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Utilisation of palm oil fibre cake biomass for magnetic nanoparticle biocomposites as adsorbents in Sasirangan liquid waste treatment

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ABSTRACT

Palm kernel fibre (fibre cake) is a waste product from the palm oil industry that has potential as a source of natural fibre. This study aims to utilise fibre cake to produce magnetic nanoparticle biocomposites as adsorbents for reducing the lead (Pb^{2+}) content and Total Suspended Solids (TSS) in Sasirangan liquid waste. The research produced two types of biocomposites: one without amino groups (FCB-M) and one with amino groups (FCB-MH). Observations from Scanning Electron Microscopy (SEM) showed that magnetic nanoparticles had formed on the surface of the fibre cake. Treatment of palm oil fibres with NaOH restructured amorphous cellulose into crystalline cellulose, as indicated by a 30.25% increase in the Crystalinity Index (CrI) based on X-Ray Diffraction (XRD) analysis. The Pb^{2+} content absorbed by FCB and FCB-NH_2 was 97.84 mg/g and 101.82 mg/g, respectively, for 3 hours at pH 8. The modification of amino groups on the surface of the biocomposite resulted in a 164.85% increase in adsorption. The effectiveness of TSS reduction at pH 8 was 65.8% and 62.9% for FCB-M and FCB-MH, respectively.

1. Introduction

Indonesia is the world's largest producer of palm oil, with plantations spread across almost the entire country, particularly in South Kalimantan. The total area of oil palm plantations in South Kalimantan in 2013 reached 372,720 hectares, consisting of smallholder plantations (69,449 hectares), large private plantations (298,365 hectares) and large state plantations (4,906 hectares) [1,2]. Palm kernel press cake is a residue from palm oil extraction containing 57.9% cellulose, 18% lignin and, upon hydrolysis, 14.94% hemicellulose [3]. Given the fibre content of palm oil, palm oil fibre can be used as a raw material for the manufacture of composites [4]. A composite is a mixture of two or more materials that are mixed macroscopically to produce a new material. This means that the superior properties of the components are still clearly visible. Composite materials have many advantages, including being lightweight and having better mechanical properties [5,6].

Currently, nanometer-sized magnetic nanoparticles are a highly sought-after object for use in several industrial applications and in the development of science and technology. These magnetic nanoparticles have been widely used in several fields of research such as magnetic storage, immunoassay [7, 8], separation media and as adsorbents [9].

The synthesis of magnetic nanoparticles using the solvothermal method produces stable nanoparticles with uniform particle sizes and high magnetisation levels, giving them the potential to be developed as adsorbents and for protein purification [10]. Sasirangan fabric is a type of batik fabric unique to South Kalimantan, so the sasirangan industry is widely found in the South Kalimantan region. Many artisans do not process their waste properly, one of which is by disposing of waste directly into the environment without processing [11]. This will endanger the surrounding environment because it contains heavy metals that are harmful to health and the environment, namely lead (Pb^{2+}), which according to research [12] reaches 4.11 mg/L in the city of Banjarmasin. This concentration exceeds the quality standard which stipulates that the maximum total Pb content allowed is 0.1 mg/L.

One of the technologies used to reduce the lead content in sasirangan liquid waste is the adsorption method. Adsorption is a process that has good and economical operations. The use of biomass adsorbents in the adsorption process is an alternative because they are easily obtained and widely available in nature [13,14]. Research that has been conducted to separate heavy metals from wastewater includes chemical precipitation, filtration, ion exchange, membrane media, and adsorption methods [15-17].

Magnetic nanoparticle biocomposites are made to reduce the lead (Pb^{2+}), TSS, and dye content in sasirangan industrial wastewater. This wastewater requires special research to reduce its chemical content. One of the easiest studies to conduct is the adsorption process. Through this study, the characterisation of palm fibre cake biocomposites with magnetic nanoparticles, such as morphological structure and

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crystalline structure, can be determined. The optimum conditions of palm fibre cake biocomposites as adsorbents for lead (Pb^{2+}) adsorption in sasirangan liquid waste can be determined based on contact time, pH, and type of adsorbent, TSS and colourants used, and the reusability of the resulting magnetic nanoparticle biocomposite will be evaluated.

The conversion of local biomass waste into high-value functional materials that support the concepts of circular economy and green technology [18]. One of its unique features is the ease with which the adsorbent can be separated from liquid waste using magnets, eliminating the need for complicated filtration or centrifugation processes. This improves efficiency and practicality in actual waste treatment applications. The resulting adsorbents can be regenerated and reused, reducing secondary waste [19]. Magnetic adsorbents have the potential to remove two pollutants at once, which has rarely been studied before. Additionally, this research offers an innovative alternative to expensive commercial activated carbon.

This study has a unique contribution compared to previous studies that used cellulose-based magnetic adsorbents. Magnetic adsorbents from palm kernels have a high fixed carbon content and a dense lignocellulose structure, so that after carbonisation and activation processes, they produce porous materials with better mechanical stability [20]. The combination of a semi-amorphous carbon matrix and crystalline Fe_3O_4 particles forms a composite with uniform magnetic distribution, providing high magnetisation properties and large adsorption capacity for Pb^{2+} ions. Compared to other magnetic cellulose adsorbents, this material shows better resistance to pH changes and is capable of simultaneously adsorbing heavy metals and dyes [21] from sasirangan wastewater. Thus, this study contributes to the development of high-performance, stable magnetic adsorbents based on local biomass for the treatment of sasirangan industrial wastewater.

2. Materials and Methods

2.1. Materials

The materials used in this experiment were palm fibre, sasirangan liquid waste taken from Kampong Sasirangan Banjarmasin, ethylene glycol ($C_6H_6O_2$), anhydrous sodium acetate ($C_2H_3NaO_2$), iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), hexanediamine, sodium hydroxide (NaOH), hydrochloric acid (HCl), ethanol (C_2H_5OH) and distilled water.

2.2. Methods

Palm fibre is washed with water and dried in the open air for 7 days, then ground using a blender to produce palm fibre powder. The dry palm fibre powder (40% v/v) is soaked for 2 hours in a 1% NaOH solution (ASTM 1109-56), heated to 80°C while stirring at a speed of 150 rpm for 2 hours. It is then washed until the filtrate is neutral and dried. Ethylene glycol, anhydrous acetate, $FeCl_3.6H_2O$, and 1.6-hexanediamine are heated for 15 minutes at 60°C and stirred at 150 rpm. The mixture and dry palm kernel fibre are placed in a reactor and heated for 6 hours at 198°C. The reactor is cooled to room temperature and the mixture is washed with distilled water and 50% ethanol. The material obtained is referred to as biocomposite with amino groups (FCB-MH) and without amino groups (FCB-M). The application of biocomposite as an adsorbent to reduce the pollutant content in sasirangan wastewater is examined by reviewing the pollutant content through an isothermal adsorption process. At this stage, variations in contact time (15, 30, 60, 120, 240, and 480 minutes) and pH (4, 6, 8, 10, and 12) in the adsorption process will be evaluated for each type of adsorbent.

3. Result and Discussion

3.1. Characterisation of magnetic biocomposite adsorbents from palm oil fibres

Palm oil fibres (POF) are a type of lignocellulose and therefore have a complex structure. The morphological structure of POF, FCB-M and FCB-MH was observed using SEM analysis. Figure 1 shows palm oil fibres in the form of rods (indicated by red marks). There are two types of palm oil structures: the first is fibres with rough surfaces (Figure 1b) and the second is fibres with porous surfaces containing silica (Figure 1c), which corresponds to the composition of empty palm fruit bunches. In Figure 1, the palm fibre is still covered by lignin, hemicellulose and other components that bind cellulose. Figure 2 (a and b) show magnetic biocomposites with and without amino groups. From these figures, magnetic particles (Fe_2O_3) can be seen adhering to the entire surface of the palm fibre. The size of the magnets formed for each with and without amino groups is ±100 nm and without amino groups ±50 nm [10]. The formation of these magnets can be analysed from their physical properties, which respond to magnetic fields, and EDX component tests on each sample, as shown in Table 1. There are no Fe components in palm fibre, while FCB-M and FCB-MH contain Fe because magnets have formed in the palm fibre.

Table 1. Components of FC, FC-D, FCB-M and FCB-MH

Component	Content (% weight)			
	FC	FC-D	FCB-M	FCB-MH
C	75,27	50,47	37,54	32,44
O	24,73	49,53	17,73	12,54
Fe	0,00	0,00	65,77	75,18

Adsorption is highly dependent on the specific surface area and pore structure of the adsorbent. Biocomposites from activated palm kernels have numerous microscopic pores, thus providing more active sites for Pb^{2+} ion adsorption. The greater the surface area and pore volume, the higher the Pb^{2+} adsorption capacity [22]. Functional groups such as hydroxyl, carboxyl, carbonyl, and ether on the adsorbent surface play a role in complexation or ion exchange with Pb^{2+} metal. The addition of iron not only provides magnetic properties but also adds active sites in the form of iron oxide surfaces. Fe–O groups can interact with Pb^{2+} through ion exchange or surface complexation mechanisms [23].

The semi-amorphous structure of activated carbon provides more reactive active sites, while crystalline Fe_3O_4 provides mechanical stability and magnetism. The combination of the two creates a heterogeneous surface that supports adsorption through multiple mechanisms (physical and chemical) [24]. Morphological analysis (SEM) shows a porous texture and uniform distribution of Fe_2O_3 on palm kernel carbon. The rough, porous, and heterogeneous surface increases the contact area between Pb^{2+} and the adsorbent. SEM analysis shows that palm kernel adsorbents have a porous morphology with larger pore sizes, thereby increasing the effective surface area and providing more active sites for interaction with Pb^{2+} ions, dye and TSS particles [25].

The increasingly porous morphology correlates positively with an increase in adsorption capacity for Pb^{2+} ions. A porous, rough, and homogeneous surface indicates a large number of active sites and contact areas between Pb^{2+} ions and the adsorbent surface [26]. When the activation process is carried out properly, a more open and evenly distributed surface structure is formed, increasing adsorption capacity.

XRD analysis was conducted to determine the crystal structure of cellulose and to determine the Crystalinity Index (CrI) of palm fibre before and after the delignification process. Palm fibre containing cellulose fibre in its structure has characteristic peaks at 2 theta (o) = 16.2° (cellulose I) and 22.6° (cellulose II).

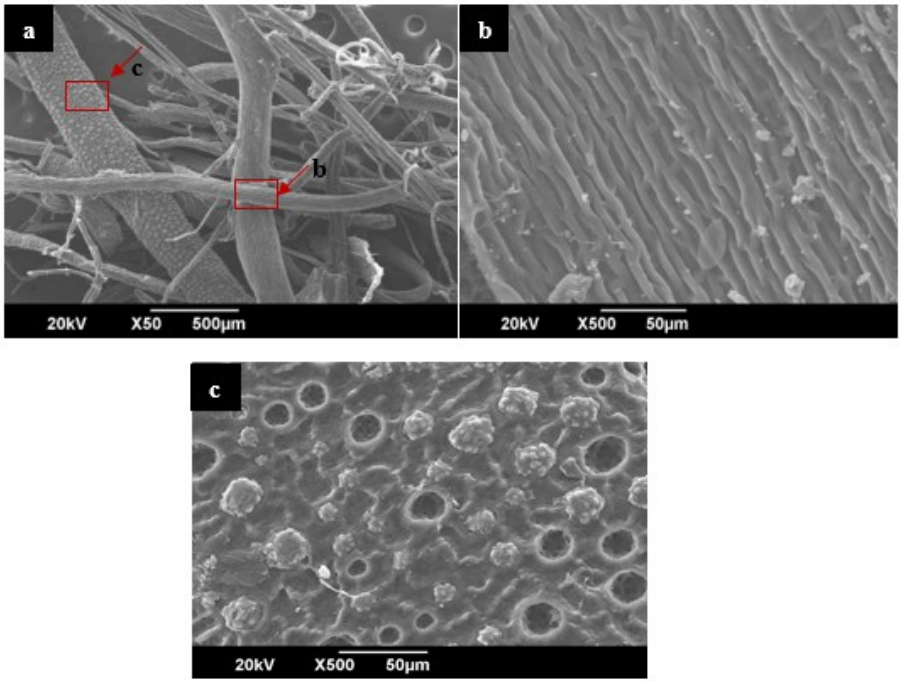

Fig. 1. SEM images of palm fibre (a) surface of palm fibre section b (b) surface of palm fibre section c (c)

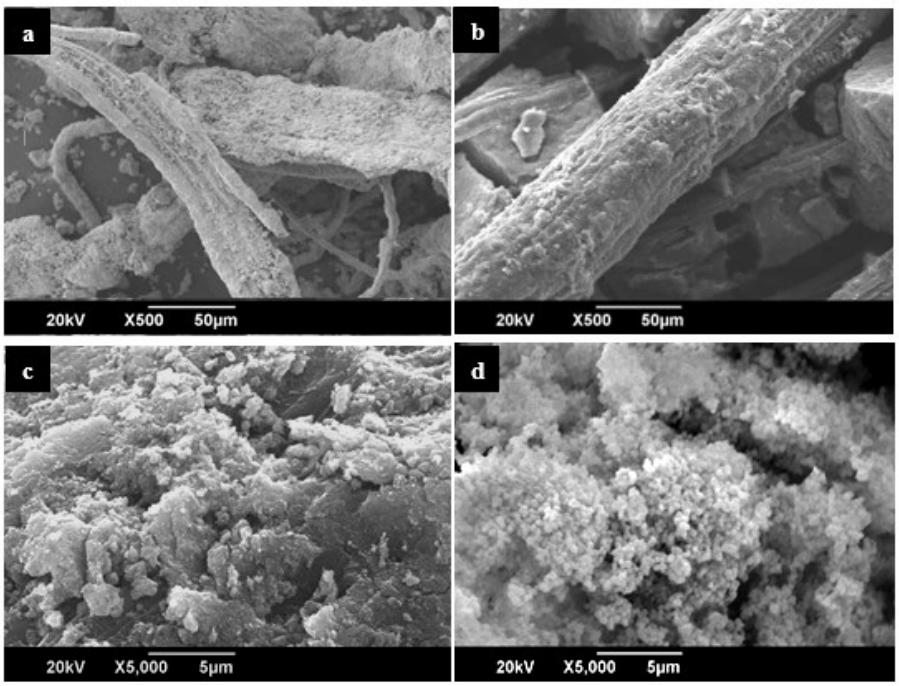

Fig. 2. SEM images of magnetic biocomposites of palm fibre without amino groups (a); with amino groups (b); magnetic biocomposites of palm fibre without amino groups magnified 5000x (c) and magnetic biocomposites of palm fibre with amino groups magnified 5000x (d)

The intensity of palm fibre at the amorphous peak characteristic of 16.2° and the crystalline peak characteristic of 22.6° are shown by Table 2. The crystalline structure of cellulose can affect the resulting product; cellulose is a parameter that determines the strength of the fibre [27]. The structure of palm oil fibres before and after treatment still contains components with amorphous (hemicellulose and lignin) and crystalline (cellulose) forms. This is due to the loss of lignin and hemicellulose content after the delignification process with NaOH. Table 2 shows that palm oil fibres

experience an increase in CrI value after the delignification process. This can be seen from the increase in CrI value from 23.73% to 34.01%. Treatment of palm fibre with NaOH can increase the amount of cellulose because it can restructure amorphous cellulose into crystalline cellulose. It can therefore be concluded that treatment with NaOH in the delignification process of palm fibre can increase the intensity or crystalline structure of palm fibre [28-30]. This indicates that palm fibre experiences a decrease in CrI value after the biocomposite manufacturing process.

XRD results show an increase in the crystallinity index of Fe_3O_4 composites, indicating better structural stability and uniform distribution of magnetic particles. The combination of porous structure and high crystallinity synergistically increases the adsorption capacity of Pb^{2+}, dye and TSS, explaining the positive relationship between the structural properties of the material and its adsorption performance. This trend confirms that surface modification and magnetic particle integration play an important role in improving adsorbent efficiency [31].

Table 2. Characteristics of FC, FC-D, FCB-M and FCB-MH

Sample	Peak Characterization		CrI (%)
	Amorph (16,2°)	Crystal (22,6°)	
FC	510	631	23,73
FC-D	244	327	34,01

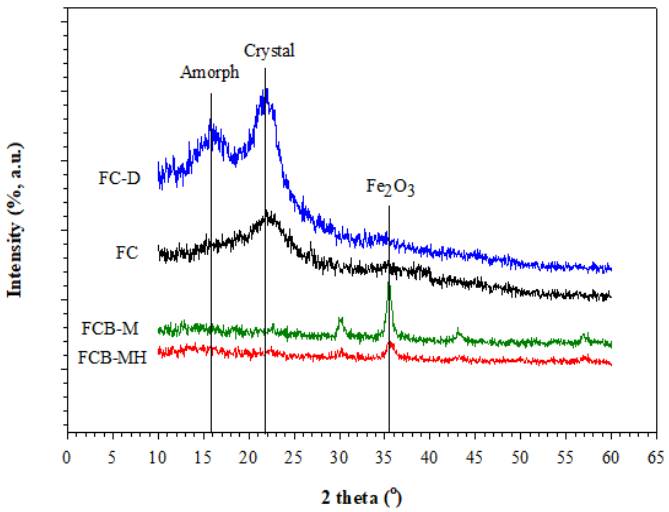

Fig. 3. X-Ray Diffraction (XRD) of oil palm straw before and after delignification and magnetic biocomposite of oil palm fibre

The decrease in intensity for this amorphous structure indicates the loss of lignin and hemicellulose content in FC-D [32]. The XRD analysis results show the formation of magnetism in palm fibre, indicated by the Fe_2O_3 peak at 36° for both FCB-M and FCB-MH. An increase in the crystallinity index after the addition of Fe_2O_3 or thermal activation indicates a better structured arrangement, which makes the surface more stable and the active sites more defined. Increased crystallinity results in improved structural stability and chemical bonding efficiency between surface groups and Pb^{2+} ions, thereby increasing adsorption capacity [26]. Adsorbents with porous surfaces and moderate to high crystallinity tend to have better Pb^{2+} adsorption, as the crystalline structure of Fe_2O_3 acts as a metal ion exchange site, while amorphous carbon provides physical adsorption sites and chemical functional groups [33].

3.2. Lead (Pb^{2+}) metal adsorption with palm oil fiber magnetic biocomposite as adsorbent

The adsorption process of Pb^{2+} metal with palm oil fiber biocomposite adsorbent was carried out for various contact times, namely 15, 30, 60, 120, 240, and 480 minutes. The contact time is the length of time required for the adsorption process of the FCB-M and FCB-MH magnetic biocomposites with the sasirangan waste solution, as shown in Figure 4.

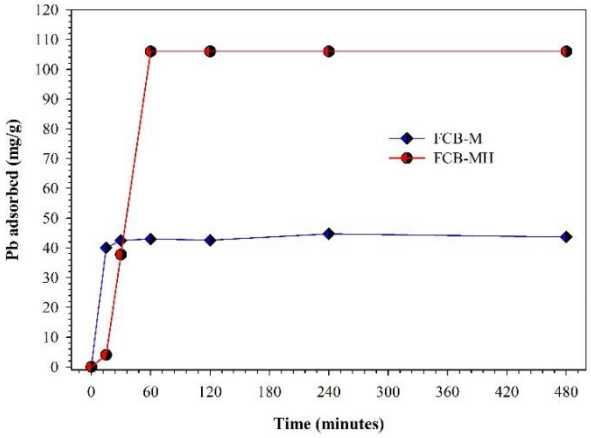

Fig. 4. Lead (Pb^{2+}) concentration after adsorption at various contact times with FCB-M and FCB-MH adsorbents with a sample volume of 50 mL, 0.1 g of adsorbent, stirring at 150 rpm and pH 6

The ability of FCB-M and FCB-MH to remove lead content increase from 0 minutes to 30 minutes to a value of 42.51 mg/g for FCB-M and 37.74 mg/g for FCB-MH. Then remained constant at 60 minutes as time increased to a value of 106 mg/g for FCB-MH. This constant value was due to the saturation condition that had been reached previously, where the entire surface of the adsorbent was filled with adsorbate particles. The adsorption capacity of Pb^{2+} increases with contact time, indicating that the adsorption process proceeds kinetically until equilibrium is reached. The rapid initial increase in the first stage indicates the availability of many active sites on the adsorbent surface, while the adsorption rate slows down, approaching a plateau because most of the sites have been filled. This trend supports an adsorption mechanism involving chemical interactions between the $–NH_2$ groups on the adsorbent surface and Pb^{2+} ions [34].

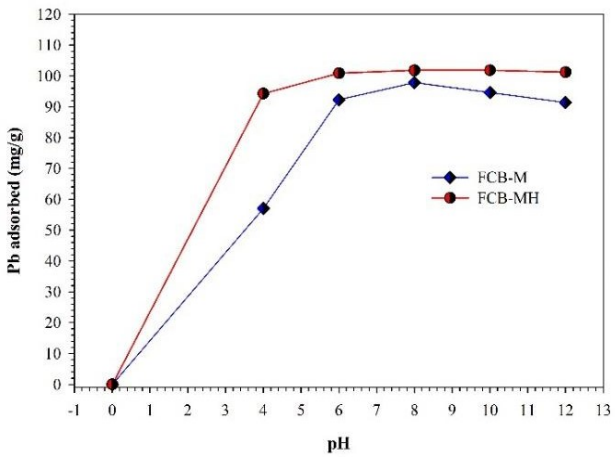

Fig. 5. Lead (Pb^{2+}) concentration after adsorption at various pH levels using FCB-M and FCB-MH adsorbents with a sample volume of 50 mL, 0.1 g of adsorbent, and a stirring speed of 150 rpm for 3 hours

For pH variations (Figure. 5), which shows pH 8 conditions for FCB-M and FCB-MH adsorbents with adsorption capacities of 97.8364 mg/g and 101.8154 mg/g, respectively. Under these conditions, a dynamic equilibrium occurs between the adsorption rate, where no more Pb is absorbed or released or redissolved into the adsorbate because it has reached the equilibrium point [35, 36]. However, for adsorbents FCB-M and FCB-MH, at pH above 8, there is a decrease in adsorption capacity. This is because the Pb metal re-ionises and is released into the solution at that pH. The coconut fibre biocomposite containing amino groups (FCB-MH) is more effective than that without amino groups (FCB-M). This occurs because the iron oxide magnet in the amino group has a smaller diameter, which can expand the surface area of the adsorbent, resulting in high permeability and stable mechanical and thermal properties. Therefore, the adsorption process can proceed more effectively [37,38].

3.3. Effect of TSS content and colouring agents on the adsorption process

In the adsorption process, TSS was calculated based on the solution conditions with FCB-M and FCB-MH adsorbents at pH 4, 6, 8, 10 and 12. Fig 6 shows that at pH 8, there was a decrease in TSS for FCB-M by 65.8% and FCB-MH by 62.9%, where the initial TSS condition was 3.03 mg/mL. The results shown in the TSS calculation provide the same pH at the most optimal adsorption capacity. Under these conditions, the degradation of organic and inorganic compounds occurs optimally, causing a large amount of suspended substances to dissolve back into the water, thereby reducing the TSS content.

Surface modification of the adsorbent with amino groups has been shown to significantly increase the adsorption capacity of Pb^{2+} metal and total suspended solids (TSS). Mechanistically, the $–NH_2$ group acts as an electron donor that can coordinate with Pb^{2+} ions through the formation of complex bonds, thereby strengthening the chemical interaction between the metal and the adsorbent surface [34, 39]. At optimum pH conditions, some of the $–NH_2$ groups are protonated to $–NH_3^+$, which increases the electrostatic attraction to negatively charged colloidal particles and organic compounds that make up TSS, thereby enhancing the coagulation and adsorption processes. In addition, amino modification also increases surface heterogeneity and expands the number of active sites [40], supporting the dual adsorption mechanism between Pb^{2+} and TSS.

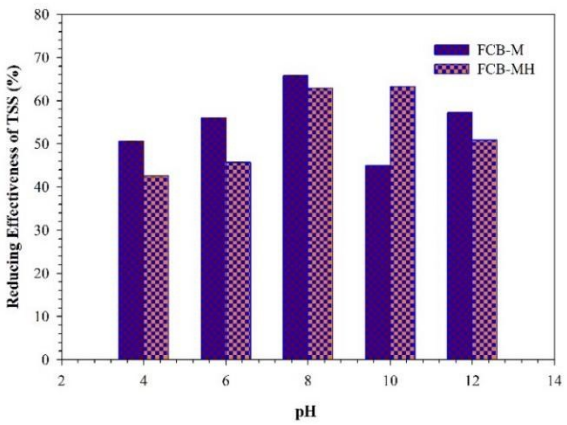

Fig. 6. Comparison of TSS reduction effectiveness with adsorbents at varying pH room temperatures, adsorbent concentration of 0.1 g, reaction time of 3 hours and stirring at 150 rpm

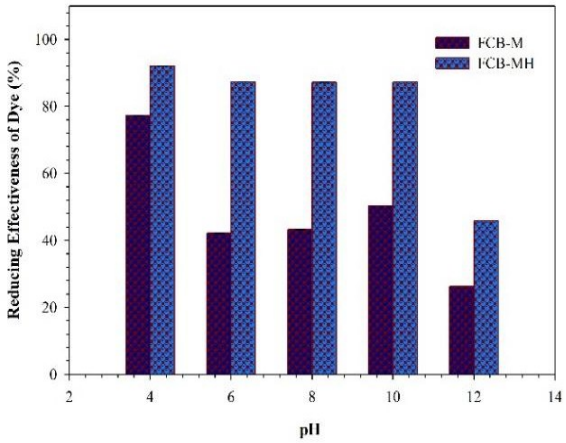

Fig. 7. Comparison of the effectiveness of dye reduction using various adsorbents at room temperature, adsorbent concentration of 0.05 g, reaction time of 3 hours and stirring at 150 rpm

The cellulose content in the adsorbent also functions to absorb colourants in waste. The effectiveness of colourant reduction can be seen in Fig 7. Colour analysis was performed using a UV-Vis spectrophotometer with a wavelength (λ) of 406 nm. The brighter the colour of the analysed material, the higher the absorbance value. Based on the colour analysis results, the colour intensity decreased at each specified pH, but at pH 8, more colourants were absorbed by the FCB-M and FCB-MH adsorbents, 81.68% and 164.85% respectively. At basic pH, the reduction of colourants is effective because the basic pH reduces the degradation process as a result of the formation of insoluble hydroxides. Similar to the reduction in TSS, the colour-forming components in the waste contain compounds that can react with the -OH and -NH2 groups in the biocomposite, so that the colourant can be bound to the adsorbent [41].

The advantage of this palm fibre biocomposite adsorbent is its magnetic nanoparticle properties, with a size ranging from 50 to 100 nm. With these properties, heavy metals (Pb) in waste will be quickly absorbed because they are attracted to these magnetic nanoparticles. In addition, this adsorbent also has the advantage of being reusable. The ease of regeneration and reuse of adsorbents is one of the important parameters in assessing the practical feasibility of an absorbent material in waste treatment. Palm kernel-based biocomposites have good regeneration capabilities with strong chemical stability and structure after several adsorption-desorption processes [42]. The magnetic properties of the adsorbent enable quick and easy separation using a magnetic field without significant mass loss. This material does not easily break down under mild regeneration conditions, supporting repeated use. Its good regeneration capacity, ease of separation, and abundant availability of raw materials make palm kernel-based magnetic adsorbents suitable for practical and economical application in the treatment of Sasirangan liquid waste containing heavy metals.

4. Conclusion

There are two types of palm oil structure, the first is fibre with a rough surface and the second with a porous surface containing silica. The amorphous structure changes to crystalline after the delignification process. There is Fe content in FCB and FCB-NH_2 because magnetism has formed in the palm oil fibre. The ability of FCB and FCB-NH_2 to remove lead (Pb^{2+}) content is constant at 60 minutes as time increases to a

value of 106 mg/g for FCB-MH and 43.0360 mg/g for FCB. Dynamic equilibrium occurs at a pH of 8 for FCB and FCB-NH_2 adsorbents, with adsorption capacities of 97.8364 mg/g and 101.8154 mg/g, respectively. The optimum condition for TSS reduction was at pH 8 with an effectiveness of 89% for FCB and 97.30% for FCB-NH_2. The optimum condition for colour reduction was at pH 8, with more colour being absorbed by the FCB and FCB-NH_2 adsorbents, at 81.68% and 164.85% respectively.

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