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Surface Modification of Natural Zeolite Using CTAB And Their Characterization

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Abstract

Natural zeolites have been widely used for various applications due to their negatively charged frameworks. The utilization of the negatively charged properties of zeolites has contributed significantly to advances in research, particularly in the treatment of heavy metal or cation-containing waste. Wastewater contains not only environmentally harmful cations or heavy metals, but also anions, such as nitrate, phosphate, sulfate, and chloride. Therefore, the zeolite was modified with CTAB to impart a positive surface charge, enabling it to effectively remove anionic species. This research was modified Klaten's natural zeolite with CTAB. Before modification, the natural zeolite was ground to 200 mesh and first activated using hydrochloric acid. Subsequently, the zeolite was modified by the addition of CTAB at a concentration corresponding to twice its cation exchange capacity. Natural zeolite, activated zeolite, and modified zeolite were characterized using XRD and FT-IR techniques. Based on the obtained results, it was concluded that the modification of Klaten's natural zeolite was successfully achieved. This is evidenced in the XRD diffractograms, where the CTAB-modified natural zeolite shows a shift in the *d*-spacing toward lower 2θ values by approximately 0.5° compared to natural zeolite and acid-activated zeolite, indicating an enlargement of the zeolite pores. Furthermore, the FT-IR spectra reveal the presence of characteristic CTAB absorption bands in the modified zeolite.

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INTRODUCTION

Natural zeolites are hydrated aluminosilicate minerals characterized by a framework structure consisting of micropores, channels, and cavities. The composition of zeolites comprises three main aspects: extra-framework cations, the porous framework, and the adsorbed phase ($Mx/n[(AlO_2)x(SiO_2)y]\cdot nH_2O$) where M is extra-framework cations, such as K^+, Na^+, Ca^{2+}, and Mg^{2+}. The variables "n" and "x" represent the number of H_2O molecules and the cation valence, M, respectively. Natural zeolites have a unique three-dimensional structure formed by $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ tetrahedra linked by oxygen. When some Si^{4+} ions are substituted by Al^{3+}, the zeolite framework becomes negatively charged. This negative charge enables zeolites to undergo cation exchange to maintain charge balance. Consequently, zeolites exhibit a high cation exchange capacity (Tran et al., 2019).

Several studies have demonstrated the potential of zeolites, including Wiyantoko and Rahmah., (2017) who measured cation exchange capacity on natural zeolite. The cation exchange capacity determination was performed by percolation, distillation and titration processes. The results obtained from physical activated and chemical activated of natural zeolite were 181,90 cmol(+)/kg and 901,49 cmol(+)/kg. Zijun et al., (2021) who employed cation exchange capacity of zeolite W from ultra-fine natural zeolite waste was 324 meq/100 g.

Acid or alkaline activation is intended to improve adsorption performance. Activation of natural zeolite is generally carried out through acid treatment. Acid activation/treatment causes demineralization, which involves the removal of metals and other impurities that obstruct the pores of the zeolite. Anghistra et al., (2023) modified natural zeolite using acid treatment (1% HF dan 6M HCl) and high-energy milling. They found that the natural zeolite underwent dealumination after activation. Norvia et al., (2016) also reported similar findings, in which natural zeolite was dealuminated through acid activation using HCl and H_2SO_4.

Zeolites have been widely used for various applications due to their negatively charged frameworks. Several studies have demonstrated this potential, including Margareta et al., (2013) who employed natural zeolites to remove heavy metals from wastewater; Fu et al., (2020) who utilized natural zeolites for ammonium removal. The modified zeolite exhibited the highest ammonium removal efficiency, which was 39.88% higher than that of the natural zeolite. Holub et al., (2016) who evaluated the ability of natural zeolites to remove cation. The Cu(II) sorption efficiency in the batch system reached 90%.

The utilization of the negatively charged properties of zeolites has contributed significantly to advances in research, particularly in the treatment of heavy metal or cation-containing waste and in the purification of surface water and groundwater (Margareta et al., 2013). However, it cannot be overlooked that wastewater contains not only environmentally harmful cations or heavy metals. Wastewater also contains various anions, such as nitrate, phosphate, sulfate, and chloride. (Hashim et al., 2019) and (Bui et al., 2018) investigated phosphate anion from river water and wastewater, (Qiao et al., 2019) and (Yin et al., 2018) investigated phosphate and nitrate anions from aqueous solution and eutrophic water, (Kalaruban et al., 2016) studied removal nitrate from water, and (Rozana et al., 2020) studied sulfate and phosphate anions from wastewater using electrocoagulation method. Therefore, modification of zeolites is required to enable their effective application for the removal of anions from wastewater.

Prior to modification, zeolites need to be activated to remove possible impurities and to increase their surface area. Zeolite activation is commonly carried out through acid treatment. Bani (2023) performed zeolite activation using a mixture of hydrofluoric acid (HF), hydrochloric acid (HCl), and NH_4Cl, which resulted in changes in the mineral composition of the zeolite. This was indicated by a reduction in the number of characteristic peaks in the XRD pattern of activated natural zeolite from Ende, decreasing from 38 peaks before activation to 34 peaks after activation, with impurities effectively removed. Mukti (2016) also conducted acid activation using nitric acid, which led to cation exchange with H^+ and dealumination. Norvia et al., (2016) perfomed natural zeolite dealumination using acid (HCl and *H_2SO_4) for catalysts.* The FTIR spectrogram results showed that the dealumination process caused significant changes in the wavenumber and Si/Al ratio. The Si/Al ratio of the zeolite increased from 4.57 to 5.41 for HCl-dealuminated natural zeolite and to 6.42 for H_2SO_4-dealuminated natural zeolite.

Surfactant-modified zeolites have been widely investigated, particularly using cationic surfactants. Darmansyah et al., (2021) successfully modified natural zeolite from Lampung with cetyltrimethylammonium bromide (CTAB), which effectively reduced the chemical oxygen demand (COD) of tapioca industrial wastewater. Trojeni et al., (2025) also reported successful modification of natural zeolite (clinoptilolite) with CTAB to enhance VOC adsorption from kerosene. Modified zeolite with CTAB has achieved optimal adsorption efficiency with conditions of pH = 3.0, adsorbent dosage of 0.4 g and contact time of 180 min.

These research findings indicate that natural zeolites have very high potential for wastewater treatment. Therefore, it is essential to utilize and enhance the potential of natural zeolite resources in Indonesia, one of which is natural zeolite from Klaten. Farisuna (2013) successfully modified zeolite and applied it for the adsorption of Pb^{2+}, nitrate anions, and methyl orange.

Surfactant-modified natural zeolite from Klaten has been proven to adsorb cations, nitrate and sulfate anions, as well as organic compounds; however, studies focusing on phosphate anions are still lacking. Therefore, this article discusses the modification of natural zeolite from Klaten and their characterization for phosphate anion removal.

METHOD

Material and Tools

The equipments used in this study were

glassware, mortar, shiever 200 mesh, oven, analytical balance Metler As 200, porcelain crucible, desicator, stirrer, vacuum pump Buchi VacR V-500, pH meter (Horiba pH meter F-52). Analytical instruments: spectrometer *Fourier Transform Infrared* (FT-IR Shimadzu 8210 PC), difractometer X-ray Shimadzu XRD 6000.

The materials used in this study were natural zeolite was collected from Klaten, Yogyakarta, HCl 37% (ρ=1,19 g mL^{-1}, Mr=36,5 g mol^{-1}), HNO_3 68%, cetyltrimetilammonium bromide (CTAB, Mr=364,46 g mol^{-1}), silver nitrate ($AgNO_3$, Mr=169,87 g mol^{-1}), KH_2PO_4 (Mr=136,09 g mol^{-1}), all of materials from Merck, aquadest, Whatman 42, pH universal indicator, filter paper and weighing paper.

Preparation of Natural Zeolite

Natural zeolite from Klaten was ground using an agate mortar and then sieved through a 200-mesh sieve. The fraction that passed through the 200-mesh sieve was used as the research sample, while the fraction that did not pass was reground and sieved again until it passed through the 200-mesh sieve. This procedure was carried out to obtain homogeneous particle size and surface area.

Acid Activation of Natural Zeolite

The 200-mesh natural zeolite from Klaten was soaked in distilled water for 24 hours at room temperature. The soaked sample was then filtered and dried in an oven at 80 °C for 24 hours, followed by grinding. A total of 100 g of the ground zeolite was heated in a fume hood at 72 °C in 150 mL of 3 M HCl solution for 30 minutes. Activation of zeolite with acid aims to remove impurity oxides and induce dealumination (Norvia et al., 2016). In addition, acid activation can affect the cation exchange capacity of zeolite due to dealumination, which makes the zeolite structure less negatively charged (Ngapa, 2017). This results in a decrease in the cation exchange capacity of the zeolite. The selection of 3 M HCl was based on the study by Farisuna (2013) and supported by findings showing that metal oxides can dissolve at a concentration of 3 M (Ngapa, 2017). After heating, the zeolite was filtered and washed with distilled water until neutral. The zeolite sample was considered neutral when no chloride ions (Cl^-) were detected in the filtrate, as indicated by the absence of AgCl precipitate upon reaction with $AgNO_3$ solution. The neutralized zeolite sample was then dried in an oven at 120 °C for 24 hours.

Modification of Natural Zeolite with CTAB

A total of 5 g of natural zeolite, previously heated with 3 M HCl, oven-dried, and finely ground, was dispersed in water and then added with 125 mL of CTAB solution at a concentration equivalent to twice the cation exchange capacity (CEC) of the zeolite. The mixture was stirred using a magnetic stirrer for 24 hours, then filtered and washed with distilled water until the filtrate was free of Br^- ions, as confirmed by the $AgNO_3$ test. The sample was subsequently dried in an oven at 80 °C for 24 hours. The zeolite modified with CTAB at 2×CEC is hereafter referred to as zeolite–CTAB.

Characterization

The characterization of natural zeolite, activated zeolite, and CTAB-modified zeolite was carried out using XRD and FT-IR techniques. Based on the XRD results, the main composition and crystallinity of the natural zeolite, as well as the changes occurring after activation and modification with CTAB, could be identified. The presence of diffraction peaks at the same 2θ positions for natural zeolite, activated zeolite, and CTAB-modified zeolite retained its original crystal structure, although changes in peak intensity were observed. Meanwhile, the FT-IR results revealed the characteristic absorption bands of natural zeolite, the changes after acid activation, and the appearance of new absorption bands after modification with CTAB.

RESULTS AND DISCUSSION

The natural zeolite used in this study was Klaten natural zeolite. Prior to use, the natural zeolite was first prepared by grinding and sieving it through a 200-mesh sieve. This process aimed to increase the surface area and obtain a more homogeneous particle size distribution. The 200-mesh zeolite was then soaked in distilled water for 24 hours to remove water-soluble impurities. After soaking, the zeolite was filtered and dried in an oven at 120 °C for 24 hours to evaporate water and polar impurities remaining on the zeolite surface, so that the pore spaces became relatively empty. The dried zeolite was then ground again.

Subsequently, the natural zeolite was activated by immersing it in 3 M HCl for 30 minutes at a temperature of 72 °C. This activation process aimed to remove inorganic impurities. The zeolite was then filtered and washed with distilled water until neutral pH was achieved. This condition was indicated by the absence of a white AgCl precipitate when the filtrate was reacted with $AgNO_3$. Finally, the zeolite was dried at 120 °C for 24 hours, reground, and sieved again using a 200-mesh sieve.

The determination of the cation exchange capacity (CEC) of zeolite was carried out on natural zeolite before and after activation. This was intended to evaluate the effect of the activation process on the cation exchange capacity of the zeolite and to determine the amount of surfactant required for zeolite modification. The results of the cation exchange capacity determination for natural zeolite and activated zeolite are presented in Table 1.

Table 1. CEC value of natural zeolite and activated natural zeolite

Sample	CEC (meq/g)
Natural zeolite	1,032
Activated natural zeolite	0,732

As shown in Table 1, a decrease in the CEC value was observed after the activation process. This indicates that dealumination occurred (Anghistra et al., (2023), leading to a reduction in the negative charge of the zeolite framework, which consequently decreased the zeolite's ability to undergo cation exchange.

The zeolite modification process was carried out using the cationic surfactant CTAB, in which an ion exchange occurs between the charge-balancing cations on the external surface of the activated zeolite and the surfactant molecules $C_{16}H_{33}(CH_3)_3N^+$.

X-Ray Diffraction Analysis

In the X-ray diffractogram of the natural zeolite (Figure 1a), diffraction peaks appeared at d (Å) = 9.516; 4.516; 4.076; 3.404; 3.269; 2.935; and 2.549 with relatively high intensities. These data are consistent with JCPDS No. 6–239, which reports d (Å) values of 9.100; 4.530; 4.140; 3.390; 3.310; 2.946; and 2.522, indicating the presence of mordenite mineral in the Klaten natural zeolite. In addition, peaks with high intensities were observed at d (Å) = 6.808; 5.976; 4.625; 4.353; 3.921; 3.834; 3.535; and 3.439. These values correspond well with JCPDS No. 25–1349, which lists d (Å) = 6.760; 5.970; 4.654; 4.346; 3.910; 3.835; 3.549; and 3.418, confirming the presence of clinoptilolite mineral in the Klaten natural zeolite. Based on the diffractogram results, it can be concluded that the Klaten natural zeolite consists of clinoptilolite and mordenite minerals.

In the diffractogram of the activated natural zeolite (Figure 1b), the same diffraction peaks as those of the natural zeolite were observed; however, the peak intensities increased after activation. This indicates that the activation process removed metal oxide impurities from the zeolite, resulting in enhanced diffraction intensities.

Following activation, the natural zeolite was modified using the CTAB surfactant. The X-ray diffractogram of the CTAB-modified zeolite (Figure 1c) showed diffraction peaks similar to those of the natural and activated zeolites. However, a slight shift of the peaks toward lower 2θ values (approximately 0.5°) was observed, accompanied by a significant decrease in peak intensities.

A shift of the diffraction peak to lower angles suggests an expansion of the zeolite lattice (Goetze et al., 2018). This results in changes in the crystal lattice parameters, which are detected as a shift of the diffraction peaks toward lower angles. This peak shift is attributed to dealumination, which increases the Si/Al ratio. Although the observed shift in diffraction peaks is not significant, it can still enhance the adsorption capacity of the CTAB-modified zeolite.

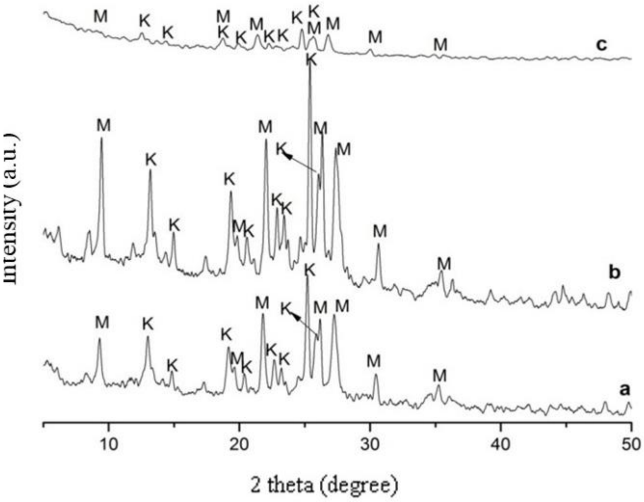

Figure 1. Diffractogram (a) natural zeolite, (b) activated natural zeolite, and (c) zeolite-CTAB.
(Additional: K=clinoptilolite, M=mordenite)

This indicates that the addition of CTAB at twice the cation exchange capacity resulted in the zeolite surface being covered by CTAB molecules, causing the mineral composition of the zeolite detected by the diffractogram to appear in relatively small amounts. This indicates that the zeolite was successfully modified.

FT-IR Spectral Analysis

The natural zeolite, activated zeolite, and CTAB-modified zeolite that had been interpreted based on X-ray diffractogram results were further supported by infrared spectral data to identify and confirm the functional groups present in the zeolite materials. The FT-IR spectra of natural zeolite, activated natural zeolite, and CTAB-modified zeolite are shown in Figure 2, and the interpretation of the obtained results was correlated with the absorption bands of natural zeolite and CTAB listed in Table 2.

In the natural zeolite, bands were observed within 500–1300 cm⁻¹, indicating the presence of aluminosilicate frameworks, which are characteristic of zeolite materials. The band at 447 cm⁻¹ corresponds to the bending vibration of Si–O/Al–O bonds. The band appearing at 794 cm⁻¹ is attributed to the symmetric stretching vibrations of O–Si–O and O–Al–O bonds, while the band at 1041 cm⁻¹ is associated with the asymmetric stretching vibrations of O–Si–O and O–Al–O bonds.

The band around 1643 cm⁻¹ indicates the bending vibration of H–O–H from adsorbed water molecules. Meanwhile, bands observed at approximately 3618 and 3448 cm⁻¹ represent overlapping asymmetric and symmetric H–O–H stretching vibrations along with hydroxyl groups present in the zeolite structure.

The FT-IR spectral of the activated natural zeolite exhibits absorption bands similar to those of the natural zeolite, although slight shifts are observed while still remaining within the same spectral ranges. The activated natural zeolite shows bands at 463 cm⁻¹, 794 cm⁻¹, 1057 cm⁻¹, 1635 cm⁻¹, 3448 cm⁻¹, and 3626 cm⁻¹, indicating the presence of aluminosilicate frameworks, Si–O/Al–O bending vibrations, symmetric and asymmetric O–Si–O and O–Al–O stretching vibrations, as well as H–O–H bending vibrations and overlapping symmetric and asymmetric H–O–H stretching vibrations.

The shift of the vibration band from 1041 cm⁻¹ to 1057 cm⁻¹, which corresponds to the asymmetric stretching vibrations of O–Si–O and O–Al–O, is highly sensitive to the dealumination process. Anghistra et al., (2023) reported that acid activation of natural zeolite induces dealumination and causes a shift of vibrational bands toward higher wavenumbers, as evidenced by shifts from 1049.28 cm⁻¹ to 1095.57 cm⁻¹ and 1080.14 cm⁻¹. Similar results were obtained by other researchers, Norvia et al., (2016) found that the wavenumber of natural zeolite increased from 1042.57 cm⁻¹ to 1072.47 cm⁻¹ for H_2SO_4-dealuminated zeolite and to 1064.75 cm⁻¹ for HCl-dealuminated zeolite.

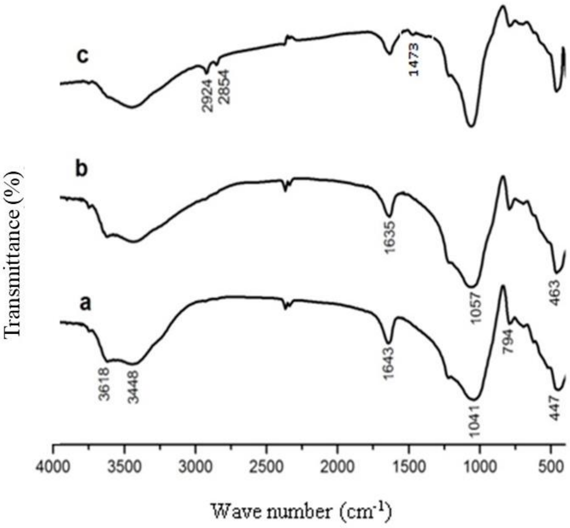

Figure 2. FT-IR Spectral (a) natural zeolite, (b) activated natural zeolite, and (c) zeolite-CTAB.

Acid activation treatment increases the distance between Al atoms, thereby reducing Al–Al interactions and enhancing the mobility of Al, which results in greater vibrational intensity. This indicates that acid treatment can reduce the aluminum content of Klaten natural zeolite through a dealumination process.

The FT-IR spectra of CTAB-modified zeolite also exhibit absorption bands similar to those of natural and acid-activated zeolites; however, several new bands appear. In the modified zeolite, absorption bands at 1473 cm⁻¹, 2854 cm⁻¹, and 2924 cm⁻¹ are observed, which correspond to methylene scissoring and the symmetric and asymmetric stretching vibrations of CH_2 groups. These bands are characteristic of CTAB, indicating that CTAB has been successfully adsorbed onto the zeolite. This indicates that modification with CTAB alters the surface characteristics of the zeolite to become more organophilic, which has the potential to enhance its ability to adsorb anionic species or organic compounds.

Table 2. Characteristic absorption bands of zeolite and CTAB

Zeolite absorption bands (cm^{-1})	**Vibration**
(420 – 500) cm^{-1}	bending vibration Si-O/Al-O
(680 – 850) cm^{-1}	symmetric O-Si-O and O-Al-O
(1000 – 1100) cm^{-1}	asymmetric O-Si-O and O-Al-O
(1630 – 1640) cm^{-1}	bending vibration H-O-H
(3000 – 3700) cm^{-1}	Overlap of asymmetric and symmetric H–O–H vibrations with hydroxyl groups of the zeolite
CTAB absorption bands (cm^{-1})	**Vibration**
2915 cm^{-1}	asymmetric CH
2850 cm^{-1}	symmetric CH
(1461–1476) cm^{-1}	methylene scissoring

CONCLUSION

This study successfully modified natural zeolite using the surfactant, CTAB, resulting in the zeolite surface being coated with CTAB and acquiring a positive surface charge. This modification was evidenced by a shift in the d-spacing toward lower 2θ values by approximately 0.5° compared to natural zeolite and acid-activated natural zeolite. In addition, the FTIR spectra exhibited absorption bands at 1473 cm^{-1}, 2854 cm^{-1}, and 2924 cm^{-1}, which are characteristic of CTAB, confirming its presence on the modified zeolite.

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