



Sustainable Magnetic Biocomposites Palm Oil Empty Fruit Bunch/Sago Dregs-Based with High Active Amine as Green Materials

Allam Naufal¹, Chairul Irawan¹, Rinna Juwita¹, Iryanti Fatyasari Nata^{1,2}, ✉

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¹Department of Chemical Engineering, Lambung Mangkurat University, Banjarbaru 70714, Indonesia

²Integrated Laboratory, Lambung Mangkurat University, Banjarbaru 70714, Indonesia

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Abstract

Palm oil empty fruit bunches (POEFB) and sago dregs (SD) are agricultural wastes that contain cellulose fibers. The one of utilization is used as raw materials for magnetic biocomposites. The objectives of this research are to determine the optimal composition of POEFB-SD fibers for the formation of amine magnetic biocomposite. The resulting biocomposite is characterized based on morphological, amine content, elemental content, crystalline structure, and functional groups. First, POEFB and SD)fibers were dried and ground to approximately 60 mesh. These fibers then underwent delignification at 80 °C for 2 h using a 1% w/v NaOH solution. A one-step solvothermal process followed, wherein different ratios of delignified POEFB and SD fibers (1:0; 1:1; 1:2; and 1:3) were added to a mixture containing ethylene glycol, FeCl₃·6H₂O, and 1.6-hexanediamine, and heated to 200 °C for 6 h. The resulting magnetic biocomposites contain Fe and amine at about 98.26% and 3.83 mmol.g, respectively. The magnetite was confirmed by X-Ray diffraction (XRD) through characteristic peaks at 36°, 43°, and 57°. Furthermore, Fourier-Transform Infrared Spectroscopy (FT-IR) revealed N–H bending vibrations and Fe–O bonds in the biocomposites at 1620 cm⁻¹ and 585 cm⁻¹, respectively. The arrangement of the matrix, iron, and amine groups on the biocomposites' is promising candidates as good adsorbents for advanced biocomposites and green materials.

INTRODUCTION

Palm oil empty fruit bunches (POEFB) and sago dregs (SD) are agricultural wastes with high cellulose content, the waste materials are not optimal to utilize yet. The cellulose contents of POEFB and SD are 44.63% and 39.50%, respectively (Hafidawati et al., 2024; Prasetyo et al., 2024). The utilization of the empty fruit bunch (EFB) is only 10% for boiler fuel, and composting then improves the high energy produced by optimization (Sukiran et al., 2020). Besides, sago stems are typically processed into flour and animal feed mixtures. The substantial cellulose content in these wastes suggests their potential as sources of natural fiber for new materials such as biocomposite.

Biocomposites, which are composite materials comprising natural polymers or biofibers as reinforcements, are notable for their degradability (Utami & Prastiwi, 2017). These materials, developed from biomass, are macroscopic mixtures of two or more substances, resulting in a new material with enhanced mechanical properties (Lestari et al., 2020; Williams & Starke Jr., 2003; Yuanita et al., 2022). For instance, magnetic composites of EFB have shown promising results as adsorbents for Cr(VI) metal, achieving an adsorption efficiency of 91.82% (Khalid et al., 2021). Similarly, SD as an adsorbent has been able to reduce color in aqueous solution up to 87.20% (Karthika & Vasuki, 2019).

The synthesis of magnetic nanoparticles using the solvothermal method via a one-step

✉ Corresponding author:
E-mail: ifnata@ulm.ac.id

process, along with surface modification with amine groups, has been successfully carried out for various natural fiber sources as biocomposites (Nata et al., 2018). Examples include rice husk fibers for Fe(III) ion adsorption (Nata et al., 2020), sugarcane bagasse fibers for Pb(II) ion adsorption (Irawan et al., 2021), and a combination of rice husk and bagasse fibers for Cr(III) ion adsorption (Nata et al., 2024). Biomass fibers have been proven effective in adsorbing metal ions and serving as a matrix for biocomposite formation.

Fe(III) impregnation on biomass fibers is advantageous due to the magnetic properties that facilitate easier separation processes (Kamel et al., 2020). Moreover, magnetic nanoparticles exhibit high adsorption capacities for metal ions and can be easily modified to meet specific material functions, boasting a large and stable surface area (Nguyen et al., 2021). Despite these benefits, there has been no research on the development of modified magnetic nanoparticle biocomposites from POEFB and SD fibers using a one-step process, highlighting the need for optimization to enhance the function and stability of these biocomposites.

This study aims to optimize the mass composition of POEFB-SD fibers and the functionalization of amine groups on biocomposites to improve the adsorption capacity. The characterization of biocomposites includes morphology, crystal structure, functional groups, and identifying elements and amines contained in biocomposites. This research seeks to develop superior adsorbents by utilizing POEFB and SD fibers as green materials for the creation of magnetic amine biocomposites.

MATERIALS AND METHOD

Materials

The materials used in this experiment were empty palm oil empty fruit bunches (POEFB) obtained from an industrial area in Indonesia. Sago dregs (SD) was obtained from the Gambut area, Banjar, South Kalimantan, Indonesia. Ethylene glycol (C₂H₆O₂, 99.8%), sodium acetic anhydride (C₂H₃NaO₂, ≥ 99%), iron (III) chloride hexahydrate (FeCl₃.6H₂O, p.a., 97%), 1,6-hexanediamine (C₆H₁₆N₂, 98%), sodium hydroxide (NaOH, 98%), hydrochloric acid (HCl, 38%) and ethanol (C₂H₅OH, 96%). All chemicals were purchased in analytical grade from Sigma Aldrich and used without purification.

Delignification of Palm Oil Empty Fruit Bunch and Sago Dregs

Separately, palm oil empty fruit bunches and sago dregs were cleaned of impurities and then dried at 80 °C for 24 h. Dry SD and POEFB were blended and formed the same size, about ±60 mesh. The powder obtained was placed in a 1% NaOH solution in a 500 mL beaker (40% v/v), heated at a temperature of 80 °C and stirred at 200 rpm for 2 h. The back solution was produced, then solid part was washed with distilled water until the filtrate pH was neutral and dried for 24 h at 80 °C. Next, the size was standardized again to ±60 mesh. Delignified SD and POEFB are symbolized by SD-D and EFB-D. Characterization was carried out on samples before and after the delignification process.

Synthesis of Amine Magnetic Biocomposites

The synthesis of amine magnetic biocomposites was carried out using the solvothermal method. Anhydrous sodium acetate (1.6 g) and FeCl₃.6H₂O (0.8 g) were dissolved in ethylene glycol (24 mL), 1,6-hexanediamine (7 mL) was added, and the mixture was heated at 60 °C while stirring at 200 rpm. The combination of SD-D and POEFB-D was added as much as 0.5 g with a mixture ratio (1:0; 1:1; 1:2; and 1:3) and left for 10 min, then put in a Teflon Stainless Steel autoclave reactor and heated for 6 h at 200 °C. The prepared biocomposites with ratio 1:0; 1:1; 1:2; and 1:3 was labeled BM-0, BM-1, BM-2, and BM-3. The resulting magnetic biocomposites were washed with distilled water and ethanol (40%, v/v) for 2 times and stored in distilled water for subsequent use.

Characterization of materials

Surface morphology of samples was observed by Field-emission scanning electron microscopy (FE-SEM, JSM-6500F, JOEL LTD, Tokyo, Japan), which treated sputter-coated samples with platinum. The elements on the sample were detected by energy-dispersive X-ray fluorescence (XRF, Shimadzu Corporation, Japan) at 20 kV voltage and 77 UA current. Fourier transform infrared spectrometry (FT-IR, Bio-Rad, Digilab FTS-3500, SpectraLab Scientific Inc., Canada) was used analyze the functional groups on the sample. The detecting element on sample was used X-Ray Fluorescence (XRF) which performed on energy-dispersive X-Ray Fluorescence at 77 UA current and 20 kV voltage. The crystal structure of

sample also identified by X-ray diffraction (XRD, Thermo Fisher Scientific Inc., UK) using copper k-alpha ($\text{CuK}\alpha$) radiation on a Rigaku D/Max-B XRD machine at 100 mA current and 400 kV voltage. X-Ray Diffraction (XRD) was carried out to determine the crystal structure of the sample after delignification.

The index of crystalline (CrI) was retrieved from Eq. (1).

$$\text{CrI} = \frac{(I_{002} - I_{\text{am}})}{I_{002}} \quad (1)$$

where CrI is the index of crystalline (%), I_{am} is the amorphous part intensity and I_{002} is the crystal part intensity.

Analysis

Analysis of amine content in biocomposites was carried out using the re-titration method (Karnitz Jr. et al., 2007). A sample of 50 mg was added to 25 mL of 0.01 M HCl solution. The mixture was stirred for 2 h at room temperature and then separated. 15 mL of filtrate

was titrated using a standardized 0.01 N NaOH solution. The group amine concentration is calculated using Eq. (2).

$$C_{\text{NH}_2} = \left[\frac{(C_{\text{HCl}} \times V_{\text{HCl}}) - (5 C_{\text{NaOH}} \times V_{\text{NaOH}})}{m \text{ sample}} \right] \quad (2)$$

where C_{HCl} represents the concentration of the HCl solution in mmol/L, C_{NaOH} denotes the concentration of the NaOH solution in mmol/L, V_{HCl} is the volume of the HCl solution in liters, V_{NaOH} is the volume of the NaOH solution used during the titration process in liters, and m refers to the mass of the sample in grams.

RESULTS AND DISCUSSION

Morphological Properties of Palm Oil Empty Fruit Bunch, Sago Dreg, and Magnetic Biocomposites

The palm oil empty fruit bunch and sago dregs are biomass materials containing cellulose with complex structures. The treatment of these

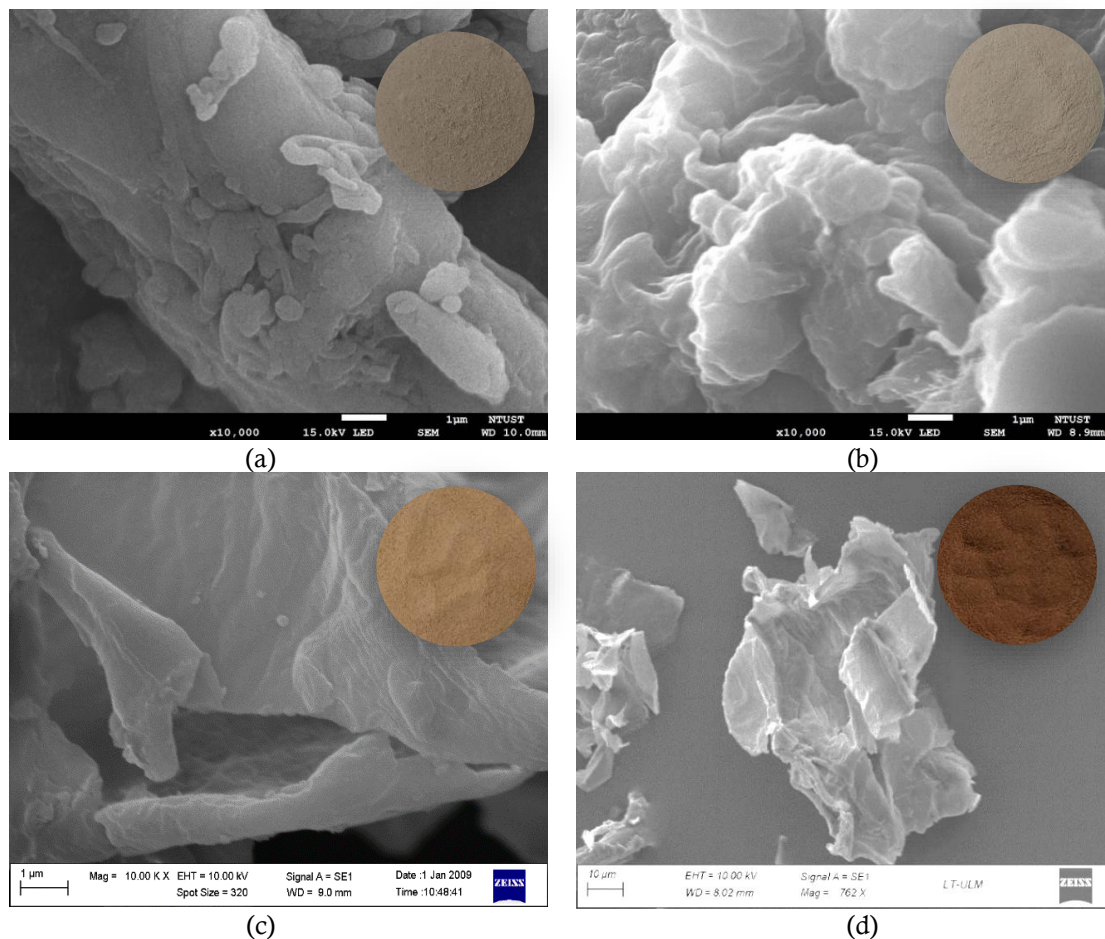


Figure 1. FE-SEM images of the morphology of POFEB (a) before and (b) after delignification (EFB-D), (c) SD before and (d) SD after delignification (SD-D).

fibers, specifically through the delignification process using NaOH, results in significant changes in their visualization, morphology, structure, and functional groups. Treatment with NaOH induced a noticeable color change in the POEFB, shifting from dark gray to a lighter gray. In contrast, SD underwent oxidation, leading to a color transformation from light brown to dark brown (Sunardi et al., 2021). These color changes are illustrated in Figure 1 (inset). Additionally, Figure 1 displays FE-SEM images that show the surface morphology of POEFB and SD before and after delignification. The surface morphology of POEFB prior to delignification (Figure 1.a) appears rough and obstructed by lignin and hemicellulose. The POEFB surface is ripped following delignification with NaOH (Figure 1.b), resulting in a more open and rough fiber structure. The SD (Figure 1.c) exhibits a compact and dense structure. However, following delignification (Figure 1.d), its morphology becomes notably more porous and less consolidated. This indicates the removal of lignin and hemicellulose components from the fiber. Figure 2 shows the morphological surface of magnetic biocomposites with variation of composition of POEFB and SD fiber. Based on those figures, there are irregular shapes with

magnetic particles along the surface. The variation of fibers did not have significant change in morphology because the same original shape of the fibers in the initial step in the synthesis of biocomposites. The size and surface functionalization of magnetic particles on biocomposites enhanced the adsorption capacity as adsorbents. The control size of magnetic using polymer that is also simultaneously functionalized by an amine group on the particle. As a source of amine groups, 1,6-hexanediamine plays a crucial role in controlling the growth of Fe_3O_4 nanoparticles during their formation from FeCl_3 . By inhibiting the incorporation of ferric ions into the nanoparticle core, it effectively restricts the growth of Fe_3O_4 , resulting in particles with more uniform and smaller sizes. This consistent particle size distribution is attributed to the deposition of 1,6-hexanediamine on the magnetic surface, which limits magnetic growth (Lee & Komarneni, 2005; Nata et al., 2023).

Physical and Chemical Properties of of Palm Oil Empty Fruit Bunch, Sago Dreg, and Magnetic Biocomposites

Varying the mass ratio of POEFB-D and SD-D in biocomposite preparation has

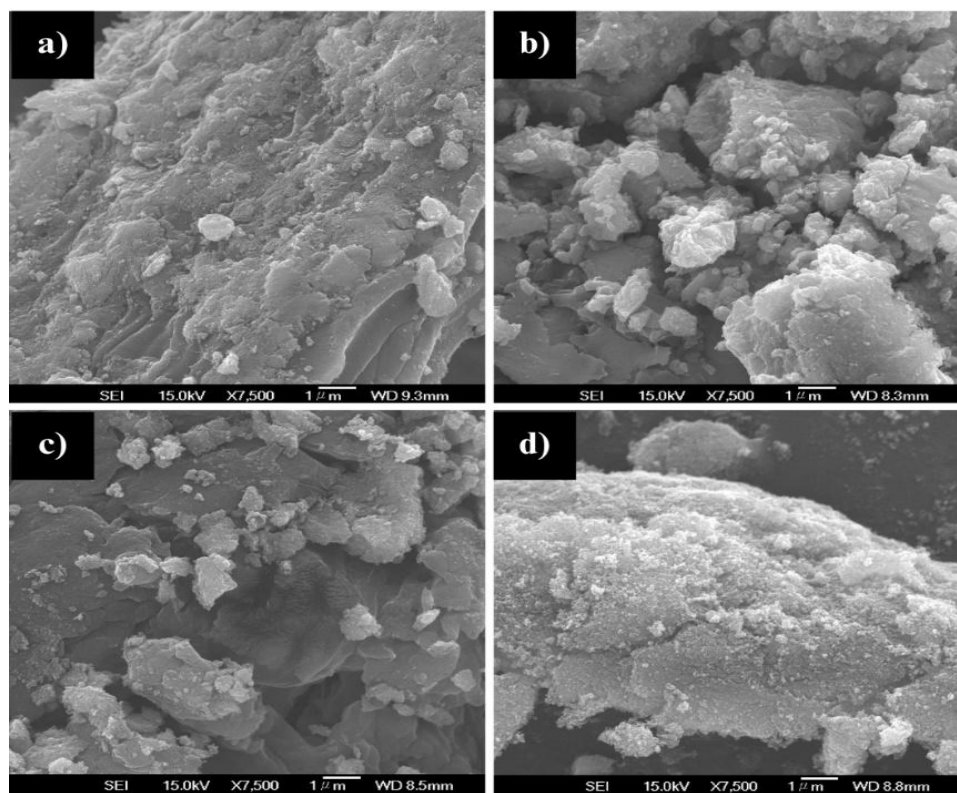


Figure 2. Images of the morphology of SD and POEFB biocomposites of a) BM-0; (b) BM-1; (c) BM-2 and (d) BM-3.

demonstrated the specific properties of these materials. As depicted in Figure 2, all magnetic were evenly distributed across the fiber surfaces. Owing to the differing structures and compositions of the fibers, the samples exhibited slight variations in amine and Fe content. The specifications for amine and Fe are shown in Table 1.

Table 1. The Fe and amine content on biocomposites.

Component	Sample			
	BM-0	BM-1	BM-2	BM-3
Fe (%)	99.09	97.48	97.71	98.26
Amine (mmol/g)	3.83	3.74	3.83	3.83

The highest Fe and amine content were found in the BM-0 biocomposite at 99.09% and 3.83 mmol/g, respectively. However, the utilization of two biomass balances makes the fiber composition ratio of 1:3 (BM-3) also contain high Fe at 98.26%. The different natural structure from these fiber makes an effect on the accesibility of iron solution during the process. Interestingly, the amine content in these biocomposites remains relatively constant. In a separate study, a biocomposite made from a 1:1

ratio of rice husk and sugarcane bagasse had an amine content of 2.63 mmol/g (Nata et al., 2022). It is shown the combination of POEFB-SD fibers showed higher Fe and amine content.

The crystalline index (CrI) and cellulose crystal structure of POEFB and SD were investigated for change in structure due to treatment. The cellulose crystals exhibited dominant peaks at 2θ angles between 20° and 80° (Hashemian et al., 2013; Prasetyo et al., 2024). Using XRD data within this angular range, the crystal intensity in the samples was measured. Whereas the XRD pattern showed notable widening, broad diffraction patterns at 2θ angles between 0° and 20° revealed the amorphous properties (Mohadi et al., 2014). Furthermore, it was observed that cellulose-containing POEFB and SD exhibited peaks at 16.0° for amorphous cellulose I and at 22.2° for crystalline cellulose II (Figure. 3), respectively. The delignification treatment of POEFB and SD fibers with NaOH solution enhanced the crystallinity of the fibers by removing hemicellulose and lignin, thereby increasing the intensity of the crystalline structure (Mohadi et al., 2014).

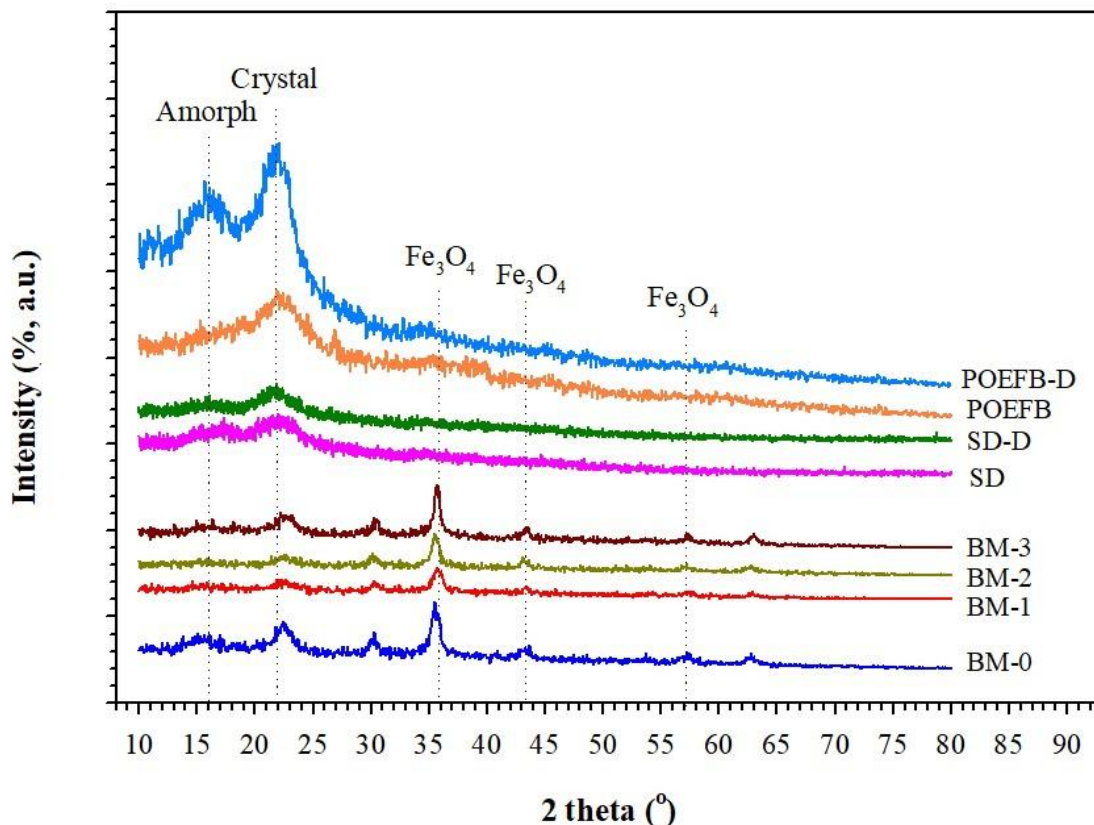


Figure. 3 XRD pattern of POEFB, POEFB-D, SD, SD-D, BM-0, BM-1, BM-2, and BM-3.

Table 2 shows the study observed a significant increase in the crystallinity index (CrI) values of POEFB-D and SD-D, reaching 36.37% and 127.02%, respectively. This enhancement in crystallinity is attributed to the formation of crystalline cellulose, as evidenced by the intensified crystal peaks. Conversely, the reduction in the polysaccharide structure was identified by the broadening of the amorphous peak (Ghali et al., 2011). The distinct crystalline and amorphous peaks serve as key indicators, reflecting the degree of organized crystalline cellulose and the presence of a less organized polysaccharide structure.

Table 2. Components of peaks characteristics of POEFB, POEFB-D, SD, SD-D

Sample	Characteristics Peak		CrI (%)
	Amorph (16.0°)	Crystal (22.2°)	
POEFB	254	320	25.98
POEFB-D	525	711	35.43
SD	175	190	9.55
SD-D	143	174	21.68

The presence of magnetic in the samples was also confirmed through X-ray diffraction (XRD) analysis. As depicted in Figure 3 for BM-0,

BM-1, BM-2, and BM-3, specific peaks corresponding to magnetite (Fe_3O_4) were observed at 36° , 43° , and 57° , consistent with the JCPDS card 39-0664 (Synytsia et al., 2022). These peaks confirm the successful formation of magnetic properties on the biocomposites. The size of magnetite is determined by its incorporation with amines, which results in smaller sizes, typically ranging from 30 to 50 nm in diameter (Nata et al., 2018). The cellulose-containing fibers have shown significant potential as materials for the development of biocomposites. XRD analysis indicates that fiber treatment enhances the CrI and prove the magnetite was formed.

The FT-IR spectra of POEFB, POEFB-D, SD, SD-D, BM-0, BM-1, BM-2, and BM-3 are presented in Figure 4. The characteristic peak at 585 cm^{-1} corresponds to the stretching of the Fe-O bond in Fe_3O_4 (Mohammadi et al., 2021), which was absent in the POEFB, POEFB-D, SD, and SD-D samples. The modification of the amine group is indicated by the peak at 1620 cm^{-1} which corresponds to the N-H bending vibration (Jeyaseelan et al., 2021). Additionally, the Si-OH bending vibration is observed at a wavelength of 1020 cm^{-1} .

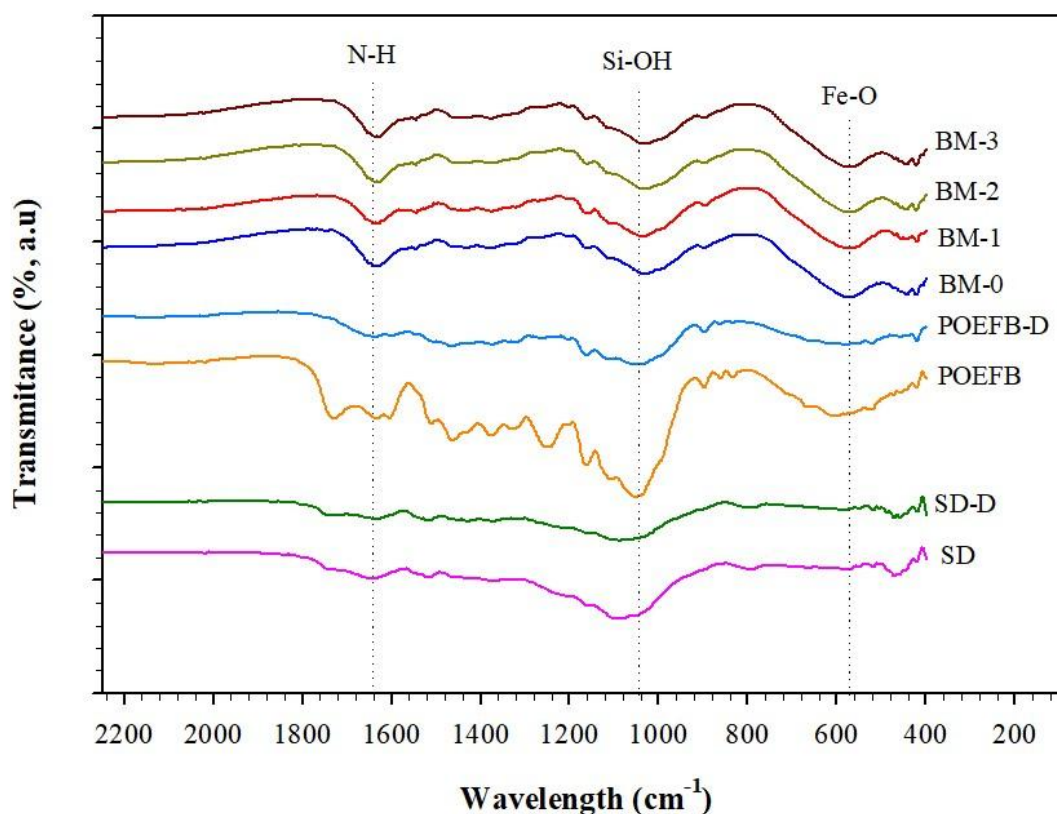


Figure. 4 FT-IR spectra of POEFB, POEFB-D, SD, SD-D, BM-0, BM-1, BM-2, and BM-3.

CONCLUSION

This study successfully demonstrated the preparation of magnetic biocomposites using a one-step solvothermal process, with POEFB and SD fibers serving as the matrix. The formation of magnetic on the fiber surfaces was confirmed through XRF, FE-SEM, and XRD observation. The optimal fiber composition was found to be a 1:3 ratio of POEFB to SD fibers. The biocomposites contain Fe and amine about 3.83 mmol/g and 98.26%, respectively. These findings suggest that cellulose-based magnetic biocomposites are promising candidates as matrices for advanced biocomposite and green materials.

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