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Physical properties and structural characteristics of alkali modified fly ash

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Abstract. Fly ash as residue from coal combustion has potential for environmental applications as an adsorbent for water pollution treatment. Adsorption performance of fly ash depends on fly ash origin and chemical treatment. Modification by the chemical treatment could increase the adsorption capacity. In this paper, fly ash was alkali modified with NaOH solution at various concentrations (2 N, 4 N, 6 N, 10 N) at a temperature of 60 °C. The physical properties and structural characteristics of unmodified fly ash and alkali modified fly ash were studied from Brunauer-Emmett-Teller (BET) surface area and pore size analysis, and also scanning electron microscope (SEM) analysis. The results showed that alkali modification could increase surface area, average pore radius, and total pore volume in fly ash. Unmodified fly ash and alkali modified fly ash could be classified as mesoporous materials and exhibited type IV nitrogen adsorption-desorption isotherms with H3 hysteresis loop according to the classification of the International Union of Pure and Applied Chemistry (IUPAC). SEM observations revealed that modified fly ash had rougher surface and more porous structure than that of unmodified fly ash. Alkali modification had changed the physical properties and structural characteristics of fly ash that supports its application as an adsorbent.

1. Introduction

Fly ash is residue from coal combustion generally at power generation plants. Fly ash can be collected especially by electrostatic precipitator. As a solid waste, fly ash has been widely utilized as raw material for zeolites and geopolymers production; substitute material in cement and concrete production; road embankments; source of silica and alumina, etc [1-2].

Fly ash contains oxides mainly from silicon, aluminium, iron, and calcium. Based on its contents, fly ash can be classified as class C fly ash and class F fly ash according to ASTM C618. Class C fly ash contains silicon dioxide, aluminium oxide, and iron oxide up to 50% and obtained from the combustion of lignite and sub-bituminous coal, meanwhile, class F fly ash contains silicon dioxide, aluminium oxide, and iron oxide up to 70% and obtained from the combustion of anthracite and bituminous coal [2].

Fly ash has also been widely utilized as an adsorbent for water pollution treatment [3-4]. Adsorption is one of water pollution treatment methods with the simple, efficient and economical process [5-6]. Compared to zeolite, the common adsorbent, fly ash is cheaper and easily obtained. Adsorption performance of fly ash depends on fly ash origin and chemical treatment. Raw fly ash generally has low adsorption capacity. Modification by the physical and chemical treatment could increase the adsorption capacity [7-8]. Fly ash modification by chemical treatment can be conducted



by acidic solution or alkali solution. The chemical treatment mostly takes place at a temperature of about 100 °C. Alkali modified fly ash has shown as more effective adsorbent than that of acidic modified fly ash [9-10]. Alkali modified fly ash has been applied as heavy metals adsorbent, i.e. (Pb(II) ion [10-11], Cd(II) ion [10], Ni(II) ion [12], Cr(VI) ion [13], Cu(II) and Zn(II) ions [14].

In this paper, fly ash from power generation plants in East Java, Indonesia was alkali modified with NaOH solution at various concentrations at relatively low temperature, i.e. 60 °C. The physical properties and structural characteristics of unmodified fly ash and alkali modified fly ash were studied from Brunauer-Emmett-Teller (BET) surface area and pore size analysis, and also scanning electron microscope (SEM) analysis. The results would provide useful information for fly ash utilization as adsorbent.

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2. Experimental

2.1. Materials

Class F fly ash was obtained from power plants in East Java, Indonesia. Fly ash contained major oxides i.e. Al₂O₃ (22.62%), CaO (14.80%), Fe₂O₃ (9.45%), and SiO₂ (42.38%) based on X-Ray Fluorescence (XRF) analysis. Commercial sodium hydroxide flakes (purity of 98%) was used as alkali modifier for fly ash.

2.2. Alkali modification of fly ash

Fly ash was sieved with 100 mesh standard sieve and then modified with alkali solution i.e. 2 N, 4 N, 6 N, and 10 N NaOH solutions, respectively, with solid to liquid ratio of 1:8 at a temperature of 60 °C for 8 hours. Modified fly ash was washed with deionized water and dried at 110 °C in the oven for 12 hours.

2.3. Analysis

The physical properties of unmodified fly ash and alkali modified fly ash were determined by Brunauer-Emmett-Teller (BET) surface area and pore size analyzer using Nova 1200e Quantachrome Instruments. Structural characteristics of unmodified fly ash and alkali modified fly ash were observed by scanning electron microscope (SEM) using JEOL JSM-6510LA Instruments.

3. Results and Discussion

The results showed that alkali modification could increase surface area, average pore radius, and total pore volume in fly ash as shown in table 1. The increase of NaOH solution concentration was in line with the increase of fly ash surface area. The highest surface area of alkali modified fly ash was obtained with 10 N NaOH solution, i.e. 18.693 m²/g.

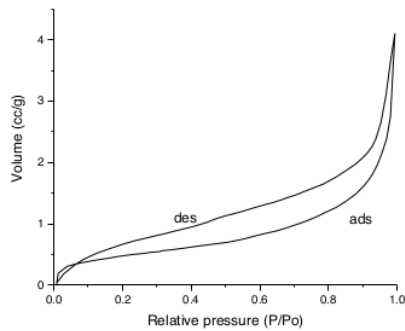
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Table 1. Physical properties of unmodified fly ash and alkali modified fly ash

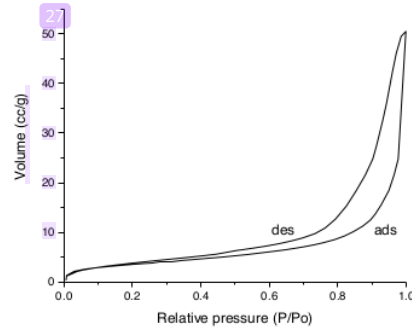
Material	Surface area (m ² /g)	Average pore radius (nm)	Total pore volume (cc/g)
Unmodified fly ash	1.745	7.270	0.006
Modified fly ash with 2 N NaOH solution	13.029	11.986	0.078
Modified fly ash with 4 N NaOH solution	11.122	16.938	0.094
Modified fly ash with 6 N NaOH solution	14.941	21.289	0.159
Modified fly ash with 10 N NaOH solution	18.693	12.892	0.120

Based on average pore radius, unmodified fly ash and modified fly ash could be classified as mesoporous materials. Mesoporous materials contain pores 2-50 nm in width, meanwhile, microporous materials and macroporous materials contain pores < 2 nm in width and > 50 nm in width, respectively [15-16]. Mesoporous materials have been reported to be widely used as adsorbent [16-18].

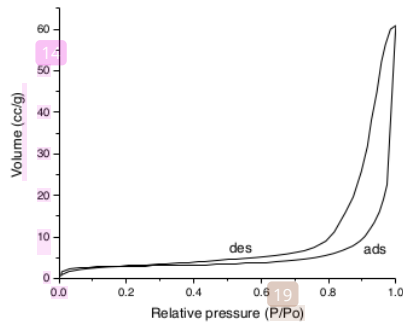
In contrast to the surface area, the highest average pore radius and total pore volume of alkali modified fly ash were obtained with 6 N NaOH solution, i.e. 21.289 nm and 0.159 cc/g, respectively. Alkali modification of fly ash could change fly ash structure into a zeolite structure. This structural change would be easier with an alkali solution having a higher concentration. Alkali modification of fly ash with 10 N NaOH solution might cause more zeolite structure that had high surface area but slightly lower average pore radius and total pore volume than that of alkali modification of fly ash with 6 N NaOH solution [19].



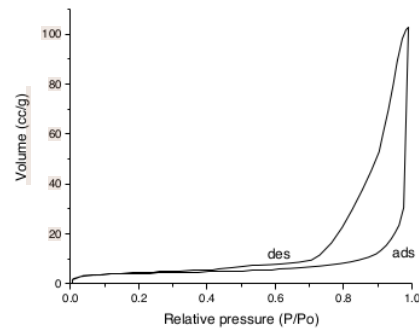
(a)



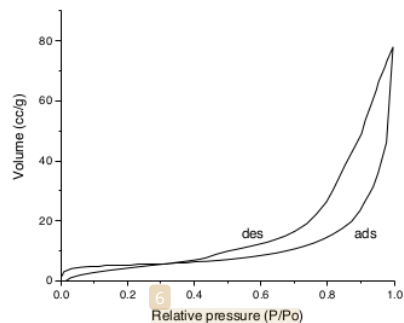
(b)



(c)



(d)

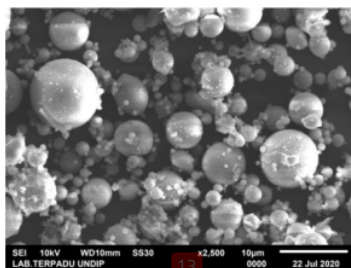


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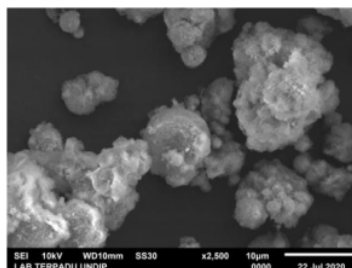
Figure 1. Nitrogen adsorption-desorption isotherms for unmodified fly ash (a) and modified fly ash with 2 N NaOH solution (b), 4 N NaOH solution (c), 6 N NaOH solution (d), 10 N NaOH solution (e).

The nitrogen adsorption-desorption isotherms of unmodified fly ash and alkali modified fly ash showed type IV isotherm with H3 hysteresis loop according to the classification of the International Union of Pure and Applied Chemistry (IUPAC) as shown in figure 1. Type IV nitrogen adsorption-desorption isotherms are given from mesoporous materials that have a hysteresis loop, indicating monolayer-multilayer adsorption and capillary condensation [15-17]. The type of nitrogen adsorption-desorption isotherms hysteresis loops was H3 type with characteristics of the pores in the material in the form of slits [18, 20].

The appearance of hysteresis loop on unmodified fly ash was slightly different than that on alkali modified fly ash. At unmodified fly ash, the hysteresis loop began at a low relative pressure (P/P_0), namely about 0.1, indicating the presence of micropores [19]. Meanwhile, at alkali modified fly ash, the larger hysteresis loops were at a higher relative pressure (P/P_0) indicating there were many mesopores [21]. This result was in accordance with the higher average pore radius of alkali modified fly ash compared to unmodified fly ash.



(a)



(b)

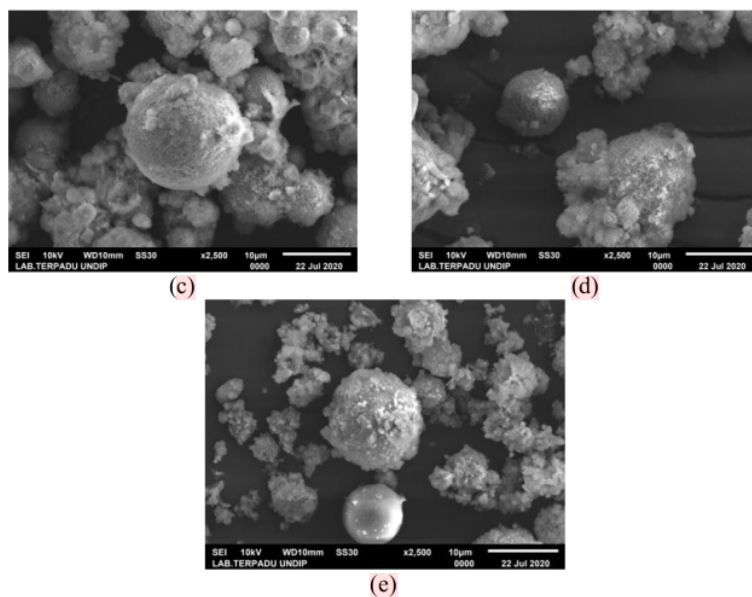


Figure 2. Scanning electron micrographs of unmodified fly ash (a) and modified fly ash with 2 N NaOH solution (b), 4 N NaOH solution (c), 6 N NaOH solution (d), 10 N NaOH solution (e).

Scanning electron micrographs of unmodified fly ash and alkali modified fly ash with 2500x magnification are shown in figure 2. There was a significant difference of structure between unmodified fly ash and alkali modified fly ash. Unmodified fly ash contained smooth spherical particles, whereas alkali modified fly ash contained particles with a rougher and more porous surface. Alkali solution had damaged smooth surface on fly ash so that its surface became rough and porous. This observation confirmed that alkali modification has changed the surface structure of fly ash so that alkali modified fly ash had a higher surface area, average pore radius and total pore volume than that of unmodified fly ash. Similar observations were reported by previous studies [11-14].

It seems that data obtained from alkali modification of fly ash can support the application of fly ash as adsorbent. Adsorbents are generally characterized from their physical properties such as surface area and porosity. The increasing surface area and porosity can increase adsorption capacity due to existence of more adsorption sites [22-24].

4. Conclusion

Alkali modification of fly ash with NaOH solution had been studied. Alkali modification could increase surface area, average pore radius, and total pore volume in fly ash. From BET surface area and pore size analysis, unmodified fly ash and alkali modified fly ash could be classified as mesoporous materials. Both unmodified fly ash and alkali modified fly ash exhibited type IV nitrogen adsorption-desorption isotherms with H3 hysteresis loop according to the classification of IUPAC. SEM observations revealed that modified fly ash had rougher surface and more porous structure than that of unmodified fly ash. Alkali modification had changed the physical properties and structural characteristics of fly ash that supports its application as an adsorbent.

Acknowledgements

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