



Feasible recycling of industrial waste coal fly ash for preparation of anorthite-cordierite based porous ceramic membrane supports with addition of dolomite



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ABSTRACT

Recycling of industrial solid state waste is always the priority solution in the prospect of waste management. This work demonstrated a new method converting coal fly ash (CFA) industrial waste to anorthite-cordierite-based porous ceramic membrane supports (ACCMS), through high-temperature sintering with additions of dolomite mineral. Characterization of as-prepared ACCMS included phase composition, microstructure, pore structure, N₂ and water permeability, and mechanical properties. From dilatometric measurements, the densification temperature of a sample with 28.43 wt.% dolomite addition was extended to 1100 °C compared to 1060 °C for that of a sample without any dolomite. For samples sintered at 1150 °C, their open porosity increased from 25.2 ± 0.5% to 46.5 ± 0.2% when dolomite addition increased from 0 to 28.43 wt.%. It is suggested that this level of dolomite addition significantly inhibited the sintering behavior of CFA.

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1. Introduction

Coal is still one of the most important energy sources. Appropriate disposal of coal fly ash (CFA), the by-product produced after the combustion of raw coal in power plants, is a serious issue of concern. If not properly treated, it can disrupt ecological cycles, pose soil and water hazards and cause heavy air pollution such as the haze and smog occurring in recent years. In 2005, China produced 100 million tons of CFA, which was just less than India (118 million tons per year) and accounts for about 14% of global CFA production. In 2012, China accounted for 50.2% of the world coal consumption and the estimated amount of annual worldwide production of CFA is about 750 million tons. By 2015, the annual CFA generation in China is estimated to be 580 million tons. Despite its huge production, only ~45% of CFA has been recycled for other applications [1–4]. Traditionally, CFA can be a cement additive in construction

and other civil engineering purposes [5,6]. Also CFA is diverted to landfill which can increase environmental taxes, as well as causing heavy metal leaching in soils and other environmental concerns [7].

In addition to those traditional disposal and reuse methods, increasing attention is being focused on recycling CFA by other novel and high-value methods. CFA, a typical mineral mixture, can be represented by the various SiO₂–Al₂O₃–MeO systems, where MeO is alkaline (Na₂O, K₂O), alkaline-earth (CaO, MgO) and transition metal oxides (Fe₂O₃, MnO, etc.) [8,9]. Because of its multiple components and its large specific surface area, some studies have been done to realize its application in improving soil quality, including texture, nutrients, bulk density and water holding capacity [10–12]. In addition, CFA is used as an adsorbent to remove heavy metal and toxic ions from mineral wastes after simple treatment [13–15], and as starting material to synthesize microporous zeolites [16–18] and ceramic materials such as mullite, cordierite and anorthite [19–21]. Moreover, porous ceramic membrane supports have been prepared from inorganic raw materials such as chamotte [22], clay [23–25], quartz sand [26] and kaolin [27–29], which have components similar to CFA. In previous work, coal fly ash was used as a raw material to fabricate mullite (3Al₂O₃·2SiO₂) [30,31] and

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cordierite ($2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$) [32] ceramic membrane supports through controlling the phase assemblage and pore-structure with addition of Al-source materials (bauxite) and Mg-source materials ($4\text{MgCO}_3\cdot \text{Mg}(\text{OH})_2\cdot 5\text{H}_2\text{O}$). In this study, a new approach is proposed to recycle CFA with the addition of dolomite in order to prepare porous ceramic membrane supports.

Dolomite ($\text{CaMg}(\text{CO}_3)_2$) is a natural mineral with abundant reserves around the world (i.e., North America, North-Western and Southern Europe, North and West Africa, the Middle East, and the Far East [33]). It has been found in Liaoning, Neimenggu, Shanxi, Jiangsu, Fujian, Hunan, Hubei and Guangxi in China, with reserves of over 4 billion tons [34]. The thermal decomposition of dolomite follows two stages: $\text{CaMg}(\text{CO}_3)_2$ firstly decomposes into CaCO_3 , MgO and CO_2 ; then CaCO_3 converts into CaO and CO_2 [35–37]. A mixture of MgO and CaO, namely doloma, is usually obtained when dolomite is calcined above 900°C .

Thus, the addition of dolomite is expected not only to adjust pore-structure by inhibiting the sintering of CFA with the as-produced doloma, but also to form a Ca-rich phase (anorthite, $\text{CaO}\cdot \text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$) in support materials. Anorthite possesses low thermal expansion coefficient ($4.82 \times 10^{-6} \text{K}^{-1}$) and high thermal shock resistance, which is beneficial for its application, especially at high temperatures, as a ceramic support material. In addition, the release of CO_2 from dolomite during sintering indicates that dolomite could be a pore forming agent. Previous work conducted by Bouzerara et al., [38] and Zhou et al., [39] indicated that the addition of doloma or dolomite has a positive effect in enhancing of the porosity of supports sintered from kaolin mineral. However, no work has been done to investigate the effect of dolomite addition on porous supports prepared from CFA.

In this work, dolomite was directly added to CFA to fabricate a porous ceramic membrane support by an *in situ* sintering reaction, with the aim of improving porosity and permeability while providing acceptable biaxial flexural strength. The influence of dolomite on the structure and properties of porous ceramic supports are discussed in detail on the basis of dynamic sintering behaviour, phase evolution, porosity and pore size distribution, microstructure, gas and water permeation flux, and mechanical properties.

2. Material and methods

2.1. Starting materials

Industrial waste coal fly ash (CFA) obtained from Ningbo power plant (Ningbo, Zhejiang Province, China) and dolomite (Hebei Xinlei Mineral Powder Processing Plant, China) were used as the starting raw materials for the fabrication of porous supports. Dolomite in this work was used as pore forming agent, sintering inhibitor and reactant. A series of ceramic supports containing dolomite were prepared by adding 0 wt.%, 15 wt.%, 28.43 wt.% and 40 wt.% of dolomite to CFA, and were labeled as D0, D15, D28.43 and D40, respectively. The value of 28.43 wt.% for the dolomite additive is calculated from the stoichiometric reaction of aluminum present in the CFA to synthesize anorthite and cordierite from the CaO and MgO in the dolomite.

2.2. Membrane support fabrication

CFA and dolomite were separately wet ball-milled in dehydrated ethanol for 12 h with zirconia balls by using a planetary ball milling machine at a constant speed of 500 rpm. Then the CFA and dolomite were wet-mixed by planetary ball milling again for 3 h. After drying at 90°C for 24 h, the mixture was uniformly mixed with organic binder PVA-1750 (5 wt.% solution) in an alumina mortar followed by uniaxially pressing into cylindrical pellets (20 mm in diameter

and 1–2 mm in thickness) at 190 MPa. Then the green samples were sintered at temperatures from 1100°C to 1200°C in an electrically heated muffle furnace in air for 2 h. The heating rate was 5°C min^{-1} from room temperature to 200°C and 3°C min^{-1} up to final temperature. A holding time of 30 min at 450°C was employed to remove the organic binder. For comparison, the supports made from CFA only (namely D0) were also prepared using the same processing conditions described above.

For dilatometric measurements, porous rectangular bars ($40 \text{ mm} \times 7 \text{ mm} \times 3\text{--}4 \text{ mm}$) were fabricated by uniaxial cold-pressing at a pressure of 190 MPa.

2.3. Characterization

Chemical compositions of CFA and dolomite were analyzed by quantitative X-ray fluorescence spectroscopic analysis (Axios-Advanced, PANalytical Corporation, Netherlands). The particle size distribution of CFA and dolomite were determined by a laser particle size analyzer (Mastersizer 2000, Malvern Instruments Ltd., UK) using water as dispersing medium. The phase compositions of the starting materials (CFA and dolomite) and sintered supports were characterized by XRD (DX-2700, Dandong Haoyuan Instrument Co., Ltd., China, $\text{Cu K}\alpha$ radiation, working voltage 40 kV, working current 50 mA, scanning speed of $5.0^\circ (2\theta) \text{ min}^{-1}$). XRD semi-quantitative analysis based on the normalized reference intensity ratio (RIR) method was employed to determine the phase contents. Morphologies of the starting materials and sintered supports were observed by a scanning electronic microscope (SEM-EDS, S-4800, Hitachi LTD., Japan).

The dynamic sintering shrinkage behavior of green rectangular bars of the microporous supports was measured in a horizontal dilatometer (DIL 402C, Netzsch, Germany) with an operating temperature from room temperature to 1200°C and heating rate of $10^\circ\text{C min}^{-1}$. The percentage diametral shrinkage of the sintered samples was measured using a vernier caliper. Thermal and mass changes of CFA and the mixtures (CFA and dolomite) were evaluated by thermogravimetric analysis (TGA) and differential thermal analysis (DTA) (TGA-DTA, STA449C, Netzsch, Germany). Open porosity and bulk density were measured in distilled water in accordance with Archimedes' principle. Pore size distributions of the sintered supports were measured by a pore size distribution analyzer (PSDA-20, Nanjing Gaoqian Function Materials Co. Ltd., China).

Biaxial flexural strength (BFS) tests of sintered samples (10 samples for each composition) were conducted using a universal testing machine (AGS-X, Shimadzu Ltd., Japan) on the basis of ISO-6872. A crosshead speed of 0.1 mm min^{-1} and a preload of 5 N were utilized along with a home-made test jig with a support radius of 5.0 mm. Then BFS values were obtained from the recorded maximum loads at fracture using the following equation given by Timoshenko and Woinowsky-Krieger [40]:

$$\text{BFS (MPa)} = \frac{P}{t^2} \left\{ (1 + \nu) \left[0.485 \ln \left(\frac{a}{t} \right) + 0.52 \right] + 0.48 \right\} \quad (1)$$

where P is the maximum load (N), ν is Poisson's ratio (here $\nu = 0.31$ for anorthite-based materials), a is the radius of three-ball support circle (mm), and t is the thickness of measured sample (mm).

To confirm the reliability of biaxial flexural strength results, Weibull analysis was employed to check the discreteness by fitting linear plots of $\ln \ln [1/(1 - P_f)]$ as a function of $\ln \sigma$, where σ is the biaxial flexural strength and P_f is the fracture probability defined by the equation $P_f = i/(N + 1)$, where i is the rank in the biaxial flexural strength values from the smallest to the largest and N is the total number of samples in one set used in the Weibull analysis (here $N = 10$) [41].

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