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Adsorption Study of Crystal Violet Using Natural Zeolite of Indonesia

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ABSTRACT. Indonesia has a lot of Natural Zeolite potential, one of which is Ende's natural zeolite(EZ) from Nusa Tenggara Timur Province, which is known to have the main components of mordenite, clinoptilolite, and quartz. EZ can be used as an adsorbent for Crystal Violet(CV) dye waste, which is often used as a dye in the textile industry, veterinary medicine, and dermatology, that can be teratogenic, carcinogenic, and mutagenic. EZ were then tested under three conditions: 100 mesh size(EZ), nano-modification with planetary milling using alumina balls (200 rpm/12 hours)(EZN), and recrystallization(EZM). Each EZ were characterized by XRF, FTIR, SEM, XRD, and Zeta Potential measurements. Optimum conditions for CV adsorption using 0.01 gram of each EZ were achieved at pH 6 and a contact time of 90 min using a volume of 10 mL solution, with the batch method. The CV adsorption process followed the pseudo-second-order adsorption kinetics model and the Langmuir isotherm model with a maximum adsorption capacity of 151.327 mg/g and an R^2 value = 0.9635 for EZ, 74.166 mg/g and an R^2 value = 0.9128 for sample EZN and a capacity the maximum adsorption was 311.357 mg/g and the R^2 value = 0.9752 for sample EZM. The thermodynamic study indicated that the process was spontaneous and endothermic and that the degree of orderliness of the system increased.

Keywords: Adsorption, Crystal Violet, Mechanochemistry, Natural Zeolite.

INTRODUCTION

Crystal violet (CV) compounds are non-biodegradable properties because they contain complex aromatic compounds that are difficult for microbes to decompose. In addition, dye waste is harmful to human health and biota living in polluted water bodies. In general, these organic compounds are teratogen, carcinogenic, and mutagenic, thus posing a serious threat to human health. (Ferreira et al., 2015a) Owing to poor biodegradability, approximately 20% of the total dyes remain in the effluent during the production process, which is not in accordance with the Environmental Health Law in Indonesia. Therefore, various methods have been developed to remove CV dyes, especially from water/wastewater (Singh et al., 2011). In previous studies, various methods have been developed to remove dyes from wastewater, including photocatalyst methods, solvent extraction, membrane filtration, ion exchange, electrochemistry, adsorption, and bio removal. However, the adsorption technique is considered a more effective liquid dye waste treatment because it is simpler, economical, and environmentally friendly (Zhou et al., 2014). One alternative adsorbent is the utilization of Indonesia's natural resources, one of which is the use of Indonesian natural zeolite, which will contribute to natural development. Natural zeolite is very abundant in Indonesia, and from research data and exploitation showing as many as 447,490,160 tons of zeolite deposits in 20 locations (Kusdarto, 2015; Razzak et al., 2013).

One of the natural zeolites that can be used as adsorbents is natural zeolite originating from the Ende district, Nusa Tenggara Timur province. Ende's zeolite (EZ) are light-dark green in color, fine-grained, compact, fractured, layered, and contain clinoptilolite and mordenite minerals, and the results of XRD analysis showed that EZ is a mixture of clinoptilolite and mordenite. This is evidenced by the characteristic peaks in the Joint Committee on Powder Diffraction Standard (JCPDS) data with high intensities appearing at angles of 25.60°, 26,25°, and 27.67° for mordenite, and the clinoptilolite intensity appearing at angles of 9.74°,13.38°, and 29.07° (Dala Ngapa & Embu Ika, 2022; Kurniawan et al., 2020).

Ende zeolite (EZ), which is dominated by clinoptilolite, has a three-dimensional structure and pores or spaces that can be filled with cations, either to adsorb metal cations or organic compounds (Atikah, 2017). CV is also a known dye as basic violet 3, gentian violet, and methyl violet 10B. Its IUPAC name is N-[4-[bis[4-dimethylamino)-phenyl]-methylene] -2,5-cyclohexadien-1-ylidine]-N-methylmethanaminium chloride (molecular formula $C_{25}H_{30}N_3Cl$ and molecular weight 407,98), and it belongs to the class of triarylmethane dyes. It is a

cationic dye whose absorption maximum ranges from 589 nm to 594 nm. CV is used as a pH indicator (yellow to purple with a transition at pH 1.6) (Mittal et al., 2010). As the EZ surface is negatively charged, the reactive and cationic dye is easily captured on the adsorbent surface by electrostatic attraction between the dye molecules and adsorbent thus maximize the adsorption (Sultana et al., 2022).

EXPERIMENTAL SECTION

Materials

Natural Zeolite from Ende district, Nusa Tenggara Timur province, NaOH 50% was analytical reagents and purchased from Sigma Aldrich. SiO_2 was analytical reagents and purchased from HJSIL®. $NaAlO_2$ and Crystal Violet (gentian violet) was all analytical reagents and purchased from Merck. Deionized water was used in all experiments.

Preparation of Ende Zeolite (EZ)

Ende natural zeolite exists in the form of boulders with an average size of 10-15 cm Indonesia (Dala Ngapa & Embu Ika, 2022). The natural zeolite was then crushed using a jaw crusher to form flakes, which were sieved through a 100 mesh sieve. The zeolite powder, which was passed through a 100 mesh sieve was then separated and milled using a Planetary Mill (Retsch PM 400). Natural zeolite powder (300 g) was ground in a 500 mL planetary container by adding balls made of alumina measuring 20 – 120 mm with a mass of 1:2 at a speed of 200 rpm for 6 and 12 h. Thus, nanoparticle zeolite later called Ende's zeolite nano (EZN). The particle size of EZN was then characterized using the Particle Size Analyzer (PSA) instrument.

Modification of Nano Ende Zeolite (EZM)

1 g of nano-zeolite, was added to 0.1175 g of $NaAlO_2$, and 0.75 g of 50% NaOH in a beaker (mixture 1). While 1.75 g of SiO_2 (fumed silica) was mixed with 22.5 ml H_2O and stirred in a mortar (mixture 2). The two mixtures were stirred until smooth and then placed in an autoclave and oven at 170°C for 24 h. The synthesized zeolite was filtered using a Buchner funnel and dried in an oven (Fajar et al., 2020).

Characterization of Materials

The crystal structures of zeolite were characterized using X-ray diffraction (XRD). The XRD patterns were collected on a Bruker D8 Advance diffractometer using Cu Kα (λ = 1.5418) as the X-ray beam with Ni as a filter (Kadja et al., 2021). XRD diffractograms were recorded from 2θ = 5° to 50° at intervals of 0.02°. The degree of crystallinity (α) of the sample was determined by calculating the relative peak intensity of the zeolite before and after recrystallization, as follows (Kurniawan et al., 2017) : The chemical compositions of the samples were calculated using EDAX X-ray fluorescence (XRF) analysis. Measurements were performed using a PANalytical Axios MAX spectrometer with a voltage of 40 kV (Kadja et al., 2020). Also, a Scanning Electron Microscope (SEM), was used to observe the microstructure at 10,000x magnification (Rohayati et al., 2017).

$$\text{Crystalinity} = \frac{\text{Number of peak areas of sample after recrystallization at } 2\theta\ (5°\text{-}35°)}{\text{Number of peak areas of initial sample at } 2\theta(5°\text{-}35°)} \quad (1)$$

The surface area, total pore volume, and pore size distribution were determined volumetrically by the physisorption of nitrogen at a normal boiling point temperature (77.4 K) in static mode using a Micromeritics Gemini VII Surface area and porosity instrument. To calculate the specific surface area, the Brunauer–Emmett–Teller (BET) equation was applied using adsorption isotherms, while the pore size distribution was calculated from desorption using the Barret–Joyner–Halenda (BJH) model. Pretreatment (degassing) of the samples was performed under vacuum (10^{-2} Torr) at 150°C (Elaiopoulos et al., 2010). Zeta potential analyzer (HORIBA SZ-100) was used to measure the zeta potential over the pH range of 2-8.

Batch Adsorption Experiment

To investigate the adsorption capacity of the adsorbents (EZ, EZN, and EZM), the effect of the experimental parameters namely pH (4–9), contact time (1–180 min), and adsorbent dose (2–12.5 mg) on the CV removal was studied in batch mode. The stock solution was prepared by dissolving the accurate amount of CV (99%) in methanol and deionized water solutions (1:1, v:v), and the working solutions were made up by dilution. In adsorption isotherm and thermodynamic studies, 10 mg of adsorbents were added to 10 ml of CV solution with the initial concentrations ranging from 10 to 750 mg l^{-1}). The solutions were placed for 90 min at 298, 303, and 313 K to reach adsorption equilibrium, respectively. In the study of adsorption kinetics, 10 ml solution with the initial CV concentration of 10 mg l^{-1} was reacted with 10 mg of each of adsorbents in a batch experiment for various contact time (1–180 min), and the residual amount of CV in the solution was measured using UV/Vis spectrophotometer at 589nm. The removal percentage and adsorption capacity of adsorbed CV into EZ were calculated as follows:

$$q_e = \frac{(C_i\text{-}C_e)V}{m} \quad (2)$$

$$\%\ \text{Removal} = \frac{(C_i\text{-}C_e)}{C_i} \times 100\% \quad (3)$$

where q_e is the adsorption capacity ($mg.g^{-1}$), C_i is the initial concentration of CV ($mg.L^{-1}$), C_e is the equilibrium concentration of CV ($mg.l^{-1}$), V is the volume of solution (l) and m is the sorbent mass (g). (Sultana et al., 2022)

RESULTS AND DISCUSSION

Materials Characterization

XRF Analysis

The characterization was carried out to determine the elemental composition of natural zeolite adsorbents, namely by XRF analysis which is presented in **Table 1.** In the **Figure 1**, it is known that Ende's natural zeolite has a composition of the main elements of O, Si, and Al. Meanwhile, small amounts of Na, Mg, K, Ca, and Fe.

XRD Analysis

XRD analysis was carried out to determine the level of zeolite crystallinity and to identify the type of zeolite, an analysis was carried out using XRD, the results of which can be seen in **Figure 2.**

The results of XRD analysis showed that EZ were a mixture of clinoptilolite and mordenite. This is evidenced by the characteristic peaks in the (Joint Committee on Powder Diffraction Standard (JCPDS) data with high intensities that appear at angles of 25.57°, 26.61°, and 27.76° for mordenite, and the intensity of clinoptilolite appears at angles of 20.88° and 22.22°. The peaks with the highest intensity belong to mordenite, indicating that mordenite is a type of natural zeolite with high abundance (Septian & Sugiarti, 2019). Furthermore, no significant changes were observed in these three main XRD patterns, suggesting that the modification and recrystallization processes did not alter the fundamental crystal structure of the zeolite.

FT-IR Spectroscopy Analysis

Followed by characterization using IR spectroscopy to determine the physical properties of the adsorbent, with the results obtained presented in **Figure 3.**

Based on the IR spectrum, it can be observed that the appearance of the absorption band at 1052 cm^{-1} indicates an asymmetric stretching vibration of Si–O and Al–O from the alumina silicate framework. In the area of 797 cm^{-1}, the stretching vibrations of halogen compounds from C–F. In the region of 3465 cm^{-1}, the -OH stretching vibration of Si-OH. In the region of 1637 cm^{-1} is the -OH bending vibration of Si–OH. In the 451 cm^{-1} region, there is a bending vibration of Si–O and Al–O (Prasetyo & Soegijono, 2018). The spectra of EZ, EZN, and EZM showed no significant differences in peak positions or functional groups, indicating that the nano-modification and recrystallization processes did not alter the fundamental chemical structure of the zeolite framework.

Table.1 XRF Analysis of Ende Natural Zeolite Elements

Oxide	Wt%	At%
Na_2O	7,09	7,26
MgO	1,18	1,86
Al_2O_3	11,37	7,08
SiO_2	76,99	81,35
K_2O	1,54	1,03
CaO	0,94	1,06
Fe_2O_3	0,90	0,36

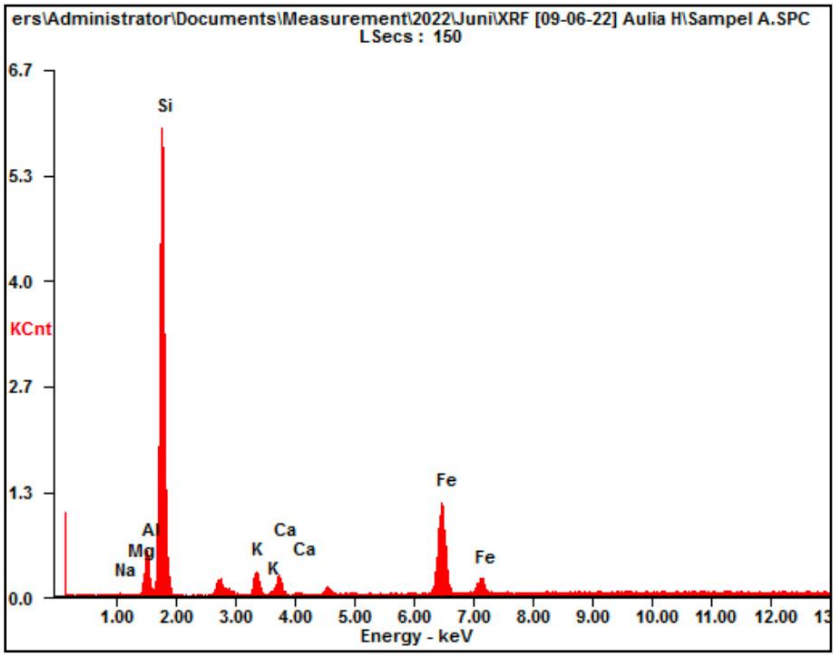


Figure 1. XRF patterns of Ende's zeolite(EZ)

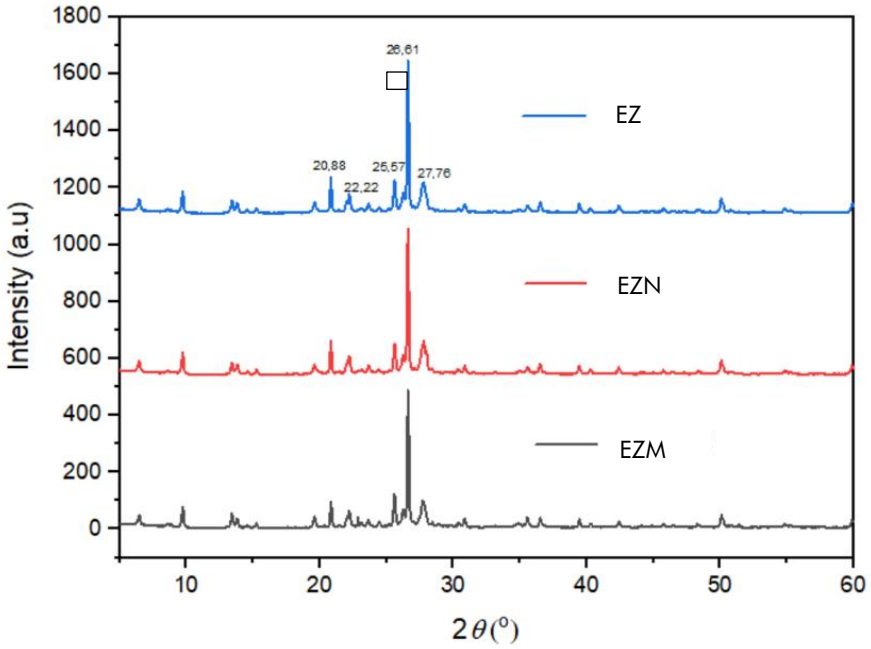


Figure 2. XRD patterns of Ende's zeolite(EZ) (a), Ende's zeolite Nano(EZN) (b), and Ende's zeolite Modification(EZM) (c)

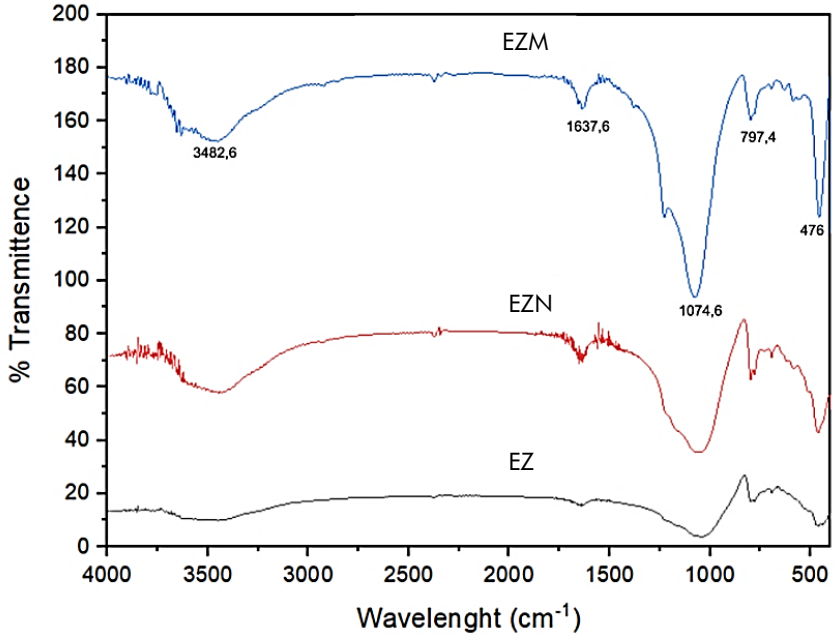
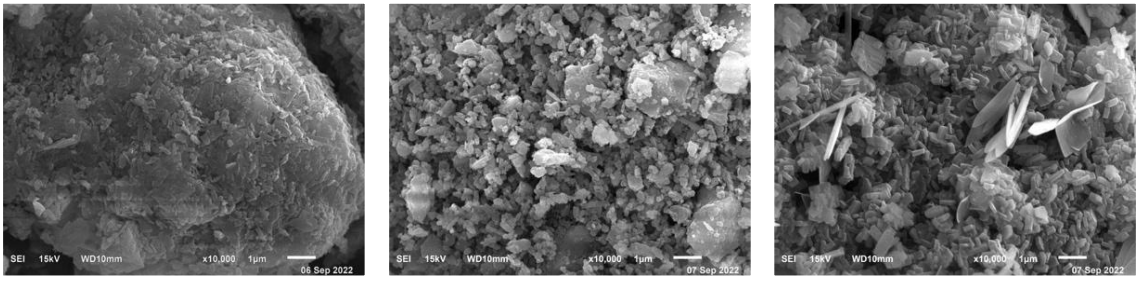


Figure 3. FT-IR Spectra of Ende's zeolite(EZ) (a), Ende's zeolite Nano(EZN) (b), and Ende's zeolite Modification(EZM) (c)

SEM Analysis

Ende's zeolite is dominated by mordenite crystals, which is a low silica zeolite that has the chemical formula $(Ca,Na_2,K_2)Al_2Si_{10}O_{24}.7H_2O$ and has an orthorhombic structure. SEM analysis was performed to determine the size and surface morphology of zeolites. SEM images of EZ, EZN, and EZM are presented in **Figure 4**.

The SEM images showed clear morphological differences among the samples. The raw zeolite (EZ) had relatively large, irregular particles with rough and compact surfaces, indicating agglomerated grains and partially blocked pores. After nano-modification (EZN), the particles became smaller and more uniform, suggesting reduced agglomeration and increased surface area. The recrystallized sample (EZM) displayed a more ordered and well-defined structure compared to EZ and EZN. The smaller particle size and more regular crystal shape indicate that the modification and recrystallization improved the zeolite's textural properties, which may enhance the accessibility of adsorption sites and improve dye adsorption.



(a) (b) (c)

Figure 4. SEM image with 10,000x magnification (a) EZ (b) EZN, and (c) EZM

PSA Analysis

To determine whether the nanomodification of Ende's natural zeolite was successful, PSA analysis was carried out to determine the average size of the zeolite that had been ground using a planetary mill. Measurements were made at a temperature of 24.9°C and a dispersing medium viscosity of 2.041 mPa·s.

From **Table 2**, it can be seen that the milled adsorbent b has a size of ± 700 nm. The results also indicate that increasing the milling time reduced the particle size, confirming that the mechanical milling process effectively decreased the particle dimensions and successfully produced a nano-modified zeolite.

It was concluded that the modification treatment of EZ produced a larger surface area and pore size. Judging from the change in size of the EZ with a surface area of 9.27 m^2/g it increased quantitatively to 44.35 m^2/g in the EZM. Also, the increase in the pore size of EZ from 2.18 nm to 4.1 nm in EZM. Generally, a certain increase in the surface area leads to better adsorption performance for nano zeolite adsorbents after modification (Hor et al., 2016)

N_2 Adsorption Analysis

The next characterization is N_2 adsorption (BET) analysis was used to determine the surface area and pore size of each adsorbent, as shown in **Table 3**.

Adsorption Activity Studies

There are 3 differences in the treatment of the zeolitic adsorbent which can be seen in **Table 4**.

Effect of pH

The adsorption behavior of Crystal Violet (CV) was evaluated as a function of solution pH because the dye is highly sensitive to environmental pH conditions. The effective pH working range for CV adsorption was found to be between pH 3 and 9. Spectral analysis showed that at pH 1–3, CV exhibited a characteristic wavelength at 629 nm. At alkaline conditions (pH 9–10), the color intensity of CV decreased, and the dye became colorless at pH 11–14. For the adsorption experiments, five pH values between 4 and 8 were selected.

Table 2. Size of the Ende's zeolite Nano(EZN)

EZN	Z-average
Milling 6 h	786,9 nm
Milling 12 h	748,4 nm

Table 3. N_2 Adsorption (BET) analysis

Adsorbent	Surface area	Pore size	Micropore volume
EZ	9,2784 m^2/g	2,1817 nm	0,00185 cm^3/g
EZN	10,7110 m^2/g	2,1852 nm	0,001638 cm^3/g
EZM	44,3517 m^2/g	4,1062 nm	0,05362 cm^3/g

Table 4 Results of treatment of each adsorbent

	EZ	EZN	EZM
Treatment	Jaw Crusher (100 mesh)	*Planetary Milling* 12 hour Ball Mass : 150 g Sample Mass : 300 g	Recrystallization
Yield	-	98,43%	67,76%

Furthermore, the adsorbent will be referred to as EZ, EZN, and EZM.

Only minor differences in adsorption capacity were observed across pH 4–8; therefore, pH 6 was chosen for subsequent analyses. Overall, the adsorption capacity did not change significantly across the investigated pH range, indicating that EZ is capable of effectively adsorbing CV over a relatively broad pH interval.

Adsorption Isotherms

The adsorption isotherm model was used to describe the distribution of adsorbate between the solid adsorbent and the solution at equilibrium and constant temperature. This model helps interpret and predict the adsorption mechanism. The most commonly applied isotherm models include Langmuir, Freundlich, Sips, and Temkin. In this study, non-linear fitting was employed because several previous studies have reported that linearization of isotherm equations can introduce errors and lead to inaccurate interpretation, as indicated by plots that do not form a true straight line. With the development of computer-based analysis, model curves can be fitted directly to experimental data to obtain more reliable parameters (Tonk & Rápó, 2022). Prior to isotherm analysis, the effect of contact time was also evaluated. The adsorption performance improved and reached its optimum at approximately 90 minutes, indicating that equilibrium was approached within this period; therefore, this contact time was used for further adsorption experiments. The non-linear fitting results of the adsorption models are presented in **Table 5.**

Seeing from the correlation coefficient (R^2) data that has been tabulated in **Table 5.** experimental results for the adsorption of CV dye following the Langmuir isotherm model, as showed at **Figure 6.** The q_{max} value indicates the amount of absorbed adsorbents per gram of adsorbent, if seen from the q_{max} of the EZM's adsorption capacity is greater compared to EZ, and EZN. This is caused by an increase in the volume of micropore of EZM. The higher volume of micropores of the recrystallized parent is due to the conversion of the distillation phase into the mordenite crystal. (Kurniawan et al., 2017).

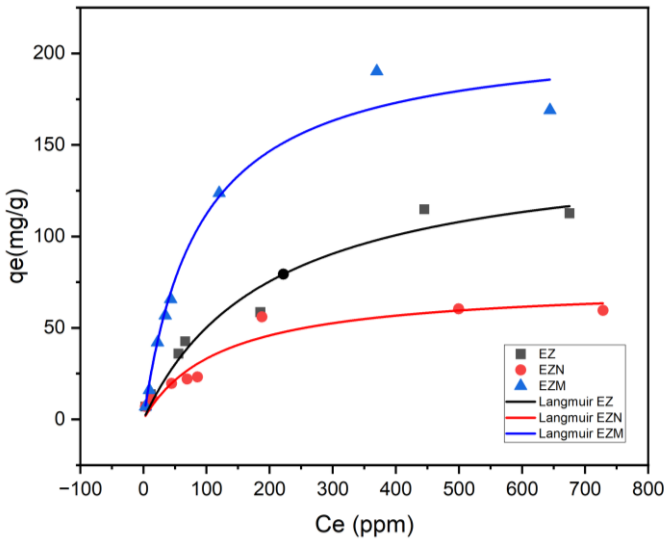


Figure 6. Non-fitting Langmuir Isotherm curve of EZ, EZN, and EZM

Table 5. Isotherm adsorption parameters

Model	Parameter	EZ	EZN	EZM
Langmuir	R^2	0.9635	0.9128	0.9752
	K_L (L mg^{-1})	0.000497	0.0081	0.00728
	q_{max} (mg g^{-1})	151.327	74.166	311.357
Freundlich	R^2	0.9595	0.8641	0.8900
	K_F (mg g^{-1})(L mg^{-1})	5.2042	5.0487	14.2066
	n	2.0661	2.5584	2.4538
SIPS	R^2	0.8787	0.8957	0.8295
	K_S (L mg^{-1})	1.2902	0.0068	3.7498
	n	103.54	0.9107	89.701
	q_{max} (mg g^{-1})	76.8223	74.923	148.984
Temkin	R^2	0.8493	0.8061	0.9208
	K_T (L g^{-1})	0.1951	5.0487	0.1971
	b_T(J mol^{-1})	21.165	11.321	37.293

The value K increasing indicates that the adsorption process is running better. Langmuir's isotherm model assumes that the adsorption runs homogeneously, the process occurs on a monolayer layer and assumes adsorbents occupy one side of the absorbent and there is no more adsorbing process to run on that side.

Adsorption Kinetics

Adsorption kinetics is a very important parameter in the design of the adsorptive system and is necessary to select the optimal operating conditions for the batch adsorption process. (Ferreira et al., 2015b). The kinetic model of the adsorption kinetics of the CV dye solution on the natural zeolite adsorbent was tested using the pseudo-first-order one using **Equation 4**, and pseudo-second-order kinetic models using **Equation 5**.

$$\log(q_e - q_t) = log q_e - \frac{k_{ad}}{2{,}303} \times t \quad (4)$$

$$\frac{1}{q_t} = \frac{1}{k_2\, q_e} + \frac{t}{q_t} \quad (5)$$

Based on the data of the correlation coefficient (R^2) in **Table 6**, it can be concluded that the kinetic model of pseudo-second-order is more accurate to describe the adsorption of CV dyes by the three adsorbents. Similar results were also obtained in previous studies (Jumaeri et al., 2017), which explains that the adsorption of CV dyes is best described by the kinetical model of the pseudo-second-order. According to **Figure 7,** the plot of q_t versus $t^{1/2}$ give a straight line for the respective model to be applicable.

Table 6. Adsorption kinetic parameters

	Pseudo Orde 1		
	R^2	q_e (mg/g)	k_1 (min^{-1})
EZ	0.7772	6.2105	0.0114
EZN	0.6923	4.1161	0.008
EZM	0.9561	15.881	0.0079
	Pseudo Orde 2		
	R^2	q_e (mg/g)	k_2 ($g.mg^{-1}\ min^{-1}$)
EZ	0.9978	7.2034	0.189
EZN	0.9997	8.1462	0.1127
EZM	0.9954	8.9079	01042

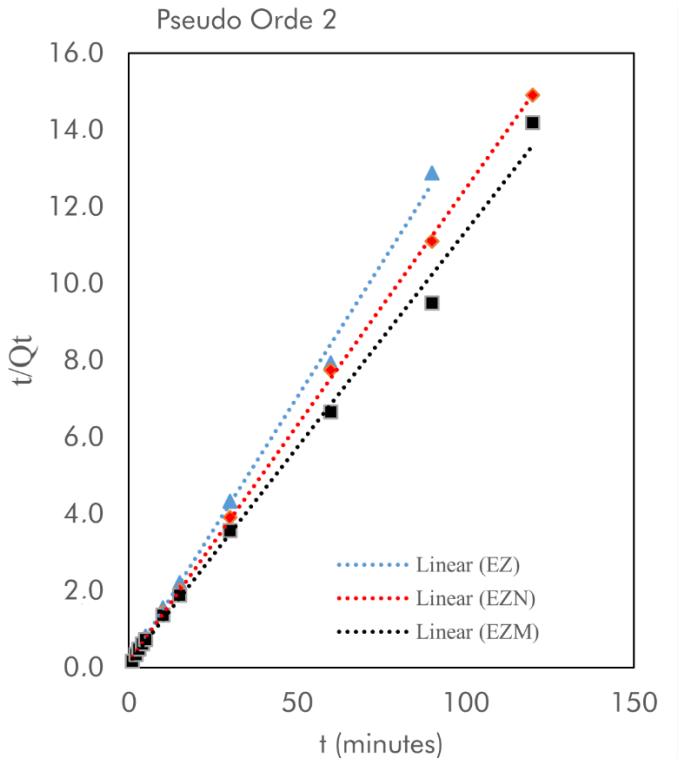


Figure 7. Curve *fitting* pseudo-second-order

This is explained despite the mechanism of adsorption in complex zeolites due to the structure of pores, surface loads inside and outside, heterogeneity and imperfection of crystals on their surfaces, but the zeolite's properties are mainly related to its ability as an ion exchange (Calzaferri et al., 2000).

Thermodynamic Studies

Adsorption thermodynamics can be used to provide information about the absorption mechanisms that occur in adsorbents and adsorbents based on the thermodynamic parameters ΔH°, ΔS°, and ΔG°. While the thermodynamic equilibrium constant (K_c) can be calculated using the Langmuir constant (K_L),

$$K_c = Mw \; x \; 55{,}5 \; x \; 1{,}00 \; x \; K_L \qquad (6)$$

Thermodynamic parameters can be derived from the plot of the value of ln K_c versus 1/T as seen in **Figure 8.** Thermodynamic parameters can be obtained from the linear equations produced by slope graph and intercept of the van't Hoff plot using ln K_c versus 1/T. The results of the calculation of thermodynamic parameters are shown in **Table 7.**

According to **Table 7**, a positive ΔH° value confirms that the reaction occurs endothermically. This indicates the occurrence of a monolayer adsorption which corresponds to the fact that the absorption of CV dyes by natural zeolites is a process of ion exchange. (Annadurai et al., 2008).

A positive ΔS° value indicates an increase in the degree of freedom derived from the adsorption of the CV dye molecules. This can be explained by several possibilities, one of which is due to the release of inorganic cations from the hydrated CV dye which leads to an increased overall entropy (Rytwo et al., 2006). Another source explains that the increase in Entropy can also be caused by the appearance of irregularities occurring in a solid interface that reflects the principle of additional translational entropies that occur because the solvent molecule that was initially absorbed into the natural zeolite pores was replaced by the CV dye. (Almeida et al., 2009).

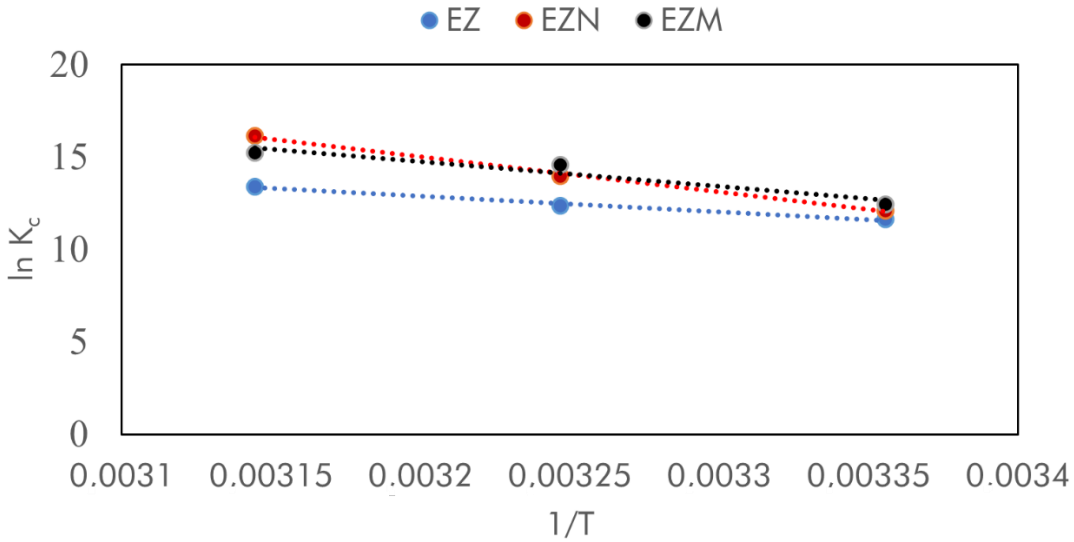


Figure 8. Plot *ln Kc* vs 1/T

Table 7. Thermodynamic parameter

T (K)	ΔG (kJ mol^{-1})	ΔH° (kJ mol^{-1})	ΔS° (J K^{-1} mol^{-1})
	EZ		
298	-28.817	70.191	331.80
308	-31.723		
318	-35.471		
	EZN		
298	-30.027	158.96	633.61
308	-35.819		
318	-42.723		
	EZM		
298	-30.865	110.757	477.07
308	-37.349		
318	-40.331		

A positive ΔS° indicates the occurrence of an increase in the degree of freedom derived from the ΔH° value and a positive ΔS° resulting in a negative ΔG° value, which indicates that the adsorption process is going on spontaneously, this parameter is meant as the energy required for a CV dye to bind to a zeolite.

CONCLUTIONS

Regarding on the adsorption of Crystal Violet, natural zeolite from Ende showed adsorption capacity much greater once it modified by mechanochemistry. The adsorption of Crystal Violet depending on the pH and the maximum adsorption occurred at pH 6. The adsorption isotherm study revealed that the adsorption of Crystal Violet followed a monolayer adsorption mechanism on a homogeneous surface, as described by the Langmuir model. The kinetics study indicated that the adsorption process followed the pseudo-second-order kinetic equation. The thermodynamic data showed that the adsorption process on the surface of Ende's Zeolite was spontaneous and exothermic. These results indicate that nano-modified and recrystallized natural Ende zeolite functions as an efficient adsorbent for removing Crystal Violet dye from aqueous media.

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DECLARATIONS

Conflict of interest The authors attest that there are no conflict of interest and financial, personal, or other relationships with other people, laboratories, or organizations worldwide.

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