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Research Paper

Selective Removal of Anionic and Cationic Dyes Using Magnetic Composites

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Abstract

Water is one of the most basic human needs, and dyes are one of the sources of water pollution. Since adsorption has proven to be effective in removing contaminants, it is the most widely used technique. In this adsorption, a LDH Zn-Al /magnetic biochar composite was used for dye removal. Zn-Al LDH, magnetic biochar, and LDH Zn-Al/biochar magnetic composite were successfully synthesized, based on XRD and FTIR studies. XRD analysis of the Zn-Al LDH material shows diffractions of (003), (006), (101), (012), (015), (107), and (110) around the 2θ angle at 10.29°, 20.07°, 29.59°, 32.12°, 34.02°, 48.06°, and 60.16° which are characteristic of LDH materials. In magnetic biochar and LDH Zn-Al/magnet biochar composites diffraction (220), (311), (422) and (440) at 2θ around 24.9°, 35°, 63° and 68.4° in these materials indicate the characteristics of carbon-based materials from biochar. FTIR analysis showed the appearance of a vibration peak at 1404cm^{-1} indicating the presence of C$-$H groups contained in biochar. The characteristic double-layer hydroxy (M$-$O) vibrations below 1000 cm^{-1} also indicated that the composite preparation process had been successful. The study's results show that cationic dyes are more easily adsorbed than anionic dyes. Specifically, the LDH Zn-Al/Magnetic Biochar composite more extensively absorbs the cationic dye malachite green.

Keywords

Composite, Magnetic, Biochar, Anionic Dyes, Cationic Dyes

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1. INTRODUCTION

Textile dyes are soluble substances and are widely found in various industrial wastes such as printing, cosmetics, dyeing, textiles, plastics, pharmaceuticals and so on (Semwal et al., 2023; Wang et al., 2020). The presence of these color pollutants will pose a serious threat to human health and aquatic ecosystems (Wang et al., 2023b; Vinsiah et al., 2020). The benzidine content of the dyes can cause effects on the eyes, digestion, skin irritation, blood clotting, and respiratory disorders to health (Semwal et al., 2023). Due to their harmful properties, dyes need to be removed to ensure human health and the health of aquatic ecosystems (Yu et al., 2023).

There are many ways to remove dye pollutants in the environment including using coagulation (Sudirgo et al., 2023), photocatalyst (Wang et al., 2023a), ion exchange (Aftab et al., 2023), chemical precipitation (Manalu et al., 2023), membrane filtration (Liu et al., 2023), and adsorption (Aghaei et al., 2023). Of the above methods, adsorption method is one of the most promising methods due to its simple operation, efficiency, low cost, and wide material sources (Akdemir et al., 2022; Li et al., 2020; Wang et al., 2023a). Various types of materials have been applied as adsorbents in the dye adsorption process, including using layered double hydroxide (He et al., 2023; Ahmad et al., 2023).

Layered double hydroxide (LDH) is generally synthesized in various ways, including using anion exchange, hydrothermal, and co-precipitation methods. The co-precipitation method involves mixing two or more salt solutions by adding sodium hydroxide to lower the pH of the solution (Xu et al., 2023). LDH with unique properties such as large surface area, ion exchange ability, and memory effect structure makes this material very effective as an adsorbent in dye wastewater treatment (Ahmad et al., 2022). According to Amri et al. (2023), LDH has poor structural stability when used in the adsorption process because the coating is easily peeled off during the application process, resulting in reduced efficiency in the regeneration/reuse process in the adsorption process. Therefore, it is necessary to improve the structure by compositing with carbon-based materials so that the structure becomes more stable. Research conducted by Manalu et al. (2023) improved the structure of

Ni/Cr LDH by compositing carbon-based materials, namely cellulose, which increased the adsorption capacity from 100 mg/g to 129.87 mg/g.

In addition to using cellulose carbon materials, other types of carbon materials such as biochar can also be used in improving the structure of LDH. Biochar (BC) is a porous structure made through a pyrolysis process that is rich in carbon with abundant and diverse functional groups, large surface area, and good sorption affinity (Su et al., 2024). In this study, Zn/Al LDH composite with magnetite biochar carbon (Zn-Al/MBC) based material from rice husk will be applied to anionic and cationic dye pollutants. The magnetization process is carried out with the aim to help facilitate the adsorption process, so as to reduce the risk of damage to the material surface and make the adsorption process more efficient. LDH Zn/Al, Magnetic biochar, and LDH Zn-Al/magnetic biochar composite (Zn-Al/MBC) material was synthesized and characterized using XRD and FT-IR, to see the success of material preparation. Furthermore, to determine the most selective dye, anionic and cationic dye selectivity was carried out.

2. EXPERIMENTAL SECTION

2.1 Chemical and Instrumental

The research made use of components sourced from suppliers. These components included zinc hexahydrate ($Zn(NO_3)_2.6H_2O$) and aluminum nitrate nonahydrate ($Al(NO_3)_3.9H_2O$) obtained from Sigma Aldrich, sulfuric acid (H_2SO_4), sodium hydroxide (NaOH) sourced from EMSURE® ACS, sodium carbonate (Na_2CO_3) from EMSURE® ACS, sodium nitrate ($NaNO_3$), sulfate heptahydrate ($FeSO_4.7H_2O$), ammonia (NH_3), hydrogen peroxide (H_2O_2), hydrochloric acid (HCl) from MallinckrodtAR®, deionized water (H_2O), biochar, malachite green ($C_{23}H_{25}ClN_2$), congo red ($C_{32}H_{22}N_6Na_2O_6S$), methylene blue ($C_{16}H_{18}N_3SCl$), rhodamine b ($C_{28}H_{31}ClN_2O_3$), met hyl orange ($C_{14}H_{14}N_3NaO_3S$) and direct yellow ($C_{35}H_{24}N_6Na_4O_{13}S_4$).

The tools used in this research are stirring rod, separating funnel, hotplate, filter paper, analytical balance, oven, pH meter, shaker and a set of glassware. Equipment used for material characterization using XRD (Rigaku Miniflex-6000), FT-IR spectrophotometer (Shimadzu Prestige-21). UV-Vis spectrophotometer (Biobase BK-UV 1800 PC) is used to measure the amount of dye adsorbed on the adsorbent.

2.2 Synthesis of Zn-Al/LDH

The synthesis of the Zn-Al layered double hydroxide involved combining 100 mL of a 0.75 M solution of $Zn(NO_3)_2.6H_2O$ with 100 mL of a 0.25 M solution of $Al(NO_3)_3.9H_2O$. This mixture was then slowly added with 50 mL of a 2 M NaOH solution. To adjust the pH NaOH solution was used until it reached a value of 10 followed by stirring at a temperature of 80°C for a duration of 4 hours. As a result a solid precipitate formed and was subsequently filtered and washed with water to eliminate any impurities. The precipitate was then dried in an oven. Subjected to analysis using techniques including XRD and FTIR spectrophotometer (Mohadi et al., 2023; Fitri et al., 2023).

2.3 Preparation of Magnetic Biochar

Magnetic biochar is prepared by adding 1 g of $FeCl_3$ to 3 mL of distilled water, which is then added to 3 mL of distilled water with 0.6 g of $FeSO_4.7H_2O$ and then the mixture is added to 1 g of biochar and stirred for 3 hours and they are used. The mixture was then slowly poured with 3.5 mL of NH_3 solution and stirred at 75 °C for 30 min. The resulting solution was transferred to a 100 mL hydrothermal stainless steel autoclave. The mixture was then heated to 150 °C for 3 hours. The resulting magnetite biochar solids were then filtered and dried at 40 °C. The solid material was then characterized using various techniques including XRD and FTIR spectrophotometer (Ahmad et al., 2022).

2.4 Synthesis of composite Zn-Al/Magnetic Biochar

A total of 15 mL of 0.75 M Zn solution was mixed with 15 mL of 0.25 M Al solution. Then, the pH was adjusted to 10 with NaOH solution. The mixture was stirred for one hour until homogeneous and the gel formed. Then, 3 g of biochar was added. The reaction was kept at 80 °C for 72 h, resulting in solution A. After that, 2 g $FeCl_3$ was added into 3 mL of distilled water and mixed with 1.6 g $FeSO_4.7H_2O$ dissolved in 3 mL of distilled water, then stirred at room temperature for 3 hours. The resulting mixture was then slowly added 7 mL of NH3 solution and stirred at 75°C for 30 min, giving compound B. It was added to solution A and stirred for 30 min and heated at 150 °C for 72 h. The resulting solid was filtered, washed with distilled water, and dried in an oven at 100 °C for 24 h. The dried Zn-Al/magnetite biochar composite was crushed and then subjected to XRD and FTIR spectrophotometer (Ahmad et al., 2022; Mohadi et al., 2023).

2.5 Selectivity to Anionic and Cationic Dyes

Selectivity tests were carried out by mixing anionic and cationic dyes. A total of 20 mL of anionic and cationic dyes with a concentration of 50 mg/L. The dye mixture was added 0.02 grams of each adsorbent, then measured the wavelength in the range of 400-700 nm using time variations of 0, 4, 6, 8 and 10 minutes (Wibiyan et al., 2023).

3. RESULTS AND DISCUSSION

3.1 Adsorption Selectivity of Cationic Dyes

The mixed dyes, namely malachite green, methylene blue, and rhodamine b, were used with the same concentration. This study aimed to determine the adsorption ratio of cationic dyes using Zn-Al LDH adsorbent, magnetic biochar, and Zn-Al LDH/Magnetic Biochar composite. The dye mixture was added with 0.2 g of material and shaken for intervals of 0, 4, 6, 8, and 10 minutes. The wavelength was measured at each minute to determine the most selective dye adsorbed on each adsorbent. Figure 1 displays the wavelengths of malachite

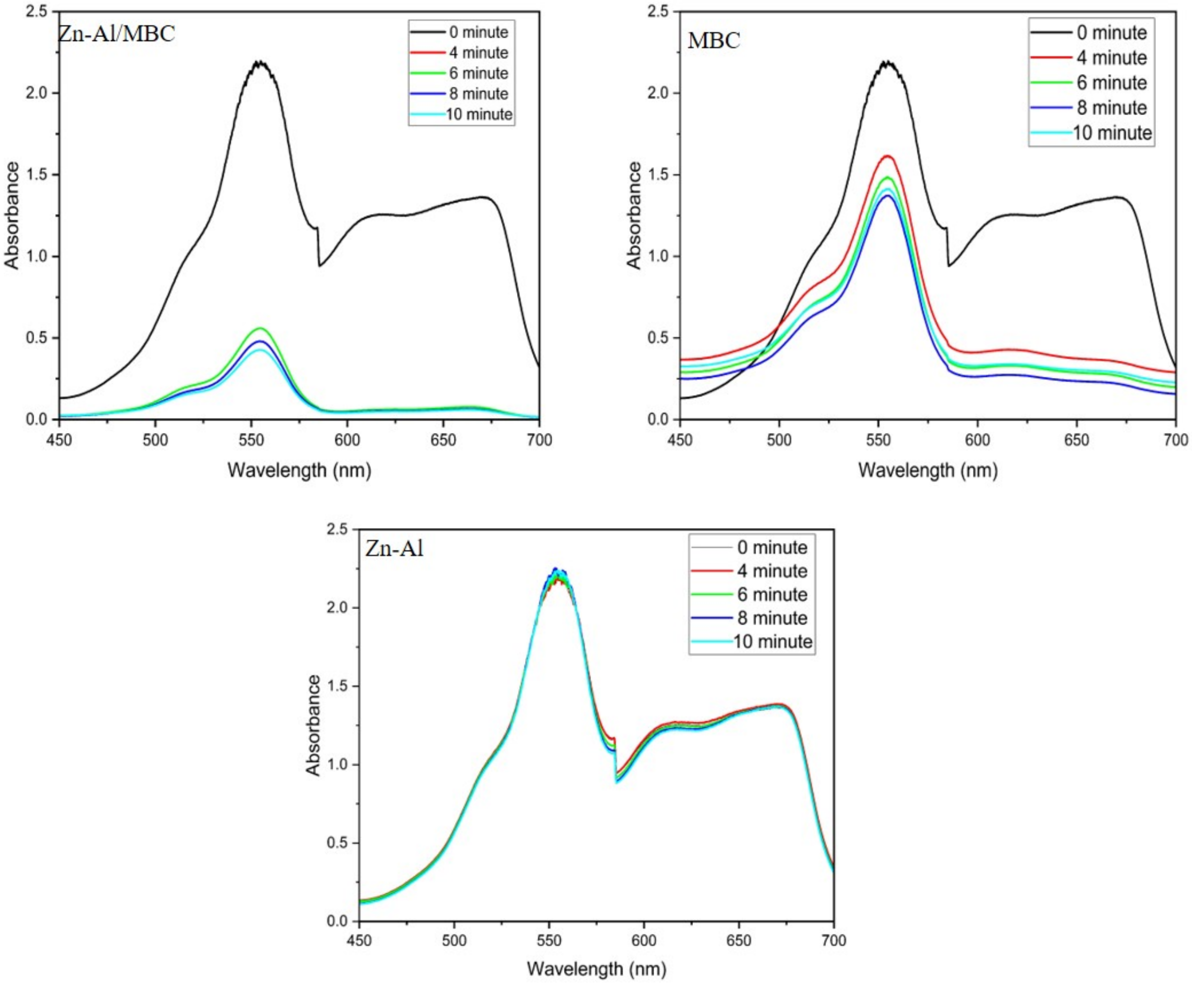

Figure 1. Selectivity of Cationic Dyes

green at 620nm, methylene blue at 665nm, and rhodamine b at 550nm.

Figure 1 displays the results indicating that malachite green is the best adsorbed dye using the LDH Zn-Al/Magnetic Biochar (Zn-Al/MBC) composite adsorbent. At minute 10, the absorbance of malachite green at a wavelength of 620 nm was 0.1, which is significantly lower than that of methylene blue and rhodamine b dyes. The selectivity of cationic dyes to each adsorbent used shows that the Zn-Al/Magnetic Biochar (Zn-Al/MBC) LDH composite has superior adsorption power compared to other adsorbents. According to Manalu et al. (2023), malachite green, a cationic dye with a simpler structure than other dyes, exhibits superior adsorption.

3.2 Adsorption Selectivity of Anionic Dyes

The anionic dyes congo red, methyl orange, and direct yellow were selectively adsorbed using the same concentration. The wavelength of congo red was 499 nm, methyl orange was 450 nm, and direct yellow was 380nm. Zn-Al LDH adsorbent, magnetic biochar, and Zn-Al LDH/Magnetic Biochar composite were used for this selectivity. The dye mixture was added with 0.2 g of material and shaken for intervals of 0, 4, 6, 8, and 10 minutes. The wavelength was measured at each minute to determine the most selective dye adsorbed on each adsorbent. The results showed that the most selective dye of the three anionic dyes used was direct yellow.

Figure 2 shows that the most selective adsorbed direct yellow dye experienced the greatest adsorption on each adsorbent at a wavelength of 380 nm. The Zn-Al/Magnetic Biochar (Zn-Al/MBC) LDH composite demonstrated superior adsorption selectivity compared to other adsorbents, with an absorbance of direct yellow reaching 0.4 at minute 10. Ni'mah et al. (2020) found that activated carbon is effective in adsorbing direct yellow due to its large surface area and pore structure that can capture dye molecules.

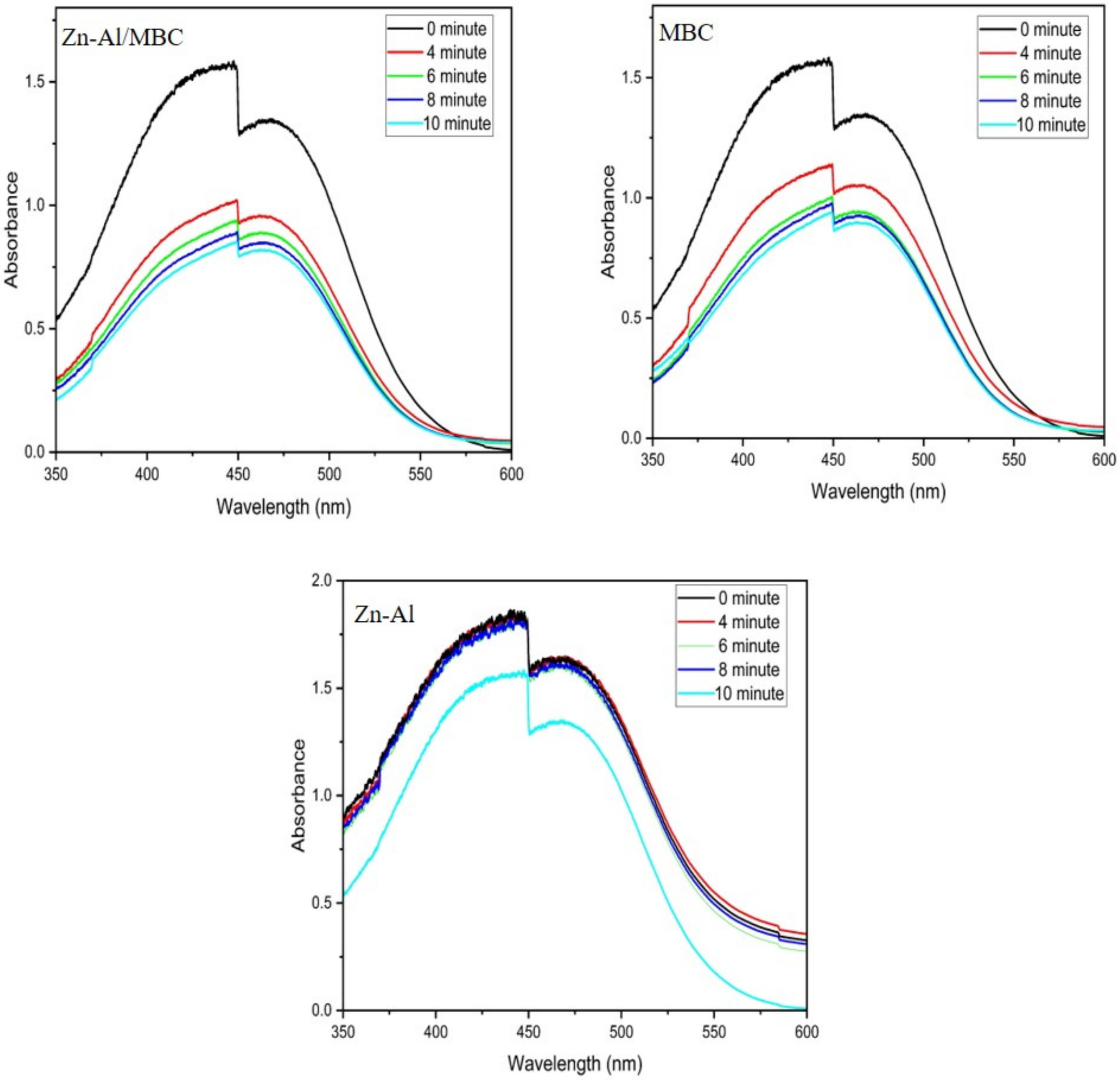

Figure 2. Selectivity of Anionic Dyes

3.3 Adsorption Selectivity of Anionic and Cationic Dyes

This study investigates the adsorption of a mixture of dyes, including malachite green, methylene blue, rhodamine b, congo red, methyl orange, and direct yellow, at equal concentrations. The aim is to compare the adsorption of anionic and cationic dyes using Zn-Al LDH adsorbent, magnetic biochar, and Zn-Al LDH/Magnetic Biochar composite. The dye mixture was added to 0.2 g of the material and shaken at intervals of 0, 4, 6, 8, and 10 minutes. The wavelength was measured at each minute to identify the dye that was most selectively adsorbed on each adsorbent. Figure 3 shows the wavelengths of malachite green at 620 nm, methylene blue at 665 nm, rhodamine b at 550 nm, congo red at 500 nm, methyl orange at 450 nm, and direct yellow at 415 nm. Figure 3 shows that malachite green is the most efficiently adsorbed dye using the Zn-Al LDH/Magnesium Biochar composite, with a significant decrease in absorbance to 0.35 at 620 nm after 10 minutes compared to other dyes.

The Zn-Al LDH/Magnetic Biochar composite exhibits better adsorption capacity than other adsorbents for both anionic and cationic dyes. During the adsorption process of the dye mixture, it was observed that cationic dyes are more efficiently adsorbed. Specifically, the cationic dye malachite green is better adsorbed due to its simpler chemical structure compared to other dye structures (Manalu et al., 2023). This is attributed to the differences in chemical properties, molecular structures, and intermolecular interactions between cationic and anionic dyes. Malachite green and other dyes are organic molecules

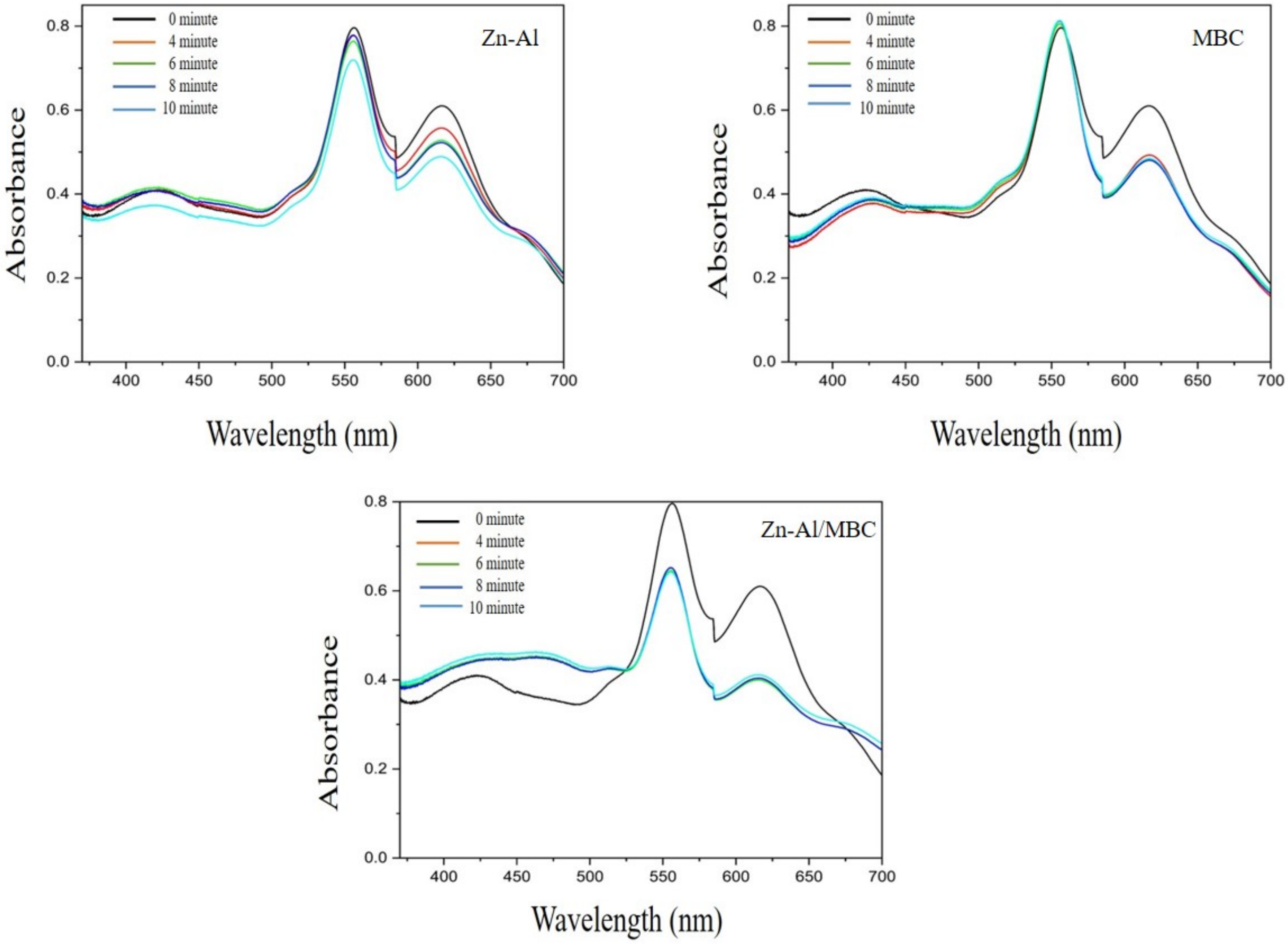

Figure 3. Selectivity of Anionic and Cationic Dyes

with distinct chemical structures. Anionic dyes, such as congo red (Ahmad et al., 2023), typically contain azo compounds in their structure, making them more resistant to degradation. Malachite green has functional groups that interact well with the material surface, resulting in a stronger and more selective chemical interaction between the malachite green molecules and the adsorbent material.

Cationic and anionic dyes have different molecular structures that can result in differences in their interaction strength with the adsorbent material. Intermolecular interactions, such as Van der Waals forces, hydrogen bonding, and electrostatic forces, play a crucial role in adsorption. Malachite green is more efficiently adsorbed than other dyes due to its stronger attraction to the adsorbent material. According to Stefancu et al. (2021) and Wang et al. (2020), malachite green adsorbs more effectively due to the differences in chemical properties, molecular structures, and intermolecular interactions between cationic and anionic dyes.

Based on the results of selective adsorption for anionic and cationic substances, it is evident that the Zn-Al LDH/Magnetic Biochar composite has superior adsorption capabilities compared to magnetic biochar and Zn-Al LDH. This is supported by the XRD and FT-IR characterisation results. The XRD and FT-IR analyses demonstrate the successful production of effective adsorbent materials, including Zn-Al LDH, magnetic biochar, and magnetic Zn-Al LDH/biochar composite. The JCPDS No. 48.2023 data for Zn-Al was compared to the diffractogram peaks of layered double hydroxides in Zn-Al LDH, magnetic biochar, and magnetic Zn-Al LDH/biochar composite materials (Siregar et al., 2021; Fitri and Ardiansyah, 2023). Figure 4 displays the characterization data of Zn-Al LDH, magnetic biochar, and Zn-Al LDH/magnetic biochar composite.

XRD patterns were used to identify typical diffractogram patterns of the Zn-Al layered double hydroxide materials with 2θ angles. The diffraction patterns exhibited peaks at 10.29°, 20.07°, 29.59°, 32.12°, 34.02°, 48.06°, and 60.16°, which were assigned to planes (003), (006), (101), (012), (015), (107), and

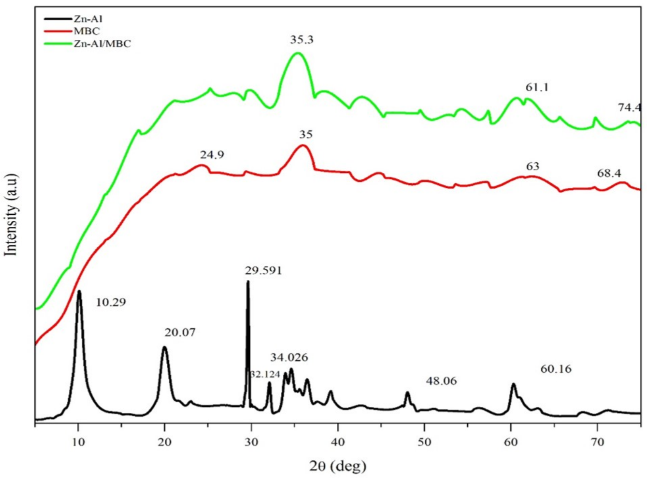

Figure 4. X-Ray Diffractogram of Adsorbents

(110). The Zn-Al material structure was constructed based on the diffraction peaks, following JCPDS file No. 48-1023. Palapa et al. (2019) reported that the Zn-Al layered double hydroxide material has anions in the interlayer, as indicated by the double diffraction peak at an angle of 60°. The diffraction peaks of planes (220), (311), (422), and (440) are observed at 24.9°, 35°, 63°, and 68.4°, respectively, as shown in Figure 4. The Zn-Al coated double hydroxide composite/magnetic biochar exhibits diffraction angle peaks at 35.3° and 61.1° for the (311) and (110) planes, respectively. This indicates the successful production of magnetic biochar and Zn-Al/magnetic biochar double hydroxide composites using JCPDS file No.19-0619 (Ahmad et al., 2023).

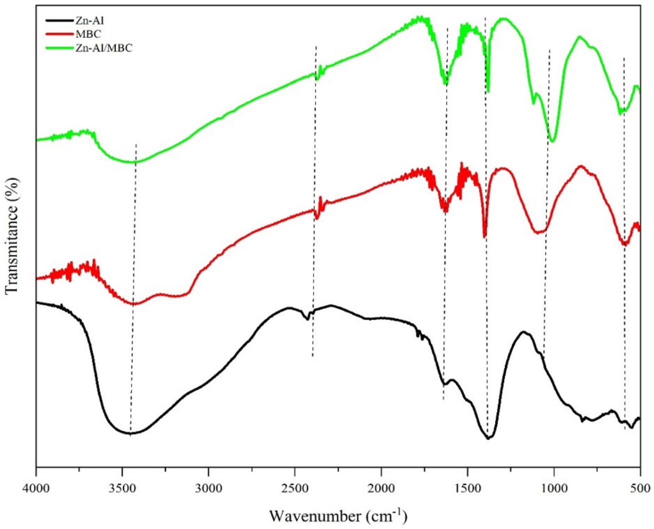

Figure 5. X-Ray Diffractogram of Adsorbents

Figure 5 shows the FT-IR spectra that support the successful synthesis of Zn-Al LDH material, magnetic biochar, and LDH Zn-Al/magnetic biochar composite. The vibrational peaks at wave numbers 3441, 3425, and 3448 cm^{-1} indicate the presence ofO−H groups derived from water molecules in Zn-Al, magnetic biochar, and Zn-Al/magnetic biochar LDH composites. The presence of C≡C is indicated by the wave numbers 2376 cm^{-1} and 2422 cm^{-1} on Zn-Al, magnetic biochar, and LDH Zn-Al/magnetic biochar composite. Additionally, the wavelengths of 1620 cm^{-1} and 1635 cm^{-1} in LDH on LDH Zn-Al/magnetic biochar composite and magnetic biochar indicate the presence of a C=O carbonyl group (Siregar et al., 2022).

The presence of M−O vibrations in the form of Zn-O and Al-O is indicated by waves 609-840 cm^{-1}, while wave number 1381 cm^{-1} indicates the presence of NO_3^- group vibrations in Zn-Al layered double hydroxides. The magnetic biochar exhibits carbon-hydrogen (C−H) groups at 1404 cm^{-1}, carbon-oxygen (C−O) vibrations at 1095 cm^{-1}, and magnetic Fe-O at 586 cm^{-1}. The presence of aliphatic amines is indicated by the wavelengths of 1110-1120 cm^{-1} in the LDH Zn-Al/magnetic biochar composite, while the wave number of 580 cm^{-1} indicates the presence of M−O vibrations on Zn-O and Al-O. Therefore, it can be concluded that the synthesis of magnetic biochar, magnetic Zn-Al/biochar LDH composite, and Zn-Al LDH has been successfully carried out (Ahmad et al., 2023; Rahmadan et al., 2021). The crystal structure of the magnet and high carbon content in the LDH Zn-Al/Magnetic Biochar composite (Zn-Al/MBC) resulted in a higher adsorption capacity and a more efficient adsorption process.

4. CONCLUSION

The study's results show that cationic dyes are more easily adsorbed than anionic dyes. Specifically, the LDH Zn-Al/Magnetic Biochar composite more extensively absorbs the cationic dye malachite green. This is supported by the XRD results, which show a well-defined crystal structure, and the FT-IR results, which indicate the presence of activated carbon.

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