



A novel approach to extract SiO₂ from fly ash and its considerable adsorption properties



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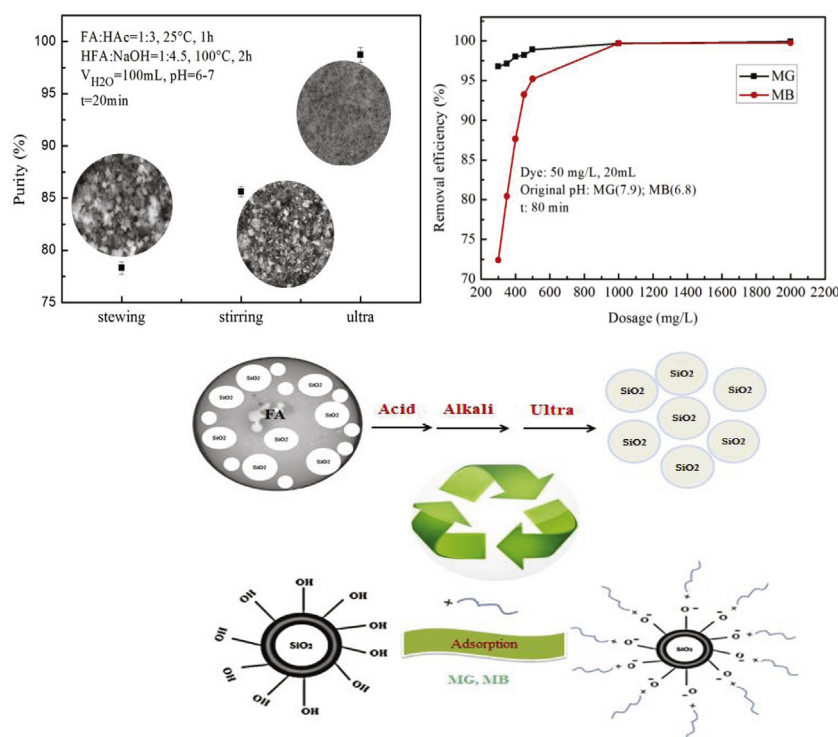
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HIGHLIGHTS

- A HAc/NaOH/ultrasonication-assisted joint process was applied to extract SiO₂ from FA.
- This joint process was feasible to separate Si/Al effectively.
- High purity (98.65%) and high yield (51%) of SiO₂ were obtained.
- The extracted SiO₂ exhibited better adsorption prospect to dyes.
- This research extended the application and recycling of FA solid waste.

GRAPHICAL ABSTRACT



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ABSTRACT

Silica extracting from fly ash (FA) becomes a key technology for its comprehensive utilization of waste. Here, high-purity SiO₂ was extracted from FA via organic acid/inorganic alkali/ultrasonication-assisted joint process and characterized by scanning electron microscopy, transmission electron microscopy, Brunauer-Emmet-Teller, X-ray fluorescence spectroscopy, X-ray diffraction, and X-ray photoelectron spectroscopy. The optimal extracting conditions were optimized: with acetic acid (HAc) leaching at 25 °C for 1 h and FA/HAc of 1:3; then with 15%

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NaOH dissolution at 100°C for 2 h and FA/15% NaOH ratio of 1:4.5; cooled and introduced water (5.42 g/100 mL); ultrasonication-assisted for 20 min. The final yield and purity of SiO₂ were 51% and 98.65%, respectively. This joint method could be applied in several various FA raw materials and the extracted SiO₂ exhibited better adsorption prospect to cationic dyes of methylene blue and malachite green, which extended the application and recycling of FA.

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1. Introduction

Fly ash are widely used in cementitious products [1,2], construction areas such as highway road bases [3], and efficient sorbents for removal of heavy metals, organics and dyes from water [4]. However, despite so many positive uses, the rate of production of FA is much larger than its consumption. There are lots of coal-fired power plants situated in the place where disposal sites are difficult to choose. So the limited availability of space requires very strict management to satisfy related regulatory bodies. Moreover, FA has negative effect on the environment. The disposal of FA is a more significant problem than ash produced from burning of municipal solid wastes because of its volume. So disposal directly of FA into the environment should be prohibited [5].

FA, usually with hollow sphere and activated sites, the main components are silica, alumina, ferrous oxide, calcium oxide and varying amounts of carbon and smaller amounts of other elements [6,7]. So extracting silica from FA could not only realize comprehensive utilization of FA to solve all kinds of negative issues brought about by FA, but also extend the production of silica [8]. Moreover, it can also decrease the Si/Al ratio in FA and realize silica and alumina separation. To improve aluminum extraction efficiency, a two-step acid leaching process was applied to dissolve aluminum and aluminum-bearing crystals with sulfuric acid, and >97% aluminum dissolving were obtained [9]. Moreover, the leaching process produced lots of by-products, sodium silicate solution and amorphous silicon dioxide, which contained nearly all the silicon from the coal ash. The response surface methodology was used to optimize the extraction conditions of silica and alumina from FA, the extracts containing Si and Al ions were successfully converted to zeolites by hydrothermal treatment at 95 °C for 72 h [10].

Currently, some researches have been reported in the silica enrichment and preparation for various forms of silicon products from FA. Acid methods [11–13] have been widely investigated. Typically, FA has been dissolved using inorganic acid solvent (such as HCl or H₂SO₄) to get aluminum salt solution at certain conditions, in this step, SiO₂ and Al₂O₃ have been separated roughly, then a series of subsequent processing has been followed and SiO₂ and Al₂O₃ have been purified. Drieux et al. [14] mixed the grinding FA and hydrochloric acid at certain temperature, then roasting ground to separate Si and Al to get highly purified SiO₂ and Al₂O₃ simultaneously. However, large amount of waste acid in the cyclic process were produced, which was harmful to the environment. Alkaline methods [15,16] included limestone sintering process, lime soda sintering process and alkaline dissolving. FA was treated with alkaline cosolvent or direct alkali solution to realize the separation of Si/Al. Wu et al. [17] studied the dissolution of silicon and aluminum from FA at different conditions and the results showed that 29.33% dissolution rate of SiO₂ was obtained at 950 °C with 2–3 mol/L alkali, and digesting at 120–130°C with pressing. Si and Al were separated preliminary, with low SiO₂ productivity more complex process and difficult administration. Acid/alkali combination method have attracted more attention [18,19]. Wu et al. [20] prepared high-purity Al₂O₃ and superfine SiO₂ (99.5%) with acid/alkali process. The consumption of acid and alkali and waste emission in the process were limited and the energy consumption was high. In addition, ammonia sulfate and calcination were also used to activate the non-reactive substance for extracting the silica [21–25]. Park [23] used the ammonia sulfate to extract the high concentrated silica from FA with low temperature sintering method and the recovery percentage of aluminum can

reach 94.36%. Jin et al. [24] studied the phase transformation of calcination products of FA and sulfate. But there were some disadvantages, such as large amount of FA residue, complex process and equipment, which limited its application for industrialization [25]. Moreover, several authors prepared SiO₂ nanostructures or xerogels with the organic acid. Arenas et al. [26] synthesized silica xerogels with high surface area using acetic acid as catalyst, and the influence of acetic acid on the pore structure and surface area of silica prepared by the sol-gel method was investigated. Elsagh [27] synthesized silica nanostructures and optimized the synthesis conditions, and the effect of molar ratio of reactants including acids and silica pre-matter was studied. However, the research was few and limited, and the above formation of SiO₂ all used organic precursors such as tetraethyl orthosilicate.

In view of the above shortcomings of traditional extraction method, here we studied the preparation of high-purity and fine-grained SiO₂ from inorganic precursor FA, a kind of solid waste residue, inorganic alkali/inorganic alkali/ultrasonication-assisted joint process firstly. The structural and morphological properties of SiO₂ were investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), Brunauer-Emmet-Teller (BET), X-ray fluorescence spectroscopy (XRF), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS). The effect of acid type, HAc dosage, acid leaching temperature, alkali dissolution temperature, and NaOH dosage on the purity and yield of SiO₂ were systematically investigated. The universality of this joint method was also investigated with various FA raw materials. On one hand, this method had some advantages: less consumption of materials (acid-base), low energy consumption with mild temperature, low cost with simple equipment and operation, and high purity of product. On the other hand, organic acid (acetic acid, HAc) was used in the process As a frequently used non-toxic acid, HAc can be removed facilely from the reaction system by calcined treating. Meanwhile, ultrasonication-assisted process was also studied in the extracting of SiO₂. Moreover, its adsorption capacities in dye removal from wastewater were studied. Considering the negative charged surface of the fresh prepared SiO₂, two basic dyes with cationic dominant groups, methylene blue (MB) and malachite green (MG) were chosen as model pollutants in the experimental adsorption test, the removal efficiencies at different conditions were evaluated. The possible adsorption mechanism was also proposed.

2. Experimental

2.1. Materials

The starting adsorbent material, raw FA, was collected from the electro-filters from two power plants located in Shaanxi Province. Raw FA was grinding fully for 10 min using a laboratory Plenary Rapid Grinding Miller (XM-4, Minsk, China), then sieved using standard sieves of 30 μm evenly with a fine grained particle size. All the organic acids and other reagents were analytical grade and used without any further purification. Distilled water was used throughout.

The chemical structures of MB and MG were displayed in Table 1.

2.1.1. Acid leaching step

FA was mixed with inorganic acid (HCl) or organic acid (HAc, oxalic acid, or citric acid) in a mass ratio 1:3, then transferred it into a temperature-controlled reaction kettle with a function of adjusting the temperature at 25 °C for 1 h with constant stirring. Then the mixture was

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