, the C-O bond type might have been formed after the reaction with STONVENT and CO_2 . At location D, sharp transmittent peak observed in range of 1000cm^{-1} to 1100cm^{-1} . Ordinary fluoroalkanes bond presents within the range might denote the presence of halogen group in that solvent.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

The objectives set for FYP II has been achieved in the given time frame:

1. To perform physical properties analysis (density, surface tension and refractive index) of the STONVENT.
2. To study the effect of temperature change (up to 80°C) on the properties of STONVENT.
3. To study the effect of concentration change on the properties of STONVENT.
4. To compare the results obtained with conventional solvent, Diethanolamine (DEA).
5. To study the characteristic of STONVENT before and after absorbing CO₂ through Fourier Transform InfraRed (FTIR) Analysis.

Overall results obtained shows that an **overall decrease in all the measured system properties** with an **increase in temperature**. However, an **overall increase in all system properties** with an **increase in concentration** in each solution tested.

The **comparison** between the **experimental data obtained and the literature data** need to be done in order to **measure the accuracy and the validity** of the equipment and method used in the project work. The smaller the AAD value calculated, the accurate the data measured from experimental work.

Lastly, the **characterization of STONVENT** before and after absorbing CO₂ need to be studied in determining **changes of components** in the solvent after the absorption process which also **reflects the ability of STONVENT absorbing carbon dioxide gas** and have the **potential to be used commercially in the industry**.

5.2 RECOMMENDATIONS

The recommendations to improve this research project are as follows:

1. To analyze another physical properties; viscosity of STONVENT in order to have a complete data to be used in the designing part later.
2. To further perform chemical and thermal analysis for STONVENT.
3. To compare STONVENT with other conventional alkanoamines and blended amines so that a proper comparison can be done.

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