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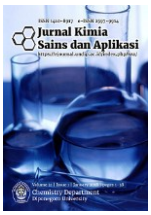

Zeolite/*Hydrilla verticillata* Composite for the Adsorption of Naphthol Blue Black Dye

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Abstract

In this study, a zeolite/*Hydrilla verticillata* composite adsorbent was prepared and characterized, and its performance was evaluated for the adsorption of Naphtol Blue Black (NBB) dye. Natural zeolite sourced from Bayah, Banten, was used as the base material. The composite was synthesized using the wet impregnation technique. Characterization was conducted using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). FTIR analysis showed absorption bands at 2919 cm^{-1}, 514 cm^{-1}, and 1014 cm^{-1}, indicating the successful formation of the zeolite/*Hydrilla verticillata* composite. XRD results revealed that the Bayah natural zeolite contains modernite and heulandite crystal phases, with an average crystallite size of 9.3 nm calculated using the Scherrer equation. SEM images indicated that the zeolite surface was largely covered by *Hydrilla verticillata* particles. The adsorption experiment showed that the composite adsorbent achieved a 34.07% removal efficiency of NBB dye. The adsorption process followed the Freundlich isotherm model, with an R^2 value of 0.9995 and a maximum adsorption capacity of 13.59 mg/g.

1. Introduction

Indonesia is a developing country in the fields of industry and technology. The rapid growth of these sectors has led to a significant increase in environmental pollution affecting the air, water, and soil [1]. Among the growing industries in Indonesia, the textile industry plays a crucial role, producing high-quality products that contribute substantially to the country's foreign exchange earnings [2]. Each year, approximately 800,000 tons of synthetic dyes and pigments are used in the textile industry, with 10–15% of these dyes being discharged into water bodies. This discharge causes environmental pollution and directly and indirectly threatens human health [3, 4].

One of the dyes commonly used in the textile industry is naphthol blue black (NBB), which contains azo functional groups (-N=N-) and benzene rings. These chemical structures make NBB highly soluble in water and resistant to biological degradation [3, 5]. Small and medium-sized enterprises (SMEs), particularly in the batik sector, are significant sources of dye-laden wastewater with high concentrations of synthetic dyes that can negatively impact the surrounding environment [6].

Reactive dyes, such as NBB, methylene blue, rhodamine B, and congo red, are generally derived from azo and benzene compounds. These dyes are non-biodegradable and readily dissolve in water [5]. Wastewater containing hazardous and toxic synthetic dyes can degrade environmental quality, particularly if discharged without proper treatment. It can disrupt photosynthesis, harm aquatic organisms, and pose serious risks to human health, including carcinogenic and genotoxic effects [1]. Due to their stability, synthetic dye compounds are difficult to degrade naturally, making them particularly harmful to aquatic environments.

Considering the environmental hazards posed by NBB dye, wastewater containing this compound must be treated before being discharged into the environment. Various treatment methods have been developed for dye-contaminated wastewater, including adsorption [7], coagulation [8], electrodecolorization [9], and photocatalysis [10]. Among these, adsorption has been reported as an effective technique for reducing dye concentrations in water. The advantages of the adsorption method include safety, the absence of harmful side effects, operational simplicity, efficiency, ease of use, and low cost [11]. Adsorption involves the use of adsorbent materials to remove hazardous substances from wastewater. Commonly used adsorbents include activated carbon, silica, chitosan, zeolite, bentonite, and clay [12].

Natural zeolite is an abundant mineral resource characterized by low production costs, good thermal stability, and a high surface area [13]. Globally, particularly in Banten Province, natural zeolite is available in significant quantities. In Banten, the estimated reserves of natural zeolite are approximately 34 million m^3, or around 68–81.6 million tons [14], with the main element composition of O, Si, Al, and the composition of other elements, namely Fe, K, Ca, and Mg [14]. In Bayah District, Lebak Regency, Banten, natural zeolite is typically mined as a multipurpose industrial mineral [15]. However, its potential applications in chemical and physical processes remain largely underutilized [16].

Bayah natural zeolite shows potential as an adsorbent for reducing pollutant concentrations in wastewater. The primary minerals in Bayah zeolite include mica, plagioclase, and quartz, with mordenite and clinoptilolite identified as the dominant zeolite types [17]. Zeolite can be further developed as an adsorbent material due to its ion exchange capabilities, molecular sieving properties, and catalytic activity [18]. Several studies have explored the use of natural zeolite in dye wastewater treatment, including zeolites from Sukabumi [19], Ende [20], Wonosari [21], and Bandung [22]. Ngapa and Ika [20] reported that natural zeolite, primarily composed of the mordenite phase, can be utilized as an adsorbent for methylene blue and methyl orange dyes.

Hydrilla verticillata is an aquatic plant known for its adaptability and ease of growth in various environmental conditions. It has been studied and recognized as a potential biomaterial for biosorption. Plant-based biosorbents serve as alternative adsorbents in wastewater treatment, owing to active functional groups facilitating adsorption. Several plant species have been explored for their ability to treat wastewater containing dyes and heavy metals, such as *Ulva lactuca* [23], yellow *Peltophorum pterocarpum* (copperpod flowers) [24], and *Hydrilla verticillata* [25, 26]. *Hydrilla verticillata* exhibits a strong capacity to absorb heavy metals, making it a suitable candidate for adsorbent applications [27]. It has also been used as a biosorbent material for congo red dye [28].

The high binding capacity of *Hydrilla verticillata* is attributed to the presence of polysaccharides, proteins, and lipids in its cell walls, which contain functional groups such as amino, hydroxyl, carboxyl, and sulfate, serving as active adsorption sites [25]. Furthermore, *Hydrilla verticillata* contains oxygen and carbon elements, as evidenced by the presence of carboxyl groups in its main structural components [29]. Given its composition, *Hydrilla verticillata* is considered effective for treating wastewater containing reactive dyes such as NBB.

Considering the advantages of zeolite and *Hydrilla verticillata* as adsorbents, this study aims to develop a composite material combining the two. A composite is a combination of two or more materials designed to enhance the properties or performance of the individual components [30]. Although zeolite has been widely used as an adsorbent, it has limitations in dye adsorption due to its relatively low pore volume and surface area. Incorporating plant-based biomaterials can help address this limitation by introducing additional active functional groups. Biomaterials are capable of attracting pollutants through the presence of functional groups such as carboxylate, hydroxyl, amino, carbonyl, phosphate, and sulfonate [31].

Previous studies have developed composite adsorbents using combinations such as zeolite/marine algae [32], zeolite/*Acanthopora spicifera* [31], and green seaweed/zeolite [33], which have shown improved dye adsorption capabilities. Other examples of zeolite-based composites include zeolite modified with organic materials such as chitosan [34, 35] and nanocellulose derived from banana leaves [36].

This study aims to enhance the adsorption capacity of zeolite by incorporating plant-derived biomaterials, specifically *Hydrilla verticillata*. Due to its abundance, low cost, and ease of availability, the zeolite/*Hydrilla verticillata* composite presents a promising alternative adsorbent for dye wastewater treatment. The composite is expected to significantly improve zeolite's effectiveness in reducing the concentration of NBB dye in wastewater.

2. Experimental

2.1. Tools and Materials

The tools used in this study included laboratory glassware, a 100-mesh sieve, hotplate magnetic stirrer, an ultrasonicator, a digital analytical balance, a Thermo Scientific Heratherm OGS 60 oven, an Eppendorf 5430 R centrifuge, a JLabTech Daihan Labtech Co., LTD shaking incubator, a Thermo Scientific Orion AquaMate 8100 UV-Vis spectrophotometer, a Bruker Alpha II Eco ATR-FTIR, a Rigaku Miniflex 600 XRD, and a Thermo Scientific Phenom Pure Gen 6 SEM. The materials used were naphthol blue black (Amido Black 10B) dye, distilled water, Bayah natural zeolite, and *Hydrilla verticillata*.

2.2. Preparation of Zeolite/*Hydrilla verticillata* Composite

The zeolite/*Hydrilla verticillata* composite was prepared using the wet-impregnation technique [32, 37].

Initially, natural zeolite was ground and sieved through a 100-mesh screen. *Hydrilla verticillata* was thoroughly washed with clean water, cut into small pieces, and oven-dried. Subsequently, 1 g of ground zeolite and 1 g of dried *Hydrilla verticillata* were combined and stirred magnetically at 500 rpm for 60 minutes. To enhance the impregnation of the plant material onto the zeolite surface, the suspension was further treated by ultrasonication for 180 minutes. The resulting composite was filtered, repeatedly washed with deionized water, and dried in an oven at 60°C for 24 hours.

2.3. Sample Characterization

Sample characterization was performed using FTIR, XRD, and SEM analyses. FTIR-ATR (Attenuated Total Reflectance) was employed to identify the functional groups present in the sample. XRD analysis was conducted to determine the crystalline phases and degree of crystallinity. The X-ray source used for XRD was a Cu anode with a wavelength of 1.54 Å. SEM analysis was used to examine the surface morphology of the sample. SEM imaging was performed at a magnification of 10,000×, with a working distance ranging from 7.268 mm to 7.546 mm, and the accelerating voltage set to 5 kV.

2.4. Adsorption Study

The study used three types of adsorbents—zeolite, *Hydrilla verticillata*, and the zeolite/*Hydrilla verticillata* composite—to evaluate the effectiveness of the composite in the adsorption of NBB dye. In each experimental variation, 20 mL of NBB solution was used. To assess the adsorption efficiency of the zeolite/*Hydrilla verticillata* composite, 20 mg of adsorbent was added to 20 mL of a 10 ppm NBB solution. The concentration of the filtrate after adsorption was measured using a UV-Vis spectrophotometer at a wavelength of 622 nm (λ = 622 nm). The percentage of pollutant removal, or adsorption efficiency, was calculated using Equation (1) [38].

$$\%Removal = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

Where, C_0 is the initial dye concentration (mg/L), and C_e is the equilibrium concentration after adsorption (mg/L).

The adsorption isotherms in this study were modeled using the Langmuir and the Freundlich isotherm equations. The Langmuir isotherm model is expressed by Equation (2) [39].

$$\frac{C_e}{Q_e} = \frac{1}{K_L Q_{max}} + \frac{C_e}{Q_{max}} \quad (2)$$

Where, Q_e is the adsorption capacity at equilibrium (mg/g), C_e is the equilibrium concentration (mg/L), Q_{max} is the maximum adsorption capacity (mg/g), and K_L is the Langmuir constant. The Freundlich isotherm model is described by Equation (3) [40].

$$\log Q_e = \log K_F + \frac{1}{n} \log C_e \quad (3)$$

Where, Q_e is the adsorption capacity at equilibrium (mg/g), C_e is the equilibrium concentration (mg/L), K_F is the Freundlich constant indicating adsorption capacity, and $1/n$ is the heterogeneity factor.

3. Results and Discussion

3.1. Characterization of Zeolite/*Hydrilla verticillata* Composite

3.1.1. FTIR

FTIR characterization was conducted to identify the functional groups present in zeolite, *Hydrilla verticillata*, and the zeolite/*Hydrilla verticillata* composite adsorbents. The FTIR spectra of zeolite (Z), *Hydrilla verticillata* (HV), and the zeolite/*Hydrilla verticillata* composite (ZHV) are presented in Figure 1.

For the zeolite sample, a prominent absorption band was observed at 1007 cm^{-1}, corresponding to the Si–O stretching vibration, which shifted to 1014 cm^{-1} in the ZHV composite [31]. Absorption bands at 786 cm^{-1} and 909 cm^{-1} are attributed to Si–O–Al linkages and octahedral Al–OH bending vibrations, respectively. A peak at 519 cm^{-1}, associated with the Si–O–Si stretching of zeolite, shifted slightly to 514 cm^{-1} in the composite. The absorption peaks within the 400–800 cm^{-1} region are generally related to metal oxide vibrations [32].

Figure 1 shows the FTIR spectrum of *Hydrilla verticillata*, confirming the presence of amine, carboxyl, and other functional groups. The FTIR results indicate a carbonyl (C=O) stretch from ester groups at 1611 cm^{-1} [25]. The bands at 2919 cm^{-1} and 2851 cm^{-1} correspond to the symmetric stretching vibrations of CH_2 and CH_3 groups, typically associated with alcohol compounds [41]. The presence of carboxyl groups is supported by absorption bands at 1544, 1402, and 1314 cm^{-1}.

Additionally, amine groups are identified by the absorption band at 1236 cm^{-1}, attributed to N–H bonding. A broad absorption at 3269 cm^{-1} corresponds to O–H stretching, likely due to asymmetric N–H_2 stretching in amine groups. The absorption band at 1019 cm^{-1} is associated with CH_3 wagging vibrations, originating from various functional groups present in the plant cell wall. Additionally, a band at 538 cm^{-1} indicates the presence of C–N–S scissoring vibrations, commonly found in polypeptide structures [42].

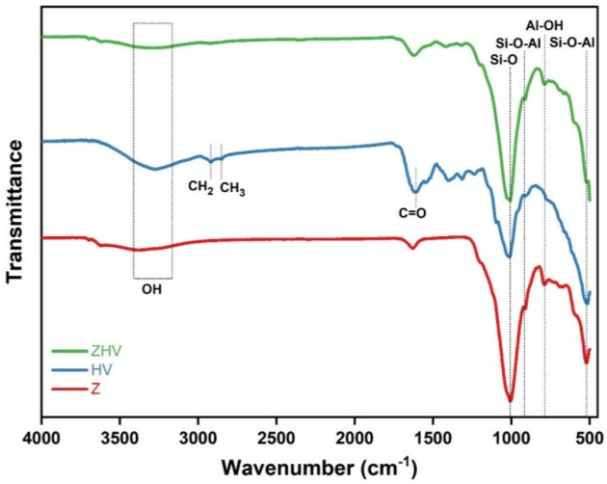

Figure 1. FTIR spectra of zeolite (Z), *Hydrilla verticillata* (HV), and the zeolite/*Hydrilla verticillata* composite (ZHV)

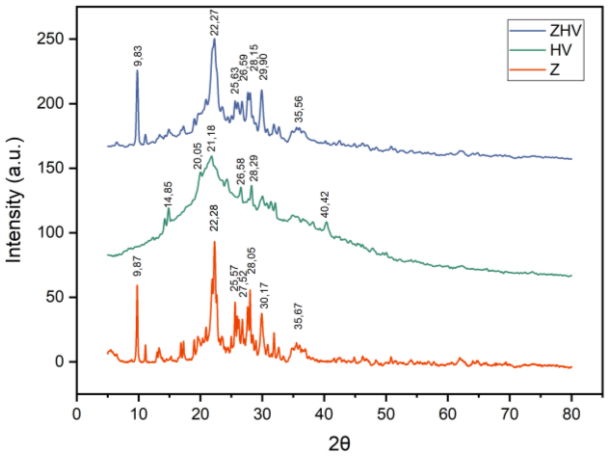

Figure 2. XRD diffractograms of zeolite (Z), *Hydrilla verticillata* (HV), and the zeolite/*Hydrilla verticillata* composite (ZHV)

The FTIR results exhibit characteristic peaks corresponding to *Hydrilla verticillata*. Additionally, the absorption peaks associated with zeolite are also present in the FTIR spectrum of the zeolite/*Hydrilla verticillata* composite, indicating successful impregnation of the plant material into the zeolite matrix. In the composite spectrum, the absorption band at 2919 cm^{-1} corresponds to the CH_2 stretching vibration, while the shifts from 519 cm^{-1} to 514 cm^{-1} and from 1007 cm^{-1} to 1014 cm^{-1} suggest interactions between the functional groups of *Hydrilla verticillata* and the active sites on the zeolite surface [33, 37].

Overall, the absorption peaks of the zeolite/*Hydrilla verticillata* composite appear within the range of 1000–1630 cm^{-1}. The observed shifts and the disappearance of certain peaks further support the formation of the composite material [33]. The presence of characteristic peaks for Si–O–Al, Al–OH, Si–O–Si, as well as amine and carboxyl groups in the FTIR spectrum of the composite (Figure 1) indicates the existence of active functional sites that may enhance dye-binding capacity.

3.1.2. XRD

XRD characterization was conducted to identify the crystalline phases present in the samples and to assess their degree of crystallinity. The resulting diffraction patterns were analyzed by comparing the observed peaks with standard reference data using the Match! software. The analysis indicates that the Bayah natural zeolite sample corresponds to the mordenite and heulandite phases. The diffraction pattern matching results for the heulandite phase are presented in Figure 2.

In Figure 2, the XRD peaks of zeolite minerals are observed at 2θ values of 9.87°, 22.28°, 25.57°, 27.52°, 28.05°, 30.17°, and 35.67°, which are consistent with the findings of Dryaz *et al.* [43] and Hamd *et al.* [32]. The XRD results reveal that Bayah natural zeolite exhibits mordenite crystal phases, specifically at 2θ values of 22.28°, 25.57°, and 27.52°, and a heulandite crystal phase at 22.28°, 28.05°, and 36.21°.

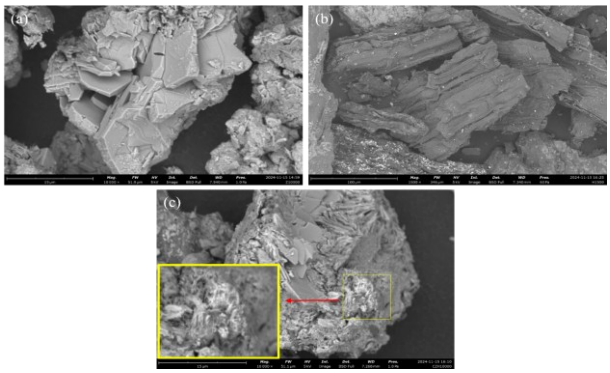

Figure 3. SEM images of (a) zeolite, (b) *Hydrilla verticillata*, and (c) zeolite/*Hydrilla verticillata* composite

The XRD diffractogram of *Hydrilla verticillata* shows main peaks at 14.85°, 20.05°, 21.81°, 26.58°, 28.29°, and 40.42°. The diffractogram of the zeolite/*Hydrilla verticillata* composite displays distinctive major peaks at around 9.83°, 22.27°, 25.63°, 26.76°, 29.90°, 31.87°, 32.72°, and 35.52°. The formation of the composite is further confirmed by the increase in the Full Width at Half Maximum (FWHM) of the 22.28° peak, from 0.22009 to 0.31281. The average crystallite size, calculated using the Scherrer equation, is 9.3 nm, confirming the nanoscale nature of the newly synthesized composite.

Based on the Scherrer equation, the crystallite size of zeolite is calculated to be 11.69 nm, while that of the zeolite/*Hydrilla verticillata* composite is 15.24 nm. This increase confirms the successful formation of the composite and indicates enhanced crystallinity, as the incorporation of *Hydrilla verticillata* promotes greater structural order. Larger crystallites generally reflect higher regularity in crystal arrangement, supporting the conclusion that the impregnation process contributes to improved crystalline quality [44]. Furthermore, the observed crystallite size confirms the nanoscale characteristics of the newly synthesized material, supporting its potential for applications requiring fine particle structures.

3.1.3. SEM

Surface morphology characterization of zeolite, *Hydrilla verticillata*, and zeolite/*Hydrilla verticillata* composites was performed using SEM. The SEM results are shown in Figure 3. In Figure 3(a), the SEM micrograph of zeolite reveals a rough surface with particle agglomeration in the form of flakes of varying sizes. The surface appears smooth with few visible pores. The flaky structure, combined with the presence of fibers, suggests that the zeolite is of the mordenite type [14].

In Figure 3(b), the SEM image of *Hydrilla verticillata* shows a hollow, irregularly folded surface with fewer pores. Figure 3(c) reflects the morphological changes resulting from zeolite treatment with *Hydrilla verticillata*. The SEM image of the composite reveals that the surface of the zeolite is almost entirely covered by *Hydrilla verticillata* particles, leading to agglomeration and the appearance of pores in some areas of the zeolite/*Hydrilla verticillata* composite.

3.2. Effectiveness of Zeolite/*Hydrilla verticillata* Composite Adsorbent, Zeolite, and *Hydrilla verticillata*

The wet impregnation technique was used to prepare the composite because it is simpler and easier than the sol-gel and coprecipitation methods. Wet impregnation involves inserting precursors into the pores of the support material, followed by stirring, which affects the surface area and composition of the composite. In this study, natural zeolite was used as the support material. Using a support material helps achieve optimum distribution in the composite, resulting in a larger surface area, high thermal stability, and increased active sites [45]. The impregnation of *Hydrilla verticillata* onto zeolite is expected to enhance the adsorptive capacity of the zeolite.

The prepared and characterized zeolite/*Hydrilla verticillata* composite was then tested for its adsorption ability in reducing the concentration of NBB dye. NBB dye solutions were prepared at various concentrations (4, 6, 8, 10, 12, 14, 16, 18, 20 mg/L), and the dye was analyzed using a UV-Visible spectrophotometer. The maximum absorption wavelength of the NBB dye was found to be λ = 622 nm. Figure 4 shows the NBB dye calibration curve. The linear regression equation obtained is y = 0.037x – 0.0055, with an R^2 value of 0.9978.

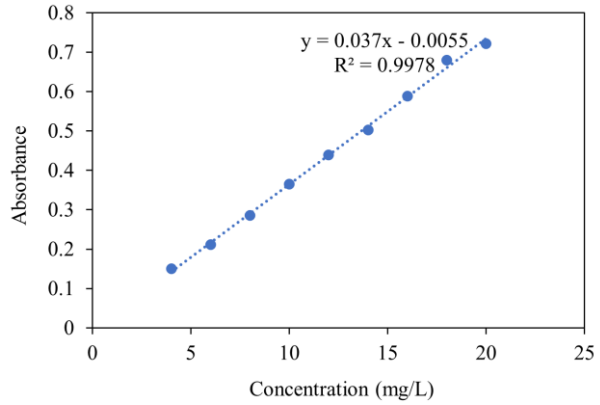

Figure 4. Calibration curve for NBB dye

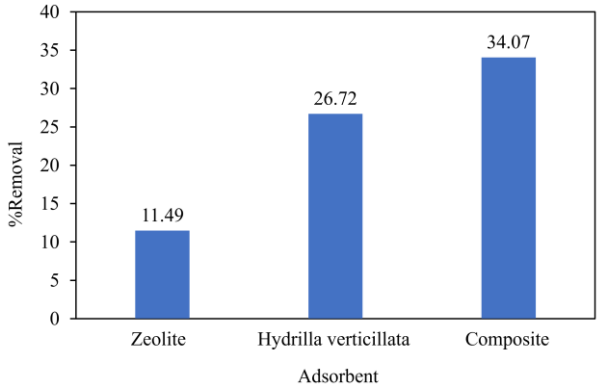

Figure 5. Adsorption of NBB dye using zeolite adsorbent, *Hydrilla verticillata*, and the zeolite/*Hydrilla verticillata* composite

In this study, the effectiveness of the zeolite/*Hydrilla verticillata* composite adsorbent was evaluated and compared with zeolite and *Hydrilla verticillata* adsorbents. Figure 5 illustrates the adsorption results for zeolite, *Hydrilla verticillata*, and the zeolite/*Hydrilla verticillata* composite. The results indicate that the zeolite/*Hydrilla verticillata* composite exhibited higher adsorption effectiveness than both zeolite and *Hydrilla verticillata*. Specifically, the zeolite/*Hydrilla verticillata* composite demonstrated the highest effectiveness for NBB dye adsorption, with the order of effectiveness being zeolite/*Hydrilla verticillata* composite > *Hydrilla verticillata* > zeolite.

The percentage decrease in NBB dye concentration during adsorption using the zeolite/*Hydrilla verticillata* composite was 34.07%. Pores and additional active sites on the composite surface, which can bind to both organic and inorganic compounds in aquatic waste, contributed to its superior performance. These active sites include acid sites, Si-O-Al, and Si-O-Si sites from the zeolite and amine, carboxyl, and polypeptide groups derived from *Hydrilla verticillata* biomass. The FTIR results further confirmed the presence of these active sites on the zeolite/*Hydrilla verticillata* composite.

The adsorption with zeolite adsorbent showed the lowest reduction efficiency, at 11.49%. This is due to the limited number of pores on the zeolite surface. The removal of impurities from zeolite cavities and the subsequent activation process can increase pore size, thereby enhancing its adsorption capacity [46]. In contrast, *Hydrilla verticillata* has pores on its surface and contains oxygen and carbon elements contributing to its ability to absorb aquatic waste, with oxygen and carbon content of 38.21% and 39.6%, respectively [29]. The decrease in NBB dye concentration during adsorption with *Hydrilla verticillata* adsorbent was 26.72%.

These results align with previous studies on zeolite/marine algae composites for reducing congo red dye concentrations, where zeolite/algae was more effective than either *Cystoseira compressa* algae or zeolite alone in a 240-minute adsorption process with an initial concentration of 20 ppm and an adsorbent mass of 20 mg [32]. The size and porosity of the adsorbent affect the surface area: larger pores provide a greater surface area, enhancing adsorption capacity. *Hydrilla verticillata*, which also has dye-absorbing properties, further increases the adsorption capacity. Adsorption is a mass transfer process on an adsorbent with a porous surface [47].

3.2.1. Isotherm Pattern and Adsorption Capacity

The adsorption mechanism can be studied by determining the isotherm pattern. The Langmuir and Freundlich isotherms are commonly used to analyze the adsorption mechanism. The isotherm model that best fits the adsorption process is selected based on the model with an R^2 value close to one [48]. Figures 6(a) and 6(b) show the curves of Langmuir and Freundlich isotherms, respectively.

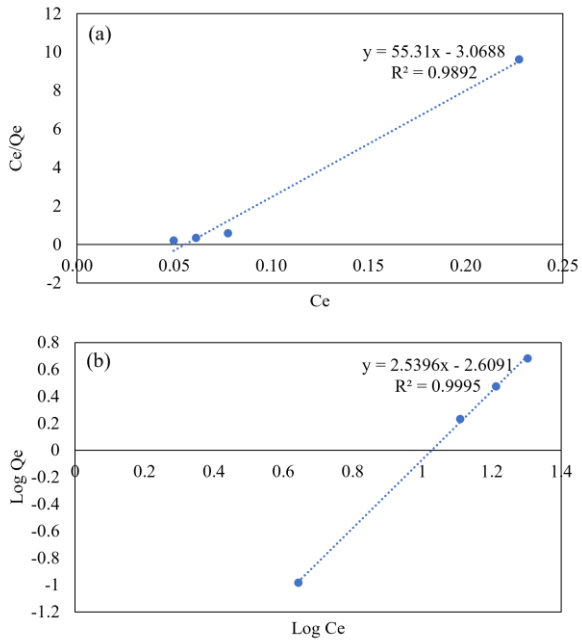

Figure 6. (a) Langmuir isotherm curve; (b) Freundlich isotherm curve

Figure 6a shows that the R^2 value for the Langmuir isotherm pattern is 0.9892, with a maximum adsorption capacity of 0.33 mg. Figure 6b shows that the R^2 value for the Freundlich isotherm pattern is 0.9995, with an adsorption capacity of 13.59 mg/g. The higher R^2 value in the Freundlich isotherm pattern indicates that the adsorption of NBB dyes with zeolite/*Hydrilla verticillata* composites follows the Freundlich isotherm model. Using natural zeolite activated with acid as an adsorbent for methylene blue and methylene orange dyes has demonstrated higher adsorption capacity due to the activation process, which removes impurities from the zeolite [20]. However, in this study, no activation was carried out on the zeolite to assess the adsorption capacity of Bayah natural zeolite without activation and modification with *Hydrilla verticillata*.

According to the Freundlich isotherm model, the adsorption process occurs through physical interactions. This suggests that a multilayer adsorption structure forms on the NBB dye adsorbed by the active surface of the zeolite/*Hydrilla verticillata* composite, which is heterogeneous [11]. The Freundlich isotherm pattern indicates that not the entire surface of the adsorbent is involved in the adsorption process, leading to multilayer adsorption due to physical interactions between the adsorbent and the adsorbate [49]. Additionally, the Freundlich model assumes that the distribution of adsorption heat and affinity is not uniform across the heterogeneous surface [50]. This model further suggests that the active sites in adsorption have high affinity, while other parts exhibit lower affinity, allowing the adsorbate to move freely and promoting multilayer adsorption [51].

4. Conclusion

Surface and pore modifications of zeolite and *Hydrilla verticillata* led to the formation of the zeolite/*Hydrilla verticillata* composite, which was evaluated as a novel adsorbent for removing NBB dye from aqueous solutions. The results demonstrated that the zeolite/*Hydrilla verticillata* composite reduced dye concentration by 34.07%. The adsorption process followed the Freundlich isotherm model, with an R^2 value of 0.9995 and a maximum adsorption capacity of 13.59 mg/g.

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