AN EXPERIMENTAL STUDY OF CARBOXYLIC ACID PREPARED BY CARBON DIOXIDE (CO_2)

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JUDUL: **AN EXPERIMENTAL STUDY OF CARBOXYLIC ACID PREPARED BY CARBON DIOXIDE (CO_2)**

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A thesis submitted in fulfilment for the award of the Degree of Bachelor in Chemical Engineering (Gas Technology)

Faculty of Chemical and Natural Resources Engineering
Universiti Malaysia Pahang

APRIL

DECLARATION

I declare that this thesis entitled "An Experimental Study of Carboxylic Acid Prepared by Carbon Dioxide (CO_2)" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ....

Name : Khairil Izuan Bin Ahmad Tarmidzi

Date :

Dedicated, in thankful appreciation for support,

encouragement and understanding

to my beloved family and friends.

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ABSTRACT

Carboxylic acid has been known as industrial chemical for many years and large amount are used in the manufacture of various product. There are several method has been used such as oxidation of primary alcohol, hydrolysis of acid derivative and carboxylation of Grignard reagent. The current situation of production of carboxylic acid using carbon dioxide as a source required a complex process which may cost higher investment. Yet, there still a lot of carbon dioxide that not fully recover and been release to the atmosphere which may influence the global warming issues. The production of carboxylic acid prepared by carbon dioxide (CO_2) has been studied using photosynthesis catalysis method. The main objective is to study the influenced of photosynthesis light and catalyst in carboxylic acid production. The study conducted using existing reactor and modified with addition of photosynthesis lamp. The carbon dioxide was injected into the reactor and reacts with calcium carbonate catalyst and let the reaction for 2 or 3 hours with difference flow rate of carbon dioxide. As the results, the acidity of the solution increased proportionally to the flow rate of carbon dioxide. The solution that been produced contains carboxylic acid and be proved through the Fourier Transform Infrared (FT-IR). It's showed that the photosynthesis catalysis method applicable to the production of carboxylic acid and the flow rate of carbon dioxide will increase the amount of carboxylic acid.

ABSTRAK

Asid karboksilik telah dikenali sebagai bahan kimia industri selama bertahun-tahun dan jumlah besar yang digunakan dalam pembuatan pelbagai produk. Ada beberapa kaedah telah digunakan seperti pengoksidaan daripada alkohol primer, hidrolisis asid derivatif dan carboxylation dari reagen Grignard. Situasi saat ini menunjukkan penghasilan asid karboksilik menggunakan karbon dioksida sebagai sumber merupakan suatu proses kompleks dan mungkin memerlukan kos pelaburan lebih tinggi. Namun, masih banyak karbon dioksida yang tidak sepenuhnya digunakan dan telah terbebas ke atmosfera yang boleh menyebabkan pemanasan global. Penghasilan asid karboksilik daripada karbon dioksida (CO2) telah dipelajari dengan menggunakan kaedah fotosintesis katalisis. Tujuan utamanya adalah untuk mempelajari pengaruh cahaya fotosintesis dan mangkin dalam penghasilan asid karboksilik Penyelidikan dilakukan dengan menggunakan reaktor yang sedia ada dan diubahsuai dengan penambahan lampu fotosintesis. Karbon dioksida yang disuntik ke dalam reaktor dan mangkin bertindak balas dengan kalsium karbonat dan dibiarkan bertindak balas selama 2 atau 3 jam dengan perbezaan aliran karbon dioksida. Sebagai hasil, keasidan larutan meningkat secara proporsional dengan aliran karbon dioksida. Penyelesaian yang dihasilkan mengandungi asid karboksilik dan dibuktikan melalui Fourier Transform Infrared (FT-IR). Ini menunjukkan bahawa kaedah fotosintesis katalisis berlaku dengan penghasilan asid karboksilik dan aliran karbon dioksida akan meningkatkan jumlah asid karboksilik.

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LIST OF SYMBOL/ ABBREVIATIONS/TERMS

CO_2	-	carbon dioxide
FT-IR	-	Fourier Transform Infrared
COOH	-	carboxylic acid
RCH_2OH	-	primary alcohol
RCOOH	-	primary carboxylic acid
H_2CrO_4	-	chromic acid
$KMnO_4$	-	potassium permanganate
KCN	-	potassium cyanide
NaCN	-	sodium cyanide
H_2CO_3	-	carbonic acid
HCO_3^-	-	bicarbonate ion
H_2O	-	Water

CHAPTER 1

INTRODUCTION

This chapter discusses brief overview of the study and discussed, the nature of the research, the problem that this paper trying to address, aims and objectives of the work and indication of how will the work progressed. Due to high emission of CO_2 into atmosphere, this research was conducted to minimize the emission by converting CO_2 into useful product such as carboxylic acid using photosynthesis catalysis method which is still new and never been claimed successful before.

1.1 Introduction of Carboxylic Acid

Carboxylic acid is among the most useful building blocks for synthesizing other molecule, both in nature and in the chemical laboratory. They are named systematically by replacing the terminal of the corresponding alkane name with oic acid. Like aldehydes and ketones , the carbonyl carbon atom is hybridized, like alcohols, carboxylic acids are associated through hydrogen bonding therefore have high boiling point.(John Mcmurry, 1997)

There are several method to produce carboxylic acid such as oxidation of primary alcohol, hydrolysis acid derivative and carboxylation of Grignard reagent. For example, the easiest acid derivatives to hydrolyze are acid chlorides, which require only the addition of water. Carboxylic acid salts are converted to the corresponding acids instantaneously at room temperature simply on treatment with water and a strong acid. For carboxylation of Grignard reagent, the Grignard reagents react with carbon dioxide (either in the gaseous form, which is bubbled through the solution, or as the solid dry ice) to give magnesium salts of carboxylic acids, which are converted to the acids themselves upon treatment with acid. (John Mcmurry, 1997)

1.2 Background of the Study

Carboxylic acids have been known as industrial chemicals for many years and large amounts are used in the manufacture of various products such as formic acid and acetic acid.For the previous research, there are several method to produce carboxylic acid such as oxidation of primary alcohol, hydrolysis acid derivative and carboxylation of Grignard reagent. For this study, use photosynthesis catalysis method. The carbon dioxide was injected into the reactor and reacts with calcium carbonate catalyst and let the reaction for 2 or 3 hours. The effect of catalyst influenced the yield of the carboxylic acid for the sample. The sample obtain from the reaction has been tested to define the composition. The functional groups of the product which is carbonyl group are identified by using Fourier Transform Infrared (FT-IR). From the studies showed that the photosynthesis catalysis method that has been use is applicable but need more of enhancement to produce high yield of carboxylic acid which can be commercialized.

1.3 Problem Statement

As a countermeasure to serious environmental problem of global warming, it is required in recent years to substantially decreasing the greenhouse effect by various so-called greenhouse gases of which carbon dioxide is one of the most notorious pollutant gases. Reducing the carbon dioxide can be accomplished by decreasing emission of the gas but can also be accomplished by increasing fixation or immobilization of the carbon dioxide gas from emission sources. (Miyazaki *et all,* 2004)

The current situation of production of carboxylic acid using carbon dioxide as a source required a complex process which may cost higher investment. Yet, there still a lot of carbon dioxide that not fully recover and been release to the atmosphere which may influence the global warming issues

There are several methods that have been done to lower the level of CO_2. One that has been studied is photosynthetic carbon dioxide mitigation. In this method CO_2 converted into carboxylic acid when react with catalyst calcium carbonate.

1.4 Objectives

The objectives of this research are to study:-

i) The synthesis of carboxylic acid production.

ii) The capability of carbon dioxide conversion by photosynthesis catalysis method

iii) Development of new green technology by using carbon dioxide as a raw material

1.5 Scope of the Study

On this research, there are focuses on two main scopes:

i. Photosynthesis Catalysis Method

   a. Experiment conduct in a reactor with presence of calcium carbonate catalyst and high intensity photosynthesis lamp.

ii. Determination of composition

   a. Analyze product composition by using Fourier Transform Infrared (FT-IR)

   b. Define the characteristic of the product.

1.6 Rationale and Significance

The rationale of this research is CO_2 is one of harmful gas if being emitted to atmosphere in large amount. Instead of emitting the gas to atmosphere, using it as feed stock to produce another valuable product is more preferred. Turning carbon dioxide into a useful feedstock chemical could help to reduce levels of this greenhouse gas in the atmosphere, as well as providing a cheap source of carbon.

Photosynthesis catalysis method is a new method that has not been acclaimed to be effective in converting CO_2 into carboxylic acid with the presence of catalyst calcium carbonate.. This head start research as an initiative in findings a new method producing carboxylic acid which efficient and cheap and in the same time environmental friendly.

CHAPTER 2

LITERATURE REVIEW

2.1 Synthesis of Carboxylic Acid

2.1.1 Hydrolysis of Acid Derivative

The easiest acid derivatives to hydrolyze are acid chlorides, which require only the addition of water. Carboxylic acid salts are converted to the corresponding acids instantaneously at room temperature simply on treatment with water and a strong acid such as hydrochloric acid (A. William Johnson, 1998)

Carboxylic esters, nitriles, and amides are less reactive and typically must be heated with water and a strong acid or base to give the corresponding carboxylic acid. If a base is used, a salt is formed instead of the carboxylic acid, but the salt is easily converted to the acid by treatment with hydrochloric acid. Of these three types of acid derivatives, amides are the least reactive and require the most vigorous treatment. (A. William Johnson, 1998)

$$RCN \rightarrow RCONH_2.$$

2.1.2 Oxidation of Primary Alcohol

The oxidation of primary alcohols is a common method for the synthesis of carboxylic acids:

$$RCH_2OH \rightarrow RCOOH.$$

This requires a strong oxidizing agent, the most common being chromic acid (H_2CrO_4), potassium permanganate ($KMnO_4$), and nitric acid (HNO_3). Aldehydes are oxidized to carboxylic acids more easily (by many oxidizing agents), but this is not often useful, because the aldehydes are usually less available than the corresponding acids. Also important is the oxidation of alkyl side chains of aromatic rings by strong oxidizing agents such as chromic acid, potassium permanganate, and nitric acid to yield aromatic carboxylic acids. (A. William Johnson, 1998)

Regardless of the number of carbon atoms in the side chain or the presence of any groups attached to them, if the first carbon in the alkyl chain is bonded to at least one hydrogen (and not to another aromatic ring), all but one of the carbons are removed, and only a COOH group remains bonded to the aromatic ring. Examples are the oxidations of toluene and 1-chloro-3-phenylpropane. (A. William Johnson, 1998)

2.1.3 Carboxylation of Grignard Reagent

Grignard reagents react with carbon dioxide (either in the gaseous form, which is bubbled through the solution or as the solid dry ice) to give magnesium salts of carboxylic acids, which are converted to the acids themselves upon treatment with acid:

$$RMgBr + CO_2 \rightarrow RCOO^{-}\,{}^{+}MgBr + HCl \rightarrow RCOOH.$$

Unlike the methods previously mentioned, this method adds one carbon atom to the carbon skeleton. A Grignard reagent is prepared from an alkyl or aryl halide; e.g.

$$, RBr + Mg \rightarrow RMgBr.$$

An alternative way to accomplish the same result is to treat the halide with potassium cyanide (KCN) or sodium cyanide (NaCN) and then hydrolyze the resulting nitrile, as mentioned above; e.g.,

$$RBr + KCN \rightarrow RCN \rightarrow RCOOH.$$

The two procedures are complementary. Although all nitriles can be hydrolyzed to the corresponding acid and all Grignard reagents react with carbon dioxide, the halide reactions are more limited. Many types of halides (including aromatic halides) do not react with NaCN or KCN. On the other hand, while Grignard reagents can be made from many of the halides that do not react with NaCN or KCN (including aryl halides), they cannot be made from halides that contain certain other functional groups, such as alcohol, carboxylic ester, aldehyde, or ketone groups. (A. William Johnson, 1998)

2.2 Physical Properties of Carboxylic Acid

Table 2.1: Physical Properties of Carboxylic Acid

Solubility	Soluble with water
Boiling Point	Higher boiling point than water
Acidity	Typically weak acid
Odor	Strong odor
Chemical Bonding	Hydrogen bonding

Table 2.1 shows the physical properties of carboxylic acid. Carboxylic acids usually exist as dimeric pairs in nonpolar media due to their tendency to "self-associate." Smaller carboxylic acids (1 to 5 carbons) are soluble with water, whereas higher carboxylic acids are less soluble due to the increasing hydrophobic nature of the alkyl chain.

Carboxylic acids tend to have higher boiling points than water, not only because of their increased surface area, but because of their tendency to form stabilised dimers. Carboxylic acids are typically weak acids, meaning that they only partially dissociate into H^+ cations and $RCOO^-$ anions in neutral aqueous solution.

Carboxylic acids often have strong odors, especially the volatile derivatives. Most common are acetic acid (vinegar) and butyric acid (rancid butter.). Carboxylic acid has a hydrogen bonding. Hydrogen bonds are easily formed when a hydrogen atom is bonded to such electronegative atoms as oxygen or nitrogen.

2.3 Physical & Chemical Properties of Carbon Dioxide

Carbon dioxide is colourless. At low concentrations, the gas is odourless. At higher concentrations it has a sharp, acidic odour. It will act as an asphyxiant and an irritant. When inhaled at concentrations much higher than usual atmospheric levels, it can produce a sour taste in the mouth and a stinging sensation in the nose and throat. These effects result from the gas dissolving in the mucous membranes and saliva, forming a weak solution of carbonic acid. (NIOSH, 2006)

This sensation can also occur during an attempt to stifle a burp after drinking a carbonated beverage. Amounts above 5,000 ppm are considered very unhealthy, and those above about 50,000 ppm (equal to 5% by volume) are considered dangerous to animal life At standard temperature and pressure, the density of carbon dioxide is around 1.98 kg/m^3, about 1.5 times that of air. The carbon dioxide molecule (O=C=O) contains two double bonds and has a linear shape. It has no electrical dipole, and as it is fully oxidized, it is moderately reactive and is non-flammable, but will support the combustion of metals such as magnesium. (NIOSH, 2006)

At −78.51° C or -109.3° F, carbon dioxide changes directly from a solid phase to a gaseous phase through sublimation, or from gaseous to solid through deposition. Solid carbon dioxide is normally called "dry ice", a generic trademark. It was first observed in 1825 by the French chemist Charles Thilorier. Dry ice is commonly used as a cooling agent, and it is relatively inexpensive. A convenient property for this purpose is that solid carbon dioxide sublimes directly into the gas phase leaving no liquid. It can often be found in grocery stores and laboratories, and it is also used in the shipping industry. The largest non-cooling use for dry ice is blast cleaning. (NIOSH, 2006)

An alternative form of solid carbon dioxide, an amorphous glass-like form, is possible, although not at atmospheric pressure. This form of glass, called carbonia, was produced by super cooling heated CO_2 at extreme pressure (40–48 GPa or about 400,000 atmospheres) in a diamond anvil. This discovery confirmed the theory that carbon dioxide could exist in a glass state similar to other members of its elemental family, like silicon (silica glass) and germanium. Unlike silica and germania glasses, however, carbonia glass is not stable at normal pressures and reverts back to gas when pressure is released. (Santoro, M. *et al 2006).*

2.4 Carbon Dioxide Conversion

C1 chemistry can no longer be equated only with syngas conversion. Nature's own carbon dioxide photosynthesis and bacterial methane conversion are also C1 conversion processes. We are far from approaching these processes for practical synthetic use efficiently. (Alpad Molnar, 1995)

Production of methane from carbon dioxide (similarly to carbon monoxide) and hydrogen is feasible process (methanation). Similarly, reduction of carbon dioxide with hydrogen to methyl alcohol can be readily carried out, and the method has been developed. (Alpad Molnar, 1995)

The main idea of this process is to be able to take carbon dioxide from atmosphere (also recycling excess carbon dioxide produced from burning fossil fuels contributing to the greenhouse effect) and hydrogen from electrolysis of seawater. (Alpad Molnar, 1995)

2.5 Carbon Dioxide Utilization

It is now well known that so called 'global warming' may become a serious threat to the global environment and human society in this century. Although there existed a lot of unknown factors in the scientific mechanism of warming and its ultimate consequences, the main cause of it is thought to be the anthropogenic emissions of carbon dioxide through combustion of large amount of fossil fuels. (Park et al., 2003)

Atmospheric CO_2 concentration is now higher than it was at any time in the past 26 million years and is expected to nearly double during this century. In order to cope with global warming problem, a variety of measures have been proposed and/ or implemented worldwide for preventing, alleviating, or for adapting to warming. As a result of this effort, carbon dioxide utilization has been making a progress and distinct contribution in conducting the strategies for carbon dioxide mitigation and finding the solution of these environmental problems by adoption of variety of technologies. (Park et al., 2003)

2.6 Characteristic of Carbon Dioxide

Carbon dioxide (chemical formula: CO_2) is a chemical compound composed of two oxygen atoms covalently bonded to a single carbon atom. It is a gas at standard temperature and pressure and exists in Earth's atmosphere in this state. CO_2 is an acidic oxide: an aqueous solution turns litmus from blue to pink. It is the anhydride of carbonic acid, an acid which is unstable and is known to exist only in aqueous solution. (Park et al., 2003)

$$CO_2 + H_2O \rightleftharpoons H_2CO_3$$

CO_2 is toxic in higher concentrations: 1% (10,000 ppm) will make some people feel drowsy. (Park et al., 2003)

2.7 Photosynthesis

Photosynthesis is a process of converting carbon dioxide into organic compounds such as sugar using the energy from sunlight. Photosynthesis occurs in green plants, algae and many species of bacteria. Organisms that use photosynthesis to create their own food called photoautotroph. Usually photosynthesis process will produce oxygen and some other organic compounds. (J.F Allen *et all,* 2006)

Photochemical systems, been studied in an effort to develop systems capable of directly reducing CO_2 to fuels or chemicals using solar energy. Transition-metal complexes have been used as both catalysts and solar energy converters, since they can absorb a significant portion of the solar spectrum, have long-lived excited states, are able to promote the activation of small molecules, and are robust. (J.F Allen *et all,* 2006)

Carbon dioxide utilization by artificial photo conversion presents a challenging alternative to thermal hydrogenation reactions which require H_2O. (J.F Allen *et all,* 2006)

2.8 Application of Carbon Dioxide by Humans

Humans use carbon dioxide in many different ways. The most familiar example is its use in soft drinks and beer, to make them fizzy. Carbon dioxide released by baking g powder or yeast makes cake batter rise. Some fire extinguishers use carbon dioxide because it is denser than air. Carbon dioxide can blanket a fire, because of its heaviness. It prevents oxygen from getting to the fire and as a result, the burning material is deprived of the oxygen it needs to continue burning. (G. Tyler Miller, 1998)

2.9 Influent Carbon Dioxide in Alkalinity

Carbon dioxide can change the pH of water. This is how it works:

Carbon dioxide dissolves slightly in water to form a weak acid called carbonic acid, H_2CO_3, according to the following reaction:

$$CO_2 + H_20 \longrightarrow H_2CO_3$$

After that, carbonic acid reacts slightly and reversibly in water to form a hydronium cation, H_3O+, and the bicarbonate ion, HCO^3-, according to the following reaction

$$H_2CO_3 + H_20 \longrightarrow HCO_3^- + H_30^+$$

This chemical behaviour explains why water, which normally has a neutral pH of 7 has an acidic pH of approximately 5.5 when it has been exposed to air. (G. Tyler Miller, 1998)

2.10 Carbon Dioxide Emissions

Due to human activities, the amount of CO_2 released into the atmosphere has been rising extensively during the last 150 years. As a result, it has exceeded the amount sequestered in biomass, the oceans, and other sinks. There has been a climb in carbon dioxide concentrations in the atmosphere of about 280 ppm in 1850 to 364 ppm in 1998, mainly due to human activities during and after the industrial revolution, which began in 1850. Humans have been increasing the amount of carbon dioxide in air by burning of fossil fuels, by producing cement and by carrying out land clearing and forest combustion. About 22% of the current atmospheric CO_2 concentrations exist due to these human activities, considered that there is no change in natural amounts of carbon dioxide. (G. Tyler Miller, 1998)

2.11 Effect of Global Warming

The last two decades the world has witnessed unprecedented effects of global warming. The increasing CO_2 in the atmosphere gave rise to warmer global temperature (IPCC, 1996). The main source of CO_2 is fossil fuel, the main power source of automobile and industry that are directly linked with economic growth and developments.

An increase in global temperature will cause sea levels to rise and will change the amount and pattern of precipitation, probably including expansion of subtropical deserts.(A.Reichler *et all,*2007). Warming is expected to be strongest in the Arctic and would be associated with continuing retreat of glaciers, permafrost and sea ice. Other likely effects include changes in the frequency and intensity of extreme weather events, species extinctions, and changes in agricultural yields. Warming and related changes will vary from region to region around the globe, though the nature of these regional variations is uncertain. (IPCC, 2007)

CHAPTER 3

METHODOLOGY

3.1 Introduction

There are several processes that we can used to produce carboxylic acid, such as hydrolysis of acid derivative, oxidation of primary alcohol and carboxylation of Grignard reagent.

The purposed of this researched is to experimentally define the capability of converting CO_2 into carboxylic acid Besides, the study is to analyze the potential of using photosynthesis catalysis method in converting green house gas into useful product. The experiment is done by using glass reactor and installed with photosynthesis lamp.

3.2 Research Methodology

The research was started with finding the related information from the journals. and reference books Next was gathering all the material needed to run experiment such as CO_2 gas with 99.97% of purity, quartz glass reaction vessel, photosynthesis lamp and calcium carbonate catalyst as summarised in figure 3.1. The conversion of carbon dioxide into carboxylic acid takes place inside the reactor. The reaction will be done in present of photo catalytic and calcium carbonate as catalyst. Product will analyze using Fourier Transform Infrared (FT-IR) to check the composition and the functional group.

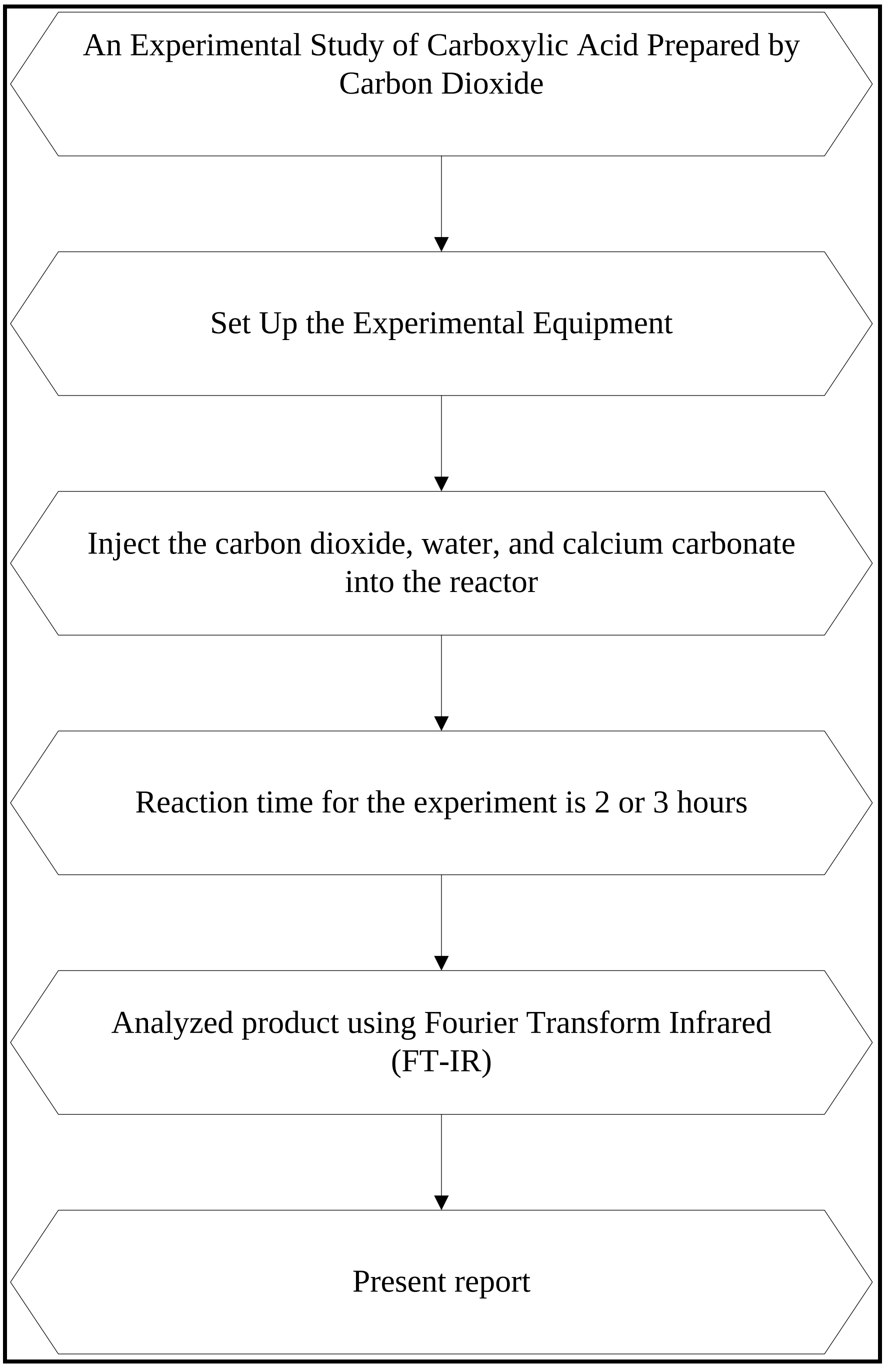

Figure 3.1: Workflow Diagram

3.3 Experimental Procedure

Operating temperature for this reaction is 60 -120 °C and operating pressure at least 1 atm. The reactor design should resist the operating condition. Experimental design describe in figure 3.2. Flow rate of carbon dioxide, water and calcium carbonate catalyst were injected into reactor. Flow rate of carbon dioxide were injected at flow rate 20 ml/hr, 40 ml/hr, 60 ml/hr and 80 ml/hr controlled by flow meter. The reaction runs for 2 or 3 hours. The reactor must be installed with an exhaust fan near the lamp to remove heat that produced by lamp. Product gain from this reaction will analyze using Fourier Transform Infrared (FT-IR).

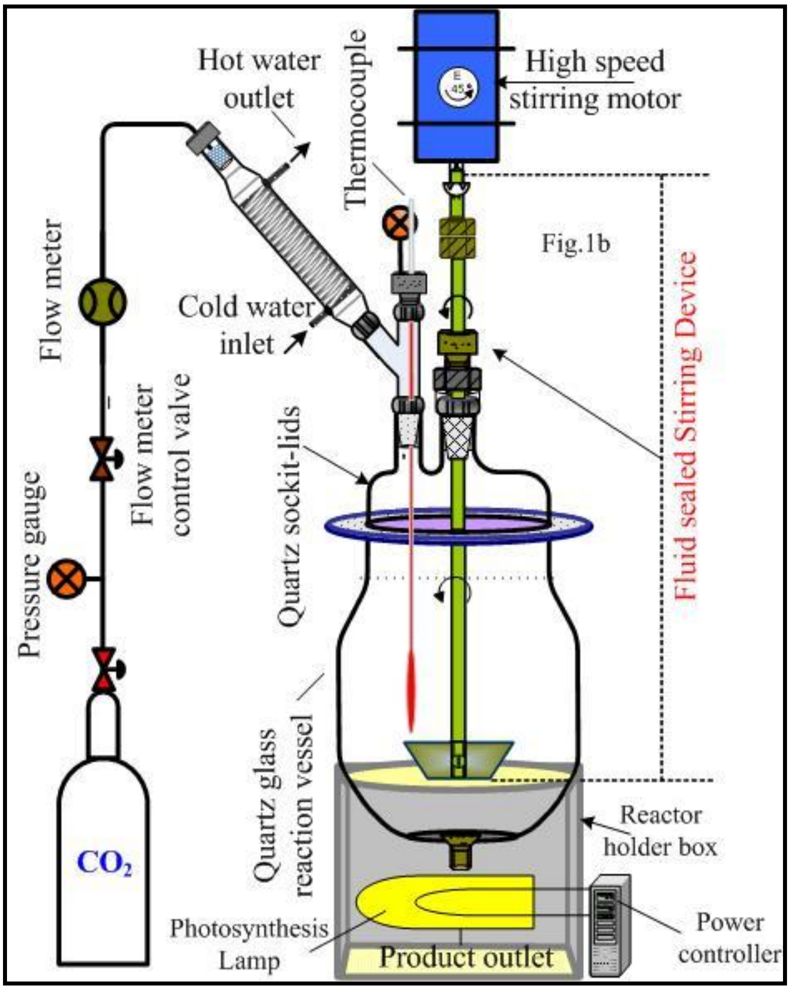

Figure 3.2 Experimental Diagram

3.4 Material & Equipment

The material that required running the experiment are high purity of carbon dioxide, high intensity of photosynthesis lamp, flow meter, quartz glass reaction vessel and calcium carbonate.

3.4.1 Carbon Dioxide

Carbon dioxide that will be used must more than 95% of purity. The purity of gas can affect the product composition. Carbon dioxide was supplied to the reactor through flexible hose and the flow rate was controlled by flow meter. The pressure of carbon dioxide from the manifold tank steps down from 200 bars to 1.5 bars.

3.4.2 Flow Meter

A flow meter shows in figure 3.3 is an instrument used to measure linear, nonlinear, mass or volumetric flow rate of a liquid or a gas. In this experiment, flow meter required to make sure the flow of CO_2 can be control.

Figure 3.3: Flow meter

3.4.3 Photosynthesis Lamp

Photosynthesis lamp required in this experiment to enhance the photosynthesis process. The types of lamp are Metal Halide Lamp with 1000 watt using single phase electric supply. Using high intensity lamp needs several devices such as ballast, ignitor and capasitor. The photosynthesis lamp was installed above the reactor around 10 cm. The light from photosynthesis lamp should direct contact to the solution. (Figure 3.2)

3.4.4 Calcium carbonate

Calcium carbonate shows in figure 3.4 is a chemical compound with the chemical formula $CaCO_3$. It is a common substance found in rock in all parts of the world. Calcium carbonate is the active ingredient in agricultural lime, and is usually the principal cause of hard water. This catalyst can be ordered via technical unit of chemical laboratory in Universiti Malaysia Pahang.

Figure 3.4: Calcium Carbonate

3.4.5 Fourier Transform Infrared (FT-IR)

FTIR shows in figure 3.5 are most useful for identifying chemicals that are either organic or inorganic. It can be utilized to quantitate some components of an unknown mixture. It can be applied to the analysis of solids, liquids, and gasses. The term Fourier Transform Infrared Spectroscopy (FTIR) refers to a fairly recent development in the manner in which the data is collected and converted from an interference pattern to a spectrum. Today's FTIR instruments are computerized which makes them faster and more sensitive than the older dispersive instruments.

Figure 3.5: Fourier Transform Infrared (FT-IR)

CHAPTER 4

RESULT AND DISCUSSION

4.1 Experimental Results and Discussions

The experiment production of carboxylic acid by using photosynthesis catalysis method is completed. The procedure that we used in this experiment is followed step by step. Table 4.1 shows the relationship of flow rate of carbon dioxide and pH number. In this study, different flow rate had been use, starting with 20 ml/hr, 40 ml/hr, 60 ml/hr and 80 ml/hr. Table above shows the value of pH number from this experiment.

Table 4.1: Flow rate of CO_2 vs. pH

Flow Rate of Carbon Dioxide (ml/hr)	pH
20	6.10
40	5.88
60	5.33
80	4.74

Figure 4.1 shows the graph of relationship between flow rate of carbon dioxide and pH number. Based on the graph, flow rate of the carbon dioxide is inversely proportional to the pH number. When the flow rate of the carbon dioxide is increase, the pH is decrease. Carboxylic acid is a weak acid because they partially dissociate in water.

Carbon dioxide is a major contributor in the production of carboxylic acid in the presence of photosynthesis lamp and catalyst. Increasing of carbon dioxide content was dissolved in water would cause an increasing of carboxylic acid production. Carboxylic acid is a weak acid and has lower pH number below than 6 More carboxylic acid produced may cause lower pH number in the solution. Proved in figure 4.1, flow rate of carbon dioxide is inversely proportional with pH number of the solution.

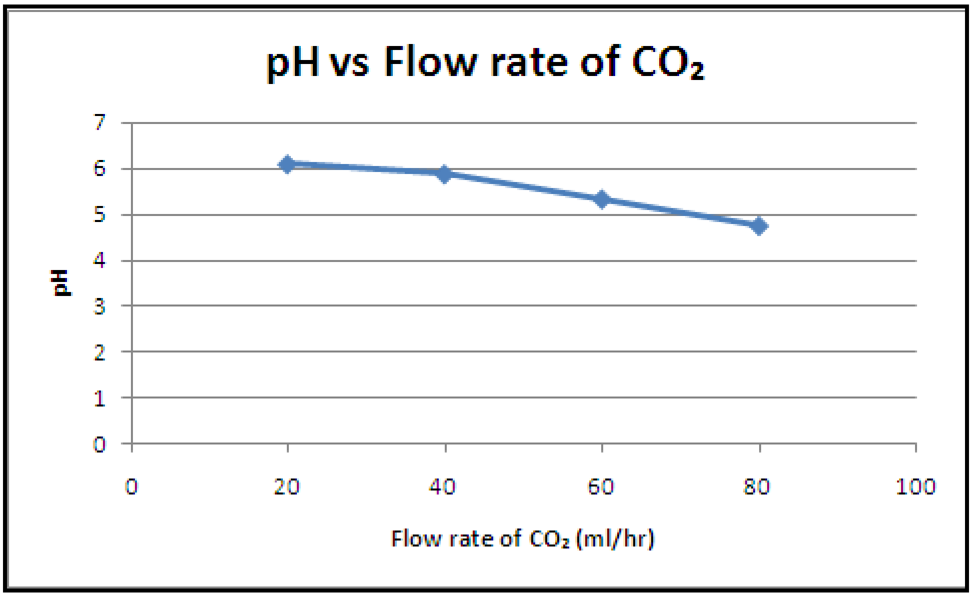

Figure 4.1: pH vs. Flow rate of CO_2

4.4 Fourier Transform Infrared (FT-IR) analysis

Fourier transform infrared (FTIR) was used in this analysis in order to determine the functional groups of the product. The functional groups of carboxylic acid will be determined based on the peak value. The wave number of the carboxylic acid represents the functional group in the product. Table 4.2 shows the table of IR Absorption of the functional groups based on their peak. The functional group of the product was analyzed by determined the wavelength.

Figure 4.2 showed the chemical equation for carboxylic acid. The ability of carboxylic acids to ionize and behave as acids is a direct function of the electronic properties and bonding order of the atoms that make up the carboxyl (COOH) moiety in the presence of photosynthesis light. Recall that this functional group consists of a carbonyl group that has an electron deficient carbon atom due to pi bonding (double bond) to electronegative oxygen.

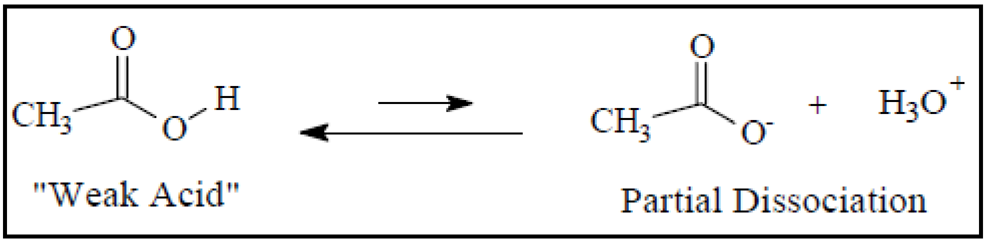

Figure 4.2 Chemical Equations for Carboxylic Acid

Table 4.2: IR Absorption

Functional Group	**Characteristic Absorption(s) (cm^{-1})**	**Notes**
Alkyl C-H Stretch	2950 - 2850 (m or s)	Alkane C-H bonds are fairly ubiquitous and therefore usually less useful in determining structure.
Alkenyl C-H Stretch Alkenyl C=C Stretch	3100 - 3010 (m) 1680 - 1620 (v)	Absorption peaks above 3000 cm^{-1} are frequently diagnostic of unsaturation
Alkynyl C-H Stretch Alkynyl C=C Stretch	~3300 (s) 2260 - 2100 (v)	
Aromatic C-H Stretch Aromatic C-H Bending Aromatic C=C Bending	~3030 (v) 860 - 680 (s) 1700 - 1500 (m,m)	

Alcohol/Phenol O-H Stretch	3550 - 3200 (broad, s)	
Carboxylic Acid O-H Stretch	3500 - 3300 (broad, v)	
Amine N-H Stretch	3000 - 2300 (m)	Primary amines produce two N-H stretch absorptions, secondary amides only one, and tertiary none.
Nitrile C=N Stretch	2260 - 2220 (m)	
Aldehyde C=O Stretch Ketone C=O Stretch Ester C=O Stretch Amide C=O Stretch Carboxylic Acid C=O Stretch	1740 - 1690 (s) 1750 - 1680 (s) 1750 - 1735 (s) 1780 - 1710 (s) 1690 - 1760 (s)	The carbonyl stretching absorption is one of the strongest IR absorptions, and is very useful in structure determination as one can determine both the number of carbonyl groups (assuming peaks do not overlap) but also an estimation of which types.

4.5 Analysis results of FT-IR

Figure 4.3 shows the result of FT-IR for the first sample. From the FT-IR graph, two types of peak appear. For the first peak is peak for C=O at wavelength 1635.60 cm^{-1} and for the second peak is peak for O-H at wavelength 3361.90. The graph shows the carboxylic acid has a strong wide band for the O-H stretch. Normally, for O-H stretch around 2500 – 3300 cm^{-1}. For O-H stretch band of carboxylic acid is so broad because carboxylic acid usually exists as hydrogen bonded dimmers.

For C=O stretch of a carboxylic acid normally is around 1760-1690 cm^{-1}. The exact position of this broad band depends on whether the carboxylic acid is saturated or unsaturated, dimerized, or has internal hydrogen bonding

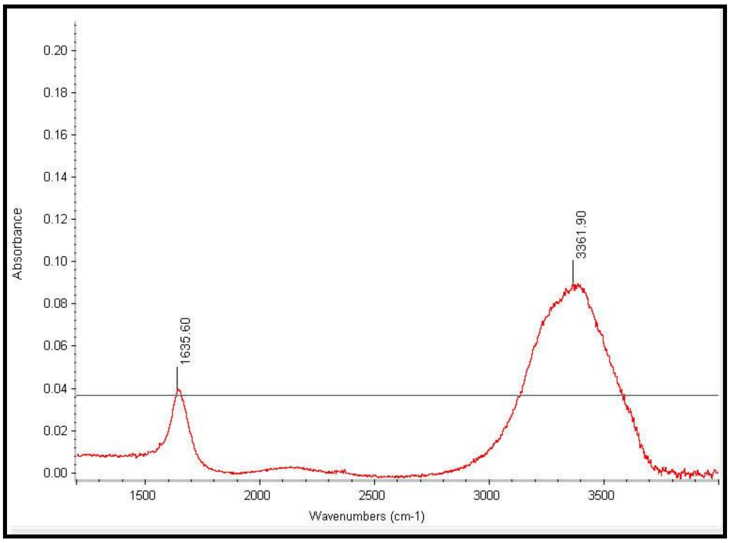

Figure 4.3: FT-IR for sample 1

Figure 4.4 shows the result of FT-IR for the second sample. From the FT-IR graph, two types of peak appear. For the first peak is peak for C=O at wavelength 1646.69 cm^{-1} and for the second peak is peak for O-H at wavelength 3395.42 cm^{-1} The graph shows the carboxylic acid has a strong wide band for the O-H stretch. Normally, for O-H stretch around 2500 – 3300 cm^{-1}. For O-H stretch band of carboxylic acid is so broad because carboxylic acid usually exists as hydrogen bonded dimmers.

For C=O stretch of a carboxylic acid normally is around 1760-1690 cm^{-1}. The exact position of this broad band depends on whether the carboxylic acid is saturated or unsaturated, dimerized, or has internal hydrogen bonding.

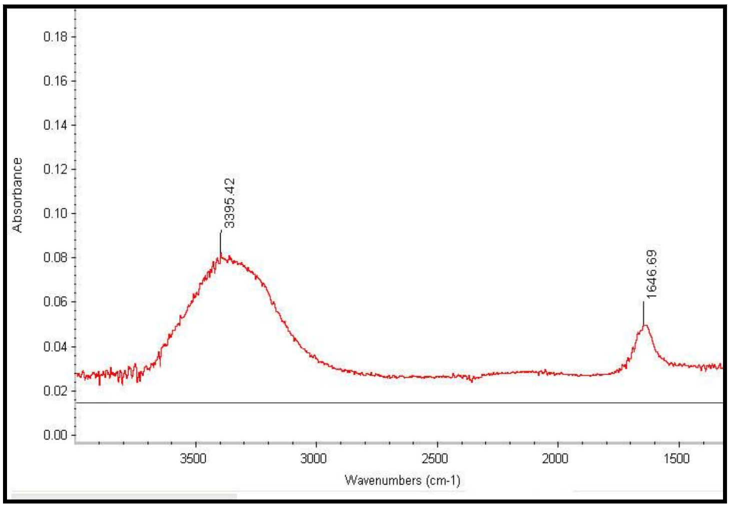

Figure 4.4: FT-IR for sample 2

Figure 4.5 shows the result of FT-IR for the third sample. From the FT-IR graph, two types of peak appear. For the first peak is peak for C=O at wavelength 1650.62 cm^{-1} and for the second peak is peak for O-H at wavelength 3401.29 cm^{-1} The graph shows the carboxylic acid has a strong wide band for the O-H stretch. Normally, for O-H stretch around 2500 – 3300 cm^{-1}. For O-H stretch band of carboxylic acid is so broad because carboxylic acid usually exists as hydrogen bonded dimmers.

For C=O stretch of a carboxylic acid normally is around 1760-1690 cm^{-1}. The exact position of this broad band depends on whether the carboxylic acid is saturated or unsaturated, dimerized, or has internal hydrogen bonding

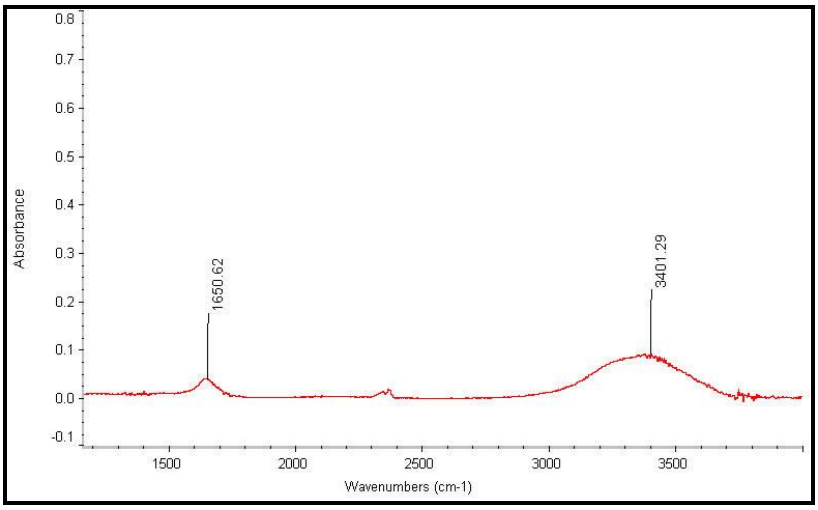

Figure 4.5: FT-IR for sample 3

Figure 4.6 shows the result of FT-IR for the fourth sample. From the FT-IR graph, two types of peak appear. For the first peak is peak for C=O at wavelength 1655.05 cm^{-1} and for the second peak is peak for O-H at wavelength 3357.09 cm^{-1} The graph shows the carboxylic acid has a strong wide band for the O-H stretch. Normally, for O-H stretch around 2500 – 3300 cm^{-1}. For O-H stretch band of carboxylic acid is so broad because carboxylic acid usually exists as hydrogen bonded dimmers.

For C=O stretch of a carboxylic acid normally is around 1760-1690 cm^{-1}. The exact position of this broad band depends on whether the carboxylic acid is saturated or unsaturated, dimerized, or has internal hydrogen bonding.

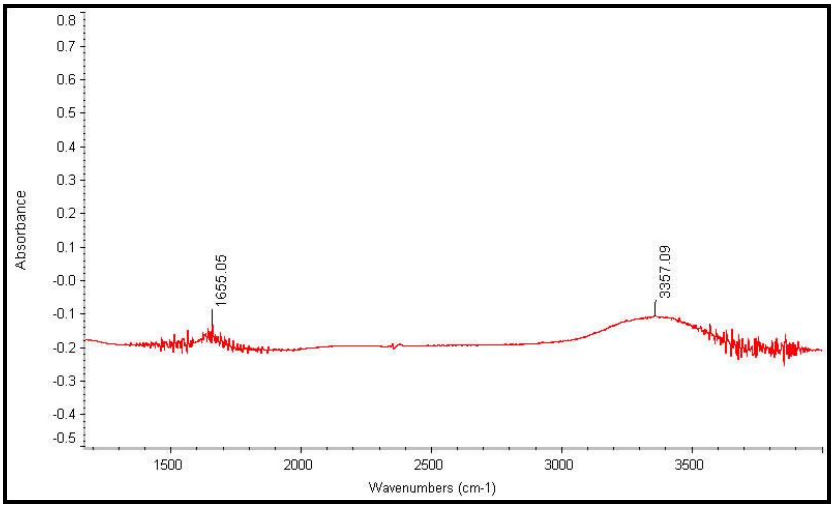

Figure 4.6: FT-IR for sample 4

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusions

Back to the objective of this research, the experiment was carry out to see whether can produce carboxylic acid using photosynthesis catalysis method After all the research have been done, and to prove the theory and new founded for those problem. The conclusions are:

I. Photosynthesis Catalysis Method is one of the methods which can be used to produce carboxylic acid. This was proven from the analysis of the product using Fourier Transform Infrared (FT-IR).

II. Flow rate of carbon dioxide will increase the acidity of carboxylic acid.

III. The objective of this research which was to study the synthesis of carboxylic acid production has been successfully achieved.

5.2 Recommendation

From this research, some recommendation can be made to improve the result of the analysis. The recommendations are:

I. Increase the reaction time for 3 until 4 hour to make the reaction more effectively.

II. Research must be conduct in close system. This reason is to make the reaction more effectively and efficient in order to produce of carboxylic acid.

III. Increase the flow rate of gas carbon dioxide (CO_2) to make the reaction faster.

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APPENDIX A

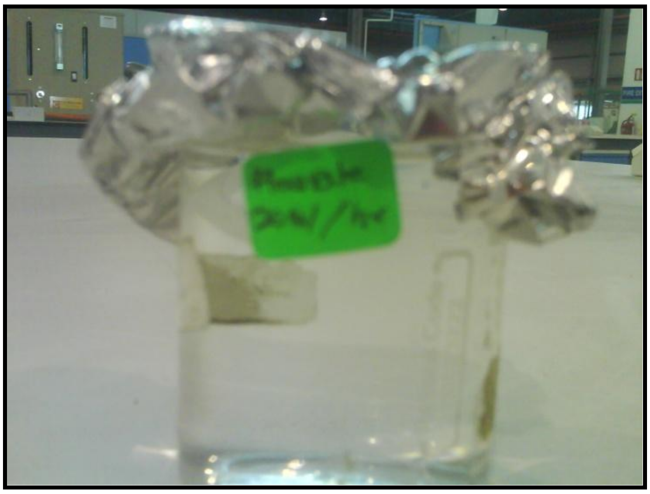
Pictures

Picture 1: Calcium Carbonate

Picture 2: Sample 1

Picture 3: Sample 2

Picture 4: Sample 3



Picture 5: Sample 4

Picture 6: High Purity Carbon Dioxide

Picture 7: Flow meter

Picture 8: FT-IR

APPENDIX B

Fourier Transform Infrared (FT-IR) Results

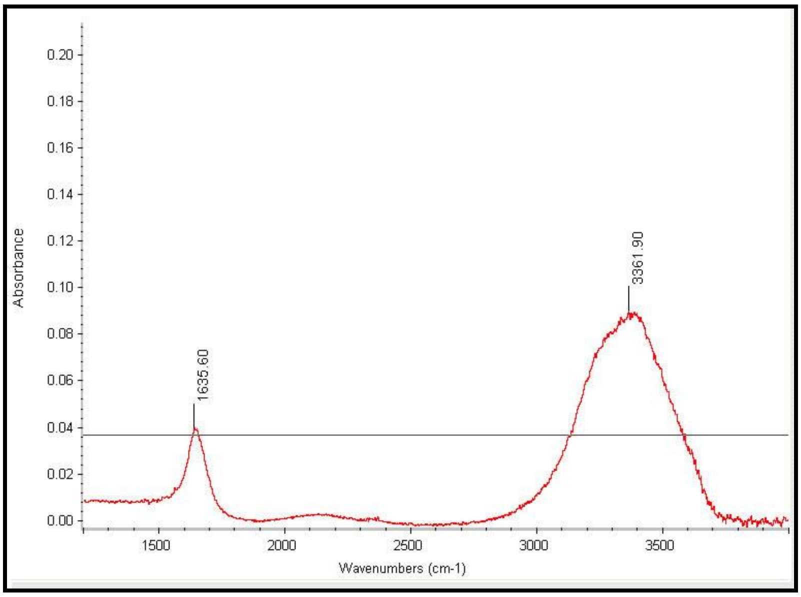


Figure 1 FT-IR results 1

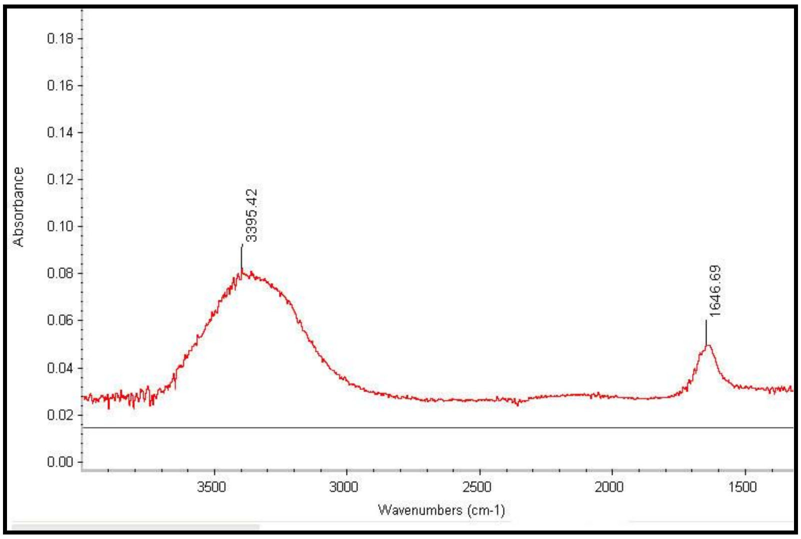


Figure 2: FT-IR results 2

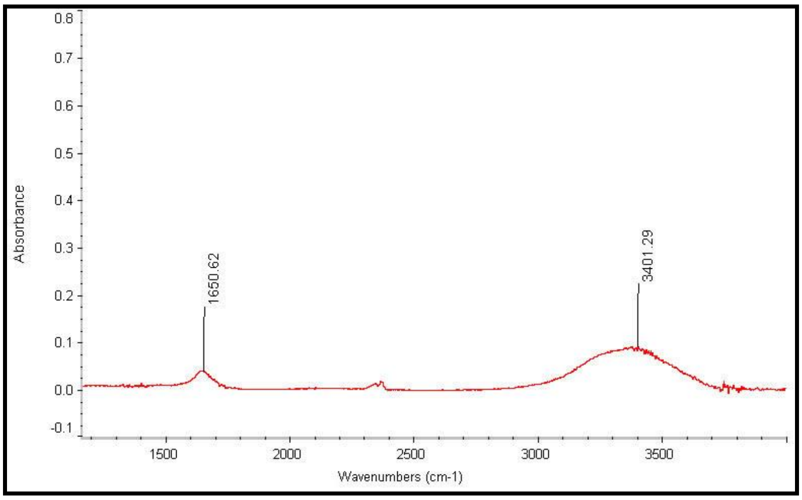


Figure 3: FT-IR results 3

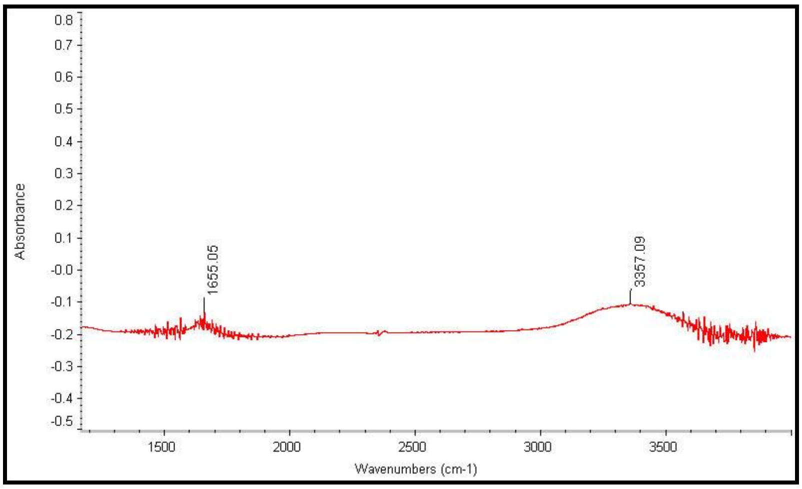


Figure 4: FT-IR results 4