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SYNTHESIS AND CHARACTERIZATION OF ADSORBENT MAGNETIC KAOLIN

Katarina Elgia, Harlia Harlia, Nelly Wahyuni*
Department of Chemistry, Faculty of Mathematics and Natural Sciences, Tanjungpura University, Pontianak, Indonesia.
*nellywahyuni@chemistry.untan.ac.id

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ABSTRACT

Kaolin as an adsorbent has difficulties separating it from the adsorbate solution because it requires centrifugation and precipitation methods, which require significant costs, time, and energy. Modification of kaolin with magnetite was carried out to increase the effectiveness of adsorption from the paracetamol solution. Kaolin-magnetite (KM) adsorbent was made by combining activated kaolin and Fe(III)/Fe(II) solution with a mole ratio of 2:1 using the coprecipitation method. The resulting activated kaolin, magnetite, and kaolin-magnetite were characterized using X-ray Fluorescence (XRF), X-ray Diffractometer (XRD), Fourier Transform Infrared (FTIR), Gas Sorption Analyzer (GSA), and Vibrating Sample Magnetometer (VSM). The presence of magnetite makes it easier to separate KM from the paracetamol solution, with 97.17% of KM being taken by the magnetic rod.

Keywords: adsorbent; kaolin; magnetite; paracetamol

INTRODUCTION

Kaolin is a clay mineral composed of hydrated aluminum silicate ($2H_2O{\cdot}Al_2O_3{\cdot}2SiO_2$) and other minerals such as kaolinite, dickite, nacrite, and halloysite [1]. The use of kaolin as an adsorbent has difficulties separating the adsorbent from the solution after the adsorption process [2]. The separation of the adsorbent from the adsorbate is usually carried out using centrifugation and sedimentation methods, which require large costs, time, and energy. Modification of kaolin with magnetite material is performed to enhance adsorption effectiveness.

Three general forms of iron oxide are common in nature, including magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3) and hematite (α-Fe_2O_3), which can be used as adsorbents for water treatment. However, iron oxide nanoparticles tend to agglomerate easily, limiting adsorption capabilities. Therefore, making metal oxide/kaolin can functionalize kaolin and stabilize nanoparticles so that both can carry out their functions properly [3]. The magnetic properties of iron oxide can be utilized to facilitate the separation of the solid phase of kaolin-magnetite adsorbent from the adsorbate solution after the adsorption process using a magnetic rod.

The synthesis of kaolin-magnetite composites produces materials with two different properties: the adsorption properties of kaolin and the magnetic properties of magnetite material composited onto kaolin [4]. Kaolin-Fe_3O_4 nanoparticles have a high magnetic response to magnetic fields. Because of its great magnetic sensitivity, kaolin-Fe_3O_4 is easily and rapidly separated [5]. Separation of kaolin-magnetite from the methylene blue solution using a magnetic

rod occurs rapidly, requiring less than 120 seconds, whereas kaolin without composite magnetite requires high-speed centrifugation for sedimentation [6]. In another study, the process of separating the magnetite-modified natural clay adsorbent from the methylene blue solution using a magnetic field takes 2 minutes, while the activated natural clay which is still dispersed takes 38 minutes to separate [4].

Kaolinite which can be found on various continents, is abundant and low-cost. Therefore, it was investigated for its adsorption mechanisms concerning drugs such as paracetamol, ibuprofen, carbamazepine, aspirin, diclofenac, and diazepam. These compounds can adsorb onto the surface of kaolinite alumina, with paracetamol showing the strongest adsorption [7]. Paracetamol is a widely recognized analgesic and antipyretic medication, that is commonly utilized by the general public. As the use of paracetamol increases, its production rises, leading to more waste. Paracetamol is one of the pharmaceutical compounds found in waste water [8].

Paracetamol is one of the active pharmaceutical compounds (PhAC). PhAC is highly toxic even at low concentrations. PhAC waste usually comes from drug production waste being thrown into the water, washing equipment used in the drug production process, and throwing expired drugs into the water, thereby reducing water quality. Waste that enters waters causes the water to lack oxygen, which can cause biota in these waters to die due to lack of oxygen [8]. The presence of paracetamol in the environment, even at low concentrations (40 ng/L) in short and long-term exposures, can potentially cause several changes related to the reproductive system of mussels. Paracetamol can cause adverse changes in gonadal tissue, including the degeneration of follicles and gametes. This poses a significant risk to the reproductive capabilities of these organisms and can ultimately impact their population [9]. Contaminants of paracetamol in seawater in Jakarta Bay and the northern coast of Central Java have been detected in high concentrations, reaching 610 ng/L in Teluk Angke and 420 ng/L in Ancol [10]. Paracetamol has also been reported to be present on the northern coast of Portugal with an average concentration of 95.2 ng/L [11]. Paracetamol is a pharmaceutically active ingredient detected with the highest concentration among other pharmaceutically active ingredients, reaching 227 µg/L in the Rio Seke River in La Paz, Bolivia [12].

Treatment of pharmaceutical waste from water can be done through several methods, namely oxidation, coagulation-precipitation, electrodialysis, ion exchange, and reverse osmosis. However, these methods require high costs and constant monitoring. Based on these methods, adsorption is the most promising technique. Adsorption can efficiently handle waste, is easy to perform, is cheap, and adsorbents can be regenerated for repeated use [13]. Paracetamol adsorption studies using KSF montmorillonite clay mineral resulted in 50% removal of paracetamol [14]; removal of paracetamol by montmorillonite was 63-67% [15]; 72% removal of paracetamol by Beninese kaolinite geopolymer [16]; and 80% removal of paracetamol and metformin from water by water-treated clay and acid-treated clay adsorbent [13].

In this study, a kaolin-magnetite adsorbent was synthesized using the coprecipitation method. The characteristics of kaolin-magnetite by X-ray Fluorescence (XRF), X-ray Diffraction (XRD), Fourier Transformed Infrared (FTIR), Gas Sorption Analyzer (GSA), Vibrating Sample Magnetometer (VSM), Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray (EDX) were studied. Subsequently, the effectiveness of separating kaolin-magnetite

from the paracetamol solution was also analyzed to see the potential for reusable adsorbents.

METHOD

Tools and Materials

The tools used in this research include 230 mesh sieves, neodymium magnetic rod (N50 grade), hot plate, magnetic strirrer bar, orbital shakers (SciLogex SK-O330-Pro), oven, centrifuge (B-One DC6015-12), sonicator (Branson 3510). The materials used in this research include kaolin from Capkala District, Bengkayang Regency, West Kalimantan; Sulfuric acid (H_2SO_4); Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$); Iron(III) sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$); Sodium hydroxide (NaOH); and paracetamol tablets.

Preparation of Activated Kaolin

Kaolin is dried for 6-7 hours under sunlight. The kaolin was crushed and decanted three times using distilled water. Subsequently, the kaolin is centrifuged at 6000 rpm for 15 minutes. The kaolinite fraction was then taken and dried using an oven at 105 ^oC for 5 hours and filtered using a 100-mesh sieve [17-18]. 50 g of kaolin was added to 500 mL of a 2M H_2SO_4 solution. The mixture was heated at 100 ^oC for 4 hours using reflux while stirring with a magnetic stirrer on a hot plate. Kaolin suspension was then cooled, washed with distilled water until the pH became neutral, and dried in an oven at a temperature of 100 ^oC for 4 hours. The activated kaolin was ground into powder and filtered using a 230-mesh sieve [19].

Synthesis of Kaolin-Magnetite

Kaolin is placed in 200 mL of water and stirred with a magnetic stirrer at 70 ^oC for 2 hours. In another container, an iron oxide solution is prepared with 200 mL of a mixture of $FeCl_3 \cdot 6H_2O$ and $FeSO_4 \cdot 7H_2O$ in a molar ratio of 2:1. The iron oxide solution is then poured into the heated kaolin suspension and stirred with a magnetic stirrer, with a mass ratio of kaolin to iron oxide of 1:1. During this process, 100 mL of a 5 M NaOH solution is added dropwise into the mixture. The precipitate is then washed with distilled water until the pH becomes neutral. The precipitate is dried at 100 ^oC for 4 hours. Kaolin-magnetite is then ground into powder and sieved using a 230-mesh sieve [20]. Additionally, magnetite without the addition of kaolin is also prepared.

Characterization of Activated Kaolin, Magnetite, and Kaolin-Magnetite

The elemental composition was analyzed by X-ray Fluorescence (XRF). The crystalline properties phase, d-spacing, and crystal size were characterized by X-ray Diffraction (XRD). The samples' function groups were analyzed by a Fourier Transformed Infrared (FTIR) spectrometer. The specific surface areas and pore properties were analyzed by nitrogen adsorption using a Gas Sorption Analyzer (GSA) instrument. The magnetization measurement of the samples was carried out using a Vibrating Sample Magnetometer (VSM). The morphology and mapping of the sample's elemental compositions (Al, Si, Fe, and O) were analyzed by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX).

Effectiveness of Magnetic Properties of Kaolin-Magnetite in Paracetamol Adsorption

A quantity of 20 mg of kaolin-magnetite is used for the adsorption of a paracetamol solution at

a concentration of 10 ppm for 30 minutes. After that, an external magnet is used to extract the kaolin-magnetite from the solution, and it is dried for 2 hours at 105 ^{o}C in an oven. The kaolin-magnetite is then weighed, and the percentage effectiveness of the kaolin-magnetite that can be extracted with the magnet after the adsorption process is calculated. This procedure is repeated until all the kaolin-magnetite has been extracted. The effectiveness calculation is shown by the following equation:

$$\%Effectiveness = \frac{mass\ of\ kaolin{-}magnetit\ bound\ magnet}{initial\ mass\ of\ kaolin{-}magnetite} x\ 100\% \qquad (1)$$

RESULT AND DISCUSSION

The XRF results in Table 1 show that the largest content in AK is Si = 62.17% and Al = 16.89%, which are the constituent elements of kaolin [21]. The iron (Fe) content in M has the highest percentage at 97.16%. The largest content in the kaolin-magnetite (KM) composite is Fe = 74.37%, Si = 13.05%, and Al = 6.24%. These compositions confirm the presence of both kaolin and magnetite within the composite material.

Table 1. The percentage (%) of elements in Activated Kaolin (KA), Magnetite (M), and Kaolin-Magnetite (KM)

Elements	Activated Kaolin	Magnetite	Kaolin-Magnetite
Fe	3.64	97.16	74.37
Si	62.17	0.42	13.05
Al	16.89	0.31	6.24
K	6.22	0	1.61
Ag	0.68	0.13	1.32
P	3.32	0.91	1.15
Ti	4.87	0	1.05
Ca	1.91	0.35	0.84
Eu	0.02	0.164	0.11
Mn	0.02	0.08	0.06

The XRD results for activated kaolin (AK) show the kaolinite indicated by peaks at 2θ = 12.33^{o}, 19.90^{o}, 24.88^{o}, and 26.62^{o}, which correspond to the diffractogram for kaolinite ($Al_2Si_2O_5(OH)_4$) in accordance with JCPDS No. 14-0164 [22]. The mineral kaolinite on Capkala Kaolin is also shown based on diffraction peaks at 2θ = 12,33^{o} (d = 7.17 Å) and 24.95^{o} (d = 3.56 Å) [23]. The XRD diffractogram for magnetite (M) shows peaks at 2θ = 35.52^{o} and 62.90^{o} corresponding to the magnetite (Fe_3O_4) diffraction peaks in accordance with JCPDS No. 01- 1111 [22] and also corresponding to the Fe_3O_4 by the American Mineralogist Crystal Structure Database (AMCSD) [20]. Additionally, characteristic peaks of iron oxide are shown at 2θ = 30^{o}, 35.3^{o}, 43^{o}, 53.3^{o}, 56.8^{o}, and 62.5^{o} in accordance with JCPDS No. 19-0629 [24]. The XRD results for kaolin-magnetite (KM) show diffractions from both AK and M peaks. Peaks at 2θ = 12.42^{o}, 20.16^{o}, 24.93^{o}, and 26.66^{o} correspond to AK, while peaks at 2θ = 35.51° and 62.84° correspond to M [22]. This is also reported, where the diffraction of Fe_3O_4-kaolin synthesized at a ratio of 1:1 is found at 2θ = 12.23^{o}, 20.83^{o}, 24.83^{o}, 26.6^{o}, 30.48^{o}, 35.625^{o}, and 62,5^{o}, providing information about the combination of kaolin and Fe_3O_4 diffraction peaks [25]. The d-spacing values for AK are 12.33^{o} (d = 7.18 Å), 19.90^{o} (d = 4.46 Å), 24.88^{o} (d = 3.58 Å), and 26.62^{o} (d

= 3.35 Å), which show slight changes in KM at 12.42° (d = 7.13 Å), 20.16° (d = 4.40 Å), 24.93° (d = 3.57 Å), and 26.66° (d = 3.34 Å). The d-spacing values for M are 35.52° (d = 2.53 Å) and 62.90° (d = 1.48 Å), for KM at 35.51° (d = 2.53 Å) and 62.84° (d = 1.48 Å). The peak values and d-spacing in the KM composite show no significant changes compared to those in the AK and M peaks. This indicates that the M phase is unlikely to be within the AK interlayer but is attached to the surface of activated kaolin, suggests the completeness of the KM composite's crystal structure, and indicates that the magnetite phase is likely present on the surface of activated kaolin [26].

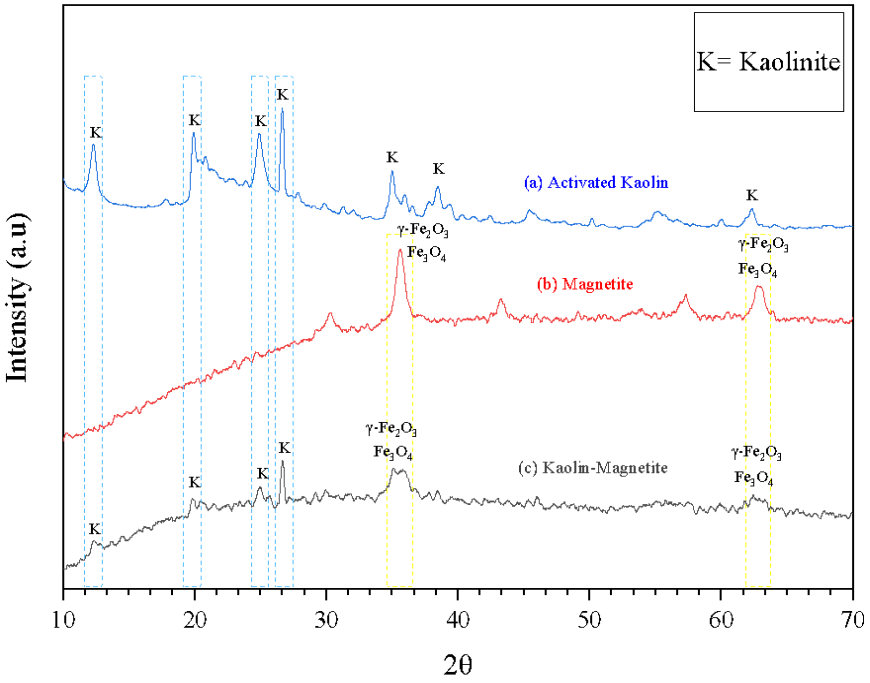

Figure 1. Diffractogram of Activated Kaolin (AK), Magnetite (M), and Kaolin-Magnetite (KM)

The GSA results show that AK has the largest surface area and the smallest porosity compared to M and KM, which is shown in Table 2. The decrease in surface area from AK to KM can occur due to the addition of iron oxide during the synthesis process of the KM composite. This is thought to be due to the addition of a high concentration of magnetite, namely 50%. The addition of high concentrations of Fe_3O_4 can cause the collapse of the kaolin structure and particle aggregation, which allows a decrease in the surface area of the particles [3]. The incorporation of porous iron oxide into kaolin increases the pore size in KM to 4.22 nm compared to AK, which is 3.65 nm. This indicates the role of iron oxide in enhancing the porosity of the kaolin composite [6]. The addition of M to AK causes a small portion of the AK surface to be covered, which subsequently reduces the surface area to 111.61 m^2/g, an increase in pore size to a pore volume of 0.24 cc/g, and an average pore radius of 4.22 nm.

Kaolinite, which is a clay with a 1:1 structure, has a small cation exchange capacity and does not swell. It is rarely reported that the structure involves intercalation by metal oxides. These different structures indicate that the MNP/kaolinite material was mostly prepared using the coprecipitation method [27]. This also shows that magnetite forms on the surface of kaolinite, as evidenced by the results of the diffraction angles of AK and KM, which are not significantly

different, and the absorption band of AK resembling that to KM [26].

Table 2. GSA data results of Activated Kaolin (AK), Magnetite (M), and Kaolin-Magnetite (KM)

Sample	Surface Area (m^2/g)	Total Pore Volume (cc/g)	Average pore radius (nm)
Activated Kaolin	115.29	0.21	3.65
Magnetite	81.03	0.29	7.04
Kaolin-Magnetite	111.61	0.24	4.22

The FTIR spectra of activated kaolin and magnetic kaolin indicate the appearance of absorption peaks at wavenumbers 3695 cm^{-1} and 3620 cm^{-1}, indicating the stretching vibration of Al-OH bonds [6], and an adsorption peak at 3446 cm^{-1}, showing the stretching vibration of OH from water molecules [25]. However, the reduced intensity of the absorption band in kaolin-magnetite indicates the possibility of Al^{+3} exchanging with Fe^{+3} on the kaolin surface. Peaks around 1000 cm^{-1} indicate the stretching vibrations of Si-O [4]. The sharp peak at 916 cm^{-1} in AK shows the stretching vibration of Al-OH, but in KM, this band weakens, indicating the interaction of Al-O in kaolinite with Fe-O bonds for KM formation [6]. Absorption peaks at 1635 cm^{-1} in AK and 1647 cm^{-1} in KM indicate the bending vibrations of H-O-H from water molecules [28]. The absorption peak at 538 cm^{-1} represents the bending vibration of Si-O-Al [29], and the peaks at 470 cm^{-1} and 472 cm^{-1} represent the stretching vibrations of Si-O [19]. The absorption peak for Fe-O in KM appears at wavenumber 430 cm-1 indicating the Fe-O vibration of magnetite [30]. Spectra in M show absorption peaks at 623 cm^{-1} representing the Fe-O vibration of maghemite, and the peaks at 580 cm^{-1} and 443 cm^{-1} respectively, indicating the Fe-O vibration of magnetite [30]. Additionally, there is an absorption peak at 3442 cm^{-1} indicating the stretching vibration of OH water molecules on the surface of Fe_3O_4, which is likely to bind with kaolin [22]. Another absorption peak appears at 1627 cm^{-1} indicating the bending vibration of OH due to water molecules absorbed by Fe_3O_4 on its surface [22].

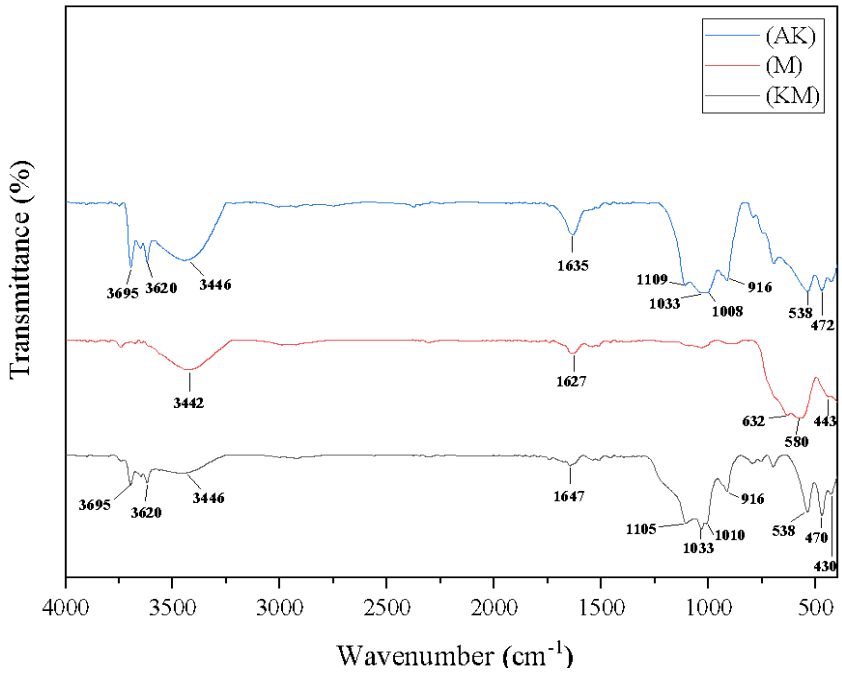

Figure 2. FTIR Spektrum of Activated Kaolin (AK), Magnetite (M), and Kaolin-Magnetite (KM)

The SEM results of activated kaolin, magnetite, and kaolin-magnetite that appear in Figure 3 demonstrate the presence of magnetite particles on the surface of activated kaolin, particularly in the KM composite. The technical morphology analysis using SEM shows that the characteristic kaolin morphology consists of heterogeneous layered sheets with heterogeneous sizes [31]. The dark spots of uneven sizes on the surface of KM indicate the formation of magnetite, resembling spherical shapes and agglomerates, which have been generated on the surface of the KM composite [26].

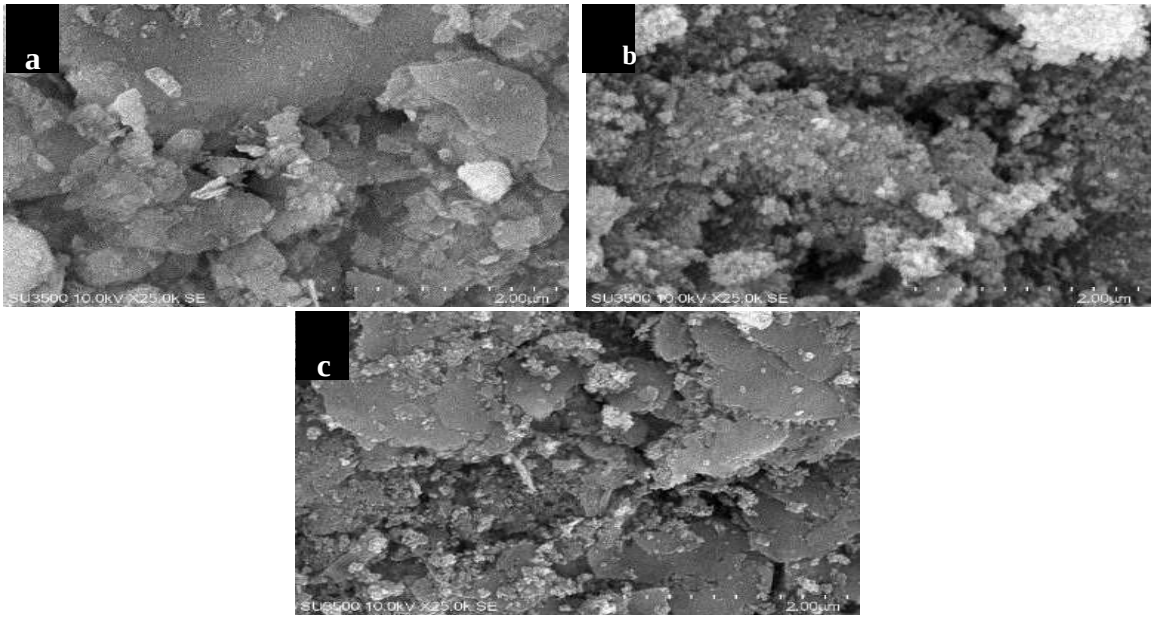

Figure 3. SEM of (a) Activated Kaolin (AK), (b) Magnetite (M), and (c) Kaolin- Magnetite (KM)

Based on the EDX mapping as shown in Figure 4, it is indicated that the AK is primarily composed of Al, Si, and O. Additionally, minimal amounts of Fe and other elements were found in this adsorbent [32]. While KM composite contains the elements O, Al, Si, K, and Fe. The Fe in the KM composite is higher compared to AK, suggesting the incorporation of Fe_3O_4 nanoparticles in the composite [32]. This indicates that Fe in the KM composite has been well-distributed on the surface of AK [33].

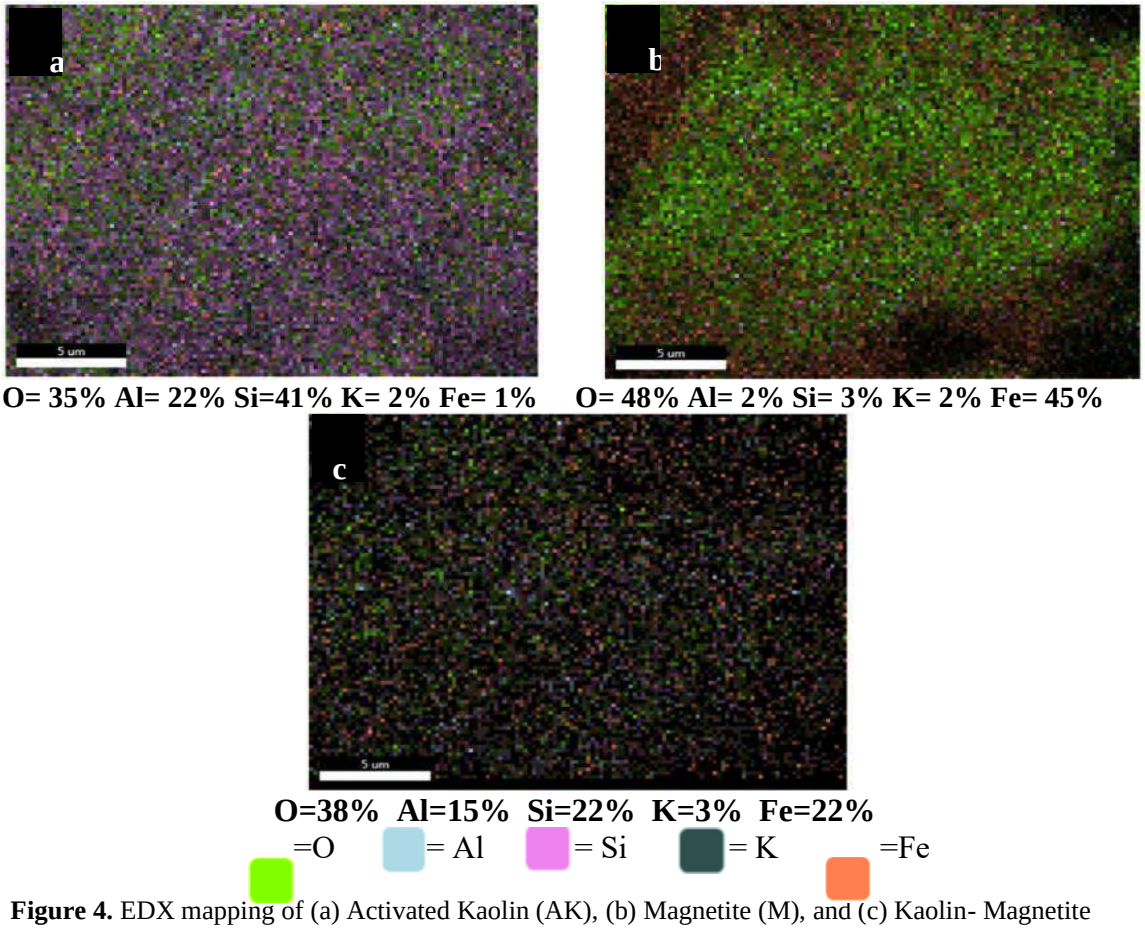

Figure 4. EDX mapping of (a) Activated Kaolin (AK), (b) Magnetite (M), and (c) Kaolin- Magnetite (KM)

The hysteresis curve analysis from VSM, as seen in Figure 5 and Table 3, indicates a decrease in the values of saturation magnetization (Ms), coercivity (jHc), and remanence (Mr) in M from values of 63.57 emu/g, 91.07 Oe, and 6.01 emu/g, respectively. When composited with AK to produce KM, these values decrease to 10.46 emu/g, 81.35 Oe, and 0.66 emu/g. The decrease in Ms value in the kaolin/Fe_3O_4 composite is due to the magnetic energy of the particles being proportional to their particle size. Hence, the decrease in magnetic strength of the particles, followed by an increase in surface area due to composite formation, causes a decrease in saturation magnetization [19]. The decrease in saturation magnetization indicates that the composite particles can be easily removed from the adsorbate using an external magnetic field after being used in the adsorption process. The decrease in magnetic strength of the kaolinite-magnetite composite is attributed to the non-magnetic nature of kaolin [6]. This is also similar to what was reported regarding the results of magnetization analysis on magnetite-modified kaolin. Even though the saturation magnetization value of magnetite-modified kaolin is significantly lower than that of magnetite (Fe_3O_4). When the magnetic properties of natural

kaolin and magnetite-modified kaolin were tested, it showed that magnetite-modified kaolin can be reassembled with magnets, while natural kaolin remains dispersed in the environment. The magnetic properties of magnetite-modified kaolin will make magnetite-modified kaolin more attractive as an adsorbent in practical applications [28].

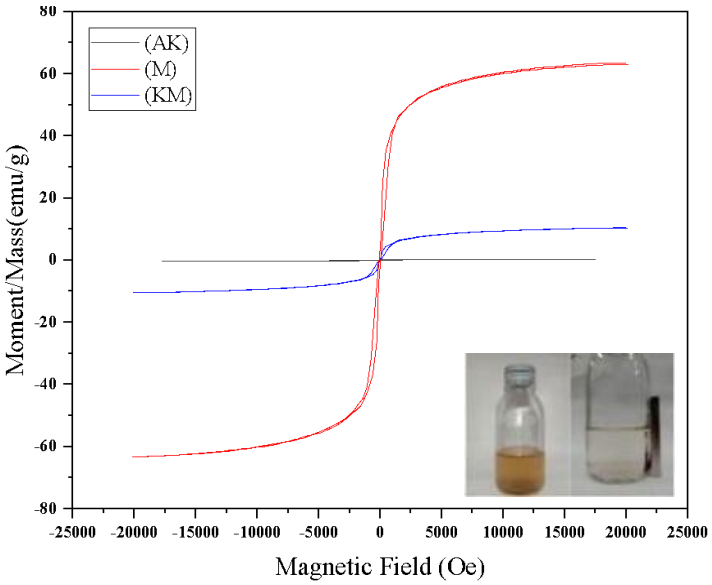

Figure 5. Hysteresis Curve of Activated Kaolin (AK), Magnetite (M), and Kaolin-Magnetite (KM)

Table 3. VSM data result of Activated Kaolin (AK), Magnetite (M), and Kaolin-Magnetite (KM)

Sample	Activated Kaolin	Magnetite	Kaolin-Magnetite
Magnetization, Ms (emu/g)	0.28	63.57	10.46
Coercivity, jHc (Oe)	613.91	91.07	81.35
Retentivity, Mr (emu/g)	0.07	6.01	0.66

Effectiveness of the Magnetic Property of Kaolin-Magnetite Adsorbent

The separation of the kaolin-magnetite adsorbent after the paracetamol adsorption process was carried out at a concentration of 10 ppm with a duration of 30 minutes. The separation of kaolin-magnetite was performed using a magnetic rod, as seen in Figure 6, and it was found that kaolin-magnetite could be separated from paracetamol in less than 1 minute. This indicates that kaolin-magnetite can respond to the magnetic field, allowing for easy and rapid separation from the adsorbate solution [4]. The separated KM was then dried and weighed, resulting in an efficiency of kaolin-magnetite separation after adsorption by the magnetic rod of 97.17%. This result is higher than the separation of magnetic activated carbon from aniline solution using a magnetic rod, which is 87% [20]. Thus, after being separated from the adsorbate, the KM adsorbent has the potential to be reused.

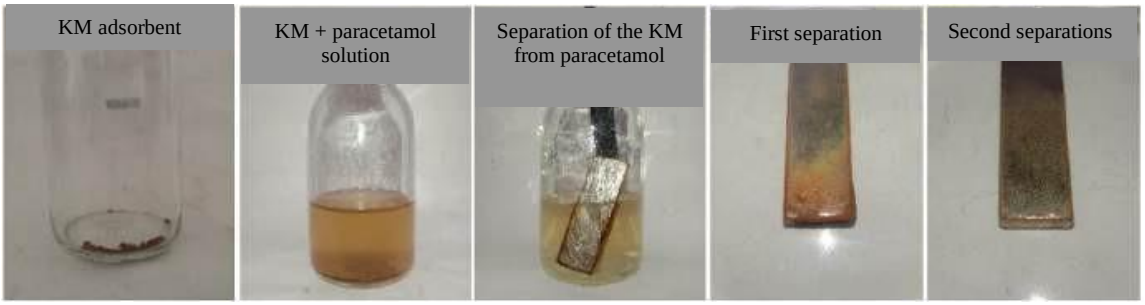

Figure 6. The separation of KM using a magnetic rod

Adsorption in this study tends to be based on the surface area, which plays a role in the adsorption process. The predicted mechanism of interaction between KM composite and paracetamol can be illustrated as shown in Figure 7.

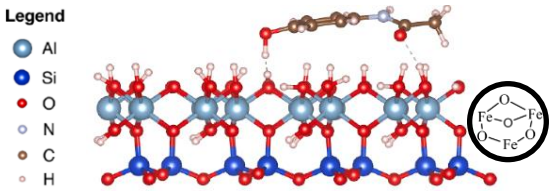

Figure 7. Paracetamol adsorption on the surface of kaolinite.

The adsorption process of paracetamol onto kaolinite's surface can form bonds between the hydroxyl (OH) groups on paracetamol and the oxygen (O) atoms on kaolinite's surface, as well as bonds between the O atoms of the amide on paracetamol and the OH groups on the kaolinite's surface [7]. According to this study, the presence of OH groups on KM is indicated by the detection of O-H groups at wavenumbers 3695 cm^{-1} and 3620 cm^{-1}, originating from the stretching vibration of Al-OH. Magnetite only adheres to the surface of kaolin, providing magnetic properties to the adsorbent and making it easily retrievable after the adsorption process using a magnetic rod.

CONCLUSION

The kaolin-magnetite composite (KM) has been successfully synthesized using the coprecipitation method. Characterized by XRF, XRD, FTIR, GSA, SEM-EDX, and VSM, indicating the presence of magnetite (Fe_3O_4) that gives magnetic properties to the KM composite. The magnetic properties of the KM composite enhance its ability to be separated from the paracetamol solution, with an efficiency of 97.17% of KM retrieved by the magnetic rod.

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