SURFACE MODIFICATION OF FLY ASH FROM ASAM-ASAM COAL POWER PLANT USING STEARIC ACID AS HYDROPHOBIC INORGANIC MATERIAL

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Abstract

Abundant coal reserves make this material a substitute fuel choice, especially for industry. The use of coal carries a high risk due to incomplete combustion and produces fly ash products. Fly ash cause pollution and health risks as well as environmental contamination when they are released, deposited, or leached into the ecosystem over short or long periods of time. The high content of silica and alumina in fly ash can be utilized and modified into new materials with added value. This research aims to modify the surface of fly ash using stearic acid as a hydrophobic inorganic material. Fly ash from Asam-asam Coal Power Plant was characterized by using XRD and modified by immersing in stearic acid (2,4,6, and 8%) and 98% ethanol. The result showed that the contact angle increases when fly ash is modified on the surface using stearic acid. The contact angle increases with increasing stearic acid concentration. The highest contact angle was obtained at a stearic acid concentration of 8%, and the lowest at 2% was about 112.9 and 102.2, respectively. The fly ash composition was primarily silica and alumina, which were crystalline, as confirmed by XRD. These findings provide several aspects of fly ash and its potential as a candidate material for environmental remediation and waste management.

Keywords*: coal, fly ash, modify, stearic acid, surface*

Introduction

The increment in population growth leads to enhanced demand for the energy sector. In many countries, the energy sector especially for electricity, was fulfilled using coal power plants to generate and produce the electricity. The use of coal causes many issues for the environment and sustainability. Coal combustion produces a high amount of CO_2 that is released into the environment, and it impacts air pollution as well as global warming. According to the latest estimates, over 30% of the world's CO_2 emissions comes from coal-fired power plants. According to the International Energy Agency (IEA), the combustion of coal to produce electricity and heat was responsible for over 14.6 gigatonnes of CO_2 in 2021, or roughly 42% of all CO_2 emissions associated with energy. Then, the coal combustion also produced some by-products such as fly ash and bottom ash (Adinugroho *et al*., 2022; Paraschiv and Paraschiv, 2020; Robbani *et al*., 2023; Spath *et al*., 1999).

Fly ash and bottom ash were categorized as hazardous materials in several countries. In Indonesia, the previous regulation classified both ash as a Hazardous material. Then, based on the new regulations of the Republic of

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Indonesia Number 22, year 2021 (Government Regulation no. 22 of 2021 on Environmental Protection, Organisation and Management., 2021) fly ash and bottom ash weren't categorized as hazardous materials (Ekaputri and Bari, 2021). So, it made it easier to utilize the by-product of coal combustion power plants into value-added materials. Currently, Indonesia's Steam Power Plants produce a significant amount of fly ash by production of burning coal (Mufrodi *et al.*, 2010).

Due to its regulation, the utilization of fly ash will be easy for supporting waste management in the power plant area. In the other hands, fly ash is a good source of silica and alumina for the synthesis of zeolite materials because of its high silica and alumina content. One of the benefits of using fly ash for zeolite synthesis is that it can be utilized directly, without needing to be pre-treated, because fly ash is abundantly available from coal power plants. Even if fly ash is obtained in a variety of compositions from different coal power plants, it can still be used for zeolite synthesis. Consequently, it can produce several zeolite framework types because of the variations in silica and alumina ratios. As reported by International Zeolite Association (IZA), zeolite had more than 200 of their framework or types. However, by adjusting the alumina and silica ratios and adding external sources of both substances, the desired zeolite can be produced. Zeolite-like materials can be used as carriers for metal catalysts, adsorbents, or other functional materials because of their porous, cavity-filled, and highly thermally stable solid structure (Sutrisno *et al.*, 2016) Several research studies have been conducted to transform fly ash into value-added materials (Alyatikah *et al.*, 2022; Ambrus *et al.*, 2019; Singh and Gupta, 2014; Wang *et al.*, 2021). Iqbal *et al* reported the transformation of fly ash into geopolymer materials that act for heavy metal immobilization (Iqbal *et al.*, 2022). Afterward, the fly ash also can be transformed into membrane materials for wastewater treatment (Agarwal *et al.*, 2020; Aprilianti *et al.*, 2021; Diana *et al.*, 2023). Boycheva *et al* also reported the transformation of fly ash into zeolite for carbon capture technology (Boycheva *et al.*, 2021). It indicates that fly ash can be utilized in many sectors and is easy to modify due to the presence of silica and alumina content, and the fine particles that lead to the higher surface area of fly ash (Risdanareni *et al.*, 2017).

As an above explanation, there was a limited report on transforming fly ash as a hydrophobic sorbent (Ge *et al.*, 2018; Lestari and Asrizal, 2022). Due to the higher surface area of fly ash, it was promising to be developed as a hydrophobic sorbent material. By modifying the surface, it is possible to control material's wettability and its interaction with liquids by altering its surface-free energy, which can change the hydrophobic and hydrophilic properties. A previous study reported the utilization of $CaCO_3$ from limestone that was modified using stearic acid as a hydrophobic sorbent for oil recovery, then Bidgoli *et al* developed chitosan-based oil sorbent to solve the issue of oil spill in the environment. Anuzyte *et al* (Anuzyte and Vaisis, 2018) have developed natural organic sorbent-based biomass through physical and chemical modification for combating oil spill issues. The surface grafting approach made it simple to turn hydrophilic materials into hydrophobic surfaces; it's used a self-assembly monolayer (SAM) mechanism that was ideal for the adsorption of low surface free energy molecules on the surface of porous materials. SAM mechanism can be divided by physical and chemical bonding between the hydrophobic agent and the substrates. If the interaction only physical adsorption, it had the lower stability compared to chemical bonding mechanism. The hydrophobic sorbent used in the wide application, and the oil spill or oil sorbent were very interesting fields towards supporting environmental sustainability.

Saturated fatty acid was the well-known surface modificatory agent for obtaining the hydrophobic surface (Wojas *et al*., 2021; Yang, *et al*., 2023; Zhang *et al*., 2023). Stearic acid is a saturated fatty acid obtained from animal and vegetable fats and oils. It is widely utilized due to its low cost and ease of production (Nguyen *et al*., 2021). Stearic acid is a monofunctional molecule made up of a long chain of 18 carbon atoms at the hydrophobic end and a carboxyl group at the hydrophilic end. As a result, its amphiphilic character makes it useful as an active interface agent. Moreover, it is employed to safeguard hydrophilic materials that are susceptible to moisture due to its strong hydrophobicity, which is a result of its lengthy carbon chain. In addition, the stearic acid molecules have the ability to self-assemble into micelles in an aqueous solution, resulting in the formation of a turbid hydrocolloid. The surface energy is reduced, water absorption is reduced, and the agglomeration of filler particulates is prevented when the filler surface is modified with stearic acid (Chen *et al*., 2020; Nguyen *et al*., 2021). The hydrophobic surface-based fly ash can be used in many sectors for wastewater treatment, i.e., cooked oil treatment and any other oil spill issues because of its simplicity, cost-effective, and eco-friendly (Sakthivel *et al*., 2013; Yu *et al*., 2023). However, natural-based materials like fly ash have significant limitations, including low adsorption capacity and poor selectivity (Bai *et al*., 2023). Therefore, synthetic natural-based-oil-absorbing material are being investigated in order to increase their oil-absorbing ability.

Research Methods

Materials

The experiment was carried out using stearic acid ($C_{18}H_{36}O_2$) (p.a), fly ash from Asam-Asam coal power plant, technical grade of ethanol 96%, and distilled water.

Surface modification of fly ash and its characterization

Firstly, the coal characteristic was informed by the CoA data from Asam-Asam coal power plant. Then, the fly ash was characterized using X-Ray Diffraction to determine the crystalline phase. The fine powder of fly ash was immersed in stearic acid solution and ethanol 96% with various concentrations, i.e., 2, 4, 6, and 8 % (w/v) for 6 hours (sample was denoted as FA-S2, FA-S4, FA-S6, and FA-S8). The surface properties of all developed samples were determined using Water Contact Angle (WCA) analysis.

Results and Discussion

The primary energy source in steam power plants is coal. Coal has characteristics that can be shown from the amount of calorific value. The higher the caloric content of coal, the better the quality of the coal. In addition to calorific value content, several other parameters affect the quality of coal, such as total moisture, inherent moisture, ash content, volatile matter, fixed carbon, and total sulfur. Coal quality data used by the Asam-Asam coal power plant is presented in Table 1.

Table 1. Coal characteristic (Proximate analysis (%))

Parameter	Air Dried Basis (ADB)	As Received Basis (ARB)	Dry Ash Free Basis (DAF)	Dry Basis (DB)
Total Moisture[1]		36.25		
Inherent Moisture[2]	14.84			
Ash Content[3]	3.66	2.74		4.30
Volatile Matter[4]	42.56	31.86	52.22	49.98
Fixed Carbon[5]	38.94	29.15	47.78	45.72
Total Sulphur[6]	0.28	0.21	0.34	0.33
Gross Calorific Value (Kcal/Kg)[7]	5579	4176	6845	6551

[1]ASTM D3302/D3302M-2015, [2]ASTM D3173-2011, [3]ASTM D3174-2012, [4]ASTM D3175-2011, [5]ASTM D3172-2013, [6]ASTM D4239 method A, [7]ASTM D5865-2013

The coal combustion process produces a very fine dust known as fly ash, which exits the furnace chimney. Fly ash is an excellent pozzolanic substance. Fly ash is composed primarily of silica (SiO_2), aluminum (Al_2O_3), and iron (Fe_2O_3) whose content is presented in Table 2. Based on the result of Table 2, the fly ash from Asam-Asam power plant can be categorized into F type due to the low content of CaO. The F type of fly ash was easier to modify to convert as aluminosilicate-based materials, I.e., zeolite and geopolymer.

Table 2. Fly ash composition (Ash analysis (%))[8]

Composition	Dry Basis (DB)
SiO_2 (Silica Dioxide)	46.6
Al_2O_3 (Aluminium Oxide)	10.1
Fe_2O_3 (Iron Oxide)	23.1

[8]AS 1038 Part 14.2:2013

The X-ray diffraction pattern of fly ash from Asam-Asam coal power plant is presented in Figure 1 dan this data is suitable to the composition result in Table 2. Figure 1 shows that the XRD pattern of fly ash will refer to the same dispersion peaks as the reference pattern recorded in the database that was used to identify the main mineral constituents of the material in crystalline or amorphous phase. It was observed that fly ash consists of major phases of mullite ($3Al_2O_3{\bullet}2SiO_2$) and silica (SiO_2) and a minor phase of hematite (Fe_2O_3). The diffractogram represented the presence of mullite, alumina, and hematite, at 2 tetha of 20.85°, 26.65°, 50.13°, and 59.94°; 42.93° and 67.76°; 33.52, 35.57, and 62.28°, respectively. The hematite peaks in fly ash shown are similar to the hematite peaks reported by Ilmi *et al*. (Mualliful Ilmi, Nurdini, Maryanti, and Setiawan, 2021) are diffraction patterns at 33.14, 35.60, and 62.44°.

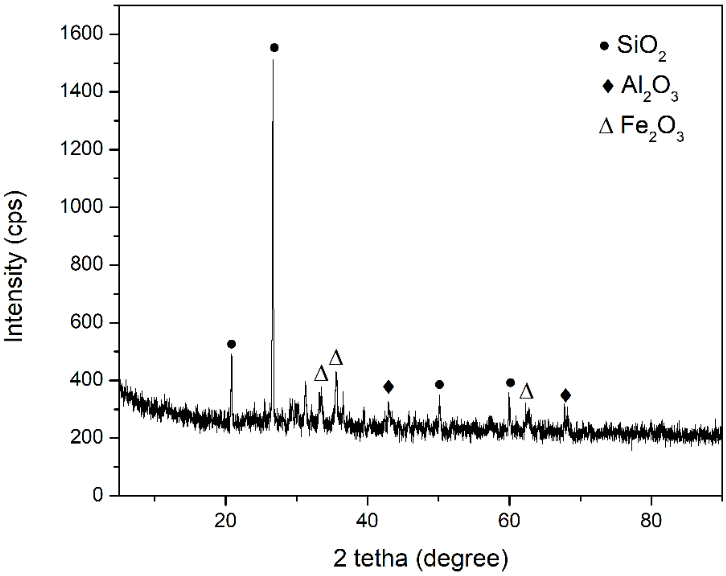

Figure 1. XRD pattern of fly ash from Asam-Asam coal power plant

A comparison of the hydrophobic properties of the 2, 4, 6, and 8% stearic acid addition variation, which is a surface test with water droplets, can be seen in Figure 2. The contact angle is the angle formed between the droplet and the solid surface in contact when the droplet is dropped. The hydrophobic characteristics of fly ash are tested by measuring the contact angle with ImageJ software, which is initially captured with a camera.

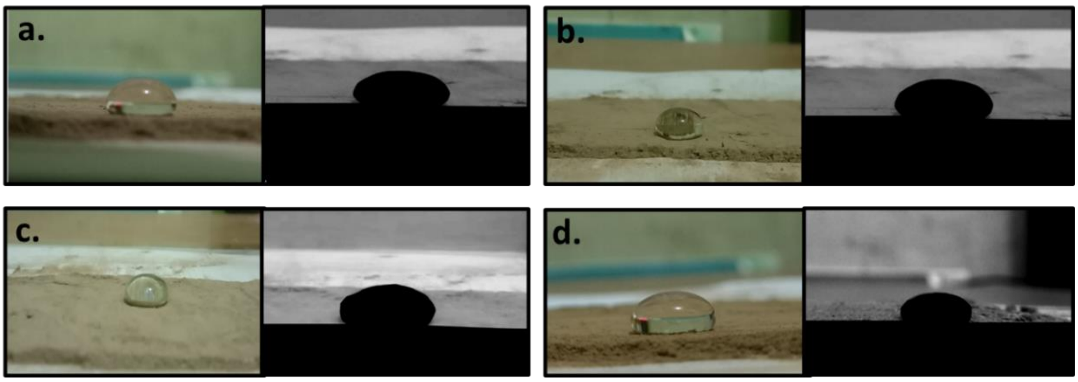

Figure 2. Measurement of the contact angle of water against a surface (a) FA-S2 (b) FA-S4 (c) FA-S6 (d) FA-S8

This contact angle measurement is important to determine whether the fly ash surface layer is hydrophobic. The hydrophobic surface has a large contact angle of 90°. Data on the effect of variations in the composition of stearic acid on the contact angle was represented in Table 3 and Figure 3.

Table 3. Average measurement results of contact angle

Sample	Average Contact Angle (°)	Surface Properties
FA-S2	102.2	Hydrophobic
FA-S4	107.5	Hydrophobic
FA-S6	108.8	Hydrophobic
FA-S8	112.9	Hydrophobic

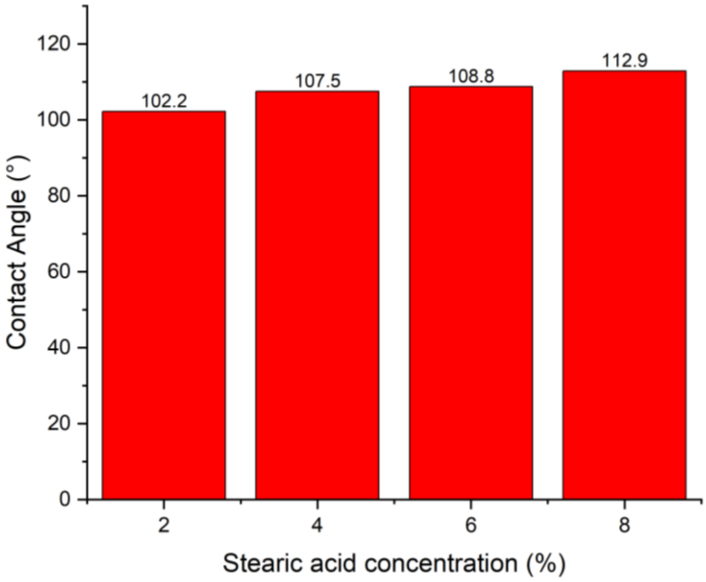

Figure 3. Comparison of contact angle data for various stearic acid concentration

The data that has been analyzed showed the changes in the results of contact angle analysis increase with changes in the concentration of stearic acid addition. Stearic acid contains a long-chain alkyl that increases surface hydrophobicity and was identified as an adhesive based on the strength of its effect on mechanical characteristics. N. Yao, *et al.*, at 2013 studied that the hydrophobic properties improved after stearic acid coating onto the fly ash surface (Yao *et al.*, 2013). The higher concentration of stearic acid obtained the higher contact angle due to increased adsorption of stearic acid molecules onto the fly ash surface via self-assembled monolayer formation. The greater amount of low surface free energy material on the surface directly enhanced the water contact angle and indicated the transformation into hydrophobic materials was done successfully. Testing the highest contact angle on fly ash with an 8% stearic acid variation of 112.9°. The lowest contact angle of fly ash with a 2% stearic acid variation of 102.2°.

Table 4. Comparison of contact angles of functional materials against water

No.	Materials and modification	Contact Angle (°)	Reference
1.	Functionalized hydrophobic sponges coating of stearic acid	143.2	(Azam, *et al.*, 2021)
2.	The surface of ultra hydrophobic glass prepared using the combination of stearic acid and TiO_2	141.0	(Wellia, *et al.*, 2020)
3.	Polyurethane sponge material modification using ZnO, Fe_3O_4+TEOS, dan stearic acid	153.4	(Putri, 2020)
4.	5052 Aluminum alloy obtained from LDH film modified with stearic acid	154.0	(Malta *et al.*, 2019)
5.	Surface modification of fly ash with stearic acid	112.9	This work

The use of stearic acid in fly ash greatly affects its antiwetting properties of water and the hydrophobicity of the coating was further improved. Table 4 presents the results of studies that have carried out surface modification using stearic acid which obtained contact angle performance in the hydrophobic range even to superhydrophobic. Fly ash soaked in stearic acid solution aims to make stearic acid molecules attached to fly ash with intermolecular forces. The long alkyl chain on stearic acid can reduce surface tension and cause transformation into hydrophobic or superhydrophobic properties.

The bonding strength of the coating to the substrate is the most important criteria for acceptable coated material performance. Sol-gel deposition and the one-layer coating process were used to create a highly hydrophobic and durable covering composed of SiO_2-TiO_2-alkylsilane. The usage of SiO_2 and TiO_2 improved the surface roughness and adhesion strength of the coatings. The hydrophobicity of the coatings increases with the length of the silane's alkyl chain (up to C=8). However, the water contact angle reduced when the C=16 alkyl-silane formulation was used, as reported by Liang *et al* (Liang *et al*., 2022).

Both factors are important to change the wettability of the surface. The lower concentration of stearic acid results in a lower contact angle, which causes the stearic molecules to be successfully grafted into a small amount, but their surface properties can be changed to hydrophobic.

Conclusions

Surface modification of fly ash using stearic acid has been investigated. The coal from Asam-asam Coal Power Plant has a high caloric value content. Based on the diffractogram obtained from the XRD analysis results, it indicates that the fly ash has a crystal structure with the presence of mullite, alumina, and hematite. The optimum concentration of stearic acid was 8% with a contact angle of 112.9, and the minimum concentration was 2% with a contact angle of 102.2. This finding exhibited that stearic acid-grafted fly ash was a promising material for an application that requires a hydrophobic surface. i.e., sorbent for oil spill issues.

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