

ORIGINAL ARTICLE

Surface Modification of Hematite using Stearic Acid as Hydrophobic Inorganic Pigment Materials

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ABSTRACT – The inorganic pigment is a material that determines the color of a paint that can be used as a coating material. Until now, the basic material for red pigment, namely hematite, has been imported to meet domestic needs. This study aims to develop a hematite-based inorganic pigment material from the mill-scale waste of PT Krakatau Steel by modifying the surface to be hydrophobic, making the pigmentation material resistant to acid attack and preventing peeling and corrosion of the material. The surface modification of hematite is carried out by reacting it with stearic acid into ethanol as a solvent. Hematite was immersed in a stearic acid solution with various concentrations of 2, 4, 6, and 8% for 6 hours; it was then separated by filtration and followed by the drying process. The hematite-stearic was characterized by X-ray diffraction (XRD), Fourier-transform infrared (FTIR), scanning electron microscope (SEM), and water contact angle (WCA). The results show that the grafting process successfully changed the properties of hematite to hydrophobic. It is also supported by the FTIR spectrum due to the presence of molecular vibration of C-H, C=O, C-O, and Fe-O. Then, the higher contact angle is 150.474° for the stearic acid concentration of 6%.

ARTICLE HISTORY

Received: 10 Dec 2024

Revised: 31 Jan 2025

Accepted: 21 Feb 2025

KEYWORDS

Hematite
Hydrophobic
Inorganic
Pigment
Stearic

INTRODUCTION

The pigment is one of the basic components in paint manufacturing; it functions as a dye and hides power in the paint. The use of pigment every year always increases in line with the growth of pigment industries such as paint, ceramics, glass, textiles, plastics, and coating [11]. One of the iron oxide pigments is in the form of hematite ($\alpha\text{-Fe}_2\text{O}_3$), which has several advantages, including being more environmentally friendly, nontoxicity, chemical stability, high strength in coloring, covering power, and good durability [12]. In general, the supply of pigment in Indonesia is still mostly imported, so it is necessary to use natural materials or industrial waste to produce hematite as a raw material for pigments in the manufacture of paints to fulfill industrial needs.

Hematite pigments are usually a source of red dye in paints [13]. However, as a coloring agent with hiding power, the pigment will also be prone to corrosion if its surface is in contact with an acidic or high-salt environment. For example, paint on a ship's hull is easily corroded due to the acidic pH of peat water. Thus, surface modification to be hydrophobic can be applied to prevent material damage due to attack by water containing acids, salts, or bases to carry out wetting on the surface of a material coated with pigments.

Yu et al. [10] carried out surface modification of inorganic pigments with stearic acid. Stearic acid is an organic acid that can be used as a surface modifier of inorganic pigment materials to become hydrophobic. The chains of stearic molecules attached to the surface of the hematite pigment by grafting give new properties to the modified material. Furthermore, long-chain fatty acids can increase their non-wetting properties, which leads to their transformation into hydrophobic or superhydrophobic surfaces. In addition, stearic acid is a green coating material that promises to enhance hydrophobic properties. If its material were leached out into the environment, it would not be a problem due to its biodegradable properties and environmental safety towards supporting sustainability [14].

Based on the previous study, a hydrophobic or superhydrophobic surface can be achieved using the length of a carbon chain around 16 [15]. Stearic acid has the potential molecule that can increase the hydrophobicity due to the longer carbon chain (C-18). The surface modification is expected to be one of the steps in developing hematite-based pigment materials that are resistant to acid salinity conditions. The aim of this research is to produce a hydrophobic inorganic pigment that is resistant to acid and corrosion by modifying the surface of hematite (produced from mill-scale industrial waste) with stearic acid as a green hydrophobic agent. This innovative study addresses the gap by utilizing industrial by-products as a sustainable pigment source, diminishing reliance on imported hematite. This study seeks to integrate surface chemical modifications that induce hydrophobicity, verified using X-ray diffraction (XRD), Fourier-transform infrared (FTIR), scanning electron microscopy–energy dispersive X-ray (SEM-EDS), and water contact angle (WCA) measurements.

EXPERIMENTAL METHOD

Materials

The materials used in this work were ethanol (Sigma Aldrich, 96%), stearic acid (Merck), and hematite supplied by the Research Center for Advanced Materials-National Research and Innovation Agency. It was synthesized by a hematite mill scale from Krakatau Steel Company, as reported by Khaerudini et al. [1].

Surface Modification of Hematite

The surface modification approach was similar to our previous report [16]. First, 10 grams of hematite solid was weighed and immersed in stearic acid solutions of varying concentrations (2%, 4%, 6%, and 8%) dissolved in 96% ethanol at room temperature. The immersion time for each concentration was 6 hours to ensure complete surface coating of the hematite. After that, the hematite-stearic and filtrate were separated by filtration, followed by drying at ambient temperature for 24 hours to ensure the ethanol evaporation. The modified hematite pigment was then characterized using X-ray diffraction (XRD) PanAlytical ExpertPro, Fourier-transform infrared (FTIR) Shimadzu IRspirit-T, scanning electron microscopy–energy dispersive X-ray (SEM-EDX) Phenom ProX-G6, and water contact angle (WCA) analysis to determine the surface properties of the material.

RESULT AND DISCUSSION

Structural Analysis using X-Ray Diffraction (XRD)

The diffractograms of hematite-stearic are shown in Figure 1. The XRD pattern represents the peak of both materials and is suitable with JCPDS No. 33-0664. The diffraction peaks at 2θ of 24.19° , 33.23° , 35.69° , 40.93° , 49.51° , 54.15° , 57.63° , 62.53° , 64.05° and 72.01° indicate that the grafting process does not change the hematite structure.

Based on the XRD patterns in Figure 1, the crystallite size can be determined using the Debye Scherrer method. Table 1 shows the results of the calculation; the crystallite size of hematite-modified stearic increases with the higher stearic acid concentration. Hematite has a crystallite size of around 77.89 nm, which significantly increased to 93.37 nm at 8% stearic acid concentration. Investigations using XRD and Debye-Scherrer calculations revealed that surface functionalization of hematite with stearic acid leads to an increase in crystallite size (Table 1), which may be attributed to several factors. Stearic acid reduces surface energy, promoting the growth of larger crystallites and enabling smaller grains to merge into more thermodynamically stable configurations. Additionally, functionalization reduces strain and lattice defects, enhances crystallite alignment, and reduces peak broadening in XRD analysis. Stearic acid may also enhance particle dispersion and orientated attachment of smaller crystallites, allowing them to combine into larger domains. Furthermore, changes in surface chemistry can influence X-ray scattering and peak broadening, potentially leading to an overestimation of crystallite sizes [17].

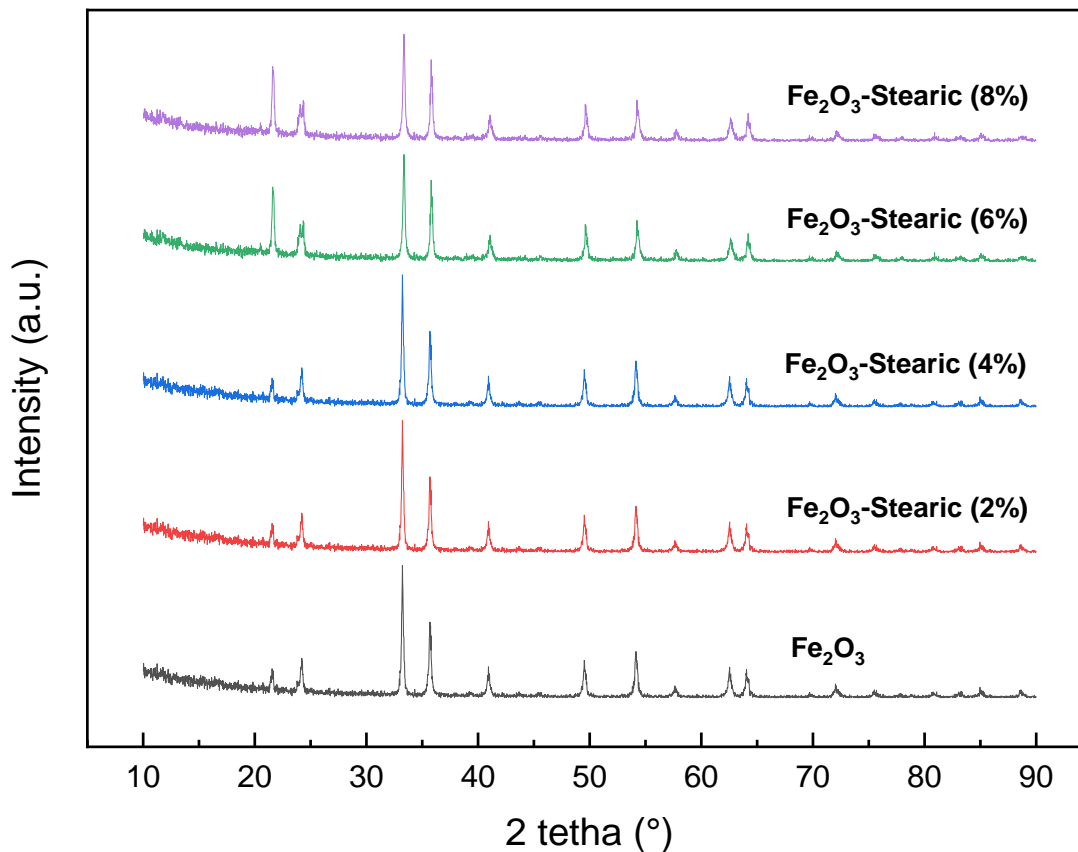


Figure 1. XRD patterns of hematite-stearic

Table 1. The crystallite size of the pigment

Materials	Crystallite Size (nm)
Fe ₂ O ₃	77.89
Fe ₂ O ₃ -Stearic (2%)	78.98
Fe ₂ O ₃ -Stearic (4%)	80.34
Fe ₂ O ₃ -Stearic (6%)	89.23
Fe ₂ O ₃ -Stearic (8%)	93.37

Fourier-Transform Infrared (FTIR) Analysis

The FTIR characterization was applied to identify the functional groups present in the hematite-stearic, as shown in Figure 2. Based on the results, it exhibits the vibration of C–H at 2965 cm⁻¹, which corresponds to the long chain (C-18) structure of the stearic molecule. The spectrum of FTIR has a different transmittance value due to the different number of stearic molecules that successfully grafted on the hematite surface.

The presence of the carboxyl functional group in stearic acid is confirmed by the C=O vibration at 1697 cm⁻¹ and the C–O vibration at 1467 cm⁻¹ [18]. Then, characteristic Fe–O vibrations at 1293 cm⁻¹, 693 cm⁻¹, and 520 cm⁻¹ indicate the presence of hematite, as shown in Table 2 [19]. The higher concentration of stearic acid might reduce the intensity of the functional group in the spectral. These spectral variations show that while increasing stearic acid concentration generally improves surface modification, excessive concentrations might cause non-uniform coverage or molecule aggregation, potentially limiting the efficiency of functionalization. An illustration of the hematite-stearic acid molecular model is presented in Figure 3 [20]. It is important to note that FTIR analysis primarily identifies functional groups and provides insight into molecular interactions within the sample.

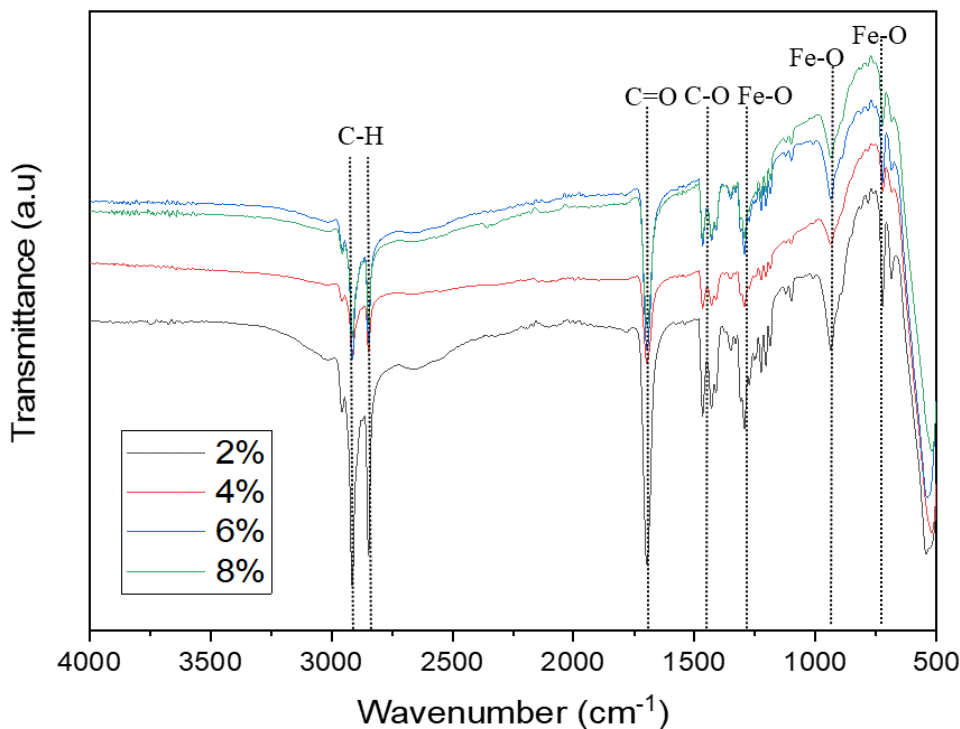


Figure 2. FTIR spectra of hematite-stearic

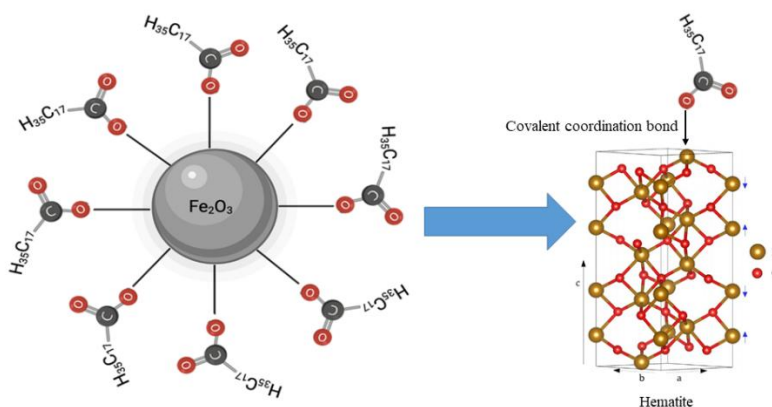


Figure 3. Illustration of the hematite-stearic molecule

Table 2. Functional group

Wavenumber (cm ⁻¹)	Functional groups
2956	C-H
1697	C=O
1467	C-O
1293, 693, 520	Fe-O

The interaction between the stearic molecule and hematite can be predicted as a covalent coordination bond, as reported by Patti et al. [21]. This mechanism offers strong stability on the hematite surface. If this material was applied as an inorganic hydrophobic pigment, it has the ability to be water-repellent and cannot react with water. This unique property demonstrated the promising hydrophobic materials.

Water Contact Angle Test

The water contact angle can be used to determine the surface properties of materials, and hematite had a hydrophilic surface based on many previous studies. As shown in Figure 4, the droplet water cannot pass through

into the material or the sample surface. It forms the droplet on the surface because the hematite was successfully transformed to hydrophobic because of the presence of stearic molecules.

The higher contact angle is 150.474° for 6% of stearic acid concentration, which means that the optimum condition for the grafting process of stearic on the hematite surface. Surface roughness and chemical composition were two important factors in determining the properties of hydrophobic surfaces. The surface was classified as hydrophobic if the water contact angle value (WCA) is more than 90°, then if the WCA value is more than 150°, it was classified as a superhydrophobic surface [22]. The lower concentration of stearic acid obtained the lower contact angle, which caused the stearic molecule to be successfully grafted into a small amount, but the surface properties can be transformed into hydrophobic.

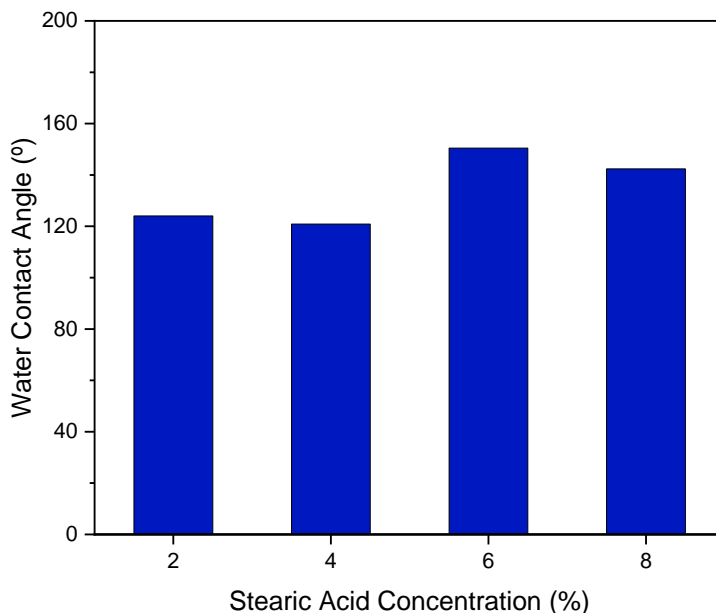


Figure 4. The contact angle of hematite-stearic

Based on their water contact angles, Table 3 contrasts the hydrophobic qualities of several inorganic materials that have been altered using different surface modifiers. Significant hydrophobicity was demonstrated by the combination of barite (BaSO₄) and TiO₂ treated with sodium octadecanoic acid, which produced a contact angle of 128.5°. A water contact angle of 110° was also shown by CaCO₃-TiO₂ modified with sodium oleate, suggesting moderate hydrophobicity. With a water contact angle of 150.47°, the hematite-derived material treated with 6% stearic acid in this work, on the other hand, demonstrated a superhydrophobic surface. Stearic acid's increased surface roughness and low surface energy are responsible for this better hydrophobicity. The findings demonstrate how well stearic acid works to increase hematite's hydrophobicity in comparison to alternative modifications, making it a material that shows promise for use on surfaces that repel water.

Table 3. Comparison of inorganic hydrophobic pigment materials

Inorganic materials	Surface modifier	Water contact angle (°)	Reference
Barite (BaSO ₄)-TiO ₂	Sodium octadecanoic	128.5	[23]
CaCO ₃ -TiO ₂	Sodium oleate	110	[24]
Hematite derived from mill scale	Stearic acid (6%)	150.47	This work

Morphology Characterization Using Scanning Electron Microscopy–Energy Dispersive X-ray (SEM-EDS)

The morphology of hematite-stearic is shown in Figure 5. Based on the SEM micrograph, it can be seen that the particle of material has irregular shape and size. The surface roughness of hematite-stearic also appears due to the irregularity of particles. Other factors that might be influenced by the surface properties are surface roughness and surface chemistry. Then, some parts of the SEM micrograph had a darker color, which caused the difference in atomic number of the element that composed the materials. The higher atomic number leads to the formation of a darker micrograph on SEM photos. Based on this explanation, the darker part of the SEM micrograph can represent the presence of Fe. Then, the different shapes and sizes of materials were not significantly affected by the grafting stearic molecules [25].

The surface morphology of stearic acid-coated hematite materials is displayed in the SEM image with various magnifications. Individual hematite particles are grouped together in the lower-magnification image, possibly as a result of surface contacts made possible by stearic acid. Higher magnification analysis reveals a heterogeneous distribution of larger faceted structures and the formation of granular particles, indicating a successful coating process. Greater in size, distinct crystallites scattered across a finer matrix suggest that stearic acid molecules may have aggregated on the hematite surface. This altered surface shape will probably improve the material's hydrophobicity, increasing its potential use in water-repellent applications. The functional characteristics of the resulting composite material are largely determined by the coating's homogeneity and the interaction between the stearic acid layer and the hematite core.

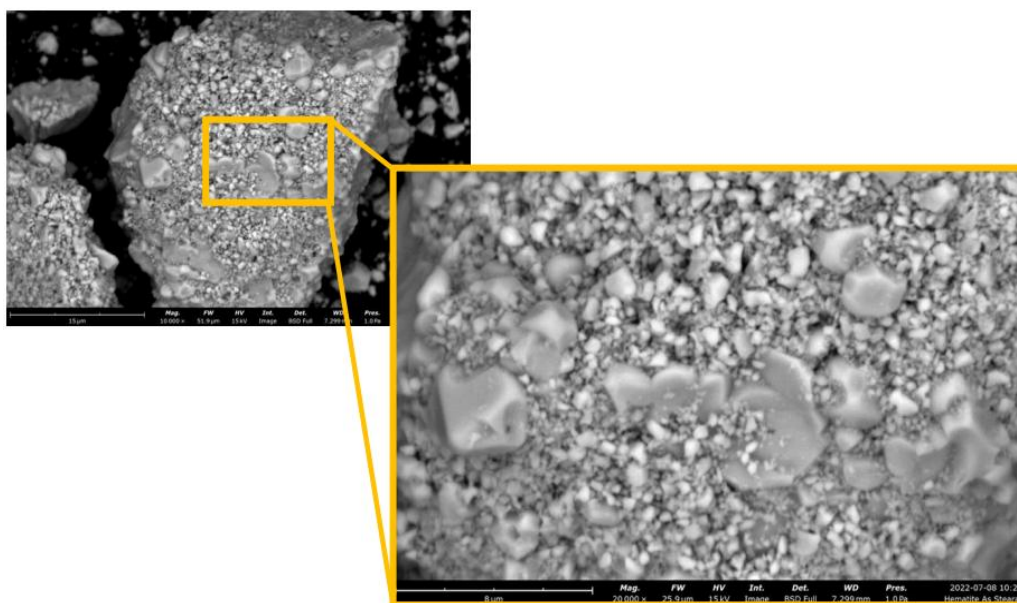


Figure 5. Microstructure of hematite-stearic (6%)

EDS data confirmed that the carbon (C) element appears on the surface of materials. Figure 6 represents the elemental mapping on hematite-stearic. The purple color confirmed the spread of the C element from stearic molecules. It can be concluded the grafting of stearic on the hematite surface was successful. On the other hand, elements from hematite, such as iron (Fe) and oxygen (O), were also detected on the surface materials. The O element had dominant intensity compared to another element, which caused the formula hematite to have a higher mol of oxygen. The Fe element had a lower intensity and C from stearic. The EDS spectra and the composition of elements are shown in Figure 6 and Table 3, respectively. As can be seen, it is 55.895% for O, 40.635% for Fe, and the lowest component is C, 3.470%.

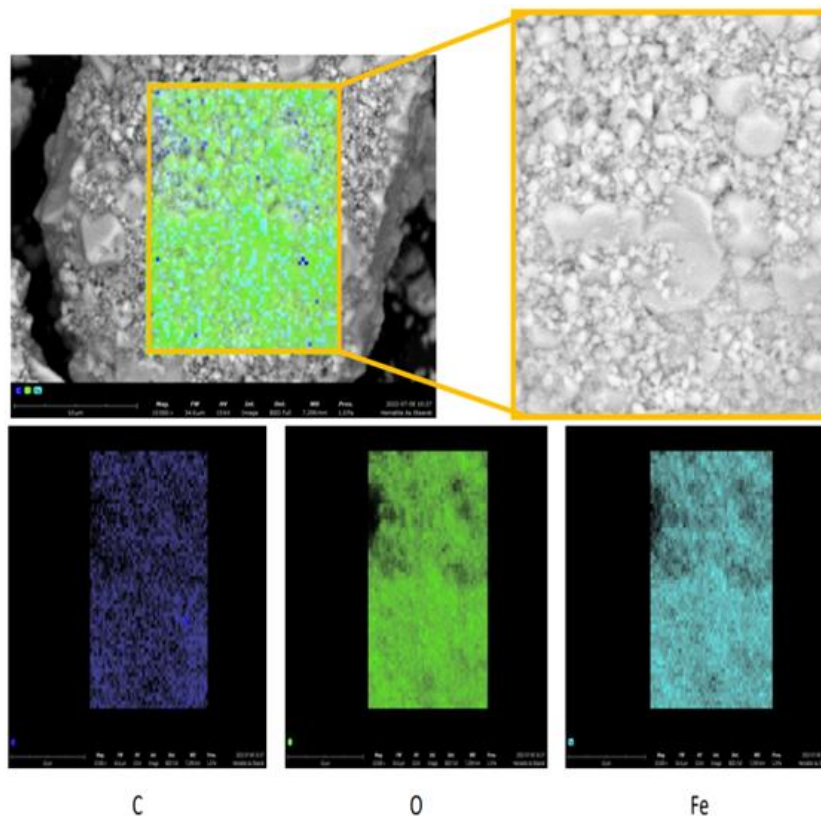


Figure 6. Elemental mapping of hematite-stearic (6%)

Table 3. Elemental composition-based EDS results in the optimum sample

Element	Percentage (%)
Carbon	3.470
Oxygen	55.895
Iron	40.635

CONCLUSION

The effective synthesis of hematite-modified stearic and the presence of a haematite structure in the X-ray diffraction (XRD) patterns indicate that the chemical and crystal structure of the hematite-derived mill scale was not altered throughout the grafting procedure. The water contact angle (WCA) data, demonstrating how haematite transforms into hydrophobic, verified that the stearic was successfully grafted onto the haematite surface. At 150.474°, the increased contact angle indicates a superhydrophobic surface at 6% stearic acid content. Higher stearic acid concentrations result in improved hydrophobicity. Nevertheless, excessive stearic acid concentrations cause uneven covering, which can reduce a material's hydrophobicity.

ACKNOWLEDGEMENT

The authors thank the Faculty of Mathematics and Natural Science, Universitas Palangka Raya, for the research grant under contract no. 668/UN24.10/PL/2022. We also thank PT Dynatech International for providing an SEM facility to characterize our samples. We also thank Reliana Romaito Sitinjak, Renita Tambunan, and Geni Kristina for their technical assistance in this work.

Credit for Authorship Contribution

A.D. contributed to conceptualization and funding acquisition. **D.S.K.** was involved in conceptualization, formal analysis, validation, and writing—review and editing. **F.A.** contributed to the methodology. **I.O.W.** and **I.S.** were responsible for visualization. **R.P.** participated in the formal analysis. **R.A.** contributed to funding acquisition and methodology. **R.M.I.** was involved in conceptualization, data curation, funding acquisition, methodology, writing—original draft, and writing—review and editing. **T.C.A.S.** contributed to data curation and investigation. All authors have read and agreed to the published version of the manuscript.

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