



Surface modification of oil fly ash and its application in selective capturing of carbon dioxide

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ABSTRACT

Oil fly ash from power generation plants was activated with 30% NH₄OH and used for selective adsorption of carbon dioxide from CO₂/N₂ mixture. The treated samples were characterized for their surface area, morphology, crystalline phase, chemical composition and surface functional groups. Energy dispersive X-ray analysis showed an increase in the carbon contents from 45 to 73 wt% as a result of leaching out metal oxides. XRD proved that chemical activation of ash resulted in diminishing of major crystalline phases of zeolite, and other alumino-silicates leaving only quartz and mullite. BET analysis showed an increase in surface area from 59 to 318 m²/g after chemical activation and the pore volume increased from 0.0368 to 0.679 cm³/g. This increase in pore volume is supported by the results of SEM, where more micropores were opened with well-defined particle sizes and porous structure. The TGA of the treated fly ash showed stability at higher temperature as the weight loss decreased with increasing temperature. For treated ash, the FTIR displayed new peaks of amine functional group. The treated ash was used for the removal of CO₂ from CO₂/N₂ mixture and the maximum adsorption/capturing capacity was found to be 240 mg/g. This capacity increases with increase in initial gas concentration, inlet flow rate and temperature suggesting the endothermic nature of the interaction between the gas molecules and the surface of the ash.

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1. Introduction

The combustion of coal and fossil fuel results in a by-product called fly ash which are normally collected by means of cyclones (mechanical devices) or electrostatic precipitators to control air pollution [1]. This material has no economical value and is one of the major waste materials in that required disposal. Approximately 50 million tons of coal combustion products were produced in 2010, about 43% of the total generated by coal burning power plants were recycled in a variety of beneficial uses [2]. However, large quantities of fly ash are produced in United States and China with 32% and 40% utilization levels, respectively [3,4]. In the United States, more than 71 million tons of fly ash are produced annually in the coal power plants but only 45% is reused in different application [5]. They are used as fillers in polymer, cementitious materials, as a replacement of Portland cement, asphalt, stabilizing agent, and wastewater treatment [6–12].

Particular attention has been given to fly ash as an economical adsorbent for its leachability of metals from waste water and removal of flue gases because of its readily availability and

abundance, another advantage of using fly ash is its ability to easily solidify after the pollutants are adsorbed [13]. The components of fly ash are basically alumina, silica, iron oxide and residual carbon [1]. Toxic constituents include arsenic, beryllium, boron, cadmium, chromium, chromium VI, cobalt, lead, manganese, mercury, molybdenum, selenium, strontium, thallium, and vanadium, along with dioxins and PAH compounds [14] but these constituents vary according to the coal type used and the degree of combustion [15]. Shawabkeh et al. studied the enhancement of surface properties of oil fly ash (OFA) by chemical treatment [16]. Bada et al. investigated the property of fly ash when subjected to chemical and heat treatment for adsorption purposes. The Chemical treatment was performed using hydrochloric acid solution and compared with untreated heat treated samples. The analysis revealed that the chemically treated fly ash has a higher specific surface area as a result of corrosion of the outer layer of the fly ash to ash which disintegrates its stable glassy layer [17]. Kishore et al. studied the effect of surface treatment of fly ash by performing adhesion experiments in order to study the behavior of polymer composite [18]. Gray et al. reported the amination of fly ash carbons possessing alcoholic and carboxylic acid moieties on the surface with chloropropylamine. They evaluated the oxidation of the carbon surface with KOH in an effort to create more sites for amine anchoring. The surfaces of carbons are easily modified by functionalization with amines

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through reaction with surface functionalities [19]. Khan et al. studied the effect of chemical modification of OFA on the rheological and morphological properties of OFA/low-density polyethylene composites. They reported improved mechanical properties for OFA/polyethylene composites as a result of the surface modification [1]. However, with the increase in the surface area due to chemical treatment ash is increasingly finding use in the synthesis of geopolymers, zeolites and adsorption [14]. The objective of this study is to functionalize this waste material (OFA) by amine group and then use the modified OFA for selective capturing and removal of CO₂ from a gas mixture. The physicochemical treatments followed in this research aimed at increasing the surface area of OFA as well as the adsorption capacity especially at high temperature. The success of such approach will eventually lead to the utilization of a pollutant (OFA) in the removal of another pollutant (CO₂).

2. Experimental

2.1. Materials

OFA samples were received from power generation plants in Saudi Arabia. It is sieved to pass 45 μm meshes, and then stored in closed capped flasks for further uses. All glassware was Pyrex, washed with soap, water and demineralized water then dried in oven at 105 °C. Ammonium hydroxide with purity of ≥99.99% and density of 0.9 g/mL at 25 °C was supplied by Sigma–Aldrich Company. The CO₂ gas was 99.999% purity and supplied by Saudi gas industries.

2.2. Functionalization of oil fly ash

The functionalization of OFA ash was performed using NH₄OH. A sample of 100 g of the ash was mixed with 300 ml of NH₄OH in a 500 ml round bottom flask and refluxed at 120 °C for 24 h. The mixture was cooled and 150 ml of ammonium hydroxide was added and allowed to soak for 24 h. It was later filtered, and half of the mixture was dried at 105 °C for 24 h in the oven while the other portion was dried at room temperature. FTIR analysis was carried out on the final product to confirm the presence of amine functional groups on to the surface.

2.3. Characterization and analysis of oil fly ash

2.3.1. Scanning electron microscope (SEM)

The scanning electron microscope (JEOL, model JSM 6400) was used to determine the qualitative characteristics and morphology of the fly ash sample. The dried sample was fixed with double side masking tape. In order to make it surface conductive, the fly ash sample was carbon coated. It was then viewed on FE-SEM at different magnification to see the surface topography of the sample.

2.3.2. Brunauer–Emmett–Teller (BET) surface area determination

The BET technique has the ability to provide information on the total volume, specific surface area, pore size and pore volume distribution of the specified specimen, and as such, is used to determine the surface properties of the sample. Micrometrics 2020 was used to determine the BET surface area using nitrogen adsorption at 77 K. A sample of 0.3210 g was degassed and dried at 150 °C for 6 h under vacuum and the specific surface area was calculated by BET equation.

2.3.3. X-ray diffraction spectrometry (XRD)

The phase identification of the fly ash sample which is in a powdery form, were determined by the X-ray diffractometry using a Phillips PW 1830 diffractometer with a copper anode. It was operated at 40 kV and 40 mA for 1 h from 0° to 80° over the range of 2θ

and High Score Plus software was used for the identification of the phases present.

2.3.4. Energy dispersive X-ray (EDX) analysis

Energy dispersive X-ray (EDX) analysis was carried for the analysis of chemical composition of treated and untreated fly ash. The as-received fly ash is mainly composed of 92.5% carbon and 5.80% sulfur and traces of Mg, Si and V typical of heavy oil combustion residue [1].

2.3.5. Fourier transform infrared spectroscopy (FTIR)

The Fourier transform infrared spectroscopy (FTIR) was used to identify the presence of functional groups attached to the functionalized fly ash. It is also used to identify either organic or inorganic compounds on surfaces of particles. The sample was analyzed using FPC FTIR Perkin–Elmer spectrophotometer. A sample of 2 mg of ash was thoroughly mixed with 1.0 g of KBr powder and the mixture was hydraulically pressed at 10 tons/m² to obtain a thin transparent disk. The disk was then placed in an oven for 4 h at 110 °C to prevent any interference with carbon dioxide or water vapor. Transmission mode was used for taking the FTIR spectra.

2.3.6. Thermogravimetric analysis (TGA)

The Thermogravimetric analysis (TGA) was carried out using SDT Q600 model combined TGA–DSC analyzer to measure the rate of change in the weight of ash as a function of temperature. Also, the thermal stability of ash at temperatures up to 1000 °C was examined in a controlled atmosphere. Samples of 5–10 mg were weighed and placed in the sample pan. The temperature was raised from 25 to 1000 °C at a heating rate of 5 °C/min with nitrogen flow rate of 10 ml/min.

2.4. Adsorption of CO₂ by treated fly ash

The adsorption column was made of quartz glass, which is 18 cm in length and with inner diameter of 1.0 cm. The tube is surrounded by a glass jacket and water circulates through the jacket to maintain isothermal conditions. The adsorption column was filled with 1.5 g of the amine functionalized ash. Nitrogen gas is first introduced to the bed at the bottom of the column to free the surface of the ash. Then, CO₂ gas enters the bed from the bottom of the column at different flow rates, while the CO₂ digital meter attached at the exit measures the CO₂ concentration as a function of time. The relative humidity was measured at the inlet and outlet of the column using the relative humidity meter. To raise the humidity of the inlet gas, the gas was passed through a water bath at 70 °C before entering the column. The experiments were repeated at different CO₂ concentrations, flow rates and temperatures for low and high humidity. Desorption experiments were performed in a similar manner by passing nitrogen to the column and the resulting CO₂ concentration was measured as a function of time.

3. Results and discussion

3.1. Synthesis and characterization of fly ash

The process in which fly ash was mixed with certain alkaline activator such as ammonium hydroxide where the mixture is cured to make a solid material is known as alkaline activation. The chemical analysis of oil fly ash used in this study is shown in Table 1. In previous works [20,21], it was found that an amorphous aluminosilicate gel was the main reaction product formed during alkaline activation of fly ash and hence the resulting product is also known as zeolite precursor. On the other hand, the alkali activation of fly ash is a process normally related to the synthesis of zeolites [22].

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