Korean Chem. Eng. Res., 59(2), 260-268 (2021)
https://doi.org/10.9713/kcer.2020.59.2.260
PISSN 0304-128X, EISSN 2233-9558

Comparative Study on Convective and Microwave-Assisted Heating of Zeolite-Monoethanolamine Adsorbent Impregnation Process for CO_2 Adsorption

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(Received 3 September 2020; Received in revised from 19 November 2020; Accepted 9 December 2020)

Abstract – Adsorption is the most promising technology used to adsorb CO_2 to reduce its concentration in the atmosphere due to its functional effectiveness. Various porous materials have been extensively synthesized to boost CO_2 adsorption efficiency, for example, zeolite. Here, we report the synthesis process of zeolite adsorbent impregnated with amine, combining the benefit of these two substances. We compared conventional heating with microwave-assisted heating by varying concentrations of monoethanolamine in methanol (10% v/v and 40% v/v) as a liquid solution. The results showed that monoethanolamine impregnation helps significantly increase adsorption capacity, where adsorption occurs as a physisorption and not as chemisorption due to the adsorbent's steric hindrance effect. The highest adsorption capacity of 0.3649 mmol CO_2 / gram adsorbent was reached by microwave exposure for 10 minutes. This work also reveals that a decrease in CO_2 adsorption capacity was observed at a longer exposure period, and it reached a constant 40-minute adsorption rate. Impregnating activated zeolite with 40% monoethanolamine for 10 minutes in addition to microwave exposure (0.8973 mmol CO_2 / gram adsorbent) is the maximum adsorption ability achieved.

Key words: Zeolite, Monoethanolamine, Adsorption, CO_2, Microwave

1. INTRODUCTION

The growing energy demand for both non-renewable and renewable energy resources is triggering an increasing amount of CO_2 accumulation in the atmosphere as a result of the combustion process. CO_2 gas in the atmosphere increased from 0.2 billion tons per year in 1990 to 50.9 gigatons of CO_2 equivalent coming from the combustion process of those energy resources [1]. Different measures have been taken into account to minimize CO_2, such as absorption using amine-based solvents [2] that are most commonly used in the chemical industry, including their development by combining them with deep-eutectic solvent [3], membrane separation [4-6], cryogenic distillation [7] and adsorption [8].

Unfortunately, the absorption process has certain drawbacks, such as the high cost of regenerating solvents, ranging from 70~80% of the total operating expenses [2, 9] to the leading cause of corrosion problems [10, 11]. Separation of CO_2 using membrane processing often requires high costs. This technology will spend \$50~\$78 per ton of CO_2, while a fee of \$40~\$63 per ton of CO_2 is expected for the adsorption process [12, 13]. The adsorption method has recently attracted attention as a promising technological approach to CO_2 capture [14, 15]. It provides low energy regeneration needs, no liquid waste, and ease of use [16, 17].

Among the development of solid adsorbent over the last few years, zeolite is an adsorbent suitable for CO_2 adsorption, as it has a high surface area, strong thermal and mechanical stability and is abundantly available [18]. Nevertheless, at elevated temperature zeolite has very poor CO_2 selectivity and decreased CO_2 capture ability [19,20]. Impregnation with amine-based solvent may resolve this issue. Some researchers have successfully performed the mixing process of various amine solvents in zeolite to improve the selectivity of CO_2 [21-23]. This solid-amine adsorbent provides lower energy consumption for regeneration. It weakens the corrosion rate of the equipment compared to the alcohol amine absorption process along with its higher CO_2 adsorption and desorption rates. Additionally, the presence of amine species in porous silica gives high affinity towards CO_2 molecule [24]. Monoethanolamine (MEA) is most commonly used for CO_2 absorption in industrial applications due to its ability to react quickly and more affordable price [2].

Some previous studies have shown that the impregnation process using an amine-based solvent with the help of convective heating requires a more extended heating period of up to 3 hours at 150 °C [10, 25]. Convective-heating is energy-intensive and time-consuming, which affects the efficiency and effectiveness of a large-scale process. Microwave-assisted heating has emerged as a promising attempt to overcome this problem by shortening the impregnation process [26], mainly due to the dielectric heating effects in microwaves [27]. Some previous researchers have demonstrated that microwave heating assists in improving the performance of zeolite-amine-based adsorbent to the CO_2 adsorption and desorption characteristics [28-30]. This work reports a comparative analysis of the MEA impregnation process in natural zeolite originally found abundantly from our local area (Malang, Indonesia) using both convective and microwave-assisted heating.

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This work is expected to give a technical assessment in solid adsorbent preparation for CO_2 adsorption process.

2. RESEARCH METHODS

2-1. Chemicals

The zeolite used as the main adsorbent in this study was natural zeolite in Malang purchased from a local supplier. Hydrochloric acid (HCl) used as the activation solvent was purchased from Merck with pro analysis grade (37%). Monoethanolamine (MEA) with the purity of 99% was supplied by Merck as a means of impregnation agent. Methanol, as a solvent for MEA, was purchased from Merck with analytical grade (99%). Potassium hydroxide (KOH) for Orsat analysis was purchased from Merck with pro analysis grade.

2-2. Adsorbent synthesize process

The first process for synthesizing solid adsorbent developed in this work was activating 100 mesh natural Zeolite powder using 3 M HCl aqueous solution for 24 hours at a ratio of 1: 3 w / v. This zeolite was then washed with demineralized water until its pH was close to the water's pH. Activated natural zeolite was then dried in oven at operating temperature of 110 °C to reduce the moisture content. Activated natural zeolite powder obtained from the previous process was granulated with the aid of sodium metasilicate as a binder on a rotary pan granulator. The granulator was operated at rotating speed of 23 rpm and blade elevation angle of 45°. The zeolite was sieved up to 10 mesh, referred to as ZAA. This ZAA was then impregnated at room temperature with the monoethanolamine-methanol solution (10% v/v) and then heated at 150 °C under conventional heating in the oven for 3 hours operating time described as ZAA-10%MEA-OV. We also employed the microwave-assisted heating method to study the effect of the heating process on the characteristics and its performance on CO_2 adsorption. Impregnated zeolite was also heated using microwave with varied operation time of 10, 20, 30, 40, and 50 minutes, which was then mentioned as ZAA-10%MEA-MC10, ZAA-10%MEA-MC20, ZAA-10%MEA-MC30, ZAA-10%MEA-MC40, dan ZAA-10%MEA-MC50, respectively. We also investigated how the concentration of monoethanolamine affects the CO_2 adsorption capacity by varying its concentration in methanol until 40% v/v and with microwave heating for 10 minutes' operation time. A schematic diagram of the apparatus for the microwave-assisted heating process is presented in Fig. 1.

All of the solid adsorbents synthesized in this work were characterized for their properties, for instance, SEM-EDX for all element contained in natural zeolite and Fourier-Transform Infrared Spectroscopy (FT-IR) Variant 1000 to determine the functional groups in our solid adsorbent.

2-3. CO_2 adsorption measurement

The CO_2 adsorption capacity was measured by continuous adsorption using the gas adsorption apparatus shown in Fig. 2 at a pressure of 1 atm and room temperature. The mixture of CO_2 and N_2 was fed in the adsorption column (Fig. 3) with the concentration varying at 40-50% by adjusting the partial pressure of the gas and the flowrate using a gas regulator. The concentration of CO_2 in the output was analyzed by Orsat analysis. The dimension of the adsorption column is explained in detail in Fig. 3.

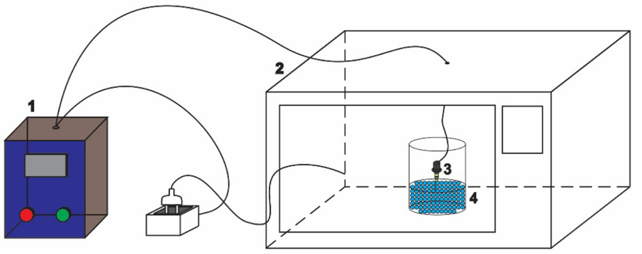

Fig. 1. Schematic diagram of the microwave heating apparatus.
1. Thermo controller
2. Microwave
3. Thermocouple
4. Zeolite

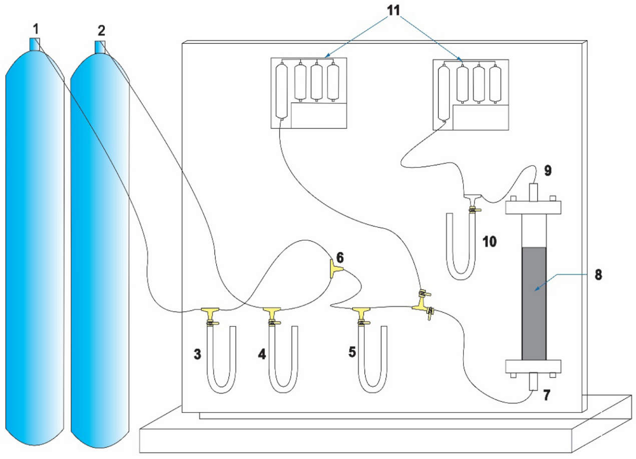

Fig. 2. Gas adsorption measurement apparatus.
1. O_2 gas cylinder
2. N_2 gas cylinder
3. CO_2 gas manometer
4. N_2 gas manometer
5. Gas feed manometer
6. CO_2 dan N_2 gas mixer
7. Gas feed nozzle
8. Adsorption column filled with a solid adsorbent
9. Gas output nozzle
10. Gas output manometer
11. Orsat apparatus

The amount of CO_2 adsorbed in each adsorbent was calculated using Eq. (1) described as:

$$q = \Sigma \frac{\rho}{m\ MW}\left[\int_0^t Fo - Ft\right]dt \tag{1}$$

where q is CO_2 adsorbed in mmol.gr^{-1}, m is the mass of adsorbent, ρ is the density of CO_2 at room temperature, MW is the molecular weight of CO_2, t is adsorption operating time, and F_0 and Ft are the inlet and outlet flow rate of CO_2 gas in adsorption column, respectively.

3. RESULTS AND DISCUSSION

3-1. Solid adsorbent physical characterization and CO_2 adsorption measurement

The results for CO_2 adsorption measurement in all the synthesized solid adsorbent in this work are presented in Fig. 4. It shows that ZA

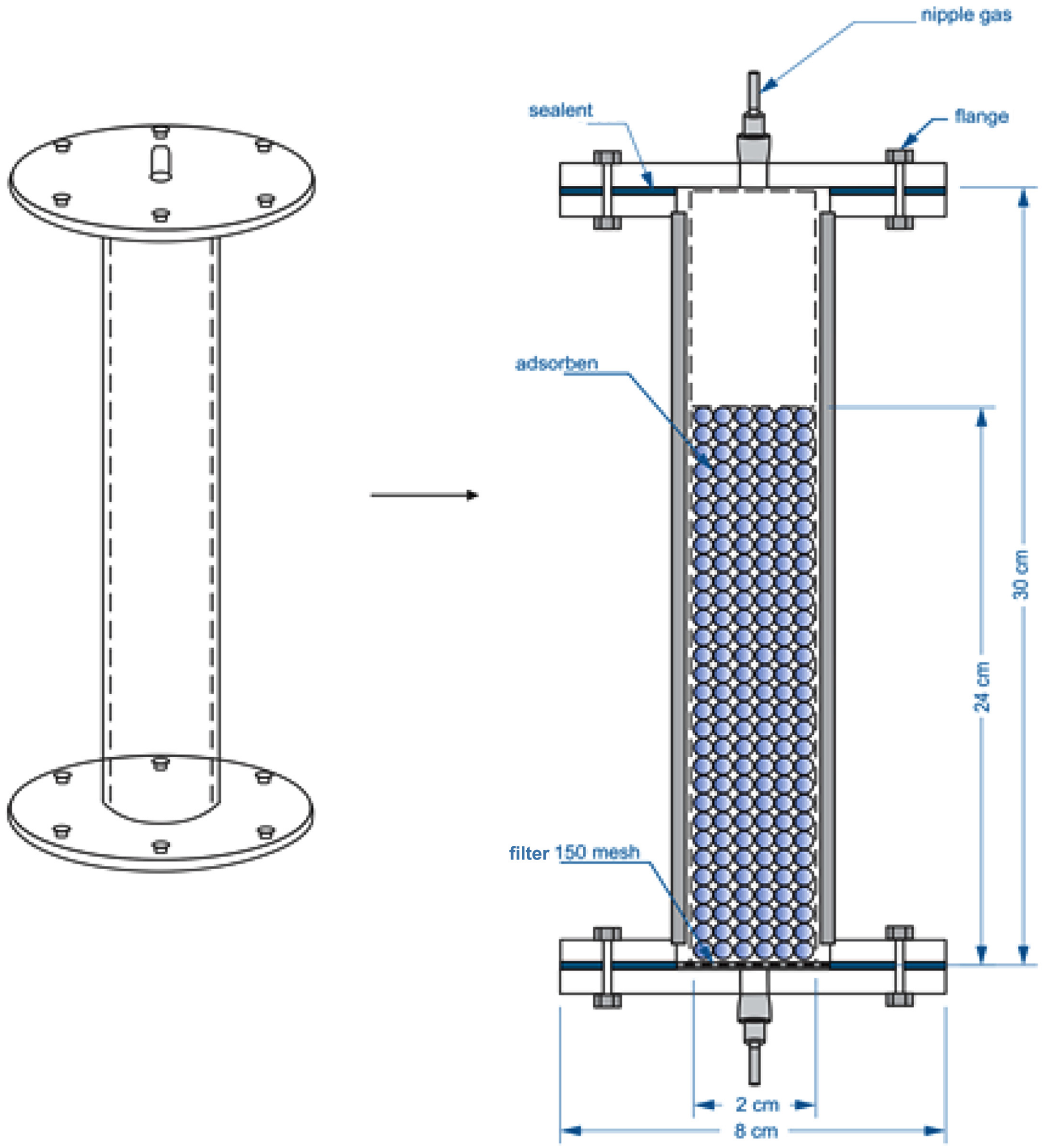

Fig. 3. Dimensions of the adsorption column.

gave the lowest capacity for CO_2 adsorption compared to other types of adsorbents and reached a saturated point at minute 50 due to the high content of metals and organic matter in zeolites. This assumption is in line with the SEM-EDX analysis for the natural zeolite composition used in this work as given in Table 1. Carbon contained in natural zeolite used in this study might come from the CO_2 in air that is adsorbed by natural zeolite before the activation process. The composition of natural zeolite used in this work is similar to natural zeolite from the Manisa-Demirci region of Turkey [31], where oxygen element is the most element contained. However, the silicon and aluminum element contained in our natural zeolite is less than that contained in Turkey's natural zeolites. The composition of natural zeolites might be influenced by the region, weather, humidity, and climate of where natural zeolite is obtained.

Table 1. The SEM EDX analysis results of natural zeolite used in this work

Element	% mass
C	10.929
O	54.155
Na	0.983
Mg	1.213
Si	14.762
Al	4.641
S	0.773
K	0.697
Ca	7.701
Fe	4.144

This metal and other elements could reduce the adsorbent surface area and pores, leading to a decreased ability of adsorbent to capture CO_2 [10]. Following chemical activation using HCl at a concentration of 3 M, the CO_2 adsorption performance improved as indicated by ZAA sample in Fig. 4. HCl aqueous solution as a solvent for the

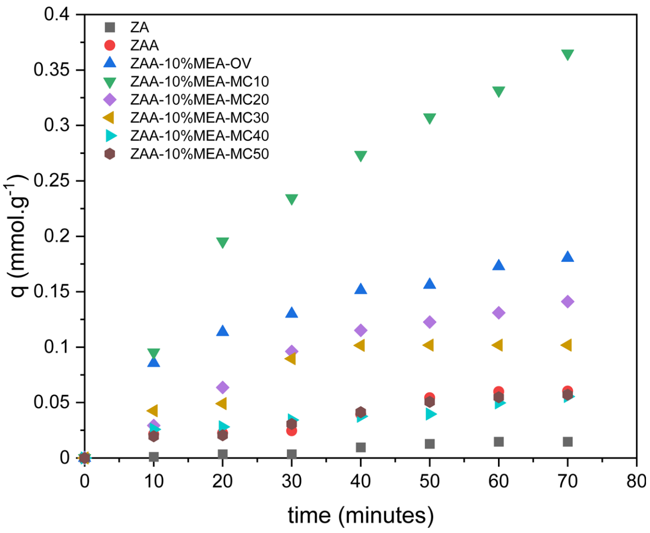

Fig. 4. CO_2 uptake for all adsorbent during the operation time of 70 minutes.

chemical activation process may dissolve some of the metals and other elements contained in the zeolite, which may increase and enlarge the zeolite pore.

The effect of chemical activation process was also observed for changes in the chemical functional group of zeolites using FT-IR peak analysis shown in Fig. 5. The results of FT-IR at wavenumbers of 3709~3608 cm^{-1} showed the presence of Si-OH groups, which was strengthened by the detection of asymmetric stretching of Si-O-Si groups in the fingerprint region at wavenumber 1037 cm^{-1} (ZA and ZAA powder) and 1040 cm^{-1} (ZAA granules) on the zeolite internal framework. Also, at a wavenumber of 1037~1040 cm^{-1}, there is an increase in the transmittance intensity for ZAA granules compared to ZA. It might be caused by the addition of sodium metasilicate in the granulation process; sodium silicate plays the role on the addition of Si atom in the zeolite framework, thus increasing the amount of silicate in the activated natural zeolite.

Fig. 5 also shows that the peaks of 2983 cm^{-1}, 2874 cm^{-1}, 2513 cm^{-1}, and 874 cm^{-1}, which were initially detected in ZA, can no longer be found in either ZAA powder or ZAA granules. This undetected peak might be caused by the decrease in carbon content group in zeolites due to chemical activation. Reactions that could occur in the activation process can be explained in Eq. (2).

$$R^+ [Al^-Si_4O_{10}] + HCl \rightarrow [Al(OH)Si_4O_{10}] + RCl \qquad (2)$$

From Eq. (2) it can be explained that in zeolites, HCl binds to the R component to form salts where R in Eq. (2) can be carbon, magnesium, potassium, sodium, magnesium, calcium, and iron. Moreover, this chemical activation process may also increase the surface area of zeolites due to H^+ ions replacing some alkaline metal and alkaline anions in zeolites. H^+ ion has smaller size than alkaline ion and alkaline earth ion such as Na^+ (1,90 Å), K^+ (2,35 Å), Ca^+ dan (1,97 Å) leading to bigger surface area of zeolite. The performance of solid adsorbent in adsorbing CO_2 was improved by the impregnation of monoethanolamine (MEA) on the surface of activated zeolite granules as indicated in Fig. 4. The highest CO_2 adsorption was reached by ZAA-10% MEA-MC10 adsorbent. The adsorbed CO_2 continued to increase until

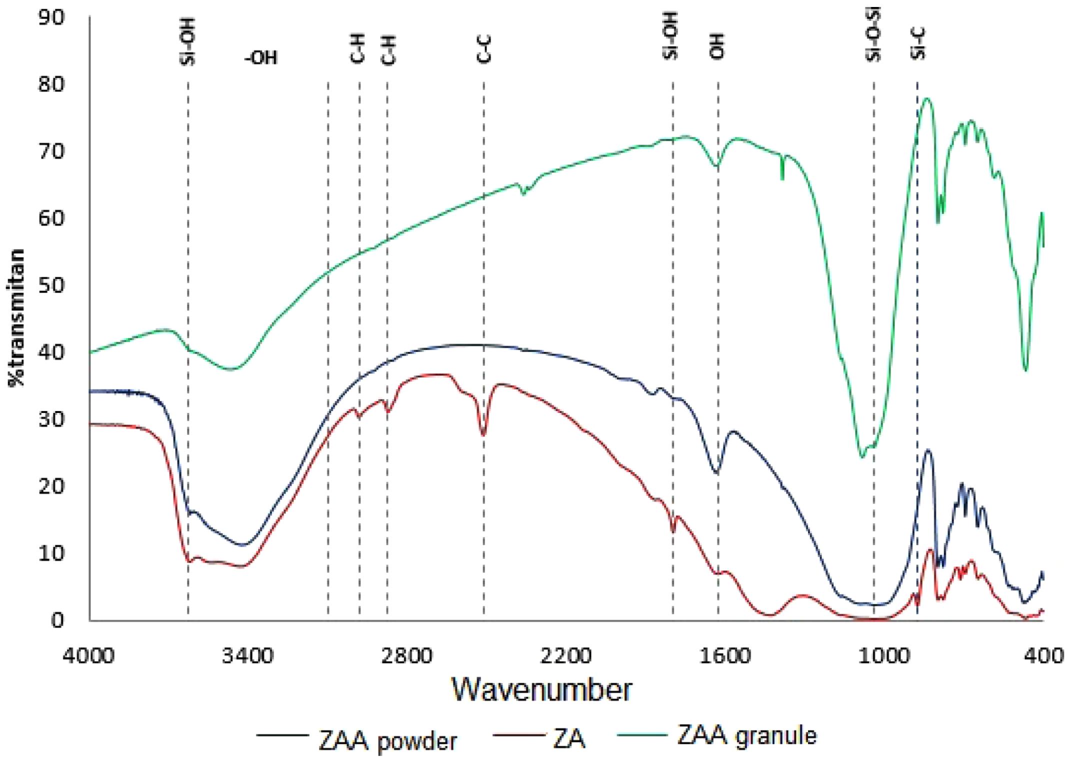

Fig. 5. FTIR analysis results for natural zeolite and activated zeolite.

it reached a breakthrough point where this point is specific to each type of adsorbent. ZA and ZAA tend to reach the breakthrough point in the 50th minute, but at the same operation time, ZAA adsorbs more CO_2 than ZA. The adsorption capacity for ZAA-10% MEA-OV, ZAA-10% MEA-MC10, ZAA-10% MEA-MC20, ZAA-10% MEA-MC40, and ZAA-10% MEA-50 has not shown a tendency to reach the breakthrough point at the 70th minute, meaning that CO_2 adsorption could still be taking place at 70 minutes. ZAA-10% MEA-MC30 sample showed that there is no significant difference in adsorption capacity at 40~70 minutes of adsorption time. It means that ZAA-10% MEA-MC30 sample reached its breakthrough point at 40 minutes.

We performed a statistical test to verify the breakthrough point in Fig. 4 by using a least significant differences (LSD) test followed by Duncan test to determine the distribution of adsorption capacity with the help of SPSS software. The results obtained from this test suggested that for ZA and ZAA, the amount of CO_2 adsorbed (*q*) has no significant mean difference at minutes 50-70. The distribution of the mean difference *q* in each time range can be obtained from the results of the Duncan test. For ZAA-10% MEA-MC30 showed that at 40-70 minutes the mean difference in *q* values is not significantly different, which indicates that at 40 minutes the adsorbent has reached the breakthrough point.

The adsorption performance of all adsorbent synthesized in this work can also be explained through % q_tCO_2 / q_0CO_2 (the inlet and flowrate of CO_2 in the adsorption column) presented in Fig. 6. In the range of 0~20 minutes, ZAA-10% MEA-MC10 has the lowest ratio of %qt CO_2 / q0 CO_2 compared to other types of adsorbents. It has a value of 91.03%, meaning that 9.97% of CO_2 can be adsorbed by adsorbent. The lower percentage of qt CO_2 / q0 CO_2 suggests that CO_2 adsorption in this region was higher than in other types of adsorbents. It demonstrates that zeolite-contained amine from impregnation processes could improve zeolite's capability to capture CO_2. However, it is observed from Fig. 6 that the ability of adsorption decreases with the operating adsorption time. The decrease in adsorption performance can be correlated with the amount of CO_2 that accumulates in the adsorbent. The longer the adsorption operates, more CO_2 will bind to the amine group in the zeolite, making it saturated.

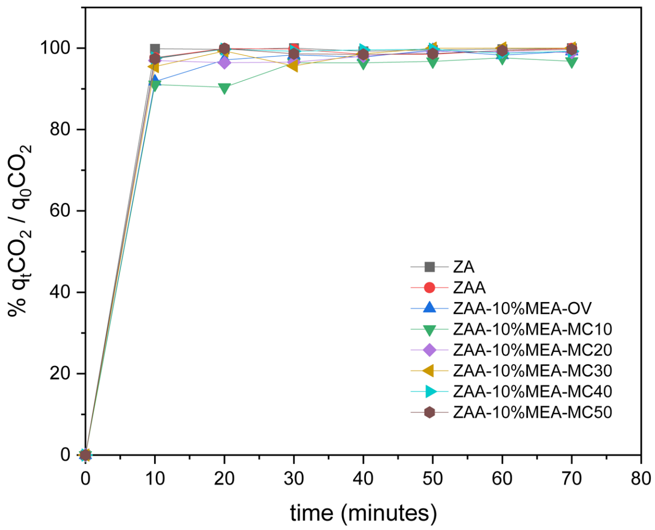

Fig. 6. CO_2 removal performance for all solid adsorbent for 70 minutes' operation time.

Table 2. CO_2 adsorbed during operation time of 70 minutes

Sample name	q (mmol/g)
ZA	0.0147
ZAA	0.0604
ZAA-10% MEA-OV	0.1806
ZAA-10% MEA-MC10	0.3649
ZAA-10% MEA-MC20	0.1411
ZAA-10% MEA-MC30	0.1017
ZAA-10% MEA-MC40	0.0555
ZAA-10% MEA-MC50	0.0571

The amount of CO_2 adsorbed for each adsorbent at 70 minutes of adsorption time is presented in Table 2, which shows that the zeolite adsorption capacity after chemical activation increased by five times than natural zeolites. Microwave heating tends to improve the adsorption capacity of MEA impregnated zeolite compared to the convective heat using an oven for two-fold, as indicated in Table 2.

The exciting phenomenon was observed for the length of microwave heating for MEA impregnation process in the activated zeolite. The highest CO_2 adsorption capacity was obtained in the addition of MEA with microwave exposure for 10 minutes. The adsorption capacity of adsorbents with microwave exposure decreased until the microwave exposure time reached 40 minutes and then it reached a constant value.

The decreasing ability of CO_2 adsorption was caused by the release of a number of amine groups from the zeolite surface as indicated by FT-IR analysis in Fig. 7. At wavenumbers of 3454 cm^{-1}, 1631 cm^{-1}, and 1384 cm^{-1}, lower transmittance was observed for ZAA-10% MEA-MC40 compared to ZAA-10% MEA-MC10. The transmittance of NH_2 and hydroxyl groups can be observed at the same wavenumber, that is, 3500~2927 cm^{-1}. The peaks at 1637 cm^{-1}~1571 cm^{-1} and 1323 cm^{-1} can be indicated as N-H and C-N bonds. The declining transmittance frequency can be detected for ZAA-10% MEA-MC40 at a wavenumber of 3454 cm^{-1}. It is assumed that there was a number of amine groups, which tends to release from the zeolite surface.

Energy transfer through microwave exposure does not occur by conduction or convection as such energy is transformed into heat from friction and collisions between molecules due to dipole rotation and ionic conduction. Microwave exposure will also affect the rotation and oscillation of molecules where the movements will cause friction between the particles, generating heat. Molecular rotation and oscillations that occur due to exposure to microwaves affect the energy or effective temperature of a particular compound on a surface, thereby causing the decreasing boiling point of MEA. It will lead to the MEA evaporating at a temperature lower than 170 °C. Molecules that rotate and oscillate due to microwave exposure cause heterogeneous heating, where the temperature inside the material is higher than on the surface due to

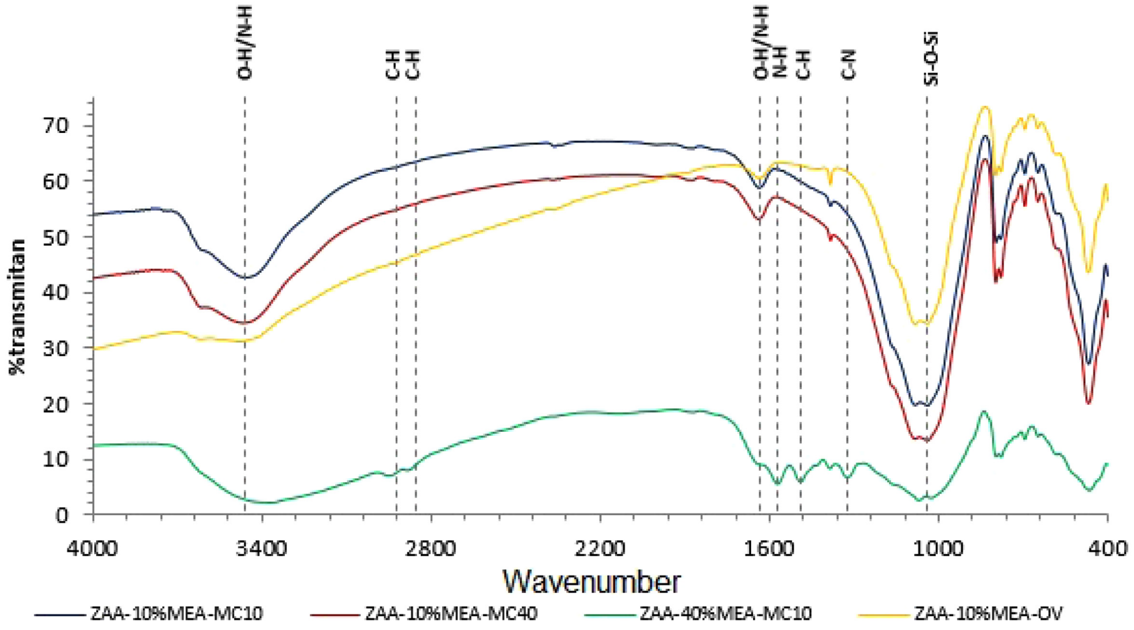

Fig. 7. FTIR analysis results for MEA-impregnated zeolite.

molecular vibrations that coincide.

This energy transfer mechanism can cause a decrease in the CO_2 adsorption capacity of adsorbents prepared by microwave-assisted heating. Heterogeneously distributed temperature causes the amine group in the zeolite to evaporate. It is suspected that the temperature inside the zeolite is higher than the boiling point of MEA (170 °C), causing the amine group to evaporate. Longer exposure time can increase the tendency of the bonds between SiO-MEA breaking, causing the amine groups to be lost from the surface of the zeolite. The decreasing performance of zeolite for adsorbing CO_2 was also encountered by Zhu and Chen [32].

The comparison between convective and microwave heating was also analyzed. For ZAA-10% MEA-MC10 and ZAA-10% MEA-OV with the same amount of impregnated monoethanolamine solution, there was a difference in CO_2 adsorption capacity due to the heating process during convection for 3 hours, causing MEA content in zeolites to evaporate partially. It is proved by FT-IR analysis where at the wavenumber of 3300~3500 cm^{-1}, there was a decrease in transmittance frequency as detected from Fig. 7.

3-2. The effect of MEA concentration on the CO_2 adsorption performance

The increasing concentration of MEA is directly proportional to the increase in adsorption ability. With the same adsorption operating time, it can be indicated that ZAA-40% MEA-MC10 can absorb CO_2 higher than ZAA-10% MEA-MC10 (Fig. 8). This can be explained that it was the result of the increasing amount of MEA impregnated on the surface of the zeolite. The increasing concentration of MEA up to fourfold should be able to enhance its performance with the same magnitude. However, from Fig. 8, the improvement of CO_2 uptake can only reach 58%. This is because of the impregnation using 10% of MEA solution, and it was suspected that most of the Si-OH groups had bonded to the MEA group and only a small portion of Si-OH groups had not reacted. It is alleged that the reaction between the Si-OH groups on zeolites has occurred entirely at a MEA solution at a concentration of less than 40%. The reaction and mechanism of MEA impregnation on the surface of zeolite are presented in eq. (3) and (4) and Fig. 9, respectively.

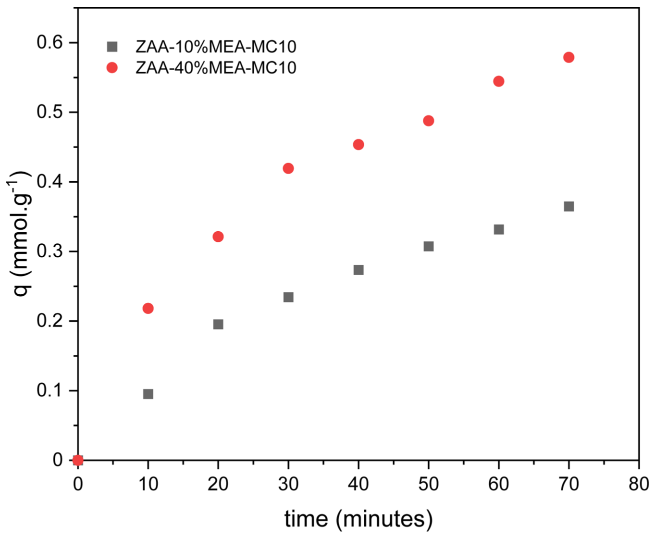

Fig. 8. The comparison of CO_2 uptake between zeolite with MEA impregnated at the concentration of 10% and 40%.

$$Si-OH+CH_3OH\ Si-O-CH_3+H_2O \tag{3}$$

$$Si-O-CH_3+HO-CH_2-CH_2-NH_2\ Si-O-CH_2-CH_2-NH_2 \tag{4}$$

It also can be explained in Fig. 7 that the impregnation process using MEA with the concentration of 40% causes changes in peak intensity at wavenumbers 3375 cm^{-1}, 1571 cm^{-1}, and 1490 cm^{-1} and

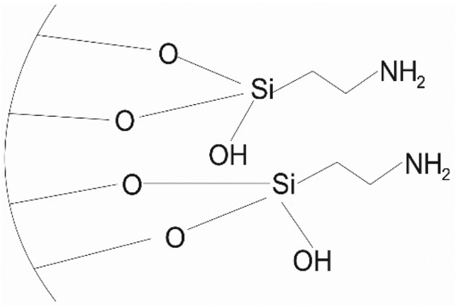

Fig. 9. Reaction and mechanism of impregnation of monoethanolamine on zolite surfaces.

1323 cm^{-1}, which were not initially detected in ZAA-10% MEA-MC10 and ZAA-10% MEA-MC40. It can be explained that impregnation using a 10% MEA solution resulted in lower NH_2 intensity in the adsorbent compared to impregnation using 40% MEA solution. At wavenumbers 2950 cm^{-1} and 2886 cm^{-1}, there is a weak peak observed due to asymmetrical and symmetrical C-H vibration, which indicates aliphatic groups due to amine groups. This group is strengthened by a peak at 1323 cm^{-1} showing the C-N group.

The addition of MEA with a concentration up to 40% causes more amine groups to be spread over the solution; thus, it can increase the possibility of contact with the zeolite surface, thereby binding with it. The adsorption process observed in this work can only be classified as physisorption indicated by FT-IR spectra in Fig. 10. It shows that the results of the FTIR test on ZAA-40% MEA-MC10 adsorbents before and after adsorption produced identical spectra at the peaks of 1571 cm^{-1}, 1490 cm^{-1}, and 1323 cm^{-1}. It proves that the adsorption of CO_2 at 30 °C does not cause changes in the functional groups of adsorbents to form carbamate groups, carbamate acids, or bicarbonates and only takes place through physical adsorption. This phenomenon could be caused by steric hindrance effect from the MEA group on the zeolite. This effect can make CO_2 molecule difficult to react chemically with the amine group on the zeolite.

4. CONCLUSION

We successfully synthesized and produced zeolite-MEA solid adsorbent for CO_2 capture purpose using two heating mechanism processes. The heating process with the help of microwave for 10 minutes operating time could produce solid adsorbent with higher CO_2 uptake than convective heating for 3 hours' operation time for 2-fold magnitude. Longer exposure time of microwave surprisingly decreases the performance of solid adsorbent in adsorbing CO_2. The addition of MEA with concentration up to 40% could only enhance CO_2 uptake ability of solid adsorbent by 58% compared to the zeolite-MEA with MEA concentration of 10%.

ACKNOWLEDGMENTS

Authors gratefully acknowledge all support received from the Faculty of Engineering University of Brawijaya via research funding through Contract No: 123/UN10.F07/PN/2017 and support from Department of Chemical Engineering, Brawijaya University for providing facilities in conducting this research. Authors also thank Bambang Ismuyanto from Department of Chemical Engineering,

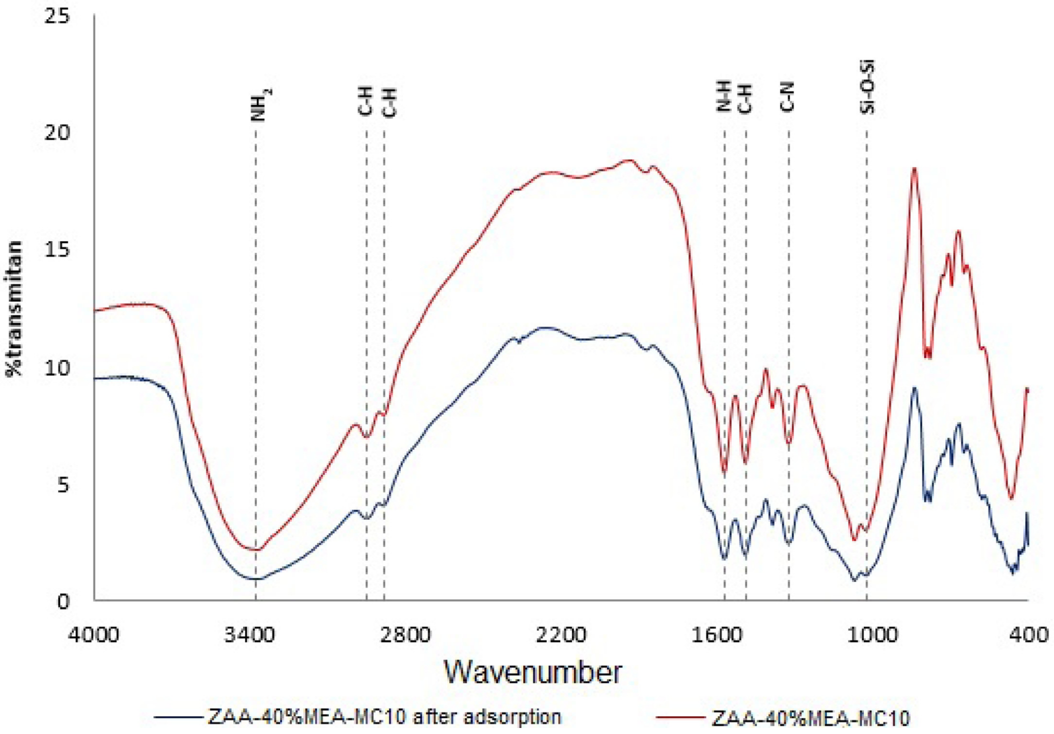

Fig. 10. FTIR analysis results for MEA impregnated zeolite at the concentration of 40% before and after adsorption process.

Brawijaya University for valuable discussions. Z.R.A would like to acknowledge Republic of Indonesia Higher Education Minister for Bidik Misi Scholarship she received for the support of her undergraduate study.

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