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Utilization of Clinoptilolite Zeolite to Reduce P-Phenylenediamine (PPD) Dye Content from Hair Color Wash Waste

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A B S T R A C T

Environmental pollution is a global problem that requires serious attention worldwide. Various human activities contribute to increased levels of pollutants, including liquid waste. One example is the hair colouring process, which generates waste that is harmful to both the environment and human health. This waste contains explicitly hazardous components such as synthetic dyes, for example, p-phenylenediamine dyes. Therefore, this study aims to utilise zeolite clinoptilolite (an adsorbent) for the absorption or removal of dyes in hair dye wash water waste. Based on the parameters of the dye content contained in the waste to determine the effect of adsorbent mass, contact time and concentration on the adsorption process. Determination of dye adsorption capacity using UV-VIS spectrophotometry. FTIR and XRD characterized Zeolite before and after adsorption. This study shows that adsorption lasts for 45 minutes using 0.6 grams of zeolite, with a dye sample volume of 20 mL for dye absorption. After the adsorption process at optimal conditions, the per cent adsorption of hair dye wash water waste was 81.78%. Analysis of the zeolite before and after adsorption using FTIR revealed a change in the spectrum, characterised by a shift in wavenumbers, indicating that the adsorption process had occurred. Characterization of zeolite using XRD showed that there was no change in zeolite structure. From these data, it can be concluded that the adsorption method can reduce the content of dyes in hair dye.

Contribution to Sustainable Development Goals (SDGs):
SDG 6: Clean Water
SDG 11: Good Health and Well-being
SDG 13: Industry, Innovation, and Infrastructure
SDG 15: Climate Action

1. INTRODUCTION

1.1. Research Background

Environmental pollution is a growing global concern that has become a significant issue in many countries. Various human activities, ranging from household scale to large industries, contribute to the increase of pollutant levels in the environment. Various types of pollutants, including heavy metals, dyes, pesticides, and pharmaceuticals, can contaminate the environment [1]. One of the activities that can pollute water is the hair colouring process, which produces hazardous waste that is detrimental to the environment and human health. One of the wastes generated through the hair colouring process is synthetic dyes, such as p-phenylenediamine dyes. This is due to its non-biodegradable nature, which can accumulate in the bodies of organisms, and it has a high level of toxicity [2].

Synthetic dyes contained in hair dyes are derived from aromatic amine compounds, such as P-phenylenediamine (PPD), also known as PPD dyes. The presence of these colorants can pose a threat to overall environmental sustainability. Therefore, effective measures are necessary to address the issue of dye pollution in wastewater, to protect both the environment and human health [3]. Paraphenyldiamine (PPD) compounds found in hair dyes have a benzene benzene structure that is difficult to biodegrade. These dyes can be harmful to human health, and the waste can enter water bodies, potentially even being consumed by humans. The most common hazards associated with these dyes are respiratory problems resulting from the inhalation of dye particles that interfere with breathing, watery eyes, sneezing, and asthma symptoms such as coughing [4].

Various methods have been developed to address the problem of heavy metal and dye pollution in wastewater, including ion exchange resin [5], membrane filtration [6], biosorption [7], and adsorption [8]. Among these methods, adsorption is considered to be the most effective due to its simplicity, low cost, and abundant availability [9]. However, zeolite, a crystalline alumino-silicate microporous material, has a three-dimensional structure with interconnected pores, making it more efficient than natural adsorbents, with a dye removal efficiency of up to 94% from wastewater [10].

Zeolite as one of the materials that can be used for adsorption dyes, has advantages including: high zeolite adsorption capacity, resistance to saturation, high selectivity to certain types of heavy metals, and ease of regeneration make it the preferred choice in a variety of applications, ranging from water purification and gas processing to the chemical separation industry [11]. Zeolite is a silica-alumina compound that has regular cavities and a tetrahedral structure, making it unique. Based on these properties, zeolites can be used as adsorbents, thermal catalysts, and catalysts with specific properties [12].

In this study, wastewater treatment from hair dye rinsing was carried out through an adsorption process using clinoptilolite-Ca zeolite as the adsorbent. The advantage of this research lies in the adsorbent used, namely zeolite. Clinoptilolite-Ca zeolite is a material that can be found naturally in the environment and is available in the hills of the Lubuk Selasih area, Solok Regency, West Sumatra Province. The treatment cost is more economical because this material is easily found and inexpensive. It can also be reused up to its maximum usage limit.

1.2. Literature Review

1.2.1. Dyes

Dyes are colored organic compounds that have chromophore functional groups such as (NR2, NHR, NH2, COOH and OH) and auxochromes such as (N2, NO and NO2). Most synthetic dyes and their degradation products have a significant environmental impact, especially on the aquatic environment, due to their widespread use and low levels of effluent treatment [3].

Paraphenylenediamine (PPD) is a commonly used ingredient in hair color formulations available in the market. PPD is a chemical compound used to give long-lasting color to hair by penetrating the hair shaft and reacting with the melanin pigment found in the hair. PPD is known for its ability to provide dark and intense color shades, which is why it is often used in permanent hair dyes [13].

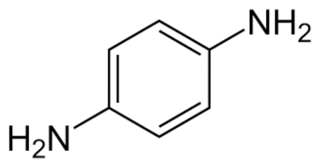

Figure 1. Structure formula Paraphenylenediamine (PPD)

1.2.2. Zeolite

Zeolites are silica-alumina compounds that have a specific pore size. The selectivity of zeolites to a reaction is due to the pore size of zeolites that can only be entered by certain specific molecules, which is referred to as *shape-selective catalysis*. Based on their properties, zeolites have been widely used as adsorbents, ion exchangers, and thermal catalysts, as well as catalyst supports [12].

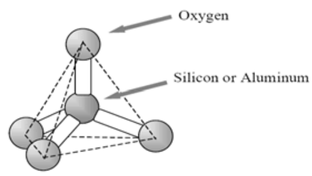

Figure 2. structure formula zeolite

1.2.3. Adsorption

Adsorption is the process of accumulation of a substance (adsorbate) at the interface between two phases (e.g., liquid-solid or gas-solid). The solid substance used for adsorption is called adsorbent [14].

Adsorption can be classified into two types: chemical adsorption and physical adsorption. Chemical adsorption occurs through the formation of strong chemical bonds between adsorbate molecules or ions and the adsorbent surface, which is generally caused by electron exchange; thus, chemical absorption is generally irreversible. Physical adsorption is characterised by weak intraparticle van der Waals forces between the adsorbate and adsorbent, and thus is reversible in most cases [14].

The adsorption capacity formula is as follows:

$$qe=\frac{(C_0-C_e)V}{m}$$

Adsorption efficiency is calculated using the formula:

$$\%\ \text{efficiency}=\frac{(C_0-C_e)}{C_0}\times 100\%$$

Where:
qe = adsorption capacity (mg/g)
C_0 = initial concentration (mg/L)
C_e = final concentration (mg/L)
V = solution volume (L)
m = adsorbent mass (g)

1.3. Research Objective

In this study, zeolite was activated using 0.1 M HCl. Furthermore, zeolite was characterized before and after adsorption using FTIR and XRD. The determination of optimum conditions was carried out by varying the initial concentration, contact time, and mass of the adsorbent.

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2. MATERIALS AND METHODS

2.1. *Material*

The materials used are Zeolite taken from Lubuak Salasiah, Solok Regency and black cream hair dye, while the chemicals used are Whatman 42 filter paper, distilled water, $Pb(NO_3)_2.2H_2O$ and $Cd(NO_3)_2.4H_2O$, 65% HNO_3 p.a solution (Merck), 50% H_2O_2 solution (Merck), 37% HCl solution (Merck), $AgNO_3$ (Merck).

2.2. *Methods*

2.2.1. *Zeolite activation*

Zeolite 320 mesh, weighed as much as 300 grams, activated using 0.1 M HCl, then stirred for 45 minutes. Then after 30 minutes the pH was measured and then the mixture of zeolite and HCl was rinsed with distilled water until the pH was neutral, then the filtrate from the zeolite was tested with $AgNO_3$ 0.05 M if a white precipitate was still formed then the zeolite was washed with distilled water until no more white precipitate was formed. After the pH is neutral, the zeolite is filtered and baked for 2 hours at 105°C [12].

2.2.1.1. *Determination of optimum conditions of hair dye*

First, the calibration curve of the standard solution was prepared by making a 1000 mg/L stock solution of hair dye, which was made by dissolving 0.1 grams of hair dye in a 100 mL volumetric flask. Then, the standard curve was created using concentration variations of 0, 200, 400, 600, 800, and 1000 mg/L in 50 mL volumetric flasks. Subsequently, the absorbance was measured using UV-VIS spectrophotometry [15].

2.2.1.2. *Effect of initial concentration*

The optimum conditions were determined by varying the concentration, contact time, and adsorbent mass. For concentration variation, where zeolite powder was put as much as 0.2 grams into an erlenmeyer and added hair dye solution with concentration variations (400; 500; 600; 700; 800; 900) mg/L. The adsorption process was carried out with a contact time of 45 minutes, followed by centrifugation for 15 minutes, after which the filtrate was measured using UV-VIS spectrophotometry.

2.2.1.3. *Effect of contact time*

The determination of the effect of contact time by adding 20 mL of hair dye solution with the previous optimum concentration and adsorbed by varying the contact time (15, 30, 45, 60, 75 and 90) minutes, then stirred and centrifuged for 15 minutes and the filtrate was measured by uv-vis spectrophotometry.

2.2.1.4. *Effect of adsorbent mass*

The determination of the effect of adsorbent mass was performed by adding 20 mL of hair dye solution with the optimum concentration obtained previously to an Erlenmeyer flask, varying the adsorbent mass (0.2, 0.4, 0.6, 0.8, and 1.0 g). The adsorption process was carried out with the optimum time determined in the previous experiment. The mixture was then stirred and centrifuged for 15 minutes, after which the filtrate was measured using UV-VIS spectrophotometry.

2.2.2. *Adsorption kinetic and isotherm models*

2.2.2.1. *Adsorption isotherms*

The adsorption isotherm models studied in this research are the Freundlich isotherm model and the Langmuir isotherm model. The adsorption isotherm parameters were determined through calculations using data on the effect of the initial hair dye concentration on the adsorption capacity. The Freundlich isotherm equation was obtained by plotting the log Ce versus log qe data, while the Langmuir isotherm equation was obtained by plotting the Ce versus Ce/qe data [16].

2.2.2.2. *Adsorption kinetics*

The adsorption kinetics models studied in this research are the pseudo-first-order and pseudo-second-order kinetics models. The adsorption kinetic parameters were obtained through calculations using data on the effect of contact time on the adsorption capacity of hair dye using zeolite. The pseudo-first-order kinetic equation was obtained by plotting data on t (minutes) versus ln(qe-qt), while the pseudo-second-order kinetic equation was obtained by plotting data on t (minutes) versus t/qt. The kinetic adsorption rate equation for pseudo first order is expressed as:

$$\ln(qe-qt) = \ln qe - k1t$$

Where qe (mg/g) is the equilibrium adsorption capacity, qt (mg/g) is the amount of adsorbate adsorbed at a certain time t, k1 (min-1) is the pseudo first order rate constant.
The kinetic adsorption rate equation for pseudo second order is expressed as:

$$\frac{t}{qt} = \frac{1}{k_2 qe_2} + \frac{1}{qe} t$$

The value of k_2 (g/mg.min) is the pseudo-second-order reaction rate constant, qt (mg/g) is the amount of adsorbate adsorbed at time t, and qe is the amount of adsorbate absorbed at equilibrium. The values of qe and k_2 are obtained from the analysis of the t/qt graph against t [17].

2.2.3. *Application of optimum conditions on hair dye wash water waste samples*

Application of natural zeolite to hair dye wash water waste was carried out by weighing the optimum mass obtained in experiment 2.2.1.4 into an Erlenmeyer flask, then 20 mL of hair dye wash water waste was pipetted into the Erlenmeyer flask. Then, the sample was stirred at the optimum time in experiment 2.2.1.3, centrifuged for 15 minutes, and the filtrate was analysed using a UV-Vis Spectrophotometer at a wavelength of 453 nm.

2.2.4. *Characterization of Zeolite*

The characterisation of zeolite before and after adsorption involves determining functional groups using Fourier transform infrared spectroscopy (FTIR) and assessing crystallinity using X-ray diffraction (XRD).

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2.2.4.1. FTIR

The determination of functional groups in zeolite was tested using FTIR. The wave number range used was 4000–400 cm^{-1}.

2.2.4.2. XRD

The crystallinity of zeolite was tested using XRD operated at 40 kV and 40 mA.

3. RESULT AND DISCUSSION

3.1. Effect of initial concentration

The effect of concentration on the dye absorption capacity using zeolite was studied with a range of 400-900 mg/L. The concentration of PPD dye significantly affects the amount of dye that can be maximally absorbed by an adsorbent. The relationship between PPD dye concentration and the adsorption capacity of the adsorbent is crucial for identifying the appropriate adsorption isotherm model.

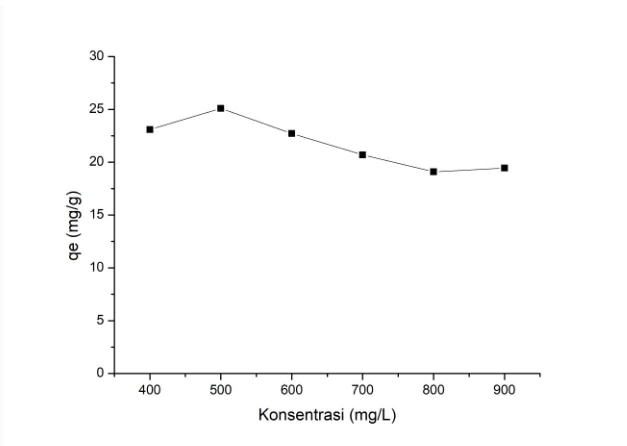

Figure 3. Effect of concentration on the adsorption capacity of PPD dye on hair dye

Figure 3 shows the effect of concentration on the capacity of dye absorption using zeolite. The results show an increase in adsorption capacity from a concentration range of 400 mg/L to 500 mg/L, from 23.0900 mg/g to 25.0900 mg/g. However, from a concentration of 600 mg/L to 900 mg/L there was a decrease in concentration from 18.6900 mg/g to 8.9700 mg/g. It can be concluded that the maximum adsorption capacity is achieved at a concentration of 500 mg/L, which corresponds to 25.0900 mg/g.

The adsorption capacity of PPD dye using adsorbent (zeolite) increases as the concentration of PPD dye increases. At low initial concentrations, the ratio between the number of zeolite active sites and dye molecules is relatively high. This allows most of the dye molecules to be effectively adsorbed on the zeolite surface. However, as the initial concentration of dye increases, the number of dye molecules per unit mass of zeolite increases. Under these conditions, the active sites on the zeolite surface become increasingly saturated, which results in a decrease in the percentage of adsorbed dye and an increase in the amount of dye remaining in solution at equilibrium [18].

The isothermal adsorption mode of dye substances is linked to the influence of initial concentration. This model explains the interaction between adsorbents and adsorbates. In this study, the Freundlich and Langmuir isotherms were used. The equilibrium between the adsorbent and adsorbate, where a single layer is formed at a homogeneous location during the adsorption process, is explained by the Langmuir isotherm model. Based on the Freundlich isotherm model, molecular interactions follow a multilayer adsorption mechanism with heterogeneous energy distribution of active sites [19].

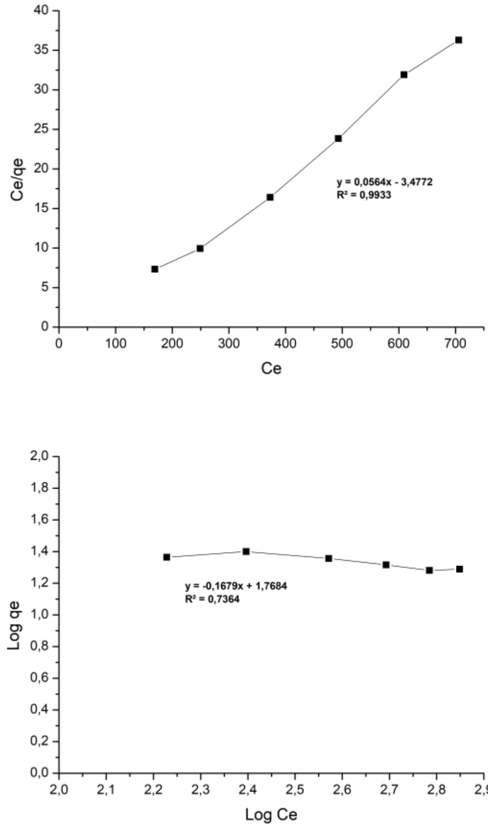

Figure 4. Langmuir and Freundlich isotherm models

Based on the two existing isotherm, the Langmuir model is most commonly used due to its assumption of a single layer of adsorbate (PPD dye) forming on the homogeneous zeolite surface. This model is considered the most appropriate, as evidenced by its higher regression coefficient (R^2 = 0.9933) compared to the Freundlich model. Additionally, the KL value of 61.6522 indicates that this method is effective, as no further active adsorption sites are available once the single layer has formed [20].

3.2. Effect of contact time

The effect of contact time on the dye absorption capacity using zeolite was studied over a time range of 15-90 minutes. The effect of contact time on the dye absorption capacity using zeolite can be seen in Figure 5.

From Figure 5, it can be seen that there is an increase in adsorption capacity from 15 to 45 minutes, reaching 50.1800 mg/g, followed by a decrease in adsorption capacity after 45 minutes. This indicates that the maximum adsorption capacity is achieved at 45 minutes. Therefore, it can be assumed that the adsorption capacity increases with increasing contact time, but decreases after reaching a certain contact time [21].

According to research conducted by [22], one of the factors that affects the adsorption process is time. The longer the adsorption time, the longer the contact time between the zeolite and the adsorbate. This results in greater opportunities for the active side of the zeolite to bind to the adsorbate. The rapid increase in adsorption capacity at the initial contact time may be attributed to the increased availability of adsorption sites on the adsorbent surface. However, as time passes, the adsorption capacity slows down, and after 45 minutes, it reaches equilibrium. This is due to the reduction in the number of adsorption sites on the zeolite surface [23]. Similar findings have been reported based on research conducted by [24], indicating that the use of small amounts of zeolite tends to result in an insignificant increase in adsorption capacity. Because the adsorbate has occupied all active sites on the zeolite surface, the addition of adsorption time will only result in a slight increase in adsorption capacity [25].

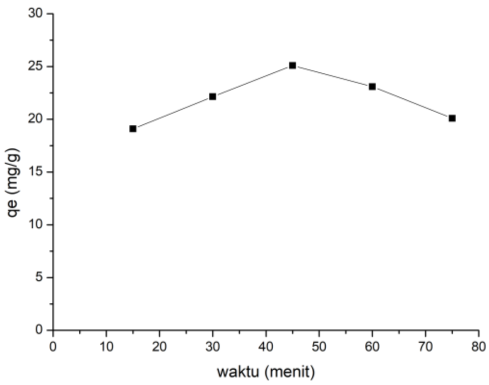

Figure 5. Effect of contact time on the adsorption capacity of PPD dye on hair dye

Data on the effect of contact time on adsorption capacity was used to obtain an adsorption kinetics model (Handayani et al., 2023). This study employed two types of kinetic models, namely pseudo-first-order and pseudo-second-order models. The linear graph of the kinetics model studied in this research is presented in Figure 6.

The first-order pseudo-model is derived from the Lagergren reaction rate equation for liquid and solid adsorption based on solid capacity. Meanwhile, the second-order pseudo-model states that the number of available sites on the adsorbent determines the adsorption capacity. Figure 6 illustrates that the second-order pseudo-kinetic model is more suitable for describing dye adsorption, with a coefficient of determination (R^2) approaching 1. Thus, it can be concluded that zeolite adsorbs dyes through chemical interactions (chemisorption) (Handayani et al., 2023).

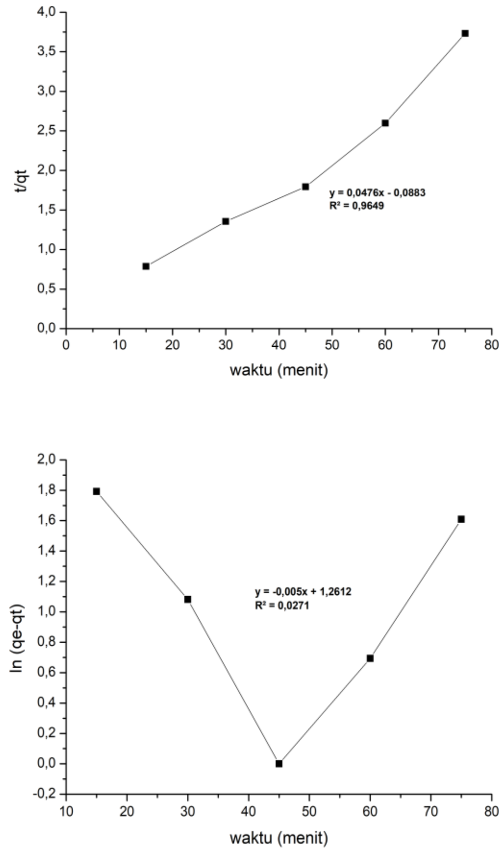

Figure 6. First and second pseudo-order models

3.3. *Effect of adsorbent mass*

The adsorbent mass is a crucial parameter that significantly impacts the adsorption performance of a material. The effect of adsorbent mass on the dye absorption capacity using zeolite was studied with a range of 0.2 - 1.0 gram.

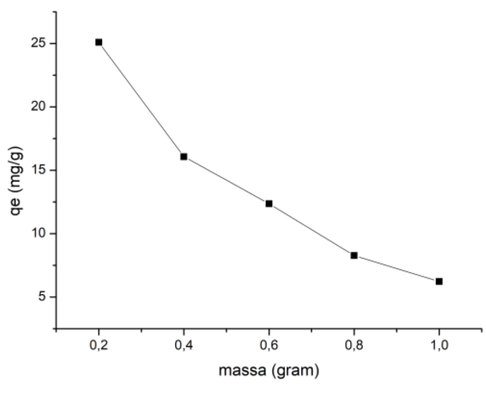

Figure 7. Effect of adsorbent mass on the adsorption capacity of PPD dye on hair dye

Based on Figure 7, the optimum amount of zeolite addition to maximise adsorption results is 0.6 grams, corresponding to 74.18%. The more the mass of adsorbent increases, the capacity of dye absorption by zeolite also increases. This is in accordance with reports from previous research [25].

Based on research conducted by [26], adsorbent mass has a significant impact on how much adsorbate is absorbed throughout the adsorbent surface. With an increase in adsorbent mass, the

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number of active sites present on the adsorbent surface will increase, but there is a decrease in adsorption capacity after 0.6 grams, this is due to the adsorbent surface becoming saturated, so there is no place available for further adsorption which causes the use of adsorbents to be no longer efficient because the amount of adsorbent is too much. This will result in greater absorbance measurements and cause the per cent adsorption to decrease [26].

3.4. Application of optimum conditions to effluent samples

The ability of zeolite as an adsorbent for the treatment of hair dye wash wastewater containing PPD dye was studied in this research. The wastewater used in this study was obtained from hair dye wash water. The application study on this wastewater was conducted using the optimum conditions for wastewater treatment, as shown in Table 1.

Table 1. Application of adsorbent to adsorption PPD dye in hair dye liquid waste

C_0 (mg/L)	Adsorbent mass (g)	C_e (mg/L)	q (mg/g)	% Adsorption
923,1000	0,6	168.1000	25.1667	81.7896

The results showed that the concentration of PPD in the wastewater was significantly reduced, from 923.1000 mg/L to 168.1000 mg/L after treatment with the adsorbent. This indicates that the adsorbent used was able to absorb 81.79% of the total PPD content in the liquid waste. This percentage indicates high efficiency in the adsorption process [27].

3.5. Characterization

3.5.1. FTIR

Fourier transform infrared (FTIR) spectrophotometry is a vibrational spectroscopy analysis method used to predict the functional groups involved during adsorption. FTIR is used to predict the functional groups involved in adsorption by analysing the shift in the FTIR spectrum between 4000 and 500 cm^{-1}. The FTIR spectra before and after adsorption are shown in Figure 8.

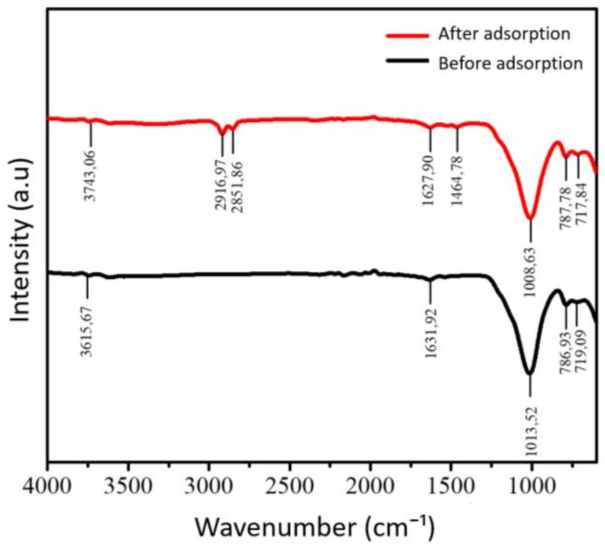

Figure 8. FTIR spectra of zeolite before and after absorption of PPD dye in hair dyes

Figure 8 illustrates that zeolite comprises several distinct types of functional groups. Based on the spectrum obtained, it can be seen that the wave number region 3615 cm^{-1} shows the absorption of streatching vibrations by the -OH group. Evidence of the presence of samples containing PPD dye adsorbed on zeolite in the 1600-1400 cm^{-1} region shows additional peaks corresponding to symmetrical and asymmetrical stretching vibrations of the N-H bond contained in the amino group of the dye. PPD is a dye that has amine groups bound to aromatic rings and heterocyclic amines. The appearance of an additional peak in the 1500-1480 cm^{-1} region is attributed to the bending vibration of the N-H bond in the NH_2 group. The peak observed at 1600-1500 cm^{-1} corresponds to the -C=C- bond vibrations of the aromatic ring of the dye [28].

3.5.2. XRD

XRD is one of the qualitative and quantitative analysis methods used to analyse the structure of zeolite powder. Characterization using XRD on zeolite was carried out to observe the structure and crystal quality of zeolite in conditions before and after the adsorption process. The diffractogram of natural zeolite analysis results is shown in Figure 9.

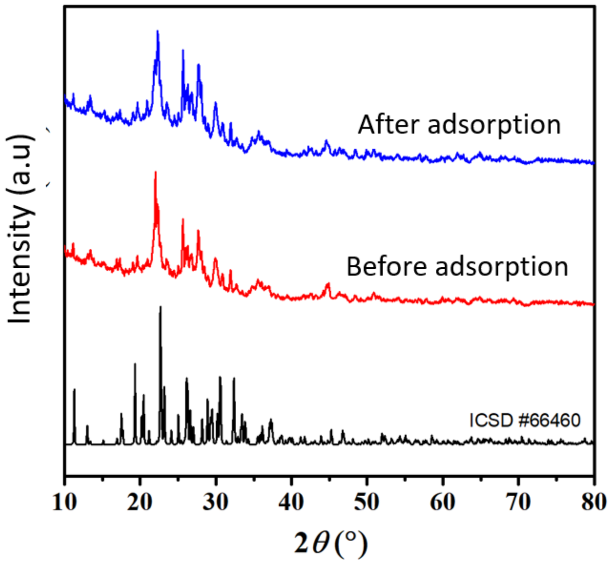

Figure 9. Diffractogram of zeolite before and after absorption of PPD dye on hair dye

XRD analysis was conducted to identify the presence of PPD dye in the zeolite by comparing the data obtained with the zeolite standard for clinoptilolite (ICSD #66460). The wave number spectra showed no significant changes before and after adsorption, indicating that the zeolite crystal structure remained stable and no new products were formed, making it safe to use in this adsorption process. XRD analysis revealed that the natural zeolite is of the clinoptilolite type. This is evidenced by the presence of peaks characteristic of clinoptilolite zeolites in ICSD (*International Centre for Diffraction Data) data, with high intensity observed at angles of 9.80 ° and 29.07°* [24].

4. CONCLUSION

Based on the research, it can be seen that zeolite is able to reduce the PPD dye in hair dye. This study demonstrates that adsorption lasts for 45 minutes using 0.6 grams of zeolite, with a dye sample volume of 20 mL for dye absorption applied to hair dye wash water waste, resulting in a hair dye adsorption percentage of 81.78%. The results of FTIR characterisation show that the absorption bands for zeolites before and after dye adsorption have the same patterns, and new peaks appear, indicating the presence of the adsorbed dye. XRD characterization results show that the zeolite spectrum is in accordance with the standard (ICSD #66460).

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