



Preliminary Studies on Magnetite/Cdots Nanocomposite: Structure, Functional Compound, and Morphology

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Received
11 August 2024

Revised
30 September 2024

Accepted for Publication
17 October 2024

Published
31 October 2024



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Abstract

This research develops Fe₃O₄/Cdots nanocomposites as an effective heavy metal adsorbent in batik industrial waste. The batik industry produces waste with hazardous heavy metal content that requires efficient processing methods. Fe₃O₄ was chosen because of its magnetic properties that facilitate separation after adsorption, while Cdots have the ability to detect and adsorb heavy metal ions. Fe₃O₄/Cdots nanocomposites are synthesized through hydrothermal methods and coprecipitation with Cdots materials sourced from orange juice and Fe₃O₄ from natural iron sand. XRD analysis confirmed the formation of the crystalline structure of Fe₃O₄ and the amorphous phase of Cdots, which are important for magnetic properties and adsorption. The FTIR spectrum shows the presence of functional groups that support interactions with metal ions. SEM test results showed particle sizes ranging from 35–65 nm, while EDX data confirmed the dominant composition of Fe₃O₄ and Cdots. This nanocomposite offers the potential as an efficient and magnetically separable batik waste treatment solution.

Keywords: Magnetite, Cdots, Nanocomposite, Adsorbent.

1. Introduction

In the era of industry 4.0, the growth of industries such as mining and textiles has a positive impact on job opportunities, but also triggers environmental problems in the form of wastewater. The textile industry, including batik production, produces waste with high levels of pH, COD, BOD, TSS, color intensity, as well as heavy metals such as Cadmium (Cd), Cobalt (Co), Copper (Cu), Nickel (Ni), Chromium (Cr), and Lead (Pb). These heavy metals are non-biodegradable and toxic, thus threatening the health of living things [1], [2]. Commonly used waste management methods are often inefficient, produce additional pollution, and have high operational costs [3], so more effective and economical alternatives are needed, such as adsorption techniques using nanotechnology materials [4], [5].

Carbon nanodots (Cdots) are carbon-based nanomaterials less than 10 nm in size, known for their unique characteristics such as high solubility in water, non-toxicity, as well as the ability to detect heavy metal ions [6]. Natural sources of Cdots include organic acids, fruit extracts, and food waste [7], [8], [9]. Some studies show that the adsorption efficiency of Cdots against heavy metals is quite high, but its separation from wastewater after the adsorption process is difficult [10], [11]. This problem makes Cdots less efficient to use on an industrial scale because of the need for expensive separation methods and complicated procedures.

Magnetite (Fe₃O₄) is a magnetic material with high biocompatibility, large surface area, and superparamagnetic performance that allows separation from wastewater using an external magnetic field [12]. The combination of Cdots with Fe₃O₄ in nanocomposite form offers additional advantages in the form of ease of separation, reuse efficiency, and strong adsorption properties [13]. Fe₃O₄/Cdots can be synthesized by hydrothermal methods, which has proven to be effective in producing high-

quality materials with high purity, without costly or lengthy costs compared to other methods such as reflux and sonication [14], [15], [16].

This research will utilize $\text{Fe}_3\text{O}_4/\text{Cdots}$ as a heavy metal adsorbent in batik industrial waste for the first time, using Cdots sourced from orange juice and Fe_3O_4 from natural iron sand in Java. These nanocomposites will be investigated in terms of structure, morphology, optical properties, and magnetic properties, as well as the influence of Cdots volume in the adsorption process. The results of the research are expected to offer an alternative solution that is efficient, easy to separate, and has the potential to be applied on an industrial scale for batik waste treatment.

2. Methods

In this study, the method used in the synthesis of Cdots is Hydrothermal which is included in the type of bottom-up synthesis method. The raw material to be used is orange juice which contains a lot of element C. Filtered orange juice, ethanol, and DI water are each 50 mL put into a beaker glass to be stirred using a 720 rpm magnetic stirrer for 10 minutes. After the stirring process is complete, the liquid is put into the autoclave reactor and then heated using an oven with a temperature of 180 C for 3 hours. The result is then stored in vial bottles and put in the refrigerator.

Then the nanocomposite synthesis process was continued using the co-precipitation method. Iron sand is extracted by the separation method using permanent magnets. The extraction process is carried out repeatedly to reduce the content of impurity compounds. The extraction process is carried out through a washing process using demineralized water, then dried in the sun. After the drying process, the sand was separated using a permanent magnet of 20 grams of Fe powder as a result of the separation was reacted with HCl 58 mL using *a magnetic stirrer hotplate* at a speed of 720 rpm at room temperature for 30 minutes. The results were then filtered using filter paper. Then, 18 mL of distiller filter results with 6 mL Cdots liquid for 30 minutes at a speed of 720 rpm then titrated with NH_4OH 25 mL in a hotplate magnetic stirrer at a speed of 720 rpm at room temperature for 30 minutes.

After the process, the resulting solution is washed repeatedly with aquades. Washing is carried out by adding aquades until the solution in the test tube is full. Then the solution is covered with plastic wrap to avoid the process of oxidation with the environment and precipitated. After the solution settles, the solution that does not contain the precipitate is discarded. The remaining precipitate is washed repeatedly in the same way up to pH 7. Next, it is filtered with filter paper, and baked at 100 C for 1 hour so that $\text{Fe}_3\text{O}_4/\text{Cdots}$ nanoparticle powder is produced.

3. Results and Discussion

The XRD data in the figure above shows the diffraction pattern of $\text{Fe}_3\text{O}_4/\text{Cdots}$ nanocomposites with peaks that correspond to the crystal structure of magnetite (Fe_3O_4) and the presence of an amorphous phase of carbon. Some of the major peaks appear at 2-theta angles around 30.1° (311), 35.5° (400), 43.1° (422), 53.5° (511), and 57.2° (440), which is consistent with Miller's index for cubic spinel structure of Fe_3O_4 , which corresponds to JCPDS reference number 19-0629. The presence of these peaks indicates that Fe_3O_4 is successfully formed in composites, which is important for maintaining the magnetic properties of the material and facilitating its separation from liquid media when used as a heavy metal adsorbent [17]. The study by Liu *et al.* (2021) also showed a similar diffraction pattern for Fe_3O_4 at 2θ around 30.1° , 35.5° , and 43.1° , indicating a stable magnetite crystal structure [18].

In addition to the peaks associated with Fe_3O_4 , there is also a wide peak region at about $20\text{-}30^\circ$ that can be attributed to the amorphous phase of carbon (C-dots) [19]. These wide peaks indicate the amorphous structure of carbon particles in nanocomposites, which are commonly found in C-dots or nanocosmic carbon materials [20]. This amorphous structure is beneficial in adsorption applications because it can provide more active sites and increase adsorption capacity [21]. These results are in line with research by Wang *et al.* (2020), which reported that the presence of C-dots with an amorphous structure increases the adsorption capacity of metal ions due to the larger carbon surface and the presence of active functional groups [22].

This diffraction pattern generated from XRD shows that the $\text{Fe}_3\text{O}_4/\text{Cdots}$ nanocomposite synthesis process successfully maintains the crystalline structure of Fe_3O_4 while introducing an amorphous carbon phase. This is important because the presence of Fe_3O_4 in crystalline form ensures optimal magnetic properties, which can facilitate the separation of materials from the solution after the adsorption of heavy metals is complete [23]. At the same time, the amorphous phase of C-dots increases

the number of active adsorption sites, which is beneficial for interaction with metal ions in waste treatment applications [24]. Previous research by Chen et al. (2020) also supports these findings, with reports that the combination of crystalline Fe_3O_4 and amorphous carbon improves the performance of adsorption and magnetic separation in waste treatment applications [25].

Overall, these XRD data show that $\text{Fe}_3\text{O}_4/\text{Cdots}$ nanocomposites have a structure suitable for application as heavy metal adsorbents. The combination of the crystalline phase of Fe_3O_4 and the amorphous phase of the C-dots provides advantages in adsorption capacity and ease of separation. With a structure that has been confirmed through XRD patterns, these nanocomposites have great potential for use in batik industrial waste treatment, where heavy metal ions must be effectively adsorbed and adsorbent materials must be magnetically segregated for more efficient waste management.

The FTIR spectrum of the $\text{Fe}_3\text{O}_4/\text{Cdots}$ nanocomposites in the figure above shows a number of characteristic peaks that signal success in the synthesis process. In an area of about 500 cm^{-1} , there is a peak corresponding to the vibration of the Fe-O bond, which is a key characteristic of the magnetite structure (Fe_3O_4) [26]. The presence of these peaks confirms that Fe_3O_4 is successfully integrated in the composite, which is important for adsorption function as the magnetic properties of Fe_3O_4 will facilitate the separation of materials after the adsorption process. Previous research also found a Fe-O peak of about 530 cm^{-1} in Fe_3O_4 nanocomposites that function as heavy metal adsorbents, which suggests that these Fe-O bonds are crucial for metal ion binding activity [13].

In the region of about $3200\text{--}3500\text{ cm}^{-1}$, there is a wide peak associated with O-H and N-H stretch vibrations [27]. These peaks indicate the presence of hydroxyl (-OH) and amine (-NH) groups on the surface of the nanocomposite, which can increase the adsorption capacity of heavy metals such as Cr(VI) and Pb(II) through hydrogen interaction and coordination bonds [28]. A study by Zhang et al. (2019) shows that the presence of hydroxyl groups and amines in carbon materials plays a role in increasing the interaction between materials and heavy metal ions, which increases adsorption efficiency [11]. These groups increase hydrophilicity and increase the number of active sites on the surface of the nanocomposite [29].

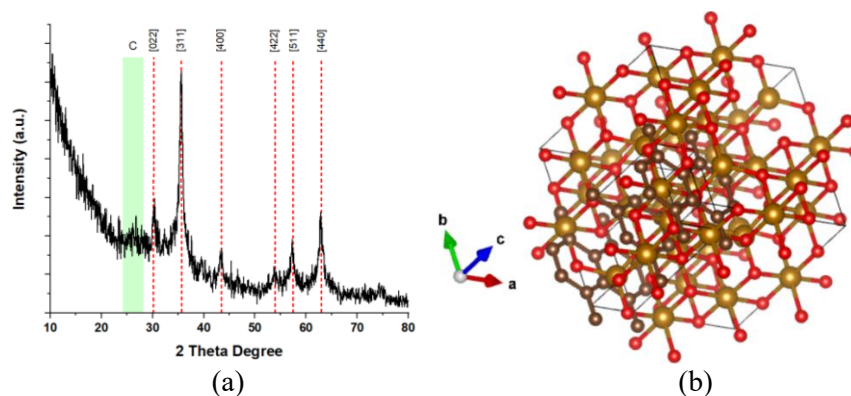


Figure 1. The Diffraction Pattern of $\text{Fe}_3\text{O}_4/\text{Cdots}$ Nanocomposite (a) and Its Crystal Structure (b).

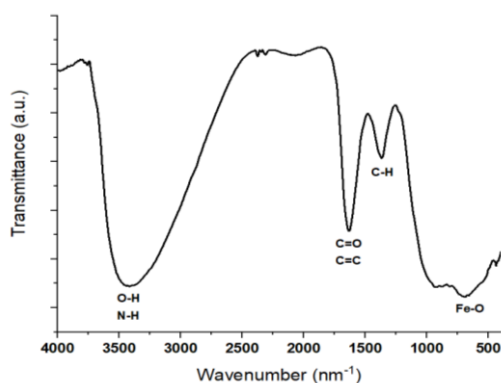


Figure 2. FTIR Spectrum of $\text{Fe}_3\text{O}_4/\text{Cdots}$ Nanocomposite.

In addition, in the region of about 1550–1650 cm^{-1} there is a peak that can be attributed to the stretching vibrations of C=O and C=C. C=O groups generally originate from partial oxidation of C-dots and can serve as active sites for binding metal ions through electrostatic interactions or complexation [30]. Meanwhile, the C=C group indicates the presence of aromatic structures in C-dots, which are often found in carbon-based materials [31]. Research from Li *et al.* (2021) shows that the presence of carbonyl groups in carbon materials increases the adsorption capacity of heavy metals because these groups have the ability to bind metals strongly, both through ionic and covalent bonds [32].

The success of the synthesis of $\text{Fe}_3\text{O}_4/\text{Cdots}$ nanocomposites is also supported by the presence of a C-H peak of around 2900 cm^{-1} , which indicates the presence of alkyl group remnants that may have originated from the synthesis process or precursor materials of C-dots [33]. This group is not directly involved in adsorption, but its existence suggests that the carbon structure in C-dots remains intact and compatible with Fe_3O_4 in composites [34]. Thus, the existence of functional groups such as Fe-O, O-H, N-H, C=O, and C=C indicates that these nanocomposites have a composition and structure that supports their application as heavy metal adsorbents in batik industrial waste.

The data shown in the SEM and EDX test results provide an important overview of the morphology and composition of the synthesized $\text{Fe}_3\text{O}_4/\text{Cdots}$ nanocomposites. Based on SEM images, it can be seen that the particles range in size from 35 to 65 nm, with a fairly even distribution, especially in the 45-55 nm range. This particle size distribution is important in heavy metal adsorption applications, as nanoparticles of uniform size tend to have a higher surface area, which is very advantageous in the adsorption process [29]. This is consistent with previous research that suggests that particle size and morphology can affect adsorption capacity in modified Fe_3O_4 -based nanomaterials [35].

The EDX data in the image shows the elemental composition of this $\text{Fe}_3\text{O}_4/\text{Cdots}$ nanocomposite, with the main contents being carbon (C), oxygen (O), and iron (Fe). This composition, with the dominant content of Fe (52.3% by weight) and oxygen (36.1% by weight), shows that Fe_3O_4 has been successfully formed and is a major component in the nanocomposite material. The presence of carbon with a percentage of 11.6% by weight shows that Cdots have also been successfully synthesized and fused with Fe_3O_4 particles. The existence of Cdots is important because Cdots can function as a matrix that improves adsorption properties and expands the surface area of the material. Previous studies have shown that Cdots can improve the stability and adsorption ability of composite materials [36].

Table 1. EDX Results of $\text{Fe}_3\text{O}_4/\text{Cdots}$ Nanocomposite.

Element	Weight %	Atomic %
C K	11.6	23.3
O K	36.1	54.2
Fe K	52.3	22.5

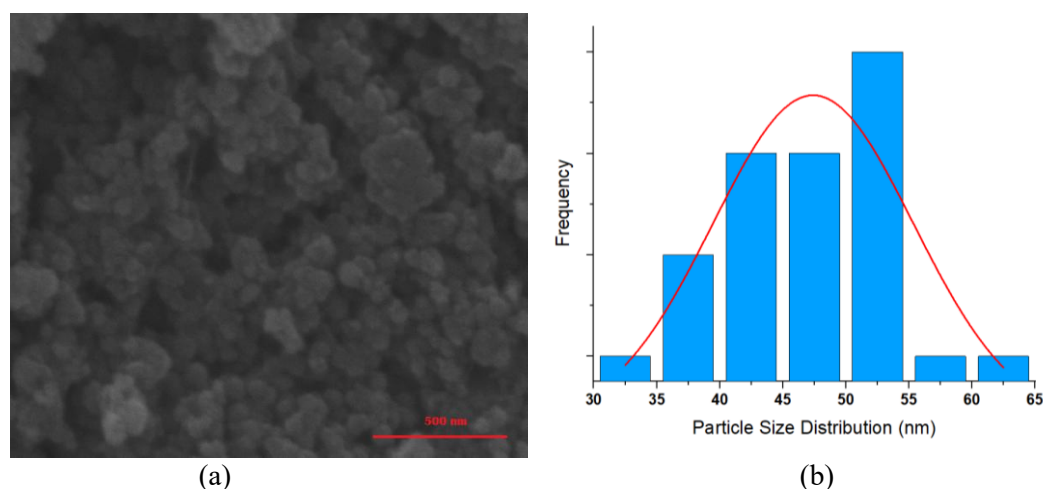


Figure 3. SEM Image (a) and Particle Size Distribution (b) of $\text{Fe}_3\text{O}_4/\text{Cdots}$ Nanocomposite.

The success of the synthesis of Fe₃O₄/Cdots nanocomposites can also be seen from the distribution of elements in the EDX data. The atomic percentage of Fe and O close to the proportion of Fe₃O₄ indicates that the composition of this iron oxide has been well formed, while the integrated amount of carbon indicates that the Cdots have successfully bonded to the surface of the Fe₃O₄ particles. In previous studies, the combination of Cdots with Fe₃O₄ was proven to increase the absorption of heavy metals, because Cdots play a role in increasing electrostatic interactions and increasing the solubility of particles in aqueous media [16].

With reference to research in the past five years, Fe₃O₄/Cdots composite materials have proven to be very effective as adsorbents for heavy metals such as Pb, Cd, and Cr in industrial waste, including batik industrial waste which is rich in hazardous heavy metals [27], [37]. The presence of nanostructures of Fe₃O₄ increases magnetic attraction, allowing for an easier sorbent separation process after the adsorption process is complete, which is a significant advantage in industrial applications [21]. Thus, the results of SEM and EDX show the success of the synthesis of Fe₃O₄/Cdots nanocomposites, which have great potential in environmental applications, especially in the treatment of batik industrial waste containing heavy metals.

4. Conclusion

This study succeeded in synthesizing Fe₃O₄/Cdots nanocomposites as heavy metal adsorbents by hydrothermal and coprecipitation methods. The results of XRD analysis showed that Fe₃O₄ was formed in the crystalline structure, while Cdots were present in the amorphous phase, which increased the adsorption capacity. The existence of functional groups such as Fe-O, O-H, and C=O identified through FTIR supports the interaction of nanocomposites with heavy metal ions, making them effective in waste treatment. SEM analysis revealed that these nanocomposites have a uniform particle size in the range of 35–65 nm, which provides a high surface area, advantageous in the adsorption process. EDX data showed the main compositions of Fe, O, and C, confirming the successful formation of Fe₃O₄ as the dominant component as well as the presence of Cdots that increased stability and adsorption capacity. This indicates that Fe₃O₄/Cdots nanocomposites have an optimal structure for application as heavy metal adsorbents. Overall, Fe₃O₄/Cdots nanocomposites have great potential as an alternative solution in the treatment of batik industrial waste containing heavy metals. The combination of Fe₃O₄ magnetic properties that allow for easy separation and high adsorption capacity of Cdots makes it an efficient and environmentally friendly material. The results of this study show that Fe₃O₄/Cdots nanocomposites can be implemented on an industrial scale for more effective and sustainable waste treatment.

Acknowledgment

This research is funded by the Ministry of Education, Culture, Research and Technology through the DRTPM research grant for the PPS-PTM scheme No. 11.6.32/UN32.14.1/LT/2024.

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