ABSTRACT

Most recent technology of removing CO_2 is by absorption system using amine-based solvent. A novel solvent is expecting may reduce the high energy consumption cost at the regeneration part of CO_2 absorption. The objective of this project is, to analyze STONVENT byproduct and characterize it by chemical and physical properties. STONVENT byproduct is the precipitate from STONVENT which already absorbs CO_2 and already reach the equilibrium condition. Among the test undergo by the STONVENT byproduct samples are pH test, refractive index test (physical characterization) , XRD and FTIR techniques (chemical characterization).From FTIR technique, observation shows some functional groups for example alkanes, alkenes, alcohol, carboxylic acid, alkynes and alkyl amine may be existed before and after STONVENT undergo CO_2 absorption. By using XRD technique, chemical compounds found in STONVENT were compared to existing international diffraction database. Further research is required to study on STONVENT and its byproduct to find its real potential as it is a new solvent.

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CHAPTER 1

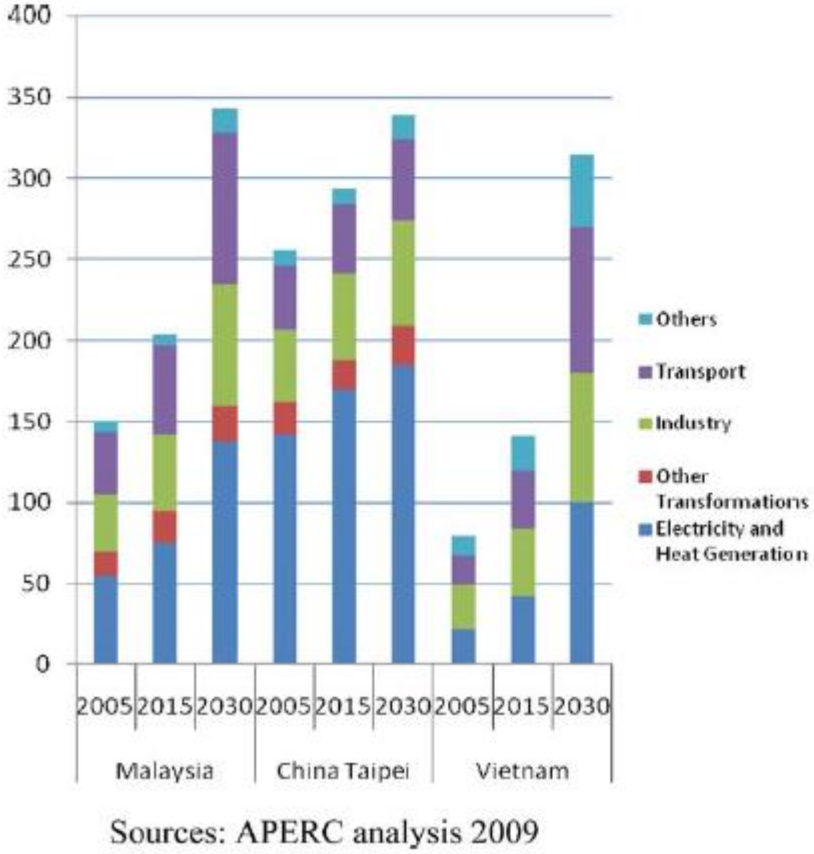
PROJECT BACKGROUND

1.1 Background of Study

Carbon dioxide (CO_2) gas is one of major distributor to green house effect. This colorless, odorless non-flammable gas is recycled back to the atmosphere by the process of photosynthesis. Not only emitted by human exhale, by deforestation, and burning fossil fuel for energy, CO_2 is also contributed by the gas emission activities from the industries all over the world, chemical and petrochemical manufacturing, and natural gas purification. This green house gas that is trap at the atmosphere will cause global warming which can increase to the earth temperature as well as give rise to sea level due to thermal expansion of the ocean, in addition to melting of land ice.

Based on temperature trend in Malaysia the state of global warming is become worse day by day. A study of the temperature trend in Malaysia found that there are magnificent rise of mean annual temperature in 100 year which ranging from 0.99 to 3.44 ° C. Besides that, the mean annual temperature regression line in 30 years before also shows high increased ranging from 1.35 to 1.6.33° C per 100 years. There are many reasons of global warming which includes the population increment, significant amount of emission, densities of energy used and the built-up of densely building. (**Ng et.al. , 2005).**

In 1990, 6 billion metric tons of “carbon dioxide equivalent’ all over the world has been released in a year, more than increase of 20%. In 2006, Malaysia rank in the third place in the South-east Asian Region with 187 million tons of carbon emissions after Indonesia and Thailand. For smaller-economies country, Malaysia contribution of carbon emission is rank into second largest country after Taipei and Taiwan for 2005 and 2015 but forecasted to be the main contributor in 2030 as in the figure below. **(Salsabila et. al.,2011)**



Sources: APERC analysis 2009

Figure 1 : Carbon Emission in Malaysia (Salsabila et. al, 2011)

Apart from the effect of CO_2 to the environment, for the gas and petrochemicals industries, the removal of CO_2 from the feed gas is crucial in order to prevent dry ice formation in the downstream low temperature equipment which could lead to equipment failure and production losses. It is also required to achieve ethane production specification. In most gas processing plant, a special unit of acid gas removal is built in order to purify the natural gas from these acidic gasses (green house gasses, GHGs) like CO_2 and hydrogen sulfide, H_2S.

To keep greenhouses gases at manageable level, large decrease in CO_2 emission through capturing and separation will be required. Examples of three main CO_2 capture technologies are post-combustion, pre-combustion, and oxyfuel combustion techniques.

Various separation technologies of removing the CO_2 gas has been discovered and explored for example chemical absorption, physical absorption process, physical adsorption, cryogenics, and membrane technology.

Post-combustion CO_2 separates CO_2 from the flue gas which fuel combustion produced. Sequestration of combustion gases is expensive in term of gas compression and storage cost therefore this technique is not feasible Low concentration of CO_2 in power-plant flue gas (typically 4% - 14%) means that a huge volume of gas has to be handled, thus lead to large equipment sizes and high capital costs. The low partial pressure of the CO_2 in the flue gas proposes a significant design challenge from post-combustion capture. A part from that, powerful chemical solvents have to be used and regeneration of the solvents to release the CO_2 will require a large amount of energy. **(Olajire, 2010)**

In pre-combustion carbon capture, fuel is reacted with oxygen or air (steam, in some cases), which produce mainly carbon monoxide and hydrogen. The product of mainly CO and H_2 is passed through a catalytic reactor. Here, the CO reacts with steam to give CO_2 and more H_2. The CO_2 is separated and the H_2 is used as fuel in a gas turbine combine-cycle plant. This technique can be applied in coal gasification, biomass and natural gas process. The equipment for CO_2 capture will be smaller as the concentration and pressure in both pre-combustion and post-combustion are high. Moreover, different kind of solvent can be used with low energy penalties for regeneration part. The main disadvantage of pre-combustion capture is that total capital cost of the generating facility is very expensive according to Olajire,2010.

For oxyfuel combustion, high concentration of CO_2 in flue gases is because of fuel is combusted in almost pure oxygen instead of air. Olajire, 2010 said that, flame temperature will be high if the fuel is burnt in pure oxygen, and some CO2-rich flue gas is then recycled back to the combustor so that the flame temperature equal to that in normal air-blown combustor. The weakness of oxyfuel combustion is the high cost of capital and energy consumption since large quantity of oxygen is needed.

Chemical absorption is chosen for low to moderate CO_2 partial pressure. Chemical absorption of CO_2 from gaseous streams in example flue gases depends on acid-base neutralization reaction using basic solvents. Weakly bonded intermediate compound is formed when CO_2 reacts with chemical solvents. The product is then broken down using heat, to generate the original solvent and produce a pure pure CO_2 stream. The example of chemical absorption technologies as stated by Olajire, 2010 are amine absorption technology and aqua ammonia process.

For physical absorption process, CO_2 is physically absorbed in a solvent and they are temperature and pressure dependent according to Henry's Law. Organic solvent is used in this process to absorb acid gas physically. The partial pressure and feed gas temperature will determine the CO_2 solubility in the solvent which will affect the CO_2 removal. Solubility of CO_2 in the solvents (absorbent) is at the best condition in higher CO_2 partial pressure and lower temperature. This technique is used commercially to remove acid gas (CO_2 + H_2S), from natural gas and to remove CO_2 from syngas in the production of hydrogen, ammonia and methanol. Example of physical absorption process is Selexol process and Rectisol process.

The physical adsorption technologies examples are molecular sieve adsorption, adsorption by activated carbon and adsorption based on lithium compound. The thermodynamic properties of a substance will determine the adsorption process which to shift from the gas phase to attach itself to a solid material. This attachment can be either physical (physisorption) or chemical (chemisorption). This technologies include the selective removal of CO2 from a gas stream to the absorbent (zeolite or charcoal), followed by regeneration (desorption), which can be achieved either by reducing pressure (Pressure-Swing Adsorption or PSA), or by increasing temperature (Temperature-Swing Adsorption, or TSA) or by passing an electric current through the adsorbent (Electrical Swing Adsorption, or ESA) or process hybrids (PTSA) or washing according to Olajire, 2011.

Cryogenics method of purification involves the separation of the gas mixtures by fractional condensation and distillation. The process happens at low temperature. This technique is a commercial process used to liquefy and purify CO2 from relatively high purity (>90%) sources. Using this technique, CO_2 can freeze out/liquefied and separated as the gases encounter very low temperature (<-73°C).

Membranes are semi permeable barriers which manage to separate substances by various mechanisms (solution/diffusion, adsorption/diffusion, molecular sieve and ionic transport). They consist of organic (polymeric) or inorganic (carbon, zeolite, ceramic or metallic) type of membranes and can be porous to non-porous. Acting as a filter, membrane can separate one or more gases from a feed mixture and generate a specific gas. Permeability that is the flux of a specific gas through the membrane, and selectivity, the membrane's preference to pass one gas species over the other will determine the membrane performance. Multiple stages or recycling necessary as membrane cannot always achieve high degrees of separation, besides it also sensitive to sulphur compounds and other trace elements. **(Olajire, 2010)**

1.2 Problem Statement

1.2.1 Problem Identification

In this reseach writer is focusing on CO_2 which is released from the industries to the atmosphere. From many technologies stated before, detail research is done in chemical absorption technology. Currently, amine-based technology is used widely for example monoethanolamine (MEA), diethanolamine (DEA) and methyldiethanolamine (MDEA). A lot of research has been made in producing the best mixture of amine including using amine with activator to produce a solvent that can absorb most CO_2 in from flue gas, so that less CO_2 will be released to the atmosphere in order to minimize the greenhouse effect.

According to Olajire, 2010, the MEA process has the following disadvantages for CO2 separation from flue gases: (1) low carbon dioxide loading capacity (g CO2 absorbed/ g absorbent); (2) high equipment corrosion rate; (3) amine degradation by SO2, NO2, HCl and HF and oxygen in flue gas which induces a high absorbent makeup rate; and (4) high energy consumption during high temperature absorbent regeneration. However, there is a lack of attention focused on environmental impact of solvents that been used in gas treating plant. Corrosion problem in certain sections of CO2 plant could be a huge disadvantage as it leads to decrease in efficiency and rising of operating cost of the process. **(Kladkaew, 2011)**

STONVENT is a new developing solvent which have significant potential to remove CO_2 by chemical absorption. So far, there is no specific chemical compound for STONVENT. A lot of test should be carried to analyze the possible environmental impact of STONVENT byproduct after it undergoes CO_2 absorption.

1.2.2 Significant of the Project

Experiments will be conducted in order to study the characterization of the STONVENT byproduct in term of its physical and chemical properties. This can be benefitted to the industries as well as to the environment if from the properties known prove that STONVENT can sustain longer in its condition compare to amine after go through CO2 removal process be commercialized.

1.3 Objective of the Project

I. To characterize STONVENT by-product by physical analysis.
II. To characterize STONVENT by-product by chemical analysis.

1.4 Scope of Study

This project will be using STONVENT byproducts which undergo absorption process. There are basically four tests that have been done upon all the samples, which are pH Test, Refractive Index Test (Atago RX-5000), Fourier Transform Infra-Red (FTIR) (EMC Standard EN 61326-1) and XRD Test. All the samples used is having different operating condition in example, the samples will be varies in temperature, column pressure, CO_2 inlet concentration and its gas flow rate. The temperature of absorption process that STONVENT undergo is varies from 30 – 50 ^{o}C with pressure 10 – 80 barG. Difference level of CO_2 inlet concentration and gas flow rate is introduced to the sample. The data collected is expected to help in characterize STONVENT byproduct.

1.5 Relevancy of the Project

Treating CO2 with alkanoamines is used widely in the industrial in controlling CO2 emission by the industry. However, they will face other problems such as corrosion and degradation of amine if using the amine-base solvent. Therefore further study must be conducted in finding other alternative solvent. In this paper, the characteristic of STONVENT byproduct will be identify which will affirm whether STONVENT byproduct properties undergo same changes like amines byproduct which become the oxidizing agent with its protonated ion of amine(**Veiwab,2009**), so that new solution can be proposed in order to overcome major problem such as corrosion and degradation of CO2 removal solvent.

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. The work (experimental work) done for the project is also depending on the availability to use equipment in the laboratory.

CHAPTER 2

LITERATURE REVIEW

2.1 Environmental Issue

Carbon dioxide (CO$_{2)}$ is a type of greenhouse gases (GHGs). According to the Intergovernmental Panel on Climate Change, (IPCC), burning fossil fuels contribute almost three-fourths of the increase in atmospheric CO$_2$. Increasing level of CO$_2$ can cause global warming effect. Among GHGs such as CO$_{2,}$ carbon monoxide (CO), nitrogen oxides (NOx), sulphur dioxide (SO$_2$), particulates and mercury, CO$_2$ has make up big portion in the atmosphere , which result in 60% of global warming effect.

Pollutant	Natural Gas	Oil	Coal
Carbon dioxide	117,000	164,000	208,000
Carbon monoxide	40	33	208
Nitrogen oxides	92	448	457
Sulphur dioxide	1	1122	2591
Particulates	7	84	2744
Mercury	0.00	0.007	0.016
Total	117,140	165,687.007	214,000.016

Table 1: Fossil Fuel Emission Level (pounds/billion BTU of energy input)

(Source: EIA)

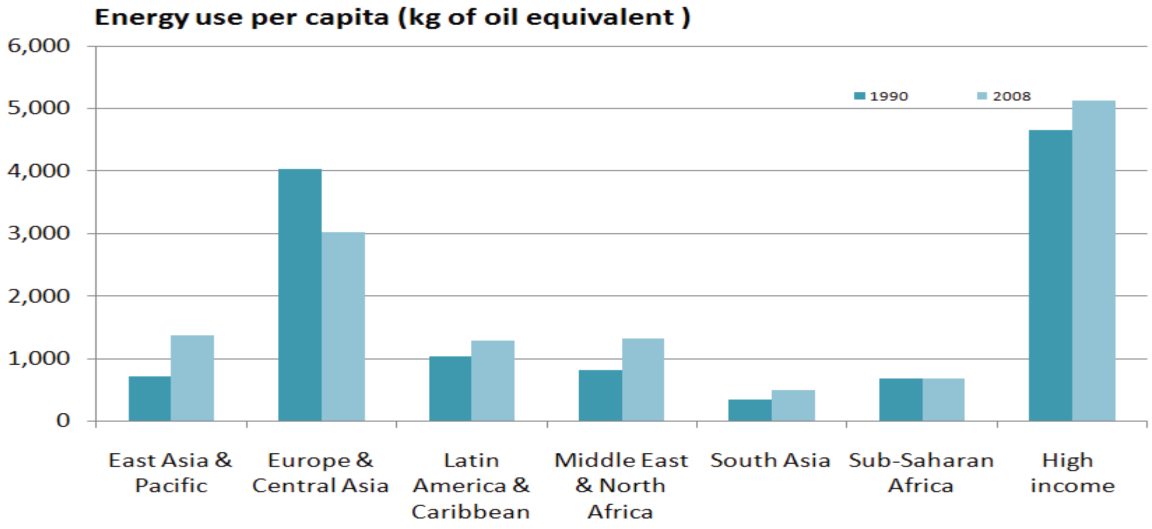


Figure 2: Energy Use per Capita

(Source: data.worldbank.org)

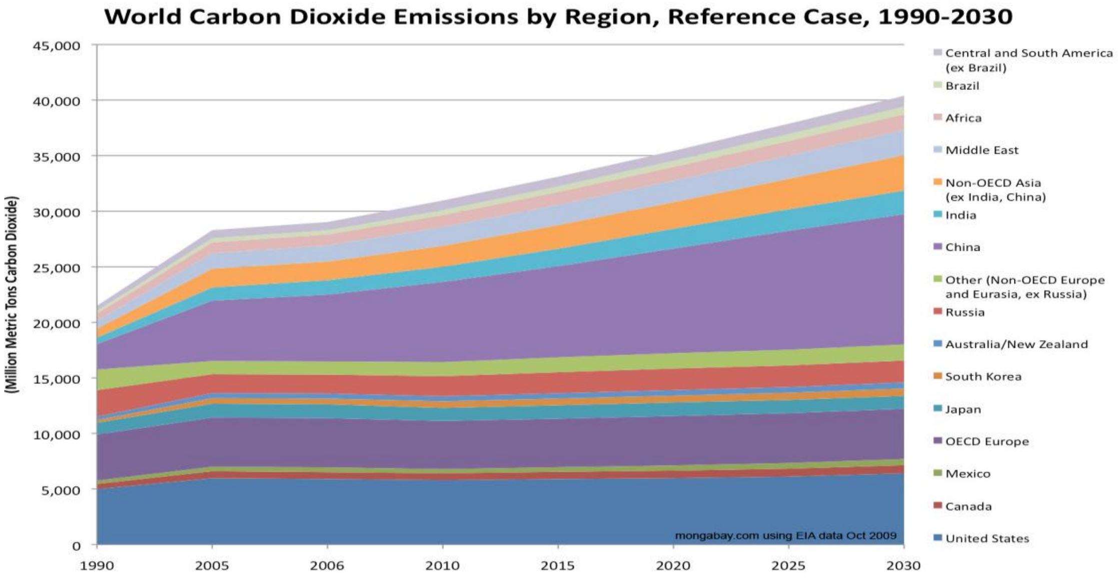


Figure 3: World Carbon Dioxide Emission by Region

(Source: rainforests.mongabay.com)

2.2 CO_2-Hydrocarbon Absorption System

From the data provided, it is very important to reduce amount of CO_2 in the atmosphere and there are many technologies available nowadays. The selection of CO_2 separation technique depends on many factors for example partial pressure of CO_2 in the gas stream, extent of CO_2 recovery required, regeneration of the solvent, sensitivity to impurities such as acid gases and particulates, purity of desired product, capital and operating costs of the process, the cost of additives necessary to overcome fouling and corrosion and where applicable is the environment impact.[**Yagi et.al.,2004**].

Cryogenic method of purifying CO_2 is happens at low temperature condition which involves the separation of the gas mixtures by fractional condensation and distillation. The gasses will be cooled down to around -73 °C so that the CO_2 can freeze out or liquefied and separated. The process has the advantage that it allow the recovery of CO_2 in liquid form which is easy to be transported or pump to the injection side for enhance oil recovery or enhance coal-bed methane.

Membranes are semi-permeable barriers able to separate substances by various mechanisms (solution/diffusion, adsorption/ diffusion, molecular sieve and ionic transport). They are available in different material types, which can be either organic (polymeric) or inorganic (carbon, zeolite, ceramic or metallic) and can be porous to non-porous. Membranes act as filters to separate one or more gases from a feed mixture and generate a specific gas rich permeate. Two characteristics dictate membrane performance; permeability, that is the flux of a specific gas through the membrane, and selectivity, the membrane's preference to pass one gas species over the other.

Chemical absorption technique is preferred for low to moderate CO_2 partial pressure. Some examples of solvent that can be used are ammonia, water, alkaline salt and alkanolamines solution. The acid gas, CO_2 will react with chemical solvents to form weakly bonded intermediate compound. Absorption of CO_2 at low temperatures (35–50°C) and relatively low CO_2 partial pressure (proportional to concentration) can occur if the solvent is highly attracted to the solute, but this high attraction between the solvent and CO_2 causes regeneration energy to be high. If the solvent isn't very attracted to CO_2, regeneration is simple, but little loading is possible. The absorption technology using amine solution or aqueous alkanolamines is regarded as the most mature technology while mixed amine have been reported to maximize the desirable qualities of the individual amines.

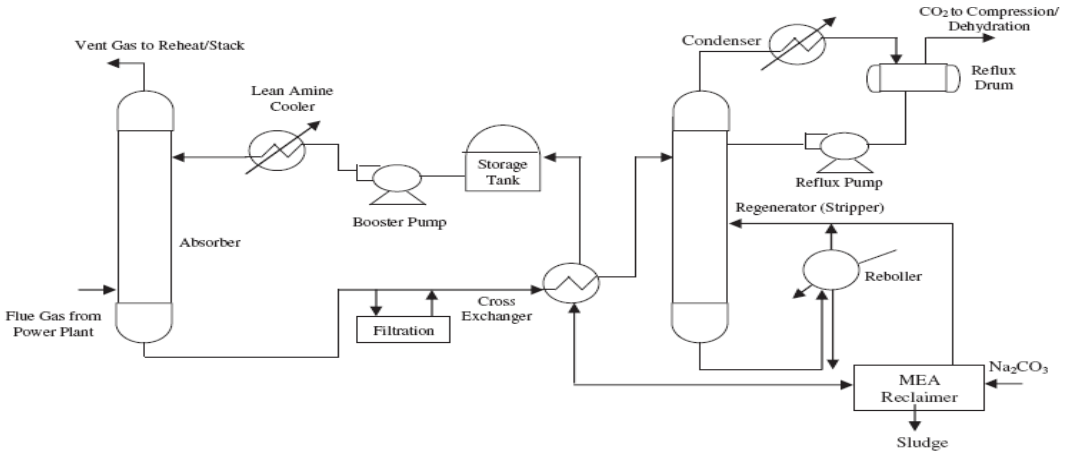


Figure 4: Process Flow Diagram of a Typical Chemical Absorption System for CO_2 Recovery from the Flue Gas (Source:Olajire,2010)

2.3 Alkanolamines Absorption

Alkanolamines are the most popular absorbents used to remove CO_2 from process gas stream. Amine fall into various classes depending on how many of the hydrogen atoms are replaced. Primary amines have only one of the hydrogen atoms in the ammonia molecule which has been replaced. The formula of the primary amine will be RNH2 where “R” is an alkyl group. Examples of primary amines are monoethanolamine (MEA) and diglycolamine (DGA). In secondary amine, alkyl groups have replaced two of the hydrogens in an ammonia molecule. The example of secondary amine is diethanolamine (DEA). Tertiary alkanolamines posses no hydrogen atom attached to the nitrogen atom, as in the case of primary and secondary alkanolamines. Sterically hindered amine have an amino group attached to a bulky alkyl group, for example, 2-amino-2-methyl-1-propanol (AMP).

Primary and secondary alkanolamines are known to react rapidly with CO_2 to form carbamate. The fundamental reaction of MEA for this process is:

$$\underset{MEA}{C_2H_4OHNH_2} + H_2O + CO_2 \Leftrightarrow C_2H_4OHNH_3^+ + HCO_3^-$$

How ever, there are some disadvantage **[Fauth et.al., 2005, Resnik et. al., 2004, Yeh et. al., 2005]** of using the primary or secondary amine, which are:

I. Low carbon dioxide loading capacity (g CO_2 absorbed/ g absorbent)
II. High equipment corrosion rate
III. Amine degradation by SO_2, NO_2, HCl, HF, and oxygen in flue gas which induce a high absorbent make up rate
IV. High energy consumption during high temperature absorbent regeneration

The primary amine has been used extensively because of its high reactivity and low solvent cost. However the maximum CO_2 loading in MEA is limited by 0.5 mol of CO_2 per mol of amine when carbamate is the final products of reaction**. [B.P Mandal et. al.,2001]**

Tertiary amines have high loading capacity (1mol of CO_2 per mol of amine) and its low heat of reaction with CO_2 lead to lower energy requirement for regeneration. **[B.P Mandal et. al.,2001].** The low reactivity is because no carbamate reaction take place due to posses no hydrogen atom attached to the nitrogen atom. The CO_2 absorption rate can be enhance by the addition of small amounts of primary or secondary amine. **[Chakravarty, Phukan, & Weiland, 1985]**

By using sterically hindered amine, the molecular configuration which attaching a bulky substitute to the nitrogen atom of amine molecule does affect the capacity of absorption and desorption temperature. The rotation of the bulky alkyl group around the aminocarbamate group will result in considerably low stability of the carbamate compound. The carbamate compound thus likely to react with water and forms free amines and bicarbonate ions.

$$2RNH_2 + CO_2 \rightarrow RNHCOO^- + RNH_3^+ \qquad (1)$$

$$RNHCOO^- + H_2O \rightarrow RNH_2 + HCO_3^- \qquad (2)$$

$$RNH_2 + CO_2 + H_2O \rightarrow RNH_3^+ + HCO_3^- \qquad (3)$$

Besides that, the sterically hindered amine is greatly low corrosive nature and does not require a corrosive inhibitor. It offers superior CO_2 absorption and regeneration process, lower degradation, lower circulation rate and has less solvent loss when compared to other amined-based system.

The CO_2 absorption or removal efficiency (η) will be determined using the following equation:

$$\eta = \left[1 - \left(\frac{y_{out}}{1 - y_{out}}\right) \times \left(\frac{1 - y_{in}}{y_{in}}\right)\right] \times 100$$

where y_{out} and y_{in} denote mole fraction of CO_2 in gas phase.

For the experiments, an aqueous solution of alkanolamine (e.g., MEA, DEA, etc.) was prepared in the feed tank by diluting the concentrated alkanolamine with deionized water to a given concentration. The total alkanolamine concentration was determined by titration with a standard 1.0 kmol/m3 hydrochloric acid (HCl) solution using methyl orange indicator.

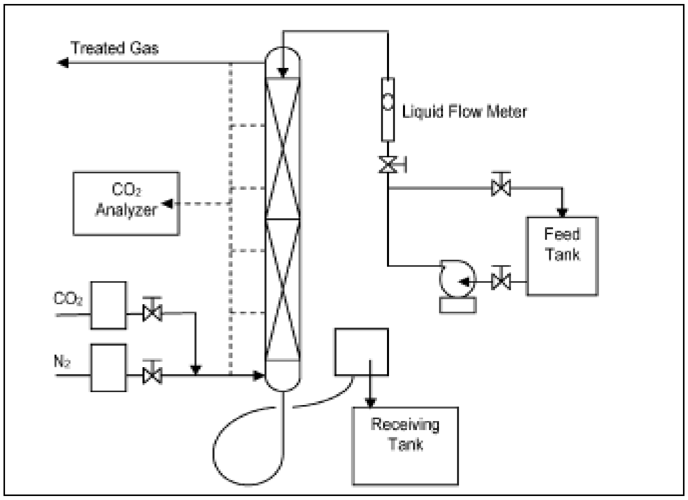


Figure 5: Example of Bench-Scale Absorption Unit

(Source: Veawab and Aroonwilas, 2004)

2.4 Adverse Effect of Amine-based Solvent

Currently, well used method of removing CO_2 in industry is by amine based process, which also used widely to remove acid gas impurities from process gas stream. Amine solvent that commercially used for natural gas sweetening operations are like monoethanolamine (MEA), diethanolamine (DEA), and N-methyldiethanolamine (MDEA), as well as mixtures of alkanolamines. Usually, the absorption operation is conducted in large scale used of chemical, this lead to emission of solvent through several ways in example during accidental spills, through exhaust gas and as degraded solvent. (**Eidi-Haugmo et. al., 2009**)

Research shows that amines corrosive from the finding of corrosion in many area in plant for example at the bottom of an absorber, the rich lean heat exchanger, most part of regeneration section and at the condenser overhead. (**Veawab, 2002**). As sulphur dioxide is one of gas that occurs in the process, it may arouse degradation of amine become worse.

2.5 Solvent Development

2.5.1 Solvent Definition

Solvent is known as a liquid or gas which can dissolves a solid, liquid, or gaseous solute to be a solution. There are various way of reaction by using solvent, for example, solvent can be a medium to combine reactants together, can act as a carrier to deliver chemical compounds in solutions in the required amount. Besides that, solvent characteristic is neither the dissolved substance nor the solvent take place in chemical change. Both will turn to the original form by physical separation method in example crystallization, adsorption and distillation. (**Ghani et. al.,2005**)

This project will focus on development of new solvent, STONVENT with unknown physical and chemical properties. If STONVENT is environmental-friendly and less corrosive, it might have bright future to be commercialized as a ‘clean’ solvent that can reduce global warming and delays the corrosion and degradation process.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

The research is focused on STONVENT byproduct characterization. The writer uses academic journals and textbooks published from year 1995 until 2011 to make sure that the information is relevant for further study and in line with the changes of technology trend besides taking account the extensive problem figures as the reference.

The characterization of STONVENT byproduct by physical analysis will require several equipments for example pH meter and refractor meter. The function of these equipments are to measure pH of STONVENT byproduct and to measure its refractive index. For characteristic of STONVENT byproduct by chemical analysis will require FTIR and XRD technique which identify types of chemical bonds in a molecule by producing an infrared absorption spectrum that is like a molecular ‘finger print’ and to characterize crystallographic structure, crystallite size (grain size) and preferred orientation in polycrystalline or powdered solid sample respectively.

At the end of each test, analysis will be made and the STONVENT byproduct characteristic will be compare with characteristic of pure STONVENT. Graphs and tables produced are expected to give clear picture regarding the result.

3.2 Project Activities

Figure 6 is as attached in the appendix.

3.3 Key Milestone / Gantt Chart

		Mei	Jun				July				August				September				
		4	**1**	**2**	**3**	**4**	**1**	**2**	**3**	**4**	**1**	**2**	**3**	**4**	**1**	**2**	**3**	**4**	
	STAGE 1:																		
1	pH Test			🟨	🟨														
2	Refractive Index				🟨	🟨													
3	FTIR Test					🟨	🟨	🟨	🟨	🟨									
4	XRD Test							🟨	🟨	🟨	🟨	🟨	🟨						
5	Data analysis							🟨	🟨	🟨	🟨	🟨	🟨						
6	**Dissertation and Presentation**											🟨	🟨	🟨	🟨				

Table 2: Milestone for the Final Year Project

3.4 Tool Required

Refractor Meter	pH Meter
Atago RX-5000	Mettler Toledo 320 pH Meter
Calibration: Using distil water, RI ~ 1.33228	Calibration: Using Buffer Solution, pH 4.01, pH 7 and pH 9.21
Accuracy: ± 0.00004	Accuracy: ± 0.01
 Atago RX-5000	 Mettler Toledo pH Meter

Table 3 : Data of Refractor Meter and pH Meter

XRD technique

Brand	Bruker
Model	D8 Advance
2θ range (°)	2° to 80°
Step rate (°/sec)	0.05
Sample type	Powder and flat surface solid

Function	To characterize crystallographic structure, crystallite size (grain size) and preferred orientation in polycrystalline or powdered solid samples. Powder diffraction is commonly used to identify unknown substances by comparing diffraction data against a database maintained by International Centre for Diffraction Data. It may also be used to characterize heterogenous solid mixtures to determine relative abundance of crystalline compounds.

Table 4 : Specific data of XRD technique for tested sample

FTIR technique

Brand	Perkin-Elmer
Model	Spectrum One FT-IR (HATR sampling accessory)
Software	Perkin-Elmer Spectrum Software
KBr Pellet	Zn – Se, 45°
Wavelength (cm^{-1})	650 to 4000
Resolution (cm^{-1})	
Sample type	Liquid and aqueous
Function	To record the interaction of infrared radiation with a sample, measuring the frequencies at which the sample absorbs the radiation and the intensities of the absorption. The identification of the sample compound is detected when chemical functional

	groups are boosted to absorb radiation at specific frequencies. The intensity of the absorption is related to the concentration of the component. Intensity and frequency of sample absorption are depicted in a two-dimensional plot called a spectrum. Intensity is generally reported in terms of percent transmittance, the amount of light that passes through it.

Table 5: Specific data of FTIR technique for tested sample

Along the testing of samples, personal protective equipment should be bring along for safety purposes for example, lab coat, full covered shoes, gloves, goggles and respiratory mask.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Result

There are two part of result which consists of physical and chemical analysis. Physical analysis is pH Test and Refractive Index Test. FTIR Technique and XRD Technique are chemicals analysis.

pH and Refractive Index Test

Sample	Liquid Temperature (Deg C)	Column Pressure (barG)	CO2 Inlet Conc (%)	Gas Flow Rate (L/min)	pH Test	RI
1	**Reference of Pure STONVENT**				13.99	1.38536
2	30	10	80	100	9.66	1.36493
3			15	50	10.82	1.37902
4			15	100	13.74	1.37857
5			80	50	10.13	1.37691
6		45	47.5	75	9.59	1.36595
7		80	15	50	9.87	1.36991
8				100	10.27	1.3755
9			80	50	9.52	1.36164
10				100	9.18	1.35986
11	40	10	47.5	75	10.52	1.37885
12		45	47.5	75	9.95	1.37201
13					8.56	1.36077
14					9.92	1.36226
15					9.81	1.36592
16					9.9	1.36868
17				100	9.03	1.36006
18			80	75	8.85	1.3612
19		80	47.5	75	9.44	1.36483
20	50	10	80	50	9.96	1.37202
21			15	50	13.45	1.37535

Table 6 : Result of refractive index test and pH test

FTIR Result

In Figure 7, both pure STONVENT (before reaction) and STONVENT byproduct (after reaction) have been analyzed its characteristic by Fourier Transform Infra-Red (FTIR) technique. The samples are analyzes base on FTIR absorption frequencies as shown in the table below.

Bond	Compound Type	Frequency range, cm^{-1}
C-H	Alkanes	2960-2850(s) stretch
		1470-1350(v) scissoring and bending
	CH_3 Umbrella Deformation	1380(m-w) - Doublet - isopropyl, *t*-butyl
C-H	Alkenes	3080-3020(m) stretch
		1000-675(s) bend
C-H	Aromatic Rings	3100-3000(m) stretch
	Phenyl Ring Substitution Bands	870-675(s) bend
	Phenyl Ring Substitution Overtones	2000-1600(w) - fingerprint region
C-H	Alkanes	3333-3267(s) stretch
		700-610(b) bend
C=C	Alkenes	1680-1640(m,w)) stretch
C≡C	Alkynes	2260-2100(w,sh) stretch
C=C	Aromatic Rings	1600, 1500(w) stretch
C-O	Alcohols, Ethers, Carboxylic acids, Esters	1260-1000(s) stretch
C=O	Aldehydes, Ketones, Carboxylic acids, Esters	1760-1670(s) stretch
O-H	Monomeric -- Alcohols, Phenols	3640-3160(s,br) stretch
	Hydrogen-bonded -- Alcohols, Phenols	3600-3200(b) stretch
	Carboxylic acids	3000-2500(b) stretch
N-H	Amines	3500-3300(m) stretch
		1650-1580 (m) bend
C-N	Amines	1340-1020(m) stretch
C≡N	Nitriles	2260-2220(v) stretch
NO_2	Nitro Compounds	1660-1500(s) asymmetrical stretch
		1390-1260(s) symmetrical stretch

Table 7: FTIR absorption frequencies

XRD Result

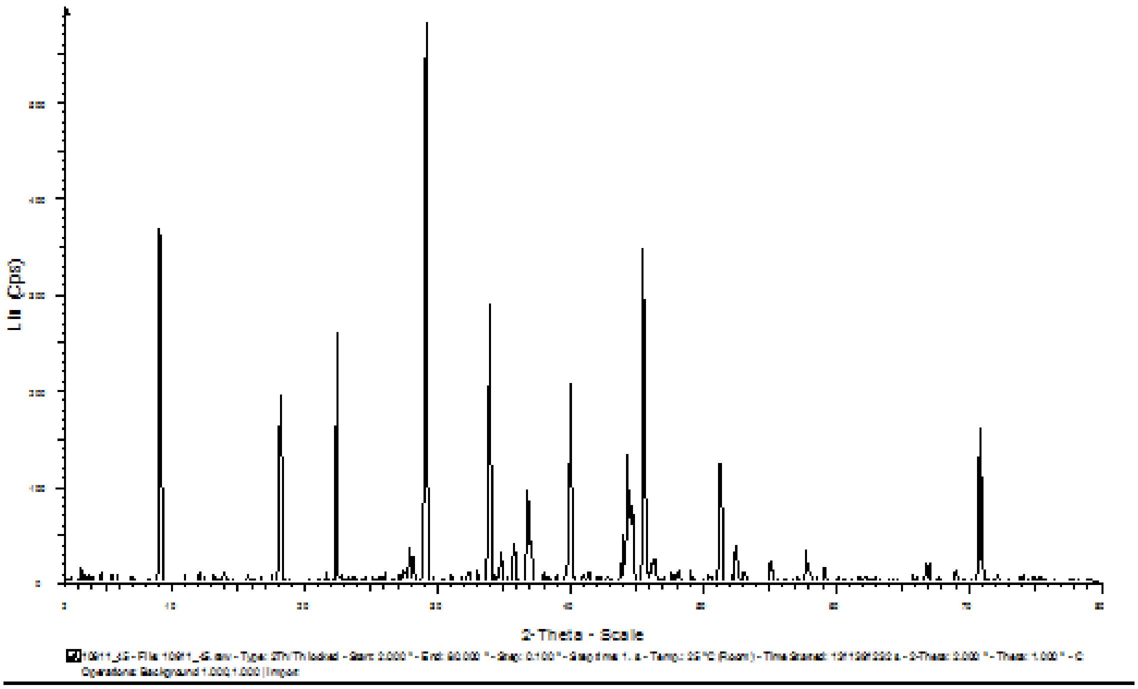


Figure 7 a): XRD Result for operating condition 30ºC, 45 Bar

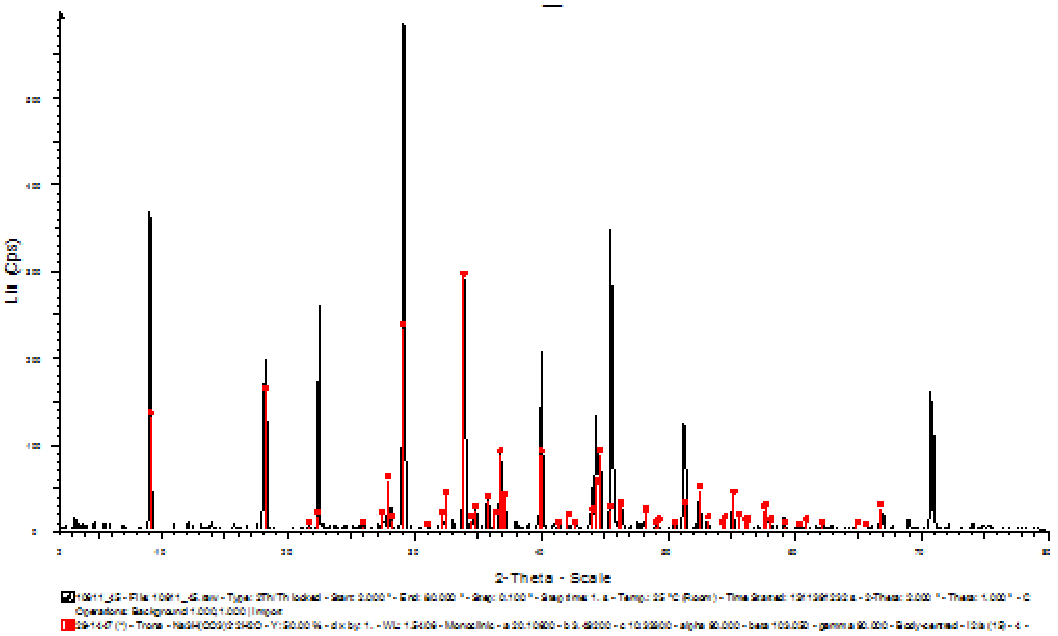


Figure 7 b): XRD Analysis for operating condition 30ºC, 45 Bar

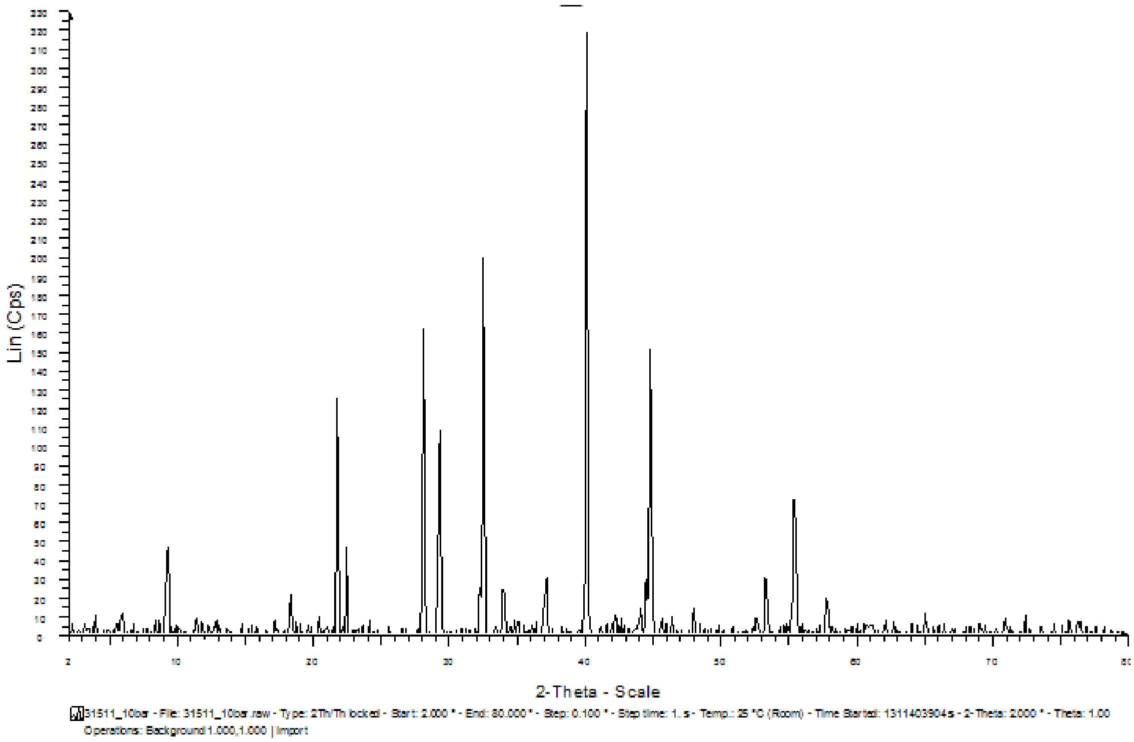


Figure 8a): XRD Result for operating condition 50ºC, 10 Bar

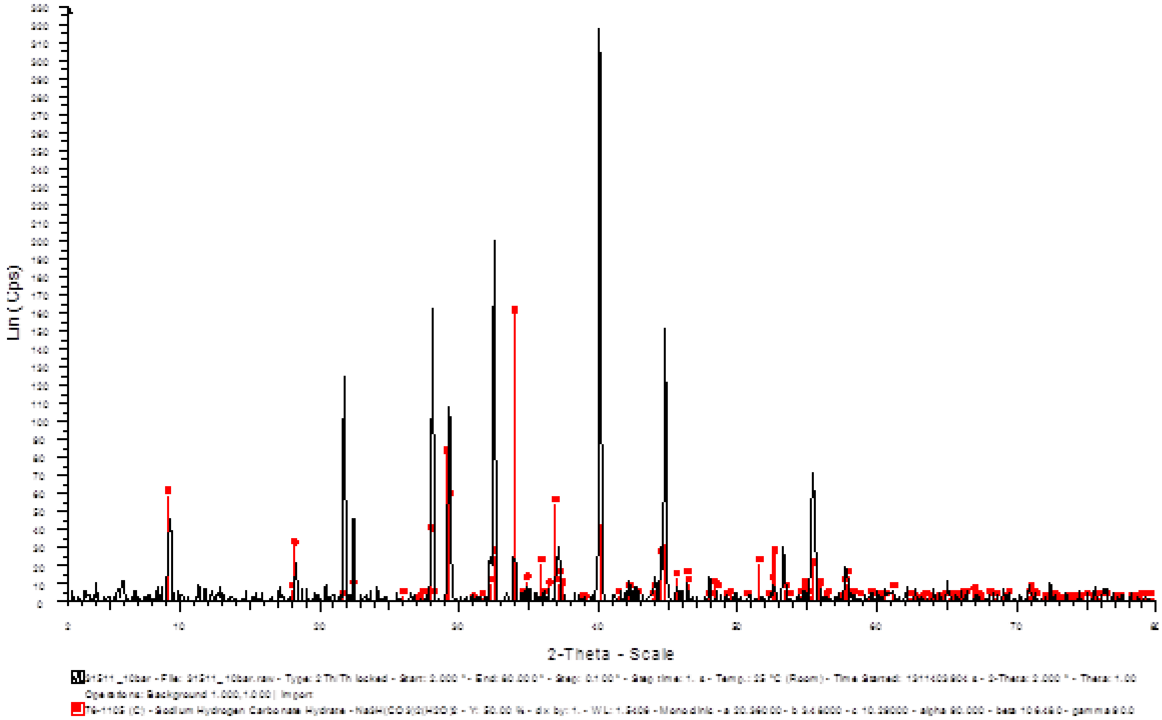


Figure 8b): XRD Analysis for operating condition 50ºC, 10 Bar

4.2 Discussion

Through the result obtained, several observations could be made:

1. When pressure increases as temperature is kept constant, the pH reading and refractive index is decreasing. Same polar can be observed when the temperature is kept 30 ºC and 40 ºC.

Sample	Liquid Temperature (Deg C)	Column Pressure (barG)	CO2 Inlet Conc (%)	Gas Flow Rate (L/min)	pH Test	RI
5	30	10	80	50	10.13	1.37691
6		45	47.5	75	9.59	1.36595
9		80	80	50	9.52	1.36164

Table 8 a): Result for Constant Temperature at 30 ºC

Sample	Liquid Temperature (Deg C)	Column Pressure (barG)	CO2 Inlet Conc (%)	Gas Flow Rate (L/min)	pH Test	Average RI
11	40	10	47.5	50	10.13	1.37691
15		45	47.5	75	9.81	1.36592
19		80	47.5	75	9.44	1.36483

Table 8 b): Result for Constant Temperature at 40 ºC

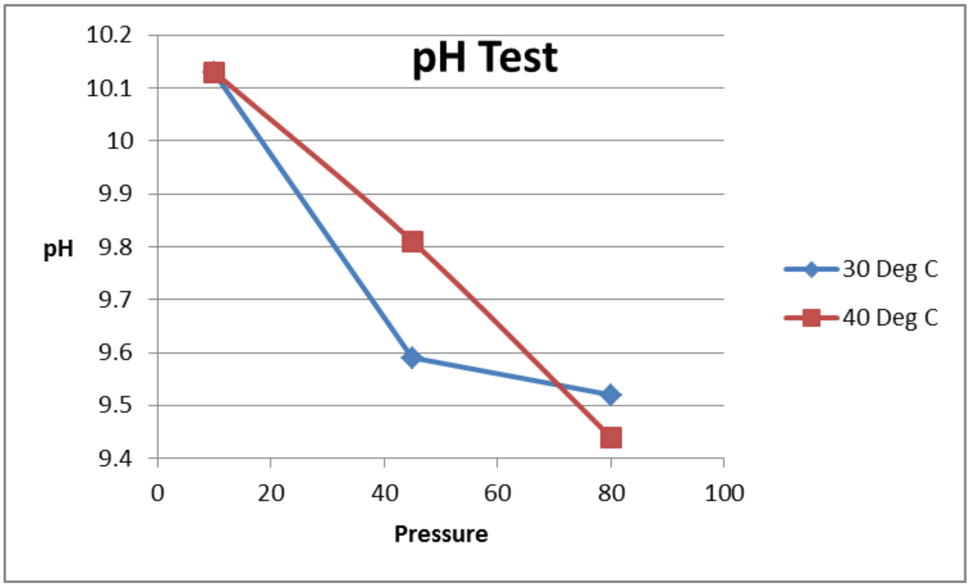


Figure 9 a): pH Test Graph for Constant Temperature

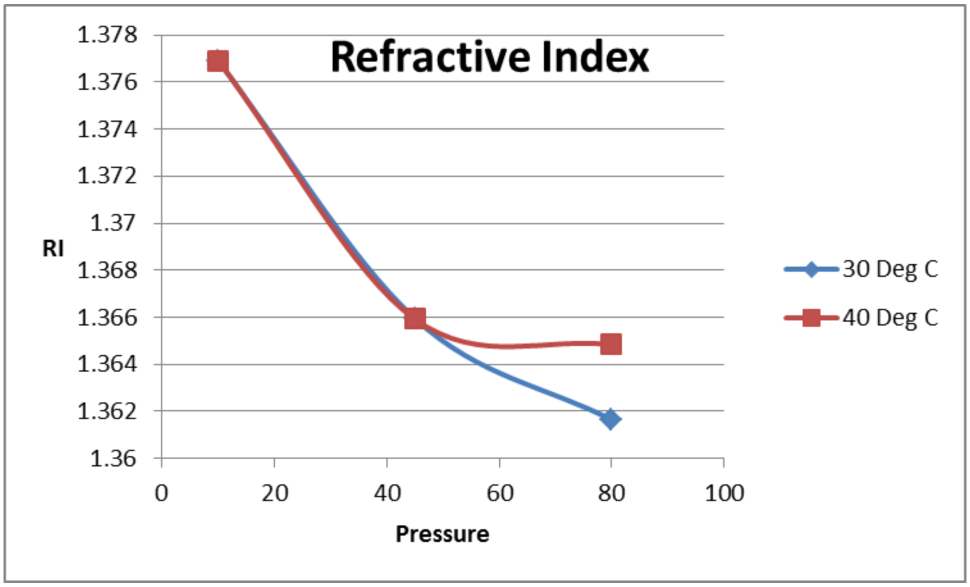


Figure 9 b): RI Test Graph for Constant Temperature

2. When temperature increase and pressure is remained constant, 10 bar G, the pH value and the refractive index is decreasing.

Sample	Liquid Temperature (Deg C)	Column Pressure (barG)	CO2 Inlet Conc (%)	Gas Flow Rate (L/min)	pH Test	RI
	Reference of Pure STONVENT				13.99	1.38536
3	30	10	15	50	10.82	1.37902
11	40		47.5	75	10.52	1.37885
20	50		80	50	9.96	1.37202

Table 8 c): Result for Constant Pressure

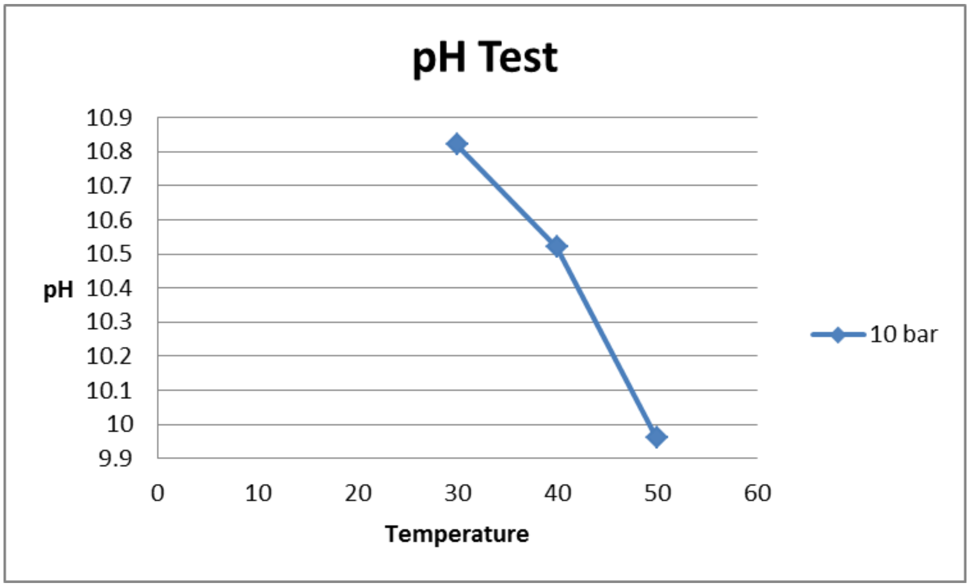


Figure 9 c): pH Graph for Constant Pressure

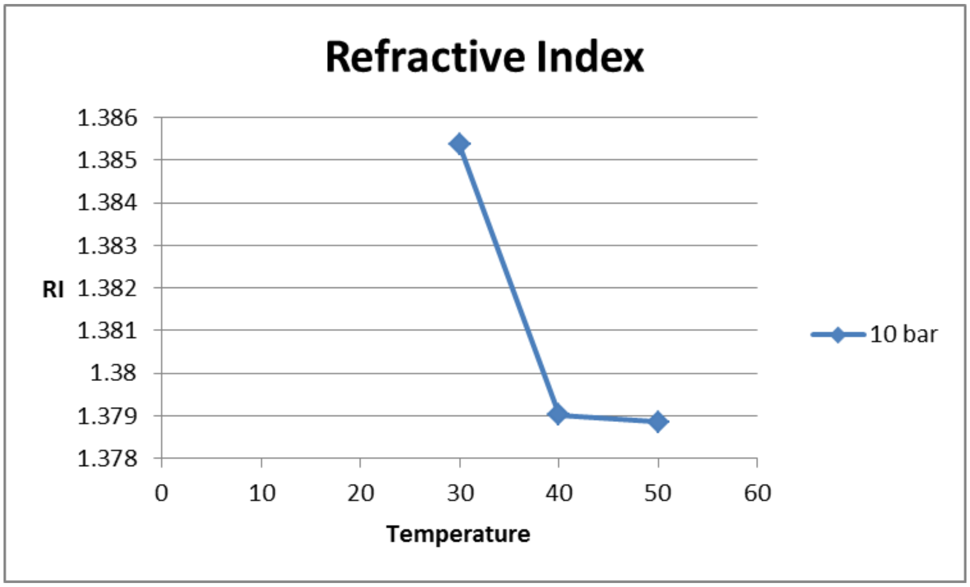


Figure 9 d): RI Test Graph for Constant Pressure

3. pH value indicates the acidity or alkalinity of a solvent. As temperature and pressure is increasing, the pH value is decreasing. This means that the solvent is becoming more acidic. It proves that more CO_2 is captured in the solvent.

4. Refractive Index indicates the concentration of active ingredient in the solvent. As temperature and pressure is increasing, the RI value is decreasing, this proves that the active ingredient of solvent is reduced after react with CO_2.

5. Before reaction, from 3200cm^{-1} to 3600cm^{-1}, the line was largely stretched and alcohol functional group with O-H bond or alkane group with C-H bond might be present. A small bend at the peak from 2400cm^{-1} to 2000cm^{-1} might exhibit the alkynes functional group, C≡C. A considerable sharp peak occurs from 1800 cm^{-1} to 1600 cm^{-1}, which comes from amine, N-H and nitro compound NO_2. A repetitive small peak found from 1200cm^{-1} to 1000cm^{-1} which probably comes from the carboxylic acid functional group, C-O bond and amine functional group with C-N bond. From 1000cm^{-1} to 650cm^{-1}, a slight sharp peak might represent the amine functional group with C-H alkenes bond.

6. For FTIR result, graph of pure STONVENT is compared with STONVENT byproduct graph which already undergone removal process, the graph are as below:

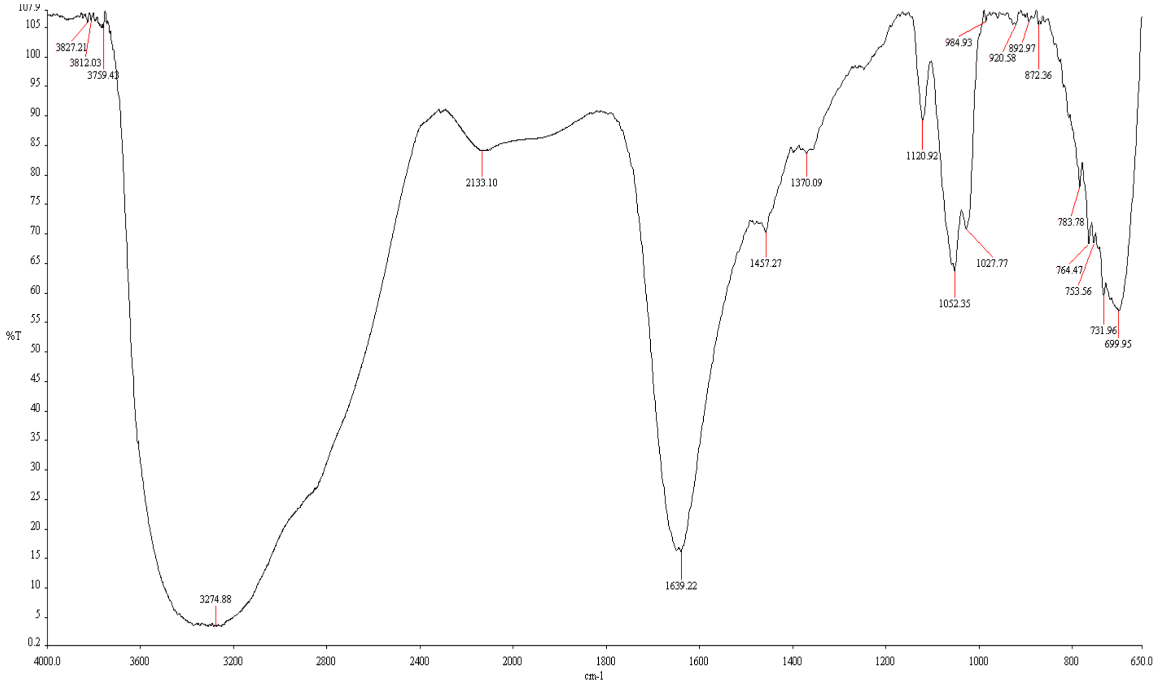


Figure 10a): Pure STONVENT

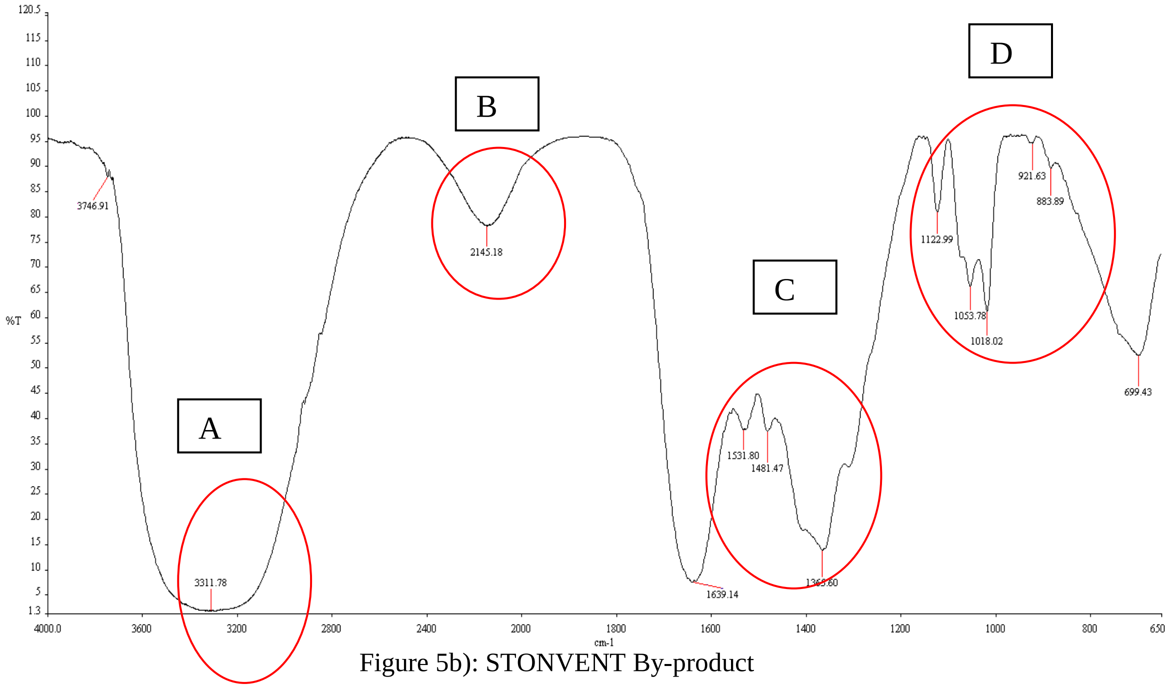


Figure 5b): STONVENT By-product

7. Before reaction, from 3200cm^{-1} to 3600cm^{-1}, the line was largely stretched and alcohol functional group with O-H bond or alkane group with C-H bond might be present. A small bend at the peak from 2400cm^{-1} to 2000cm^{-1} might exhibit the alkynes functional group, C≡C. A considerable sharp peak occurs from 1800 cm^{-1} to 1600 cm^{-1}, which comes from amine, N-H and nitro compound NO_2. A repetitive small peak found from 1200cm^{-1} to 1000cm^{-1} which probably comes from the carboxylic acid functional group, C-O bond and amine functional group with C-N bond. From 1000cm^{-1} to 650cm^{-1}, a slight sharp peak might represent the amine functional group with C-H alkenes bond.

8. After reaction, at point A, transmittance peak from range 3200cm^{-1} to 3600cm^{-1} is narrowed. Here, STONVENT experience certain loss of identity after undergone CO_2 absorption. Low concentration of O-H bond occurs here. The peak of C≡C bond which at range 2000 cm^{-1} to 2400cm^{-1} is observed to be more bending, this shows that solid STONVENT byproduct might associate to C≡C group at point B. At point C, new transmittance peak is found at range 1200cm^{-1} to 1600cm^{-1} and

consists of C-H bond, C-N bond N-H bond and NO_2 bond. At point D, alkyl amine bond C-H present within the range of $650cm^{-1}$ to $1200cm^{-1}$ which denote the presence of amine functional group in the solvent.

9. From XRD test, it is observed that both result shows the presence of Trona, $Na_3H(CO_3)_2.2H_2O$.

CHAPTER 5

CONCLUSION

At the end of this project, the conclusions are:

1. From liquid by-product analysis, it indicates that **CO_2 is more effectively** removed at high temperature and high pressure. In this project, the range of termperature is 30 to 50 Deg C, it is means the effective temperature is 50 Deg C. For the pressure, the range is 10 to 80 Bar, the effective pressure is 80 Bar.

2. The samples of STONVENT undergo changes in characteristic after reaction.

3. Solid by-products was found to consist of Sodium hydrogen carbonate hydrate, Na3H(CO3)2.2H2O compound.

RECOMMENDATION

Since the experiment is one-time run, it is recommended to perform more samples at same operating condition.

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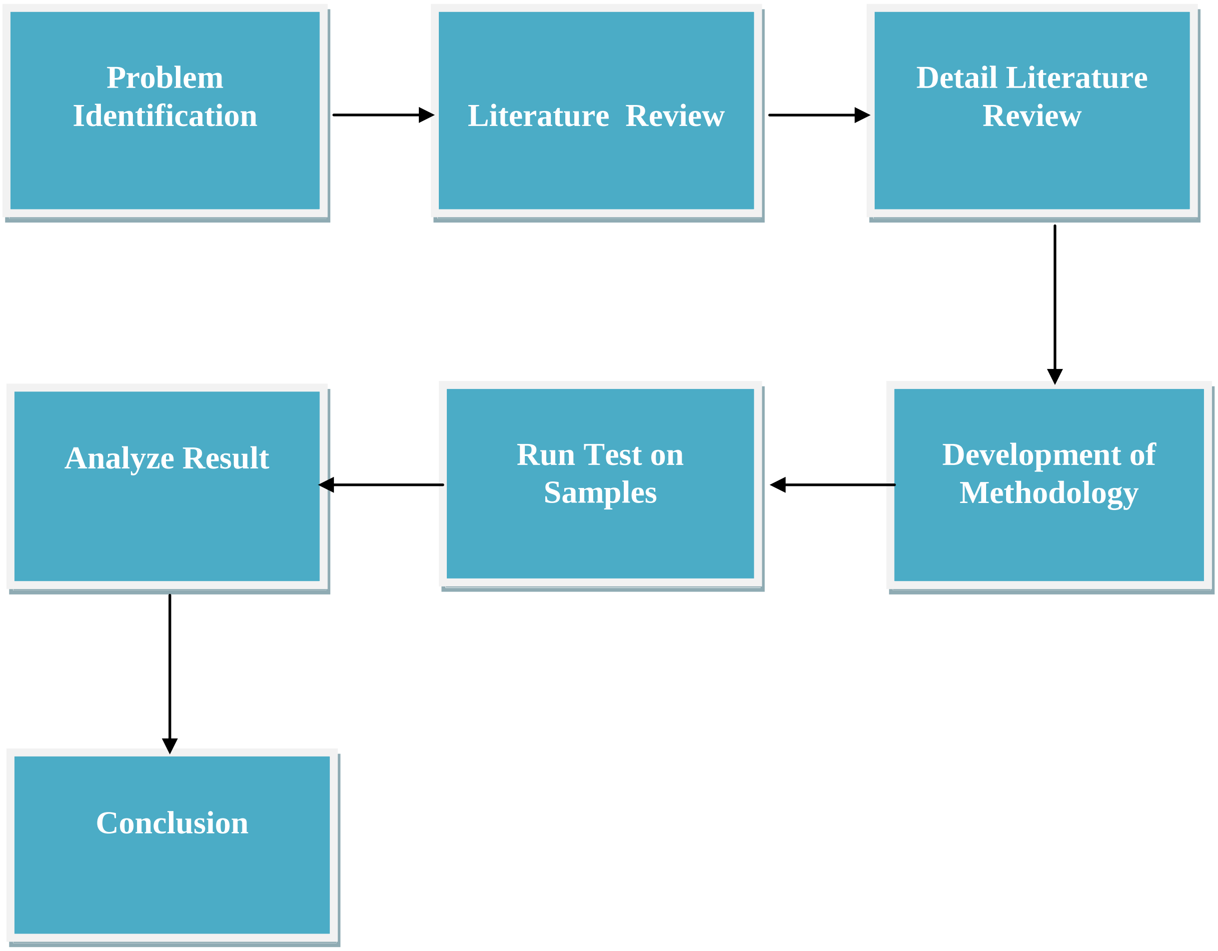
APPENDIX



Appendix 1: Figure 6 : Project Activities

Appendix 2: FTIR Analysis

Sample	Bond	Compound Type	Frequency range, cm^{-1}
1	C-O	Alcohols, Ethers, Carboxylic acids, Esters	1260-1000(s) stretch
	C-H	Alkanes	700-610(b) bend
	C-H	Alkanes	3333-3267(s) stretch
	C-H	Alkenes	1000-675(s) bend
	C≡C	Alkynes	2260-2100(w,sh) stretch
	N-H	Amines	1650-1580 (m) bend
	C-N	Amines	1340-1020(m) stretch
	C-H	CH3 Umbrella Deformation	1470-1350(v) scissoring and bending
	O-H	Monomeric -- Alcohols, Phenols	3640-3160(s,br) stretch
		Hydrogen-bonded -- Alcohols, Phenols	3600-3200(b) stretch
	NO$_2$	Nitro Compounds	1660-1500(s) asymmetrical stretch
			1390-1260(s) symmetrical stretch
	C-H	Phenyl Ring Substitution Bands	870-675(s) bend
4	C-O	Alcohols, Ethers, Carboxylic acids, Esters	1260-1000(s) stretch
	C-H	Alkanes	3333-3267(s) stretch
	C-H	Alkanes	1470-1350(v) scissoring and bending
	C-H	Alkanes	700-610(b) bend
	C-H	Alkenes	1000-675(s) bend
	C=C	Alkenes	1680-1640(m,w)) stretch
	C≡C	Alkynes	2260-2100(w,sh) stretch
	N-H	Amines	1650-1580 (m) bend
	C-N	Amines	1340-1020(m) stretch
	O-H	Monomeric -- Alcohols, Phenols	3640-3160(s,br) stretch
		Hydrogen-bonded -- Alcohols, Phenols	3600-3200(b) stretch
	NO$_2$	Nitro Compounds	1660-1500(s) asymmetrical stretch
			1390-1260(s) symmetrical stretch
	C-H	Phenyl Ring Substitution Overtones	2000-1600(w) - fingerprint region
		Phenyl Ring Substitution Bands	870-675(s) bend

Appendix 2: FTIR Analysis