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ENHANCING ZEOLITE PERFORMANCE THROUGH AMINO MODIFICATION: A REVIEW OF METHODS AND CHARACTERIZATION

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ABSTRACT

The increase in CO_2 emissions in Indonesia is primarily attributed to the expansion of the economy and ongoing industrialization, primarily driven by the operation of fossil fuel power plants. It is imperative to implement strategies aimed at reducing CO_2 emissions in the industrial sector in order to effectively address Indonesia's pollution problems. An effective method involves utilizing adsorbents, specifically zeolites, to capture and convert CO_2 into valuable fuels. In order to improve the adsorption of CO_2, the zeolite is chemically modified by adding amine groups. The objective of this literature study is to examine the impact of adding amine groups into zeolite on CO_2 adsorption. We collected various theories and data methods to compare the CO_2 adsorption and characterization of modified zeolites. The characterization of the zeolites was determined using XRD, BET, and FTIR techniques. In general, the modification of zeolite with amines has a substantial effect on its properties, particularly on the surface area and volume porosity.

***Keywords**: carbon dioxide, emission, adsorption, zeolite, amine modification*

1. INTRODUCTION

CO_2 emissions in Indonesia are increasing in line with economic expansion and industrialization, primarily due to the significant contribution of the energy sector, specifically fossil fuel power plants. Deforestation and changes in land use also play a substantial role in these emissions, primarily through the burning of forests and peatlands for agricultural purposes, such as establishing oil palm plantations. The number of industries has markedly expanded, rising from 2.756.030 units in 2010 to 4.411.516 units in 2019, reflecting heightened economic activity and societal mobility in Indonesia [1]. This surge in economic activity, propelled by factors such as industrial production growth, underscores its impact on Indonesia's carbon dioxide emissions.

Implementing strategies to mitigate CO_2 emissions in the industrial sector is crucial for addressing Indonesia's pollution issues. One effective approach is the use of adsorbents to capture and convert CO_2 into valuable fuels. Mesoporous silicates [2-3], activated carbon [4-6], covalent organic frameworks [7], and porous organic polymers [8] have undergone

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thorough examination as adsorbents for CO_2. Zeolites, precisely, are recognized for their highly ordered microporous structure and efficiency as adsorbents [3].

Zeolites are crystalline aluminosilicates that exhibit molecular sieving properties and have high porosity and surface areas. [9]. This property enables them to selectively adsorb molecules of significant practical value, such as CO_2. Their intricate structure and composition allow for precise adjustment of adsorption properties to achieve targeted separations [10]. Zeolites possess features that make them highly beneficial in industrial applications as adsorbents or catalysts. Zeolite has several advantages over other adsorbents, such as its low cost, lack of toxicity, and easy production [11]. The effectiveness of zeolites depends on their physical and chemical characteristics, which are determined by their crystal structure and chemical composition. [12]. In order to improve their ability to adsorb CO_2, scientists have conducted studies on modifications and functionalization techniques, such as amine modification.

The effectiveness of zeolites may decrease under humid conditions due to their affinity for water, which competes with CO_2 for adsorption sites. To address this issue, efforts are focused on improving the hydrophobic characteristics of zeolites, such as through amine incorporation. This approach aims to mitigate interference from water molecules and maximize the availability of active sites for CO_2 adsorption [13-14]. The modification of zeolites with amines has garnered significant scientific interest as a potential method for CO_2 adsorption at low pressures, facilitated by the synergistic interaction between amine-CO_2 and CO_2-metal sites within zeolites [15]. These modifications have the ability to activate aluminum sites, expand surface area, reduce pore size, increase porosity volume, and improve stability and regeneration capabilities.

The method utilized tetraethylenepentamine (TEPA) [16-20], monoethanolamine (MEA) [21-22], and triethanolamine (TEA) [22-23] to enhance the adsorption and selectivity of zeolites. Hence, the objective of this literature investigation is to examine the impact of adding amine groups into different types of zeolites in order to facilitate separating CO_2 gas through adsorption. Crystal structure of zeolite, the surface area, and chemical composition, and were analyzed using XRD, BET, and FTIR techniques.

2. METHODOLOGY

This literature review synthesizes various theories and methodologies gathered from numerous sources. The data are derived from existing research. This study employs a qualitative approach, focusing on gathering data regarding the adsorption capacity of CO_2 and its characterization. Subsequently, an analysis compares the collected data and draws conclusions based on the findings. The primary objectives were to assess the effect of amine groups on zeolite for CO_2 adsorption and to explore their influence on zeolite properties. The acquired data are presented graphically, with detailed table discussions reserved for subsequent sub-chapters.

2.1. The process of Conducting a Journal Research

The process of searching for journal articles involves identifying the specific keywords used to search in journal databases such as Science Direct, Google Scholar, and ACS Journal, all related to the same study topic. The selection research outcomes are derived from the analysis of the same variables and characteristics. The journal search focuses on the investigation of the amines group on the zeolite for the purpose of CO_2 adsorption. Journal selection has been prioritized internationally throughout the ten years prior to 2014. The information obtained from the journal will be gathered and synthesized into a conclusive statement.

2.2. Data Collection Methodology

Collecting information is the process of comparing data obtained from one publication with data obtained from another journal. The collecting of data is dependent on the following variables:

- The impact of amine groups on zeolite on its capacity to adsorb CO_2
- The influence of temperature and pressure on the adsorption capability of zeolite for CO_2 gas.
- The influence of amine groups on the properties of the modified zeolites

3. RESULT AND DISCUSSION

3.1. Amine Modification on Zeolite

There are two methods for integrating the amines group into the zeolite: amine grafting and amine impregnation. The amine grafting approach involves the interaction of aminosilane with the hydroxyl groups on the surface of zeolites through a base-catalyzed condensation reaction, resulting in the formation of stable Si-O-Si chemical bonds. The specific types of amines that can be utilized in this particular approach are APTMS (Aminopropyltrimethoxysilane) and TMPED (N-[3-(Trimethoxysilyl) propyl] ethylenediamine). Amine functionalized zeolites have significantly enhanced heat stability throughout the process of adsorption and regeneration. Conversely, in the amine impregnation procedure, amine molecules permeate the porous structure of a solid bed as a result of chemical attraction and differences in concentration. Various amines can be utilized for these procedures, including APTES (3-Aminopropyltriethoxysilane), TEPA (Tetraethylenepentamine), MAE (Monoethanolamine), IPA (Isopropylamine), DEA (Diethanolamine), PEI (Polyethyleneimine), DETA (Diethylenetriamine), and AEEA (N-(2-Aminoethyl)-ethanolamine). Table 1 displays the CO_2 capture capability of different zeolite-based adsorbents that have been modified with amines, along a range of temperatures and pressures.

Table 1. Summary of the CO_2 adsorption performance of modified zeolites with amines

No.	Sample	Amine type	Amine loading [% w/w]	Total loading [mmol/g]	P [bar]	T [°C]	Ref
1.	4A	IBA	0.3	2.56	1	25	[15]
2.	4A	BA	0.3	2.48	1	25	[15]
3.	4A	TEPA, impregnated	15.275	9.2	5	25	[19]
4.	4A	DEA, impregnated	11.112	9.4	5	25	[19]
5.	4A	IPA, impregnated	0.3	2.31	1	25	[24]
6.	4A	APTMS, grafted	0.3	1.05	1	25	[24]

No.	Sample	Amine type	Amine loading [% w/w]	Total loading [mmol/g]	P [bar]	T [°C]	Ref
7.	Beta-25	APTES, impregnated	40	4.7	0.15	35	[16]
8.	Beta-25	TEPA, impregnated	40	2.55	0.15	35	[16]
9.	NaY	TEPA, impregnated	-	2.11	1	75	[17]
10.	NaY	MAE, impregnated	-	1.94	1	75	[17]
11.	Analcime	TEPA, impregnated	60	1.16	1	70	[20]
12.	HZSM-5	AEEA, impregnated	55	4.44	1	48	[21]
13.	HZSM-5	MEA, impregnated	55	4.27	1	20	[21]
14.	Clinoptilolite	MEA, impregnated	2	3.58	4	25	[22]
15.	Clinoptilolite	TEA, impregnated	10	5	5	25	[22]
16.	13X	DETA, impregnated	40	1.69	1	75	[23]
17.	LTA	APTMS, grafted	-	2.3	0.15	60	[25]
18.	LTA	TMPED, grafted	-	1.4	1	60	[25]
19.	13X	PEI, impregnated	60	1.22	1	75	[26]
20.	NaA	TEPA, impregnated	60	4.02	1	70	[27]
21.	HY	ED, grafted	14.98	1.76	1	90	[28]
22	Zeolite Y	TEPA, impregnated	20	1.12	0.005	25	[29]

The application of grafting techniques using APTMS to modify zeolite 4A results in a decrease in both surface area and total pore volume [24]. This drop can be attributed to the molecular size and density of the amine type that covers the pores of the zeolite [24]. The reduction in surface area and total pore volume leads to a corresponding fall in the zeolite adsorption rate, which is measured at 1.05 mmol/g as a shown in Table 1. This modification imparts the sample with the ability to withstand high temperatures, as evidenced by a weight loss value of 18.2% [24].

Employing amines of TEPA and DEA types on 4A zeolite resulted in an increase in CO_2 adsorption by 9.2 mmol/gram and 9.4 mmol/gram, respectively [19]. This is because additional sites for CO_2 adsorption are provided, which enhance the adsorption capacity by increasing surface contacts. The interaction between zeolite and CO_2 molecules is primarily lead by van der Waals forces, which are controlled by the presence of Si and Al atoms in the zeolite structure [19]**.** These forces attract and retain CO_2 molecules on the surface of the zeolite, contributing to the adsorption mechanism [30]. Impregnation was determined to be the more appropriate method for post-combustion CO_2 adsorption compared to grafting, this is because impregnation allows for easy diffusion amines which has a small molecular size and density, into the zeolite surface pores [24]. As a result, the diffusion control mechanism becomes predominant, aiding in the enrichment of CO_2.

The quantity of amine loading in the zeolites directly affects the capacity of CO_2 adsorption. Increasing the amine loading from 20 to 60 wt% on 13X zeolite results in a corresponding increase in adsorption capacity at a temperature of 75°C at a pressure of 1 bar [26]. The maximum loading of PEI, which is 80 wt%, has the lowest adsorption capability, this scenario is due to the structure of zeolites that can clog pores to their full capacity [26].

3.2. The Influence of Temperature and Pressure on The Adsorption Capability

The effectiveness of CO_2 adsorption on zeolites is affected by the operating temperature and pressure. As shown in the Table 1, the temperature condition for testing the CO_2 adsorption was tested bellow 100°C. Lower temperatures typically enhance physical adsorption processes, such as CO_2 capture. The adsorption capacity exhibits an increasing trend with pressure until it reaches a certain threshold, beyond which it may either stabilize or drop due to the saturation of accessible adsorption sites. TEA and MEA were amine groups used to modify the clinoptilolite zeolites, and the adsorption capacity was tested with an initial CO_2 pressure ranging from 0 to 5 bar [22]. The samples all show that the zeolite adsorbed CO_2 increases proportionally to the pressure [22]. Increase in the partial pressure of CO_2 leads to a larger capacity for CO_2 adsorption, this is because there are more CO_2 molecules available for adsorption [31]. Conversely, when the temperature increases, the capacity of CO_2 to be adsorbed decreases due to the weakening of the bonds between CO_2 molecules and the zeolite material.

3.3. The Influence of Amine Groups on The Properties of The Modified Zeolites

Table 2. The summary of textural properties of zeolites

No.	**Sample**	**Amine type**	**Surface BET [m^2/g]**	**Total pore volume [cm^3/g]**	**Average pore diameter [Å]**	**Ref**
1.	4A	IBA	32*	0.125	-	[15]
2.	4A	BA	35*	0.139	-	[15]
3.	4A	IPA	33*	0.133	-	[15]
4.	4A	IPA, impregnated	17.44*	0.09	-	[24]
5.	4A	APTMS, grafted	12.74*	0.01	-	[24]
6.	4A	TEPA, impregnated	46.02	0.1390	91.07	[19]
7.	4A	DEA, impregnated	52.563	0.1802	63.42	[19]
8.	Beta-25	APTES, impregnated	37	-	-	[16]
9.	Beta-25	TEPA, impregnated	23	-	-	[16]
10.	NaY	TEPA, impregnated	172	0.148	5.64	[17]
11.	NaY	MAE, impregnated	191	0.172	6.21	[17]

No.	Sample	Amine type	Surface BET [m^2/g]	Total pore volume [cm^3/g]	Average pore diameter [Å]	Ref
12.	NaA	TEPA, impregnated	23.42	66	-	[20]
13.	Analcime	TEPA, impregnated	21.56	71	-	[20]
14.	Clinoptilolite	MEA, impregnated	24.224	0.1524	251.64	[22]
15.	13X	DETA, impregnated	35.429	0.993	58.03	[23]
16.	13X	PEI, impregnated	1.31	0.003	116	[26]

*external surface area

The modified zeolite conducted analysis using multiple techniques to obtain a comprehensive understanding of the structural and surface modifications. The X-ray Diffraction (XRD) results presented in Figure 1 illustrate that the zeolite crystal structure remains unchanged after being modified with PEI, with no significant changes in the primary crystal parameters or mineral phases. This indicates the modification does not have a direct impact on the internal crystal structure of the zeolite. Nevertheless, the surface area data acquired through the Brunauer-Emmett-Teller (BET) measurements in Table 2 indicates a variation in the surface area of the zeolite. Unmodified NaY zeolite has 212 m^2/g surface area, 0,213 cm^3/g pore volume and it has 7 Å average pore diameter [17]. After the addition of amine groups, the amine-modified NaY material exhibits a decrease in surface area and micropore volume, indicating that the pores are now occupied by the amines [30]. In contrast, modified clinoptilolite that treating it with 2 % w/w of MEA resulted in an increase in surface area and enhance porosity volume [22]. Based on the molecular volume of the amines used, which influences the surface area, pore volume, and mean pore size, TEPA has a larger molecular volume compared to DEA, while 2-MAE has a smaller molecular volume than both TEPA and DEA [17]. Thus, the zeolite that has been modified with the smallest amine exhibits the greatest surface area, pore volume, and mean pore size [17]. The variation in surface area can be attributed to modifications in pore distribution or the introduction of functional groups on the surface.

Fourier Transform Infrared Spectroscopy (FTIR) analysis has been utilized to obtain more insights of these changes by providing supplementary information about the functional groups and structural features present on the surface of the zeolite [32]. FTIR spectroscopy detects the emergence of additional functional groups or modifications to existing functional groups. In other words, while XRD analysis confirms that the crystal structure of the zeolite, the surface area and FTIR data suggest that the introduction of surface modifications or functional groups has had an important effect on the surface properties of the zeolite [27].

The LTA zeolite, which has been modified by monoamine (APTMS) and diamine (TMPED), exhibits additional peaks at 2950 and 1650 cm^{-1}, these peaks can be attributed to the stretching of C-H bonds and the bending of N-H bonds in primary amines, respectively [25]. Furthermore, an extra peak at 1530 cm^{-1}, which corresponds to the bending of N-H bonds in a secondary amine, was observed specifically for LTA-125-diamine [25]. Hence, the combination of data obtained from X-ray diffraction (XRD), surface area analysis, and Fourier-transform infrared spectroscopy (FTIR) offers a comprehensive comprehension of the impacts of modifications on zeolites. This elucidates alterations in surface characteristics and reactivity while preserving the inherent crystal structure.

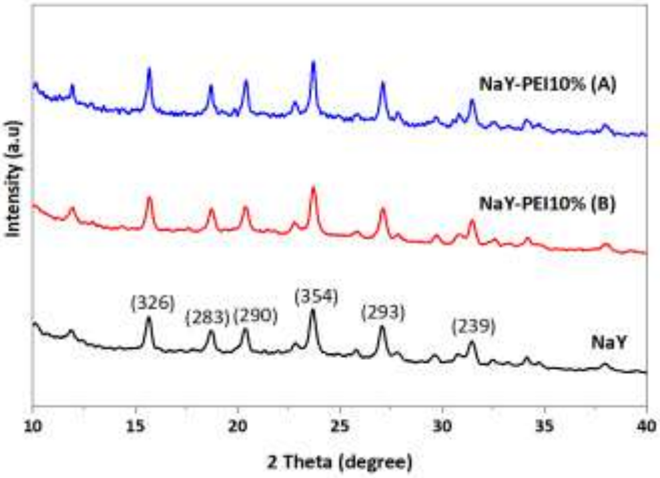

Figure 1. XRD patterns of the synthesized sample NaY and modified NaY [27]

4. CONCLUSION

The addition of amine groups to zeolites, integrated by amine grafting and impregnation techniques, enhances their thermal stability and adsorption capabilities. Research shows that incorporating amines like TEPA and DEA increases zeolites' CO_2 adsorption efficiency by expanding their surface area for interaction. CO_2 capture efficacy depends on factors such as temperature and pressure, with lower temperatures and higher pressures generally favoring greater adsorption capacities. Ongoing studies are optimizing amine loading and operational conditions to improve CO_2 capture efficiency using zeolites. Overall, amine modification significantly alters zeolite properties, impacting surface area, and volume porosity, as confirmed by XRD, BET, and FTIR analyses, thereby enhancing their suitability for applications in adsorption and catalysis.

Further research on zeolite modification with amines can focus on several key aspects. Firstly, exploring different types of amines and their optimal concentrations to enhance adsorption capacity towards gases like CO_2 and NH_3. Secondly, conducting in-depth studies on the interaction between amines and zeolite is essential to understand the involved adsorption mechanisms. Evaluating the thermal stability of amine-modified zeolites is also necessary to ensure effectiveness under various operational conditions. This research can also lead to practical applications of modified zeolites, such as CO_2 capture, gas purification, or chemical catalysis.

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