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Enhanced Copper Ion Adsorption by Rice Husk and Sugarcane Bagasse-based Magnetic Nanoparticles Biocomposites

***Iryanti Fatyasari Nata** *,1,2,3*
***Chairul Irawan** 1*
***Muhammad Istnaeny Hudha** 4*
***Aceng Lesti** 1*
***Gita Cristy** 1*
***Muhammad Hersandy Syarkani** 1*
***Allam Naufal**1*
[1] *Department of Chemical Engineering, Lambung Mangkurat University, Banjarbaru 70714, Indonesia*
[2] *Wetland-based Materials Research Centre, Institution of Research and Community Services, Lambung Mangkurat University, Banjarbaru 70714, Indonesia*
[3] *Integrated Laboratory, Lambung Mangkurat University, Banjarbaru, 70714, Indonesia*
[4] *Department of Chemical Engineering, National Institute of Technology, Malang 65125, Indonesia*
*e-mail: ifnata@ulm.ac.id

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Abstract. Rice husk and sugarcane bagasse are biomass waste by-products of agriculture activities. High cellulose content makes them a potential material to develop. Biomass utilization is more interesting to develop due to its high cellulose content and nature abundance. Cellulose from rice husk and sugarcane bagasse is utilized as fiber in producing magnetic biocomposites. The Biocomposites can adsorb Cu(II) ions in aqueous solutions. A one-step solvothermal reaction was used to synthesize biocomposites. The amine functionalization on biocomposites was also studied for adsorption performance. The magnetic particle was generated on the surface of fiber and verified by XRD as Fe_3O_4. Amine group on biocomposites is also found on 570 cm^{-1} and 1636 cm^{-1} peaks for Fe-O bonds and N-H bending, respectively. Batch isothermal adsorption employing magnetic biocomposites has optimal conditions for Cu(II) ions adsorption at pH 5 for 60 minutes, with an adsorption capacity of 118.26 mg/g. The reusability of biocomposites for the next run showed a good performance at 3rd repetitions with only a decrease in adsorption capacity of 8.81%. The rice husk and sugarcane bagasse-based magnetic nanoparticles biocomposites can adsorb Cu(II) ions and potential material to develop for wastewater treatment.

Keywords: Adsorbent, Bagasse, Biocomposites, Copper, Rice Husk

INTRODUCTION

Biomass waste in the form of rice husk and bagasse is part of agricultural activities. The sugarcane bagasse has a composition of 26%-43% cellulose, 17%-23% hemicellulose, and 13%-22% lignin, while rice husk consists of 15%-20% silica, 50% cellulose and 25%-30% lignin (Ilindra *et al.*, 2008; Oliveira *et al.*, 2016; Supranto *et al.*, 2015). Rice husk and bagasse are wastes generated from agricultural products that have yet to be

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maximally utilized. The content of each of these materials provides an opportunity to be developed (Yu *et al.*, 2021), such as carbon material for Pb(II) ion adsorption (Annissuzzaman *et al.*, 2023; Mc Lein Roger *et al.*, 2023). Cellulose from rice husks and bagasse could be employed as a fiber source in manufacturing magnetic nanoparticle biocomposites used as adsorbents in wastewater treatment. The fiber contained in rice husk and bagasse can be used as raw composite material.

The development of the composite technique is interesting to explore, as it has a good performance in physical properties as a new material. Magnetic nanoparticles with nanometer size are an interesting material to develop in wide applications in industry, science, and technology. Magnetic nanoparticles are used as functional material in research such as drug delivery (Bhatti *et al.*, 2022), cancer therapy (Dongsar *et al.*, 2023), bioseparation (Alimohammadi *et al.*, 2022), and water treatment (Irawan *et al.*, 2023). The synthesis of magnetic nanoparticles with stable, uniform, and high levels of magnification could be done by the solvothermal method, and surface functionalization has been developed and applied for adsorbed metal ions (Nata *et al.*, 2023; Nata *et al.*, 2022a; Nata *et al.*, 2022b). The functionalization of amine on magnetic nanoparticles enhances the interaction with metal ions due to the formation of $–NH_3^+$, and during synthesis, a small magnet was also produced. The small size of magnetic particles also provides a high surface area. The magnetic property of the adsorbent helps in the separation process, making it fast and easy to separate.

The combination of magnetic nanoparticles and biomass fiber produces biocomposites. Biocomposites based on biomass fiber and magnetic have been investigated for water treatment. The adsorption process is considered a practical and affordable approach for adsorbed metal ions. The ability of magnetic biocomposites was observed by other researchers for the adsorption of dye and chromium ions (Aryee *et al.*, 2022), lead and cadmium ions (Irawan *et al.*, 2023), copper ions (Nata *et al.*, 2022b), and other contaminants (Irawan *et al.*, 2021). However, no research observes copper ions that use a combination of two biomass fibers as biocomposites. On the other hand, the textile industry is a major producer of liquid waste caused by the textile refinement process, which contains chemicals, metal ions, and other contaminants that can lead to the destruction of river biota whose role is very important for life and environment (Badawi & Zaher, 2021). This study examines how well rice husk-sugarcane bagasse-based magnetic nanoparticle biocomposites adsorb Cu(II) ions. It also looks at the characterization of magnetic biocomposites.

MATERIALS AND METHOD

Materials

Rice husk (RH) and sugarcane bagasse (SB), harvested in Gambut and Banjarbaru, South Kalimantan, Indonesia, are utilized as raw materials for the fiber source. The ethylene glycol (99.8%, $C_6H_6O_2$), sodium hydroxide (98%, NaOH), 1-6 hexanediamine (99.8%, $C_6H_{12}N_2$), iron (III) chloride hexahydrate (97.%, $FeCl_3.6H_2O$), sodium acetate anhydrous (99.9%, $C_2H_3NaO_2$), hydrochloric acid (37%, HCl), and ethanol (98%, C_2H_6O) were purchased from Sigma Aldrich. The grade chemicals are pure and used without purification.

Delignification of Rice Husk and Sugarcane Bagasse

After being screened and cleaned to remove contaminants, the RH and SB were dried for 24 h at 100 °C in an oven. Individually, the fibers were sized into powder and passing ±50/60 mesh. 1% NaOH (40 % v/v) was added to the RH and SB, and they were then agitated at 100 rpm for two hours at 80 °C. Following filtration, the material was dried for 4 h at 100 °C, and the raffinate was rinsed with deionized (DI) water until the filtrate was almost pH 7. The delignification of RH and SB are called RH-F and SB-F, respectively.

Magnetic Biocomposites Synthesis

Teflon Stainless Steel Autoclave used as a reactor in the solvothermal preparation of magnetic nanoparticle biocomposites. In summary, 1.6 g of sodium acetate anhydrous, 7 mL of 1-6 hexanediamine, 0.8 g of iron (III) chloride, and 24 mL of ethylene glycol were combined and heated to 50 °C while being agitated at 150 rpm for 5 min. After 10 min, add 0.5 g of each RH-F and SB-F (at a 1:1 ratio). Following the allotted period, the mixture was placed in the reactor and heated to 200 degrees Celsius for 6 h. After the reactor was allowed to cool to normal temperature, sedimentary black solid biocomposites were formed. Wash the solid portion three times with 50% ethanol and DI water. The product is called B-MNH_2. Two types of biocomposites were created; the second type did not contain 1-6 hexanediamine (B-M). The reactivity effect of amine functionalization on biocomposites was examined using the B-M as a control. For the subsequent experiment, the B-M and B-MNH_2 were maintained in DI water.

Cu(II) ion Adsorption via Magnetic Biocomposite

In a batch experiment, biocomposites were studied for their adsorption. The Cu(II) ions solution was prepared from an artificial solution (50 mg L^{-1} as the starting concentration). The pH of solution (4, 5, 6, 7, and 8) and adsorption contact duration (5, 15, 30, 60, 120, and 240 min) were recorded. NaOH (0.1 M) and HCl (0.1 N) were used to establish the pH. 40 mg of biocomposites (B-M and B-MNH2) were added to a 200 mL solution of Cu(II) ions. Next, shake for 150 rpm at room temperature and sample for certain time adsorption. An external magnetic field collected the biocomposites following the adsorption process, and an atomic absorption spectroscopy (AAS) was used to detect the concentration of Cu(II) ions that remained in the solution. The measurement was taken twice. Desorption of B-M and B-MNH_2 loaded with Cu(II) ions in 0.1 N HCl at 150 rpm for 4 h demonstrated the recurrent use of biocomposites. After the desorption period was over, the biocomposites were rinsed with DI water until the pH of the washing water was about 7. At which point B-M and B-MNH_2 were utilized as the adsorbent for the subsequent run. The adsorption capacity of Cu(II) ions was computed using Eq. (1).

$$q_e=(C_o-C_e)\frac{V}{m} \quad (1)$$

The variables Co (mg L^{-1}) and Ce (mg L^{-1}) represent the starting and equilibrium concentrations of Cu(II) ions, V (L) denotes the volume of the solution, and m (g) denotes the amount of adsorbent.

Characterization of Material

Field-emission scanning electron microscopy (FE-SEM, JOEL JSM-6500F) was used to investigate the structure and morphology of the materials. The sample was coated with sputter platinum before observation. The identification of functional groups on materials was conducted by Fourier transform infra-red spectrometry (FT-IR, digilab FTS-3500, bio-rad). The Rigaku D/max-b XRD machine was utilized to perform X-ray diffraction (XRD) using copper k-alpha (CuKα) radiation, operating at 400 kV voltage and 100 mA current. Eq. (2) is used to determine the Crystalline Index (CrI) of the material.

$$\mathrm{CrI} = \frac{I_{002} - I_{am}}{I_{002}} \quad (2)$$

The crystalline index (%) is represented by CrI, the material's amorph intensity by I_{am}, and the crystal intensity by I_{002}.

RESULTS AND DISCUSSION

Magnetic Biocomposite Characterizations

Rice husk and bagasse are known as lignocellulosic and have complex structures, particularly for rice husk that contains silica, the fiber surface of both sugarcane bagasse (Figure 1b) and rice husk (Figure 1a) exhibit rod-like clusters according to FE-SEM observation (Ciannamea *et al.*, 2010). After delignification, the surface of each material becomes smooth because the NaOH solution damages the lignin structure during the reaction. As lignin and hemicellulose dissolve on materials, the amorphous structure transforms into crystals (Banu Jamaldheen *et al.*, 2022; de Carvalho Neto *et al.*, 2014).

Figure 2 depicts the morphological structure of magnetic biocomposites, with magnetic particles developing on the surface of B-M and B-MNH$_2$. For B-MHN$_2$, the magnetic particles were evenly dispersed across the surface (Figure 2b); on the other hand, some parts were empty of magnetic particles (red circle) for B-M (Figure 2a). The synthesis of B-MHN$_2$ presence of 1,6 hexanediamine as an amine source protects growth particles and initiates the other particles. Amine functionalization influences the distribution and size of magnetic particles.

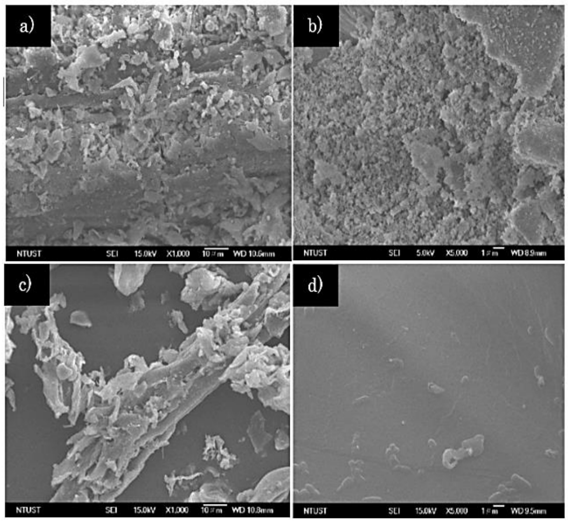

Fig. 1: FE-SEM images of (a) rice husk (RH); (b) sugarcane bagasse (SB); (c) rice husk after delignification (RH-F); and (d) sugarcane bagasse after delignification (SB-F).

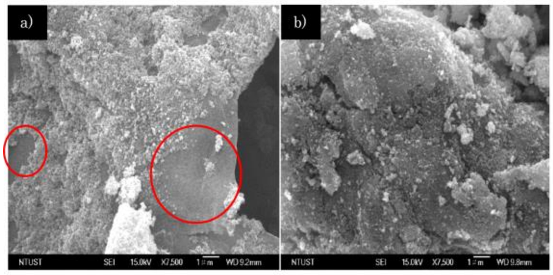

Fig. 2: FE-SEM images of (a) magnetic biocomposites without amine functionalization (B-M) and (b) magnetic biocomposites with amine functionalization (B-MNH$_2$)

This result is related to previous research for magnetic biocomposites synthesis by solvothermal method (Nata *et al.*, 2010; Nata *et al.*, 2011; Wang *et al.*, 2006). The functionalization of amine on biocomposites produced a high magnetic content and size around 30-50 nm (Nata *et al.*, 2023).

XRD analyzed the crystal structure of materials and Crystallinity Index (CrI) before and after delignification were. The Cellulose crystals shown as a dominant peak could be detected in the range of 2θ (20°- 80°) (Hashemian *et al.*, 2013). The amorphous part of cellulose is seen at an angle of 2θ (0°-20°) with broad diffraction (Mohadi *et al.*, 2014). Based on observation, the RH and SB containing cellulose fibers in their constituent structures have a characteristic peak at 18.7° (cellulose I) and 22.4° as amorphous and crystalline (cellulose II), respectively (Nata *et al.*, 2023). The delignification treatment on RH and SB increases the crystalline structure shown by higher intensity in the crystal region. It is assumed that the release of lignin and hemicellulose into the solution altered the structure of the fibers. Table 1 shows the CrI values of RH, SB, RH-F, and SB-F, which have increased CrI after delignification. The RH and SB have increased about 37.5% and 90.3%, respectively. The amorphous peak's reduced form indicates the organization of polysaccharide structures, whereas the high intensity of crystalline peak indicates the presence of crystalline cellulose (Ghali *et al.*, 2009).

Table 1. The characterization of RH, RH-F, SB, and SB-F for XRD spectra

Sample	Characteristics peak		Crystalline Index (CrI, %)
	Amorphous (18.7°)	Crystals (22.4°)	
RH	771	1.208	56.680
RH-F	686	1.218	77.551
SB	464	665	43.319
SB-F	564	1.029	82.447

Based on XRD observation, the magnetic biocomposites produced by the solvothermal method were identified as magnetite (Fe_3O_4), as evidenced by peaks at 36, 43, and 57 degrees. The detected peaks were fitted with a magnetite standard pattern (JCPDS card 39-0664). Specific for B-MNH_2, the higher intensity of Fe_3O_4 on spectra is due to the smallest particles on B-MNH_2, which affects the diffraction spectra through the sample (Nata *et al.*, 2023). The XRD pattern is shown in Figure 3.

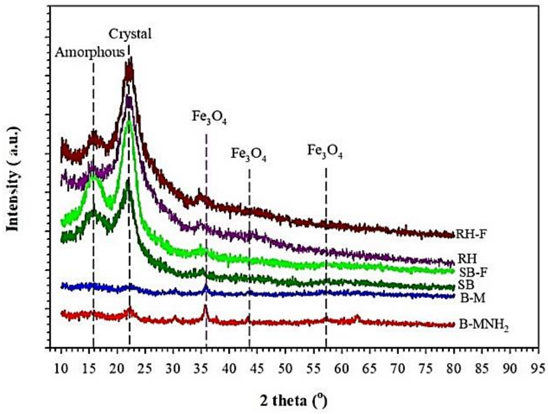

Fig. 3: X-Ray Diffraction (XRD) spectra rice husk (RH), sugarcane bagasse (SB), rice husk after delignification (RH-F), sugarcane bagasse fibers delignification (SB-F), magnetic biocomposites without amine functionalization (B-M), and magnetic biocomposites with amine functionalization (B-MNH_2).

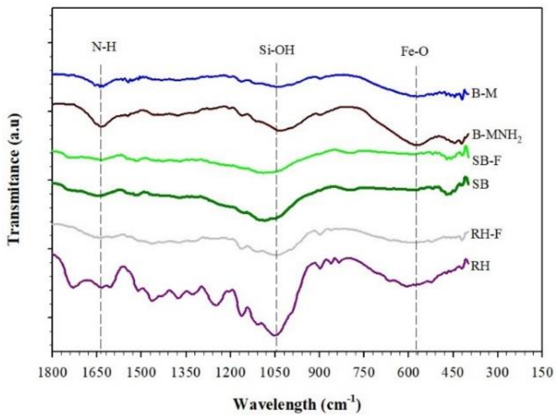

Fig. 4: FT-IR spectra of rice husk (RH), sugarcane bagasse (SB), rice husk after delignification (RH-F), sugarcane bagasse fiber delignification (SB-F), magnetic biocomposites without amine functionalization (B-M), and magnetic biocomposites with amine functionalization (B-MNH_2).

Figure 4 displays the FT-IR spectra of RH, RH-F, SB, SB-F, B-M, and B-MNH_2. The samples' functional groupings were identified and displayed at specific peaks. The magnetic Fe-O stretching in Fe_3O_4 was detected at 570 cm^{-1} for BM and BMN_2. The peak of amine group was visible for N-H bending vibration at 1636 cm^{-1}. The sharp Si-OH binding was shown at 1045 cm^{-1} (Nata *et al.*, 2023).

Adsorption Study of Cu(II) Ions by Magnetic Biocomposites

In the first 30 min of adsorption, the adsorbent's capacity to absorb Cu(II) ions rose noticeably, reaching 52.51 mg/g and 116.63 mg/g for B-M and B-MNH_2, respectively (Figure 5). The higher starting concentration in first adsorption provides the driving force needed to overcome the obstacle to the mass transfer of metal ions between the aqueous phase and the solid phase (Yu *et al.*, 2013). For B-M and B-MNH_2, the adsorption capacity was 53.42 mg/g and 118.60 mg/g, respectively, after an additional 240 minutes of adsorption time. After 60 min, the adsorption capacity becomes constant, indicating that the Cu(II) ion has reached saturation on the surface of adsorbent. Cu(II) ions adsorb via surface complex formation, ion exchange, and Cu(II) ions' electrostatic interactions with the adsorbent surface (Gómez-Pastora *et al.*, 2014). The B-MNH_2 has a higher adsorption capacity of 2.1-fold than that of B-M. A greater interaction between amine functionalized on magnetic surfaces and Cu(II) ions results in a reduced diameter (50 nm), excellent permeability, and stable mechanical and thermal properties (Nata *et al.*, 2022b).

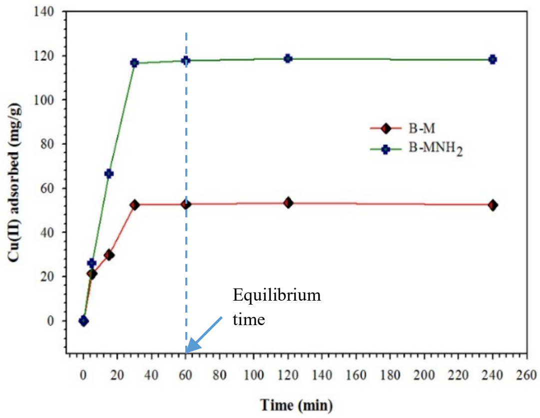

Fig. 5: The Cu(II) ions adsorption capacity on various contact times by B-M and B-MNH_2. Reaction conditions: 200 mL of sample, 0.05 g of adsorbent, stirring at 150 rpm and pH 5.

The pH of the solution influences the adsorbent's adsorption capacity, which is connected to the protonation or deprotonation of the active site surface. pH impacts degree of ionization, the adsorbent surface charge, and adsorbable species in the adsorption process (Irawan *et al.*, 2023), so that pH values can affect chemical equilibrium, both in the adsorbate and adsorbent. Figure 6 shows that B-M and B-MNH_2 can absorb Cu(II) ions.

The optimal pH solutions are pH 6 (B-M) and pH 5 (B-MNH_2), with adsorption capacities of 53.41 mg/g and 118.26 mg/g, respectively. Again, the optimal pH for adsorption is a dynamic equilibrium of the adsorption rates. The mechanism of adsorption is different in low and alkaline pH conditions. At low pH (pH 4) the absorption of Cu(II) ions is low, because H^+ ions surround the surface of adseobent, the adsorption concentration site becomes saturated, reducing the adsorption capacity. (Hamza *et al.*, 2013). As pH increases, H^+ ion concentration lowers, and the adsorbent surface deprotonates to form FeO^-. Cu(II) ion adsorption occurs due to negative charge surface and electrostatic interaction (Pipíška *et al.*, 2020).

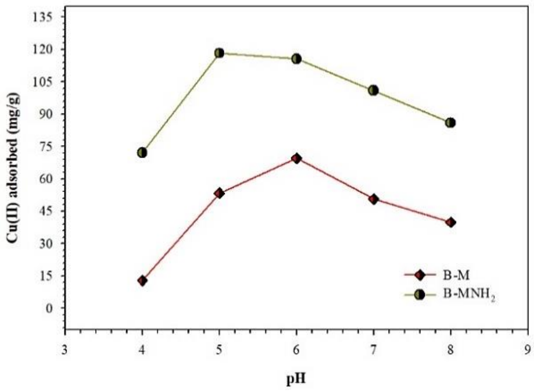

Fig. 6: The Cu(II) ions adsorption capacity on various pH by B-M and B-MNH_2. Reaction conditions: 200 mL of sample, 0.05 g of adsorbent, stirring at 150 rpm for 60 min.

Metal ions can undergo hydrolysis processes in solution at neutral pH and alkaline conditions, making them unstable in their original metal ion state and decreasing the adsorbent's ability to absorb. In addition, metal ions can precipitate as hydroxide in solution (Tian *et al.*, 2023).

Biocomposites with amine functionalization on (B-MNH_2) improved adsorption capacity and facilitated the synthesis of -NH_3^+ and water solubility of amine effects to boost hydrogen bonding and lone electron pair. This condition will increase adsorption capacity (Irawan *et al.*, 2021; Nata *et al.*, 2020b). The best setting for adsorption in this investigation is pH 5, which is consistent with results from earlier studies (Irawan *et al.*, 2019; Irawan *et al.*, 2021; Nata *et al.*, 2020a; Nata *et al.*, 2020b; Nur Afiqah Aina *et al.*, 2022).

The adsorption capacity of B-M and B-MNH_2 in interactions with metal ions was compared to another study. Table 2 presents the adsorption capability of several adsorbents. The produced biocomposite shows good adsorption ability toward Cu(II) ions, about 1.6-fold greater than Activated Coke/$MnFe_2O_4$ as an adsorbent.

Table 2. The maximal capacity of Cu(II) ion adsorption on various adsorbent.

Adsorbent	Capacity (mg/g)/pH	Reference
Amino-functionalized magnetic nanoparticles	25.77/6	Hao *et al.*, 2010
Biochar magnetic chitosan modification	65.87/6	Fan *et al.*, 2023
Activated Cake/$MnFe_2O_4$	72.71/6	Barzegar *et al.*, 2023
Nano Zeovalent Alumunium	5.89/6	Sadek *et al.*, 2023; Iin *et al.*, 2024
Modified incineration fly ash	24.20/2	Sadek *et al.*, 2023; Iin *et al.*, 2024
Biocomposite magnetic nanopartilce (B-M)	53.41/6	This study
Amine biocomposite magnetic nanoparticle (B-MNH_2)	118.26/6	This study

Reusability of Magnetic Biocomposite for Cu(II) Ion Adsorption

One of the important things in using adsorbents is the ability to reuse them in a process. This is very important because it can reduce waste. The advantage of these biocomposites is that they are separated easily from solutions with external magnetic fields. After adsorption, the adsorbent is washed with 0.1 N HCl. Washing using HCl is intended to reuse the adsorbent, where Cu(II) ion will interact with Cl^- to form $CuCl_2$. Then reused for the next run for adsorption. After three cycles, the adsorption capability of Cu(II) ion decreased by 9.37% and 8.81% for B-M and B-MNH_2, respectively. Figure 7 displays the profile of the adsorbent in the repeat utilized.

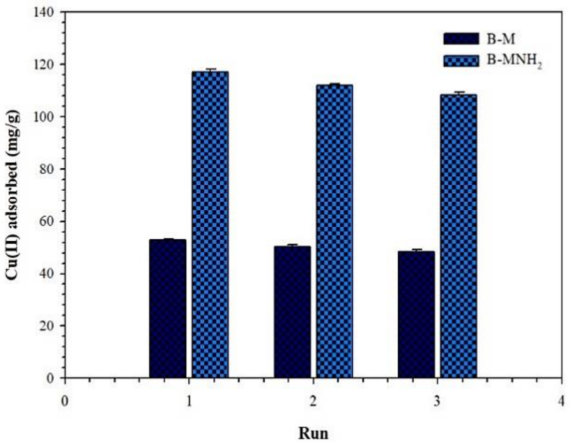

Fig. 7: Repetition of the adsorbent of BM and B-MNH_2 on the adsorption process. Reaction conditions: 200 mL of sample, 0.05 g of adsorbent, pH 5, stirring at 150 rpm for 60 min.

Figure 7 displays that after three times of application, the concentration of absorbed Cu(II) ions decreased. This is feasible due to the lower concentration of H^+ ions on the surface of biocomposites, which plays a significant role in the adsorption process. This shows that the B-M and B-MNH_2 have good performance and are still effective in being reused as adsorbent.

CONCLUSIONS

The solvothermal process successfully synthesized the rice husk-sugarcane bagasse-based magnetic nanoparticle biocomposites. The B-MNH_2 has an adsorption capacity of approximately 118.26 mg/g of Cu(II) ions at pH 5 for 60 min. B-MNH_2's reusability performance remained stable even after the third use. The prepared biocomposites are effective, efficient, and promising candidates as an alternative adsorbent for wastewater treatment.

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