Preliminary study of molecular sieve materials to alleviate problems faced by tropical peatland

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Abstract. Zeolites are crystalline aluminosilicates usable for numerous applications, including in the separation or purification gas processes, catalysts, ion exchanger and adsorbent. These benefits are due to their molecular sieve property. One of the appropriate technologies for the peat management related to C sequestration and potential peat fire suppressor is molecular sieve material, i.e., zeolite-clinoptilolites and other gas adsorbent material taken from empty fruit bunches of oil palm (EFBOP) obtained through the pyrolysis process (biochar). This study assessed the physicochemical and morphological characterization of natural zeolite-clinoptilolite type, originated from Bayah, Banten, Indonesia, and biochar as gas adsorbent to minimize greenhouse gas emissions and C sequestration. Physicochemical characteristics of 60-80; 80-100, 100-150 particles mesh size of zeolite and biochar were measured according to standard analytical procedures based on Kepmentan 261/KPTS/SR.310/M/4/2019. Mineralogical characteristics and the capacity to absorb greenhouse gases (CO$_2$ and N$_2$) were determined using SEM-EDS and Brunauer-Emmet-Teller analysis, respectively. The result indicated that 100-150 mesh zeolite had a higher CEC value (126.45 cmol(+)/kg$^{-1}$), CO$_2$ (1.0586 mmol g$^{-1}$) and N$_2$ adsorption capacity (75.6 cm$^3$ g$^{-1}$), compared to biochar originated from EFBOP 26.9 cmol(+)/kg$^{-1}$; 0.071 mmol g$^{-1}$; and 1.66 cm$^3$ g$^{-1}$, respectively. Biochar observed in this research yielded a higher pH and organic carbon content, with larger pore space. Combining zeolite and biochar provided an optimal result to deplete greenhouse gas emission i.e., 0.3662 mmol g$^{-1}$, and improved water retention as indicated by water holding capacity percentage.

1. Introduction

Climate change is widely accepted to be closely related to Green House Gas (GHG) emissions due to the warming effect of these gases in the atmosphere [1]. Methane (CH$_4$), as well as carbon dioxide (CO$_2$), and nitrous oxide (NO$_2$) are three gases contributing to heat trapping in the atmosphere. The trap was reported by IPCC [2] based on human use of fossil fuels, logging, and agricultural cultivation practices.

Protecting and restoring peatlands is crucial within the context of low carbon and circular economy. Around 400 million hectares, or about 3%, is covered by peatlands. It is estimated that about 10-12% of peatlands are located in the tropics, with coverage area of about 30 – 45 million hectares. Statistically, around 90% of Indonesian peatland are planted with oil palm. Despite this large figure, planting oil palm over peatlands is yet to achieve satisfactory yield [3]. The use of peatlands in a long-term cultivation exposes negative impacts on the environment. These include changing aerobic conditions and its rhizosphere communities [4]. Peat has its uniqueness in the structure of organic molecule that directly link to its water retention capacity [5]. Due to high content of organic materials, saturated water prohibits forming complex organic bonding. Dried peat associates with minimum water and nutrient retention.
capacity. With that sense, stabilizing peat material is vital to obtain maximum productivity, one of which is by implementing soil amelioration approach [6].

Clinoptilolite zeolite (CZ) is a type of zeolite that is hydrated in nature and is widely used in agriculture. Soil physical and chemical properties have been shown being improved with the addition of CZ, especially on acidic soil and peat areas [7]. The addition of zeolite increases the water holding capacity as well as mineral fertilizers efficiency [8]. Zeolites applied in acidic soils are invaluable for improving nutrient uptake, especially nitrogen, phosphorous, and potassium [9], leading to efficient use of the minerals. The natural zeolites, namely clinoptilolite, can increase soil P availability for plants. It can react with phosphate rock by replacing calcium with ammonia, resulting a higher P solubility in the soil [10]. Zeolite also increases water-use efficiency. Altogether, zeolite can alleviate environmental problems at the agroecosystem, thus promoting the sustainable agriculture.

Both zeolite and biochar’s inner pore are abundant, thus they are able to retain water and gas [11,12]. Previous study on oil palm plantations showed that combining molecular-sieving material with exopolysaccharide producing microbes, could improve water holding capacity and stability of soil aggregate, promoting a yield increment up to 40% [13]. Paucity of studies on zeolite combined with oil palm empty fruit bunch (OPEFB) onpeat soil, however, is observable in the literature. This preliminary study investigated the potential use of clinoptilolite-zeolite milled from Bayah, Banten, combined with biochar derived from OPEFB to reduce green-house gas (CO$_2$ and NH$_3$) emission and to increase water retention capacity of peat soil on palm plantation. We specifically observed and measured: (a) zeolite and OPEFB’s physico-chemical characterization, (b) evaluation of formulated molecular sieve material for CO$_2$, N$_2$ and NH$_3$ adsorption, and (c) observation of water holding capacity that leads to minimizing the chance of fire hazard on peatlands.

2. Materials and methods

2.1. Materials

The Microbiology and Environment Laboratory at Indonesian Research Institute for Biotechnology and Bioindustry (IRIBB), Bogor, hosted this laboratory-scale experiment in November 2020 - April 2021. In this trial, zeolite particle from Bayah, Banten, Indonesia sieved with 60-80, 80-100, and 100-150 mesh. We employed empty fruit bunches of oil palm biochar (EFBOPB), combined with zeolite, to assess their capacity in adsorbing water and green-house gas emission and their conformity level to Indonesian standard regarding molecular sieve material formula as soil ameliorant.

2.2. Methods

2.2.1. Physicochemical analyses of zeolite and EFBOPB

Chemical properties were measured according to the national standard SNI 13-3494-1994 (ICS 73.080) and Indonesian Agriculture Ministerial Decree. Measurement of parameters included organic C, Nitrogen, Phosphate (P$_2$O$_5$), Potassium (K$_2$O), cation exchange capacity, heavy metals, pH, micro-nutrients, and water holding capacity [14]. Contaminating bacteria, such as E. coli and Salmonella, were also investigated.

2.2.2. Formulation of zeolite and EFBOPB as soil ameliorant

Calcination of zeolite milled from Bayah, Banten were done at 150$^\circ$C for four hours. OPEFB biochar was obtained through pyrolysis. Zeolite and biochar ratios of 50:50 and 75:25 (% w/w) were formulated in this experiment. Inoculation of Bulkholderia cenocacapica were then applied to both formulas. Bacteria were capable to stabilize peat soil aggregate using their ability to produce exopolysaccharide, while utilizing Bayah zeolite as its carrier [15]. The isolate was grown on 250 mL liquid medium of ATCC No. 14 which consists of (L-1): 20 g sucrose; 0.8 g K$_2$HPO$_4$; 0.5 g yeast extract; 0.2 g KH$_2$PO$_4$; 0.2 g MgSO$_4$.7H$_2$O; 0.1 g CaSO$_4$.2H$_2$O; 2.0 mg FeCl$_3$; Na$_2$MoO$_4$.2H$_2$O (trace); with final
pH 7.2. Incubation period was 48-72 hours at 30°C. As much as 5% (v/w) concentration of the culture was then inoculated into those two formulas.

2.2.3. Morphological characteristics of zeolite and EFBOPB.
Scanning electron microscope (SEM) analysis was performed to capture images of zeolite and OPEFB surface particle. SEM analysis requires electron beam to thin by accelerated through 20 kV high voltage and passing through an electromagnetic lens [16]. After sputter process was completed, the cast was coated with 35 nm gold-palladium (Au-Pd) and electron micrographs were generated using a Jeol JSM-5310LV SEM. The analyses were conducted at the Physics Laboratory, managed by the Indonesian Institute of Sciences (IIS), Serpong.

2.2.4. Determination of molecular sieve material capacity in gas adsorption.
Physical characterization, i.e. pore structure, specific surface area, pore space area and volume, and average pore diameter, was done using Zielinski-Kettle approach [17], whereas gas (CO₂, N₂, and NH₃) adsorption capacity as well as specific surface area were analyzed using the Brunauer-Emmet-Teller method [18–20]. Analyses were done at the Chemical Laboratory of the Indonesian Institute of Sciences (IIS) at Serpong, Banten. The adsorption amount of nitrogen gas at P/Po ~0.99 was used to determine total pore volume (Vt) of the adsorbent. Density functional theory (DFT) method was employed to calculate micropore volume (<2 nm, Vmi), ultramicropore volume (<0.7 nm, Vultra), and mesopore volume (2 ~ 50 nm, Vme).

Gas adsorption/desorption isotherm of CO₂ and N₂ adsorption were done to determine adsorption capacity of both Bayah zeolite and OPEFB. Samples were pre-treated by heating samples at 350°C for 90 minutes under Helium (He) gas (inert) exposure. At ambient temperature, CO₂ adsorption was conducted for 30 minutes under 5% (v/v) Helium, then purged with Helium gas (inert) for 30 minutes. CO₂ desorption was carried out at 100-700°C temperature with 10°C minute⁻¹ increment followed by maintaining the temperature at 700°C for 10 minutes. The rate of gas flow was set to constant at 30 cm³ minute⁻¹. N₂ adsorption follows the same procedure with constant gas flow rate at 40 cm³ minute⁻¹. Gas adsorption analyses of two formulated combinations, 100-150 mesh zeolite and OPEFB in ratios of 3:1 and 1:1, respectively, were also determined. Adsorption measurements of formulated molecular sieve material were measured in two conditions, with and without CO₂ adsorption.

3. Results and discussion

3.1. Physicochemical properties of zeolite and EFBOPB
Alkaline reactions, occurred on both materials studied with OPEFB, gave higher pH (95) than that of zeolite (7.7) (Table 1). Nitrogen and Potassium were found higher in OPEFB, whereas zeolite delivered higher P content and CEC value. CEC value of 100-150 mesh zeolite (126.45 cmol(+).kg⁻¹) has met the Indonesian standard of SNI 13-7168-2006 (min 100 cmol(+).kg⁻¹), while the CEC value was unable to conform with OPEFB standard. As both zeolite and OPEFB serve as bio-ameliorant to soil, it is a necessity to follow to Ministry of Agriculture’s standard on inorganic soil ameliorant (Tables 1 and 2). Based on the regulation, this research discovered that microbial contaminants and heavy metals values in both materials passed the standard, except for CEC value of OPEFB. Further, zeolite acted as adsorbent while maintained the integrity of its crystal structure according to the molecular sieve material [21]. Bayah’s zeolite-clinoptilolite was able to adsorb water; thus, increased peat’s water-holding capacity. Higher mesh value of zeolites suggests higher water holding capacity (Table 1). This study discovered that when zeolite’s surface area increased, prominent reactions were on chemical and physical properties. For example, higher CEC values and an improved release of K, P, and micronutrients (Zn, Mn, and Fe) were observed on 100-150 mesh, in comparison to the ones on 60-80 or 80-100 mesh.
### Table 1. Physico-chemical characteristics of the studied EFBOPB and zeolite materials.

| Characteristics     | Zeolite: EFBOPB 60-80 mesh | Zeolite: EFBOPB 80-100 mesh | Zeolite: EFBOPB 100-150 mesh | Kepmentan: 261/KPTS/SR.310/M/4/2019 | Method         |
|---------------------|----------------------------|-----------------------------|-----------------------------|------------------------------------|----------------|
| pH                  | 9.2                        | 7.8                         | 7.2                         | 8.2                                | 7-12 pH meter |
| Organic C (%)       | 31.15                      | 0.2                         | 0.1                         | 0.7                                | 30-60 biochar |
| N (%)               | 1.4                        | 0.013                       | 0.012                       | 0.013                              | Kjedahl       |
| P2O5 (ppm)          | 0.3                        | 27.9                        | 36.1                        | 89.92                              | Spectrophotometry |
| K2O (%)             | 2.7                        | 1.54                        | 1.58                        | 1.75                               | ICP           |
| CEC (cmol(+)/kg(-)) | 26.2                      | 70.35                       | 86.97                       | 126.45                             | Min.60 Titration |
| Particle size (nm)  | 4,324.448                  | 74.3346                     | 73.5550                     | 71.4413                            | - BET         |
| E. coli (MPN g(-1))| <3                        | <3                          | <3                          | <3                                 | <3 TPC        |
| Salmonella (MPN g(-1)) | <3                    | <3                          | <3                          | <3                                 | <3 TPC        |
| As (ppm)            | Nd                         | Nd                          | Nd                          | Nd                                 | 10 ICP        |
| Hg (ppm)            | Nd                         | Nd                          | Nd                          | Nd                                 | ICP           |
| Cd (ppm)            | 0.2                        | 1.54                        | 1.58                        | 1.75                               | 2 ICP         |
| Pb (ppm)            | 4.3                        | 3.48                        | 3.34                        | 3.38                               | 50 ICP        |
| Fe (ppm)            | 584.5                      | 88.45                       | 93.24                       | 105.34                             | Min. 7 ICP    |
| Mn (ppm)            | 367.1                      | 8.96                        | 10.52                       | 30.38                              | - ICP         |
| Zn (ppm)            | 11.9                       | 1.34                        | 1.5                         | 2.06                               | - ICP         |
| Ni (ppm)            | 3.62                       | Nd                          | Nd                          | Nd                                 | Max.50 ICP    |
| Cr (ppm)            | 7.2                        | 0.20                        | 0.29                        | 0.32                               | Max.180 ICP   |
| WHC                 | 39.10                      | 43.59                       | 49.22                       | 60.50                              | -             |

*) Indonesian Agriculture Ministerial Decree.

**) CEC = Cation Exchange Capacity, ICP-AES = Inductively Coupled Plasma-Atomic Emission Spectroscopy, BET = Brunauer, Emmett and Teller, TPC = Total Plate Count, WHC = Water Holding Capacity, nd = not detected.

### Table 2. Physico-chemical characteristics of molecular sieve material formula as soil ameliorant.

| Characteristics     | Zeolite: EFBOPB 50:50 | Zeolite: EFBOPB 75:25 | Kepmentan: 261/KPTS/SR.310/M/4/2019 | Units | Method         |
|---------------------|------------------------|------------------------|------------------------------------|-------|----------------|
| pH                  | 7.8                    | 7.5                    | 7-12                               | -     | pH meter       |
| Organic C (%)       | 25.78                  | 12.57                  | -                                  | %     | Spectrophotometry |
| N                   | 0.852                  | 0.686                  | -                                  | %     | Kjedahl        |
| P2O5 (ppm)          | 0.59                   | 0.15                   | -                                  | %     | Spectrophotometry |
| K2O (%)             | 2.0                    | 1.4                    | -                                  | %     | ICP-OES        |
| CEC (cmol(+)/kg(-)) | 67.96                  | 77.22                  | Min.60                             | -     | Titration      |
| Particle size (nm)  | 388,232                | 374,189                | -                                  | nm    | BET            |
| E. coli (MPN g(-1))| <3                     | <3                     | <3                                 | MPN g(-1) | TPC |
| Salmonella (MPN g(-1)) | <3                   | <3                     | <3                                 | MPN g(-1) | TPC |
| As (ppm)            | nd                      | nd                     | 10                                 | ppm   | ICP-OES        |
| Hg (ppm)            | nd                      | nd                     | 1                                  | ppm   | ICP-OES        |
| Cd (ppm)            | 0.08                   | nd                     | 2                                  | ppm   | ICP-OES        |
| Pb (ppm)            | 3.36                   | 3.93                   | 50                                 | ppm   | ICP-OES        |
| Fe (ppm)            | 373.86                 | 238.82                 | Min.7                              | mg/100g | ICP-OES    |
| Mn (ppm)            | 216.82                 | 139.83                 | -                                  | ppm   | ICP-OES        |
| Zn (ppm)            | 8.94                   | 5.08                   | -                                  | mg/100g | ICP-OES    |
| Ni (ppm)            | 2.85                   | 1.84                   | Max. 50                           | ppm   | ICP-OES        |
| Cr (ppm)            | 6.47                   | 3.82                   | Max.180                           | ppm   | ICP-OES        |
| WHC                 | 44.16                  | 44.22                  | -                                  | %     |                |
| Water content       | 5.0                    | 5.0                    | Max.10                             | % (w/w) | -             |

*) Indonesian Agriculture Ministerial Decree.

**) CEC = Cation Exchange Capacity, ICP-AES = Inductively Coupled Plasma-Atomic Emission Spectroscopy, BET = Brunauer, Emmett and Teller, TPC = Total Plate Count, WHC = Water Holding Capacity, nd = not detected.
Smaller zeolite particle size combined with OPEFB at the ratio of 75:25 increased water holding capacity water. This finding argues that its implementation would subsequently enhance water efficiency (Table 2). The research signaled that production of soil ameliorant with formulation ratio of 3:1 between 100-150 mesh zeolite and OPEFB was possible at the laboratory scale and could be further evaluated in field experiments.

3.2. Morphological characteristics of zeolite and EFBOPB
Analysis of Scanning electron microscope (SEM) showed that Bayah’s zeolites have large specific surface area and highly porous. With this crucial characteristics, therefore, they could be engaged as a microbial carrier such as exopolysaccharide bacteria. The bacteria has a capacity to contribute into the formation of aggregates and the structure of firmer peat molecular configuration, which eventually affects water and nutrients supplies to plants. Figure 1 depicts *B. cenocepacia* which helps 100-150 mesh zeolite being more aggregated.

Diameter of zeolite pores tends to be greater with subsiding particle size. As observed, OPEFB has many large pore spaces, which are expected to function as carbon and water adsorbent, as well as retain nutrients ready for use by plants. A larger pore diameter was observed in OPEFB than in zeolite. This finding was supported by Brunaer-Emmet-Teller (BET) analysis which provided information on average pore width for each molecular sieve material i.e., OPEFB (82.5952 nm), 60-80 mesh zeolite (12.9586 nm), 80-100 mesh zeolite (15.3243 nm), and 100-150 mesh zeolite (17.0669 nm).

![Image 1](image1.png)

*Figure 1.* Scanning electron photomicrographs of a 100-150 mesh zeolite (A), inoculated with exopolysaccharide-producing bacteria (B), and EFBOPB particle (C). Mag. 500x.

3.3. Zeolite and EFBOPB capacity as molecular sieve material in gas adsorption
Zeolite and OPEFB are potential materials helping to reduce GHG emissions. This research investigated their physical properties such as scanning electron microscope (SEM) outcomes, surface area of the pores, and the capacity to absorb GHG (CO$_2$) and N$_2$ using the Brunaer-Emmet-Teller method [19,20].

Capacity of gas adsorption from OPEFB was measured with and without CO$_2$ or NH$_3$ adsorption. Net CO$_2$ or NH$_3$ adsorption was measured by differencing two measurements. Table 3 indicates that 100-150 mesh zeolites adsorbed CO$_2$ and NH$_3$ more than those of smaller particles. However, CO$_2$ and NH$_3$ adsorption ability of zeolites was inferior to that of OPEFB (Table 3). When zeolite and OPEFB were combined as formulated molecular sieve material, they were able to improve gas adsorption capacity. Underlying mechanism, however, is yet to be fully understood. In theory, with 1:1 ratio, the proportion of adsorbing capacity is about 1.24 mmol CO$_2$ g$^{-1}$ and 0.54 mmol NH$_3$ g$^{-1}$. Observations suggested about 1.67 mmol CO$_2$ g$^{-1}$ and 0.52 mmol NH$_3$ g$^{-1}$ in 3:1 ratio. It is argued that gases adsorption capacity is associated with pores volume and pores total area of molecular sieve material.
The biggest contributor to greenhouse gas emissions associated with agriculture has been nitrogen (N) fertilizers application. Atmospheric pollutant of this element is in the form of N\textsubscript{2}O emissions. One can exploit valuable microorganisms to improve soil structure, which in turn giving favorable yield level. By doing so, the use of fertilizer can be reduced and eventually leads to a reduced greenhouse gas emissions. Based on previous research [22,23], emission spanning from 1052.26 to 1209.51 kg CO\textsubscript{2}-eq ha\textsuperscript{-1} was expected with a release of nitrogen in the form of N\textsubscript{2}O between 19.11 and 22.17 kg N\textsubscript{2}O-N ha\textsuperscript{-1}. According to the Intergovernmental Panel on Climate Change [1], applying N fertilizer contributes to one percent of N\textsubscript{2}O-N emission. It is therefore, a necessity to study practical solution of reducing the N\textsubscript{2}O emissions by adsorbing emitted gas from N fertilization.

To adsorb N\textsubscript{2} gas, molecular sieve materials, in the forms of zeolite, were investigated in present research. Zeolites with 100–150 mesh yielded a superior adsorption capacity (75.6 cm\textsuperscript{3}/g STP) of N\textsubscript{2} gas by zeolite, EFBOPB, and the combination of zeolite and EFBOPB formula (50:50 and 75:25).

Table 3. Adsorption of CO\textsubscript{2} and NH\textsubscript{3} gas by zeolite, EFBOPB, and the combination of zeolite and EFBOPB formula (50:50 and 75:25).

| Molecular sieve material types | mol CO\textsubscript{2} (mmol) | Basmicity (mmol/g) | CO\textsubscript{2} | mol NH\textsubscript{3} (mmol) | Acidity (mmol/g) |
|-------------------------------|----------------|--------------------|------------------|----------------|-----------------|
| Zeolite 60-80 mesh             | 0.0336         | 1.0586             | 0.025725436 0.8380 |
| Zeolite 80-100 mesh            | 0.0539         | 1.7856             | 0.029328566 0.8729 |
| Zeolite 100-150 mesh           | 0.0721         | 2.4038             | 0.032618937 1.0290 |
| EFBOPB with adsorption of CO\textsubscript{2} or NH\textsubscript{3} gas | 0.0879         | 2.6104             | 0.095730411 2.8156 |
| EFBOPB without adsorption of CO\textsubscript{2} or NH\textsubscript{3} gas | 0.0914         | 2.5394             | 0.096908400 2.7614 |
| Zeolite:EFBOPB 50:50 with adsorption of CO\textsubscript{2} or NH\textsubscript{3} gas | 0.0678         | 2.0492             | 0.069936024 2.0630 |
| Zeolite:EFBOPB 50:50 without adsorption of CO\textsubscript{2} or NH\textsubscript{3} gas | 0.0568         | 1.7533             | 0.152950676 4.7947 |
| Zeolite: EFBOPB 75:25 with adsorption of CO\textsubscript{2} or NH\textsubscript{3} gas | 0.0554         | 1.5654             | 0.057090597 1.8240 |
| Zeolite: EFBOPB 75:25 without adsorption of CO\textsubscript{2} or NH\textsubscript{3} gas | 0.0368         | 1.1992             | 0.052505654 1.6306 |

4. Conclusions

To minimize the impact of intensive agricultural practices over peatlands, technologies capable to reduce dried peat condition and GHG emission are needed. Preliminary study using Bayah zeolite, OPEFB, and the mixture of them on CEC, water holding capacity, and greenhouse gasses (CO\textsubscript{2} and N\textsubscript{2}) indicated a potential of these as molecular sieve materials. Zeolites with 100–150 mesh size yielded a higher CEC (126.45 cmol(+)/kg), water holding capacity (60.5%), CO\textsubscript{2} (1.0586 mmol g\textsuperscript{-1}), N\textsubscript{2} adsorption capacity (75.6 cm\textsuperscript{3} g\textsuperscript{-1}) when compared to biochar originated from EFBOP, achieving 26.9 cmol(+)/kg; 39.1%; 0.071 mmol g\textsuperscript{-1}; and 1.66 cm\textsuperscript{3} g\textsuperscript{-1}, respectively. Meanwhile, biochar has higher pH, organic carbon content, and larger pore space. Combination
of zeolite and biochar provided the optimal result to reduce greenhouse gas emission, i.e., 0.3662 mmol g\(^{-1}\), and an improved water retention shown by water holding capacity percentage of around 44.2%. The results of this study indicated that Bayah zeolite and OPEFB potentially serve as molecular sieve materials, capable to retain more water and to absorb greenhouse gases. Despite this, effectiveness of this new molecular sieve materials needs to be further evaluated field-wise.

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