The Effect of Ultrasonic Treatment on the Formation of Zeolite-T and Its CO_2 Adsorption Characteristic

by

St. Antikira Novichaka

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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BACHELOR OF ENGINEERING (Hons)
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Approved by,

_____

(Dr. Yeong Yin Fong)

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TRONOH, PERAK
May 2013

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.

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ST. ANTIKIRA NOVICHAKA

ABSTRACT

Carbon dioxide is the fourth most abundant gas in the atmosphere which contributes to global warming. It has become necessary to find a way for CO_2 management. In addition, removal of carbon dioxide in natural gas stream is an essential process because it can reduce the energy content and heating value of the gas and also corrodes pipelines and storage system. Some of the strategies have been developed to overcome this problem. As one of those technologies, adsorption is the most preferable methods used due to its energy and cost efficiency. Research presented that T-type zeolite has great potential in CO_2 adsorption as it has high selectivity at either very low pressure or concentration. However, the synthesizing process requires long duration in crystallization step. Besides, the reduction of particle size is also expected in order to enhance the performance of CO_2 adsorption. Thus, this project focuses on synthesizing of zeolite-T by using ultrasonic pretreatment prior to hydrothermal method. The synthesis parameters including ultrasonic pre-treatment time and crytallization duration were studied. The influence of those parameters on the particle size and CO_2 adsorption capability were investigated. The resulting zeolite-T were characterized using several characterization methods such as X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and Fourier transfer infrared spectroscopy (FTIR). Also, the CO_2 adsorption capacity of the resulting zeolite-T were determined.

ACKNOWLEDGEMENT

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ABBREVIATIONS AND NOMENCLATURES

CO_2	Carbon Dioxide
GHG	Green House Gases
OFF	Offretite
ERI	Erionite
SEM	Scanning Electron Microscopy
XRD	X-ray Diffraction
FTIR	Fourier Transform Infrared Spectroscopy
$NaAlO_2$	Sodium Aluminate
NaOH	Sodium Hydroxide
KOH	Potassium Hydroxide
SiO_2	Precipitated Silica
IR	Infrared

CHAPTER 1

INTRODUCTION

1. INTRODUCTION

1.1 Background Study

The increased use of fossil fuels during the past 200 years contributes to the steady rise of carbon dioxide (CO_2) level in the atmosphere, the most prominent major greenhouse gas that contributes significantly to global warming (Bonenfant, Kharoune, Niquette, Mimeault, & Hausler, 2008). Some of the strategies to decline rate of emitting CO_2 to atmosphere and establish processes to recover and convert CO_2 to valuable products have been studied (Galhotra, Navea, Larsen, & Grassian, 2009). Carbon dioxide which is produced largely in petrochemical processes corrodes pipelines and storage systems within the transportation, due to its acidic property in the presence of water (Mirfendereski et al. 2008). Furthermore, the reduction of the heat and energy content of natural gas has made the separation of CO_2 from methane in natural gas purification as an important and critical process (Rad et al. 2012).

The techniques used to decrease the emission of carbon dioxide including minimizing emission from vehicles and industries, using alternative sources of energy (wind energy, tidal energy, and solar energy), gas capture and adsorption, and membrane technologies (Galhotra et al., 2009) have been widely reported. In comparison to other techniques, adsorption has the potential for removal of CO_2 by considering the high adsorption capacity and selectivity of adsorbents existed which include activated carbon, charcoal, activated alumina, silica gel, activated clay, and crystalline aluminosilicate zeolites (Shimekit and Mukhtar, 2012).

Zeolites have been reported as potential materials for adsorption process (Cejka et al. 2007). Zeolites are crystalline, porous aluminosilicates materials which are extensively used in catalysis, chemical separations and as adsorbents (Mirfendereski and

Mohammadi, 2011). Zeolites are rapidly becoming one of the promising materials in petrochemical industries for CO_2. Zeolites are capable to separate molecules based on the difference in molecular size and shape due to their well-defined pore size. Referring to the reported research work (Mirfendereski et al. 2008), zeolite types Y, X, SAPO-34 are some of the examples of zeolites that have been studied as an adsorbent to remove CO_2. However, there are limited information reported on synthesis and characterization of zeolite-T.

Zeolite T is an aluminosilicate molecular sieve which was initially introduced by Bennet and Gard (Rad, et al. 2012). Zeolite T is an intergrowth crystalline structure of offretite (OFF) and erionite (ERI) zeolite structure with the pore size of 0.36 nm x 0.51 nm (Liu et al. 2009). The pore size of zeolite-T is comparable to the kinetic diameter of CO_2 molecules which will lead to high adsorption coverage of CO_2 in zeolitic pores. Furthermore, T-type zeolite is a potential adsorbent in CO_2 adsorption process because it can adsorb relatively large quantities of adsorbate at either very low pressure or concentrations (Breck et al. 1958).

The purpose of this research is to synthesize and characterize zeolite-T as an appropriate adsorbent for CO_2 removal. Study of the synthesis parameters such as ultrasonic pretreatment and time duration during the synthesis process is performed in order to justify their significance effects on the changes of zeolite-T particle size as well as CO_2 adsorption capability. In this present work, the effect of ultrasound pre-treatment on synthesis of zeolite-T is firstly studied. The synthesized zeolite-T crystals will be characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier transfer infrared spectroscopy (FTIR). In addition, physisorption analyzer is used to investigate the CO_2 adsorption characteristics of zeolite-T.

1.2 Problem Statement

In recent years, research interest has been focused on the removal of carbon dioxide (CO_2). Conventionally, chemical and physical absorption, low temperature distillation (cryogenic method), separation through a membrane and adsorption on solid are the methods used for CO_2 removal in petrochemical industries. Despite the availability of the above mentioned processes for separation of carbon dioxide in gas stream, the adsorption process still remains attractive with a great potential due to its easy and economic method (Rad et al. 2012). The treatment of natural gas stream by adsorption with T type-zeolite molecular sieves is considered to be a promising method for separation of CO_2 because of its high stability toward acid solvents and high thermal conditions (Cichocki and Koscielniak, 1999).

In spite of this advantage, there are remaining issues need to overcome so that high quality materials of zeolite-T with high surface area for CO_2 adsorption can be produced. As reported from the literature, the crystallization time of zeolite-T is relatively time consuming as compared to the other types of zeolites (Rad et al. 2012, Musyoka et al. 2011). Hence, the reduction time is required. Besides that, the study on the effect of particles size of zeolite-T on the CO_2 adsorption capacity is remaining a new and challenging task.

1.3 Objective and Scope of Study

The objectives of this research are as follow:

a) To investigate the effect of ultrasonic treatment on the crystallization time of zeolite-T.
b) To characterize the resultant zeolite-T using different analytical tools such as X-Ray Diffraction (XRD), Scanning Electronic Microscopy (SEM), and Fourier Transform Infrared Spectroscopy (FTIR).
c) To study the CO_2 adsorption characteristic of the synthesized zeolite-T.

The research is done in order to study the feasibility of zeolite-T as an adsorbent for CO_2 removal as well as to investigate the CO_2 adsorption capability of zeolite-T as compared to the other type of zeolite.

1.4 Relevancy and Feasibility of The Project

The relevancy of this project in shortening the synthesis duration is to obtain a feasible method and reduce the crystallization time for synthesis of zeolite-T. The synthesis parameters, such as synthesis duration and the ultrasonic pre-treatment time will be varied in order to reduce the synthesis duration and particle size of Zeolite-T. The influence of those parameters on the particle size and CO_2 adsorption capability will be investigated. The characterization tests will be carried out to study the changes of properties of the resulting particles. Subsequently, the CO_2 adsorption characteristics of zeolite-T will be tested and the effect of particle size on CO_2 adsorption will be discussed. Eventually, this project can be completed as per scope before the date of completion by proper planning and implementation.

CHAPTER 2

LITERATURE REVIEW

2. LITERATURE REVIEW

2.1 Removal of Carbon Dioxide

Carbon dioxide (CO_2) is ranked the fourth most abundant gas in the atmosphere with a concentration of about 385 ppm (Galhotra et al., 2009). 40% increment of CO_2 concentration has been detected since the Industrial Revolution and is constantly increasing (Galhotra et al., 2009). Carbon dioxide is the most prominent greenhouse gases and its accumulation in earth's atmosphere contributes significantly to global warming issues. It has become necessary to minimize the emission of carbon dioxide to overcome the negative impacts in nature and society.

Carbon dioxide removal is a great interest among the researchers due to the global implications related to climate change, sustainability, and energy. In natural gas purification, selective removal of carbon dioxide is an important and critical process due to its acidity properties. Dortmund et al. (1999) reported that CO_2 removal is subjected to the rise of energy content and heating value of the natural gas. In addition, the removal of carbon dioxide will be beneficial to minimize the volume of gas to be transported in pipelines and to be stored in a storage column. This process will lead to the reduction of corrosion level in the pipelines and storage system within the gas distribution network (Delgado et al. 2007). In the other hand, CO_2 must be removed to avoid freezing in the low-temperature chillers in LNG plants (Dortmund et al., 1999). Eventually, the decrease of CO_2 in petrochemical plant gas stream will prevent the atmospheric pollution by reducing the rate of greenhouse gases emission.

2.2 The Existing Technologies in CO_2 Removal

The process of capturing CO_2 efficiently is difficult to perform. Therefore, intensive research efforts have been made in recent years to develop methods for recovering the CO_2 emitted from industrial gas streams and for storing the recovered CO_2 without discharging it into the atmosphere.

The main CO_2 separation technologies from gas stream include chemical and physical absorption, low temperature distillation (cryogenic method), separation through a membrane and adsorption on solid (Shimekit and Mukhtar, 2012). The technology most widely practiced for carbon dioxide removal is amine adsorption, but due to the high cost of maintenance caused by the high equipment corrosion rate and large amount of energy consumption during high temperature absorbent regeneration, amine plants are costly and complex. In the other hand, the polymeric membranes technology which is commercially available for the separation of CO_2 still need further development for more selective higher-flux, and cost effective membranes in order to fully exploit the use of membranes in natural gas purification (Rufford et al., 2012). For cryogenic separation, there is a need to put more concern on highly energy intensive for regeneration which significantly reduces the overall plant efficiency and also a high tendency for blockage of process equipment (Ebenezer and Gudmunsson, 2006). Meanwhile, the adsorption process has shown its potential to be an alternative method for CO_2 capture purpose because of low energy requirements and low capital investment costs (Delgado, 2007).

In recent years, attention has been focused on the adsorption processes for separation of carbon dioxide. Hence, it is growing need to prepare an adequate adsorbent with high adsorption capacity and selectivity. However, the affinity to CO_2 molecule should not be too high, because it will compromise the regeneration of the adsorbent (Shimekit and Mukhtar, 2012). Besides, the cost of the adsorbent must be as low as possible.

2.3 Zeolite : T-Type

The development of new advanced adsorbents for separation of CO_2 in industrial applications via adsorption processes is receiving great attention. Among the potential routes explored for gas separations, adsorption processes involving zeolites as the adsorbents have shown an increasing importance due to their unique properties (Cejka et al., 2007). Zeolites are a class of microporous crystalline aluminosilicate materials where the aluminum and silicon atoms are present in the form of AlO_4 and SiO_4 tetrahedral (Thompson, 1998). These types of materials which have molecular sieve properties are potential for gas separation processes because of the small pore size and their narrow pore size distribution.

The selective adsorption by zeolites is influenced by the size and shape of molecules (Breck et al., 1958). There are many types of zeolites which have different size and shape of molecules. T type zeolite is one of those which belong to an intergrowth-type zeolite of erionite (ERI) and offretite (OFF) (Liu et al., 2009). This type of zeolites has reported to have high stability towards acid solvents and high thermal conditions (Cichocki and Koscielniak, 1999). Hence, zeolite T has been investigated and used as the adsorbent for gas separation processes.

Zeolite-T has an effective pore size of 0.36 nm x 0.51 nm which is comparable to carbon dioxide with a kinetic diameter of 0.33 nm (Rad et al., 2012). This will lead to high adsorption coverage of CO_2 in zeolitic pores due to the affinity of CO_2 molecules to zeolite T. Unlike common adsorbents, zeolite T exhibits selectivity based on the size, degree of unsaturation, shape, polarity, and polarizability of the adsorbate molecule (Breck et al., 1958). Another property of zeolite T which contributes to its usefulness is that of adsorbing relatively large quantities of adsorbate at either very low pressure or concentration (Breck et al., 1958). The novel material of this type of zeolite can therefore be utilized as a selective adsorbent for CO_2 removal in gas separation.

2.4 Synthesis of Zeolite T

Conventional hydrothermal heating is the common method used for synthesizing of zeolite-T (Zhou et al., 2009). Hydrothermal synthesis is a promising method to obtain microporous crystalline particles. Aneesh et al. (2007) reported that the hydrothermal process is efficient to achieve the crystalline phase at low temperatures and easy to control the particle size and crystallinity. The particle properties such as morphology and size can be controlled via the hydrothermal process by adjusting the reaction temperature and time. In addition, the particles prepared through hydrothermal synthesis are expected to have large surface area, smaller crystallite size, and higher stability (Sayilkan et al., 2007).

However, in conventional hydrothermal heating method, the escalation in temperature is comparatively slow and less uniform due to the presence of a temperature gradient. Microwave heating has been reported as an alternative way to substitute the conventional heating (Abrishamkar et al., 2010). The application of this method can minimize the crystallization time and enhance the crystallinity of the ultimate product compared with those acquired by conventional heating. Microwave heating method has been reported as a promising method to shorten the lengthy synthesis time of zeolite-T.

Lately, there has been a fast growth in the usage of unconventional hydrothermal method by introducing sonochemical method (Abrishamkar et al., 2010). Sonochemical method is the use of power ultrasound to increase chemical reactivity. Ultrasound can enhance and change dissoulution processes, chemical reactions, nucleation and growth of precipitates (Wang et al., 2008). Musyoka et al. (2011) has reported that the application of ultrasound reduce the crystallization time instead of normal stirring prior to the hydrothermal crystallization step. Despite the enhancement of heating method in synthesizing of zeolite-T, there is still a remaining issue to find the optimum parameters in achieving desirable porosity of the particle size. Thus, the synthesis parameters such as temperature and duration time for crystallization process have to be taken into consideration.

CHAPTER 3

METHODOLOGY

3. METHODOLOGY

3.1 Research Methodology

Figure 1 shows the schematic diagram of the research flow in the present project. The research was started with synthesis of zeolite-T using hydrothermal method. Then, the synthesis parameters i.e. ultrasonic duration and crystallization time were varied. The synthesis process was repeated by varying the synthesis parameters which produced different particle size of zeolite-T. After that, characterizations tests were conducted with purpose to study the structure and the morphology of the zeolite-T. XRD, SEM, and FTIR were used to characterize the resulting zeolite-T particles. Eventually, the particles were examined for their adsorption capacity in CO_2 using BELSORP in order to study the effect of particles size on CO2 adsorption.

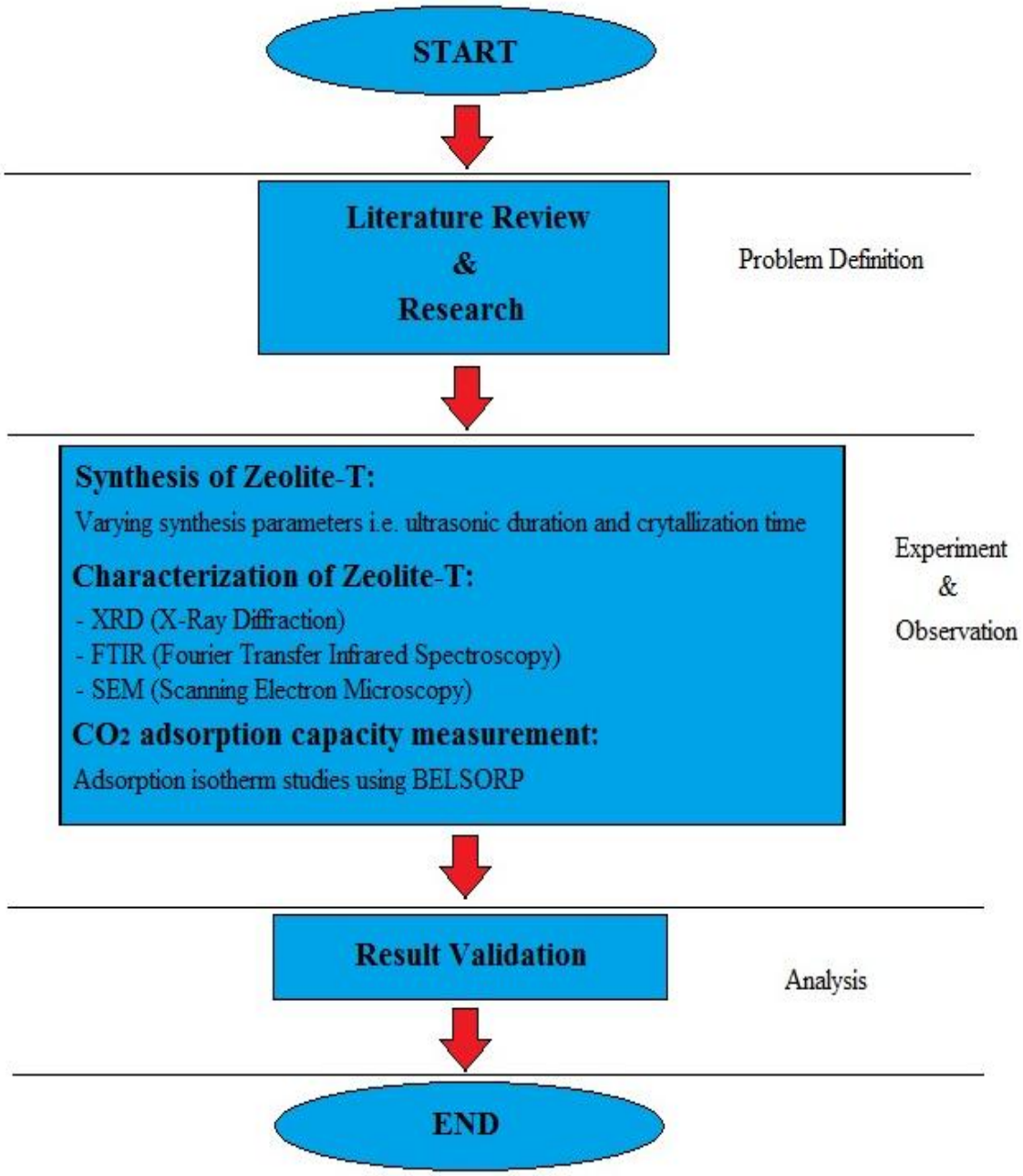


Figure 1 Research Methodology

3.2 Experimental Procedures

3.2.1 Equipment and Chemicals

The chemicals were used as received without further purification. The equipment and chemicals required for conducting the experiments are shown in Table 1.

Table 1 List of Chemicals and Apparatus

Chemicals	**Description**
Sodium Aluminate, $NaAlO_2$	Powder, Na as (Na_2O) 41% + Al as (Al_2O_3) 54%, BDH Chemicals
Sodium Hydroxide, NaOH	Pellet, 99% wt., Merck Chemicals
Potassium Hydroxide, KOH	Pellet, 85% wt., Merck Chemicals
Precipitated Silica, SiO_2	Powder, 99.8% wt., Aldrich Chemicals
Deionized Water	Aqueous, available in the lab
Equipment	**Description**
Bottle	As a container for preparing the chemicals
Stirrer	Used for preparing the aluminosilicate gel
Ultrasonic Bath	Used for the ultrasonic treatment during modification process
Teflon-lined autoclave reactor	Used for the hydrothermal growth synthesis
Oven (Memmert Isotemp Oven)	Used for the hydrothermal growth of zeolite-T and drying the synthesized crystals

3.2.2 Synthesis of Zeolite-T

Figure 2 shows the steps taken in precipitation method to synthesize zeolite-T.

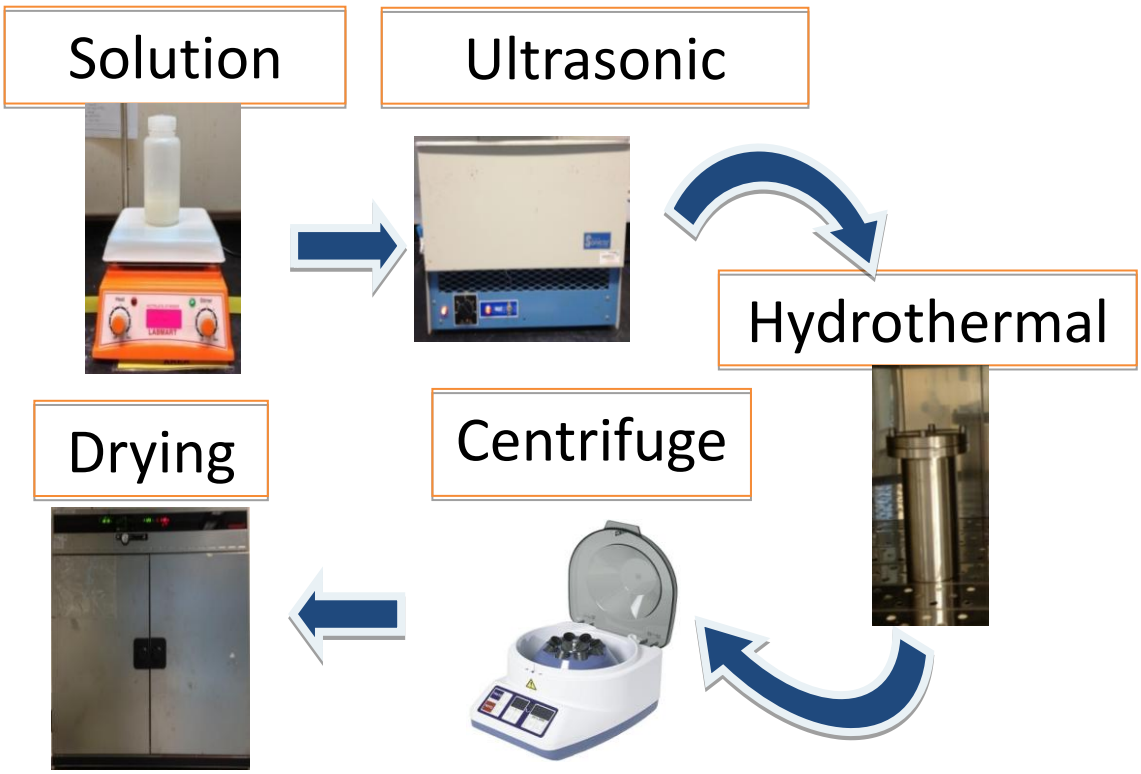


Figure 2 Flow chart of synthesizing zeolite-T

4.64 g of sodium hydroxide and 3.45 g of potassium hydroxide were dissolved in 71 g deionized water under stirring. After reached to ambient temperature, 2.71 g of sodium aluminate was added gradually to the solution and continuously stirred for 1 hour until the clear solution was achieved. Under slow stirring, 18.20 g of precipitated silica was added into the solution. This step took long time because the precipitated silica was very slightly dissolved in the aluminate solution. A homogeneous gel was formed after about 3 hours stirring. Then the gel was aged at ambient temperature for 24 hours. The obtained gel was placed in a Teflon-line autoclave reactor. The autoclave reactor was kept in an oven at 120 ^{o}C for 7 days to synthesis the zeolite crystals. During the modification process, ultrasonic treatment was done prior to the hydrothermal growth in

the oven. The duration for the treatment was 30 minutes. Also, the synthesis duration was varied from 7 days, 5 days, 4 days and 3 days. After the synthesis, the reactor was taken out from the oven. Then the crystals were centrifuged and washed with distilled water. This step was repeated three times. The zeolite-T crystals were dried in an oven at 120 ^{o}C for 2 h. The produced solid crystals were stored in dessicator prior to the characterization study and adsorption test.

3.3 Characterization Studies

3.3.1 X-Ray Diffraction (XRD)

XRD is a rapid analytical technique primarily used for phase identification of a crystalline material. X-rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor. The information that can be obtained through XRD includes characterization of crystalline materials and measurement of sample purity.

3.3.2 Fourier Transformed Infrared (FTIR)

FTIR is widely used to determine the structure of organic compound. Infrared can identify the presence of certain functional groups within a molecule. Most FTIR units operate within the range of 400-4000 cm^{-1} wave number. The wave numbers are directly proportional to energy. Functional group tends to adsorb a photon of infrared radiation. Thus, the energy increase and it is associated with bond vibration involving in the stretching and bending of bonds. FTIR analysis can be applied to small quantities of materials. The equipment used in this study was Perkin Elmer (1600 series) spectometer using the KBr wafer technique.

3.3.3 Scanning Electron Miscroscopy (SEM)

SEM creates the magnified images by using electron. SEM shows 3-dimensional images at high magnifications. The images are very detailed. The images created without light waves rendered black and white. It can be used to estimate the particle size of zeolite-T quantitatively.

3.4 CO_2 Adsorption Test

Carbon dioxide (CO_2) adsorption on zeolite-T was analyzed using physisorption analyzer, BELSORP. BELSORP is designed to identify adsorption isotherm for surface area and pore size distribution as well as CO2 adsorption capacity. Therefore, the CO_2 adsorption results can be determined and the amount of CO2 adsorbed was compared with those results reported in the literature.

CHAPTER 4

RESULTS AND DISCUSSION

4. RESULTS AND DISCUSSION

4.1 Yield of Zeolite-T

In this project, a comprehensive study towards synthesis and characterization of zeolite-T for CO_2 removal has been carried out. This chapter presents the results and discussion for main aspects of the research work. Zeolite-T was synthesized by using hydrothermal growth method with and without the ultrasound pre-treatment at different crystallization time. The ultrasonic treatment was believed to reduce the crystallization time in the process of synthesize zeolite T as it has been proven for other type of zeolite (zeolite-A) (Andaç, Tatlıer, Sirkecioğlu, Ece, & Erdem-Şenatalar, 2005). Figure 3 shows the zeolite-T produced in the present research. The characterization and adsorption results obtained were compared with the those results reported for zeolite-T from the literature (Zhou et al., 2009, Mirfendereski and Mohammadi 2011).

Figure 3 synthesized zeolite-T

4.2 Characterization of Zeolite-T

4.2.1 XRD (X-Ray Diffraction)

As the main phase of zeolite-T is offretite, the peaks of zeolite-T are assigned according to the standard XRD pattern of offretite. Table 2 summarizes the experimental conditions and the XRD results for the zeolite-T samples synthesized in the present work.

Table 2 Experimental conditions and XRD results for the zeolite T synthesized in the present work

Sample	Crystallization Temperature (^{o}C)	Ultrasonic Treatment Duration (minutes)	Crystallization Duration (day)	Product Phase (XRD)
S1	120	0	7	Zeolite T
S2	120	30	5	Zeolite T
S3	120	30	4	Zeolite T
S4	120	30	3	Zeolite T

Figure 4 shows the XRD patterns of zeolite-T crystals synthesized with or without ultrasonic treatment at different crystallization time. It was comparable to XRD pattern of commercialized zeolite-T as given in Figure 5 (Rad et al. 2012). It was observed that all the samples shows zeolite-T morphology with the peaks at $2\theta = 7.69$. These results show that by inducing ultrasonic pre-treatment, the crystallization time can be shortened while the structure of zeolite-T can be maintained with slightly decrease in crystallinity.

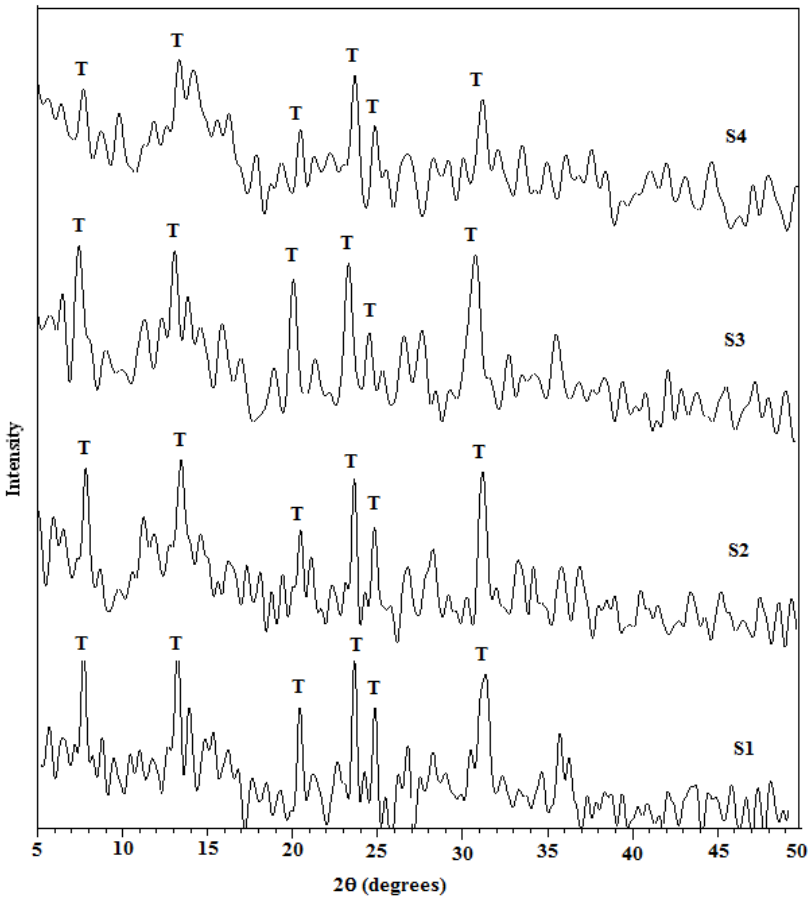


Figure 4 XRD patterns of the synthesized zeolite-T samples

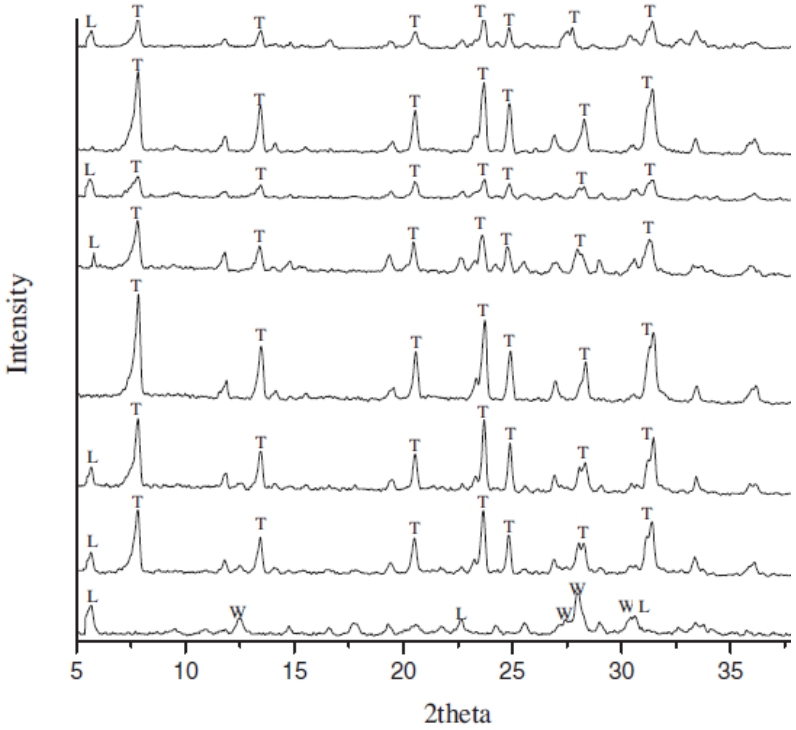


Figure 5 XRD patterns of zeolite-T (Rad et al. 2012)

4.2.2 FTIR (Fourier Transferred Infrared)

FTIR is an analytical method used to identify the presence of functional groups within a molecule. The IR spectra of the resultant zeolite-T are illustrated in Figure 6. The entire FT-IR spectra for all the samples are similar and show no obvious differences between each other. IR spectra for all samples show similar peaks at 430, 470, 575, 615, 720, 775, 1040, and 1050 cm^{-1}, which represent the major peaks for zeolite-T reported in the literature (S. Yang, N.P. Evmiridis, 1996).

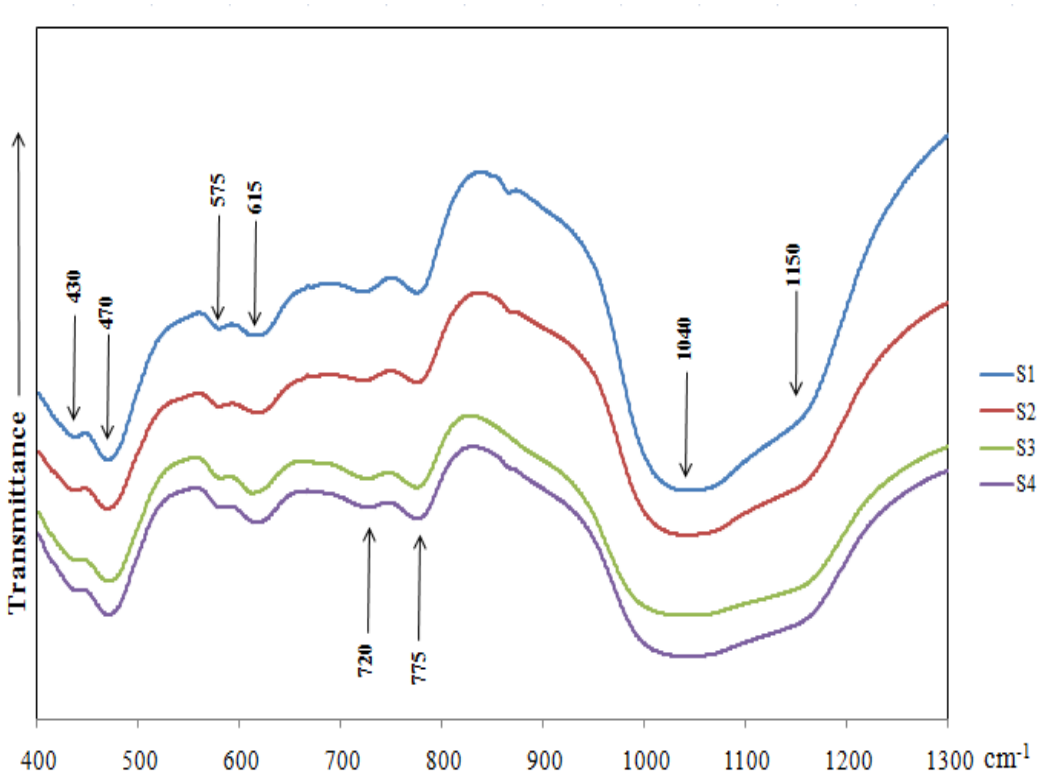


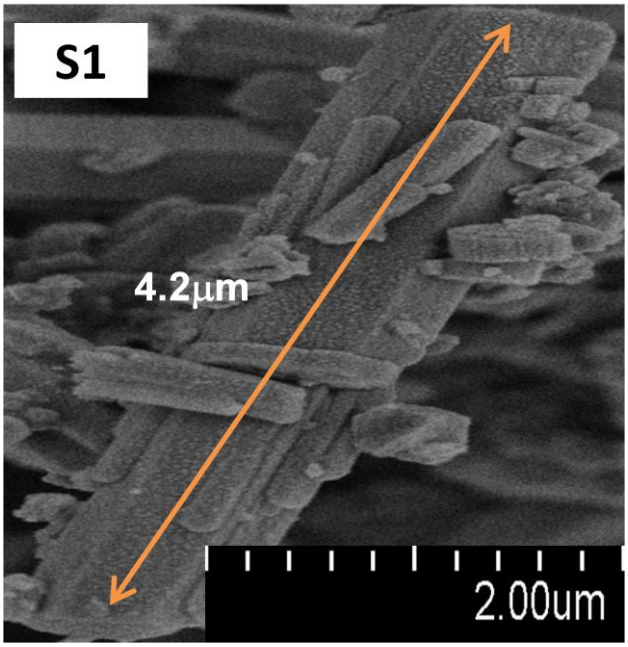
Figure 6 IR spectra of the Zeolite-T synthesized in the present work

From Figure 6, the peak around 430 cm^{-1} and 470 cm^{-1} corresponds to the T-O bonding in the zeolite-T topology. These spectra correspond to zeolite-T with offretite and erionite structure as reported in literature (S. Yang, N.P. Evmiridis, 1996). For peak around 575 cm^{-1} and 615 cm^{-1} is attributed to the secondary building units of single 6-ring.

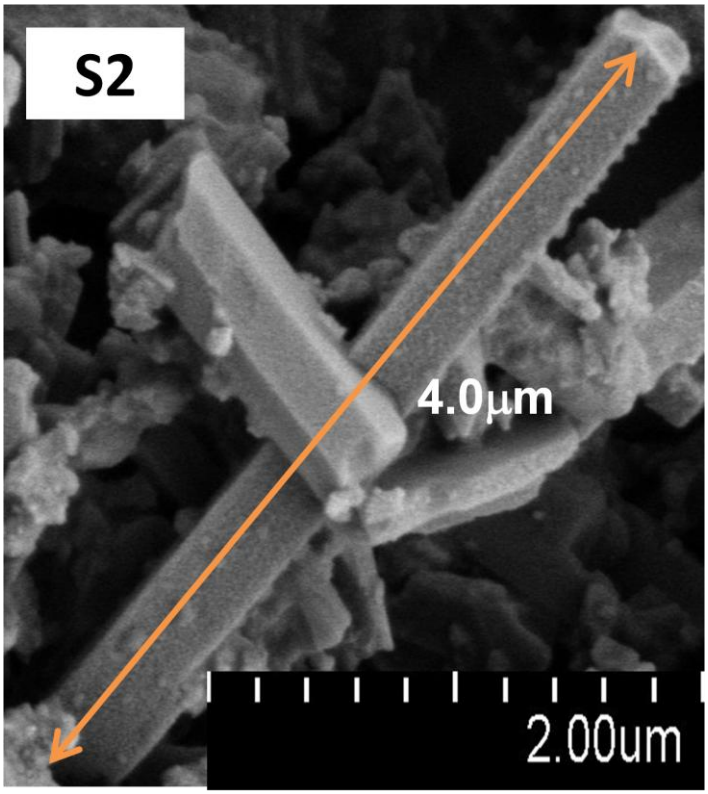
Meanwhile, peak around 720 cm^{-1} and 780 cm^{-1} is due to the symmetric stretching vibration of double ring and peak around 1040 cm^{-1} and 1150 cm^{-1} is because of the asymmetric stretching vibration. From these results, it can be concluded that all the samples synthesized in the present work showed zeolite-T framework.

4.2.3 Scanning Electron Microscopy (SEM)

The microstructure morphology of the resultants zeolite-T was observed by using Scanning Electron Microscopy (SEM). Figure 7 shows the surface morphology of synthesized zeolite-T. All the zeolite-T samples synthesized in the present work shows typical crystal morphology of zeolite-T reported in the literature (Rad et al. 2012) The SEM images confirm the formation of the rod like crystals of zeolite-T for all the samples. The structure is still maintained regardless of the reduction of crystallization time. Referring to Figure 7, the particle sizes of the zeolite-T synthesized at 7 days without ultrasonic pretreatment (S1) was 4.5 μm. It was found that the particle size of zeolite-T reduces to 3.2 μm , 3.1 μm and 2.6 μm for samples synthesized at 5 days (S2), 4 days (S3) and 3 days (S4), with ultrasonic pre-treatment, respectively. These results were consistent with the XRD results (Figure 4) whereas the slight reduction of crystallinity resulted in the decrease in particle size of the zeolite-T sample



(a)



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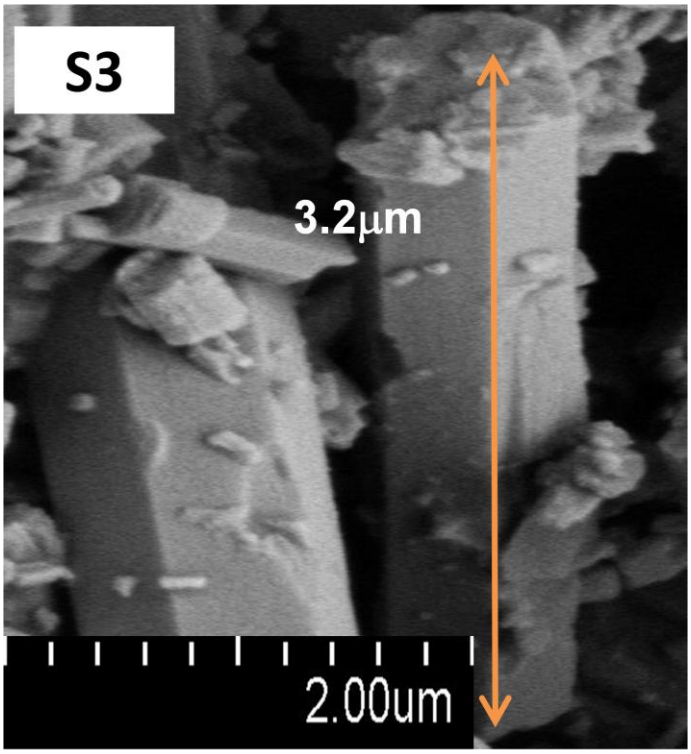

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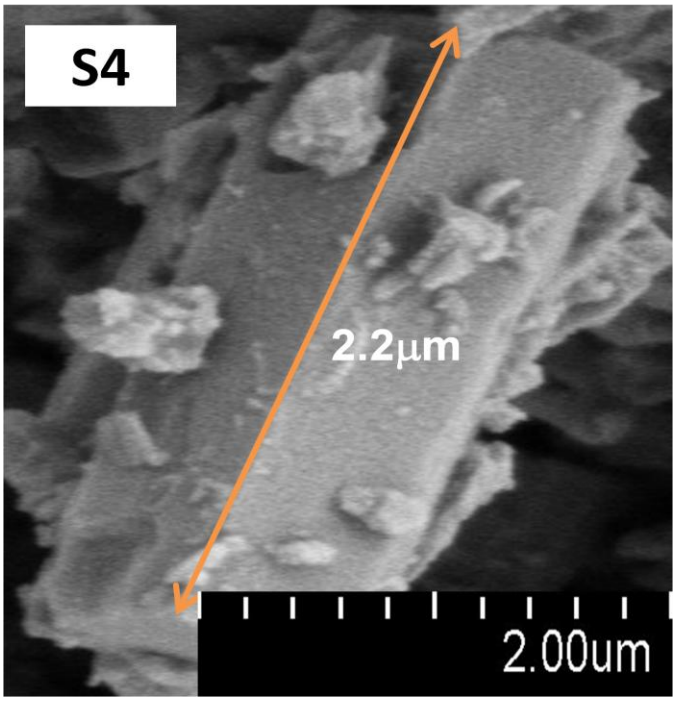

(d)

Figure 7 SEM images of Zeolite-T synthesized in the present work: (a) S1; (b) S2; (c) S3; (d) S4.

4.3 Adsorption Study

The single gas adsorption isotherms of CO_2 were obtained at constant temperatures of 298 K for pressures variable in range of 0.1 – 1 kPa. The isotherms of all the zeolite-T samples are displayed in Figure 8. The CO_2 adsorption capacity of zeolite-T for various condition crystallization parameters is presented in Table 4. It was observed that CO_2 adsorption capacity increases when crystallization time was shortened.

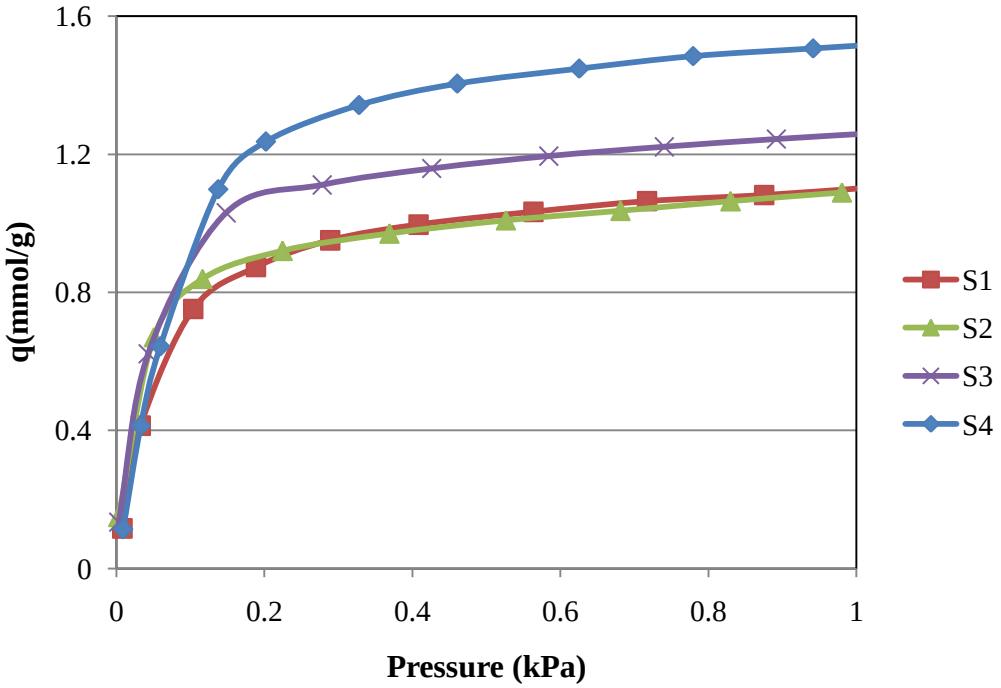

Figure 8 Adsorption of CO_2 for the all the zeolite-T samples

Table 3 CO_2 adsorption capacity of the prepared samples

Sample	CO_2 Adsorption (mmol/g)
S1	1.1039
S2	1.0894
S3	1.2634
S4	1.5628

From the results obtained, it has been proven that the ultrasonic treatment has an impact on the reduction of synthesis duration and particle size of the zeolite-T formed. This leads to the increment of CO_2 adsorption capacity which was mainly due to the increase in surface area.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5. CONCLUSION AND RECCOMENDATION

5.1 Conclusion

Zeolite-T has been synthesized within shorter crystallization time by inducing ultrasonic pre-treatment. From the results obtained, synthesized zeolite-T has shown better adsorbent characteristics and higher adsorption performance when the ultrasonic treatment was applied.

Results from XRD showed that zeolite-T was successfully synthesized for all samples with different synthesis duration. By inducing ultrasonic pre-treatment, the crystallization time can be reduced from 7 days down to 3 days. The FTIR results confirmed the IR spectra of the resultants zeolite-T over the range of 400-1300 cm^{-1}, whichwere similar to the characteristic absorbencies of the frameworks of zeolite-T reported in the literature. Meanwhile, the SEM images present the formation of the rod like crystals of zeolite-T for all the samples. The structure was still maintained regardless of the reduction of crystallization time. Furthermore, based on the BELSORP test, the ultrasonic treatment has an impact on the increment of CO_2 adsorption capacity due to the increase in surface area.

In this study, it was clearly proven that the synthesized zeolite-T was improved in terms of morphology and also the capability of adsorbing CO_2. It was because the particle size was reduced and thus created larger CO2 adsorption sites.

5.2 RECOMMENDATION

For future works, it is recommended that the duration of ultrasonic treatment is extended to study whether further reduction in synthesis duration could be achieved. It is also suggested to modify the synthesis parameters and other important factors that affecting the crystal properties of Zeolite-T, for instance, the synthesis temperature used in synthesizing of zeolite-T.

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