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Synthesis and fabrication of $MgAl_2O_4$ ceramic foam via a simple, low-cost and eco-friendly method

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ABSTRACT

The $MgAl_2O_4$ nanocrystalline powder was synthesized using naturally available egg white and inexpensive metal nitrate salts. During this process, the freshly extracted egg white was mixed with metal nitrate salt and subsequently heated at 350 °C in a pit furnace. The entire dehydration of the aqueous solution thus facilitates the low-density fluffy mass. From TGDTG results, it was observed that maximum decomposition of the precursors occurred below 600 °C. Therefore, the calcination temperature of as-synthesized powder was set at 600 °C. The $MgAl_2O_4$ bulk ceramic foam was fabricated by dispersing different loading of $MgAl_2O_4$ nanoparticles in the egg white, and then coating on polyurethane sponge prior to drying and sintering at a higher temperature. The ceramic suspensions exhibit a typical shear thinning behavior, and its viscosity was found to be significantly influenced by $MgAl_2O_4$ powder content. An optimum loading of 40 wt% $MgAl_2O_4$ nanoparticles in the egg white was found to show maximum porosity up to 90%. The obtained ceramic foam has potential applications in catalysis, absorption and sensor.

1. Introduction

Magnesium aluminate ($MgAl_2O_4$) is one of the most frequently used catalyst support materials with high melting point and excellent structural stability [1,2]. The conventional solid-state reaction method is the most commonly used route for the synthesis of $MgAl_2O_4$ refractory oxide powder [3]. However, this process is time-consuming, expensive and requires multiple grinding/heating steps. Alternatively, low-temperature solution-phase-based methods, such as citrate-nitrate route [4], flash pyrolysis [5], hydrothermal [6], auto-ignition technique [7], soft chemical method [8] complex precursor approach [9], surfactant-assisted precipitation method [10] and Pluronic P123-based template route [11], have been used to synthesize $MgAl_2O_4$ powder.

In this work, the $MgAl_2O_4$ ceramic powder is prepared using metal nitrate salts as the precursor and egg white as a gelling agent. As a low-temperature synthesis technique, the egg-white-based synthesis method has been applied to prepare a broad range of ceramic powders including Al_2O_3, CeO_2, $NiFe_2O_3$, and Bi_2WO_3 [12–15]. However, synthesis of $MgAl_2O_4$ using egg white has not been attempted yet. So, in the present investigation, we will try to synthesize $MgAl_2O_4$ ceramic powder using the egg albumin. The present method has three important aspects. First, mesoporous ceramic structure can be developed without using any complicated expensive additives (i.e. Pluronic P123 [11] or N-cetyl-NNN-trimethylammonium bromide [10]). Second, egg white is readily available and easily processed in aqueous medium. Third, the processing time is less as compared to hydrothermal process [6,10].

Bulk $MgAl_2O_4$ foams are traditionally fabricated using polymer sponge replica method and direct foaming method [16,17]. Among these two, polyurethane sponge method is the simple and most widely used process to fabricate ceramic foams. It has been successfully employed to prepare a broad range of ceramic foams, including Al_2O_3, SiC, Si_3N_4, and $MgAl_2O_4$ [16–18]. In this method, a polymer sponge template is uniformly coated with a ceramic suspension. The coated sponge is dried at a low temperature and finally sintered at high temperature. Organic binders are most frequently used in this method to prepare the ceramic slurries. Unfortunately, most the available organic binders are toxic and environmentally unsafe.

In the past, many attempts have been made to substitute the organic binders by natural additives. Jamaludin et al. described using sago as a binding agent when fabricating porcelain foams [19]. Recently, carrageen, sodium alginate, Arabic gum, and carboxyl methylcellulose have been used as thickeners in replica

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technique to prepare alumina foams [20]. However, to the best of our knowledge, egg white has never been used for the fabrication of $MgAl_2O_4$ ceramic foams. Therefore, it is important to use egg white to prepare $MgAl_2O_4$ foams. Egg white is a source of natural protein and has been used as a binder in food industries. In this present work, we will try to use environmental friendly water soluble protein, egg white, as a binder and polyurethane sponge as the sacrificial template to prepare $MgAl_2O_4$ ceramic foams.

2. Experimental

2.1. $MgAl_2O_4$ powder synthesis

The egg white was separated from egg yolk and homogenized with the help of a magnetic stirrer. Then, homogenized egg white solution was mixed with deionized water in a ratio of 60:40. Subsequently, acetic acid was added dropwise to the egg white solution to maintain the pH value at 7.5. The stoichiometric amount of $Mg(NO_3)_2{\cdot}6H_2O$ and $Al(NO_3)_3{\cdot}9H_2O$ was added into the resultant solution followed by heating at 350 °C for 2 h using a pit furnace. The precursor solution turned into fluffy mass after the evaporation of water. Finally, the low-density dried gel was collected, crushed, and calcined at 600 °C for 2 h in the air atmosphere to obtain the nanocrystalline powder.

2.2. Characterization of powder

Decomposition behavior of as-prepared powder was tested using a thermal analyzer at a heating rate of 10 °C min^{-1} in the air (TA TGQ50). XRD analysis was carried out using a powder diffractometer (PANalytical High-Resolution XRD PW 3040). The morphology of the calcined powder was studied using FESEM (Merlin ZEISS). The texture properties of the calcined powder were estimated using the N_2 adsorption and desorption method (Quantachrome Autosorb-1). Fourier transform infrared spectroscopy was done using Bruker (TENSOR 27).

2.3. $MgAl_2O_4$ slurry and porous ceramics preparation

The $MgAl_2O_4$ powder calcined at 600 °C for 2 h was used below as a starting material for the fabrication of bulk ceramics foams. Ceramic suspensions were prepared using freshly extracted egg white as the binder, $MgAl_2O_4$ (40–50 wt%) as starting powder, 1-octanol (2.5 mL per 50 mL) as the froth controlling agent and ammonium polyacrylate (Darvan 821A) as dispersing agent. The above ceramic suspensions were taken in polypropylene containers, and ball milled for 8 h using zirconia balls. Commercially available sponges were submerged into the ceramic slurries and kept for a few minutes, making sure that sufficient amount of $MgAl_2O_4$ powder was absorbed within the template. Then the excess amount of slurry was removed by mechanical squeezing. The soaked foams were dried and sintered at 1600 °C for 2 h.

2.4. Characterization of $MgAl_2O_4$ slurries and foams

The rheological properties of the suspensions were studied using a parallel plate rheometer (AR 1000 TA instruments). The thermal analysis of the green foam was investigated using a thermal analyzer (NETZCH TG 209F). Morphology of the sintered foams was studied using scanning electron microscope (Vega II-LUS, TESCAN).

3. Results and discussion

In the present investigation, egg white acts as a gelling agent and inhibits the agglomeration of nanoparticles. The long

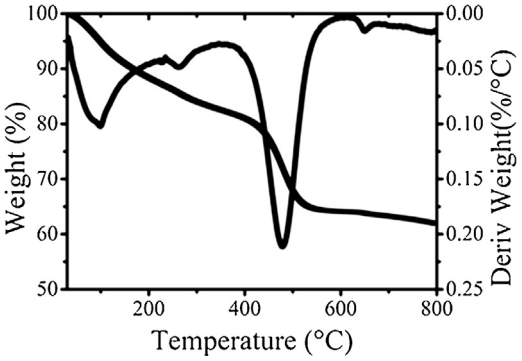

Fig. 1. TG–DTG curves of thermal decomposition of $MgAl_2O_4$ as-synthesized powder.

polymer chains present in egg white entrap the metal ions [21]. Dispersed metal ions containing albumin egg suspension turned into nanocrystalline powder on heating. Fig. 1 shows the TG–DTG curves of the as-synthesized powder. The initial peak appears at the low temperature below 200 °C, due to the release of the physically adsorbed water. The prominent peak at about 480 °C belongs to the decomposition of nitrates, burning of organic species present in the precursor salts and egg white. Therefore, a sharp decrease in weight (18.9%) was observed in the TG curve, which is due to the repetitive combustion reaction during the decomposition process. A small peak at 650 °C in DTG curve can be attributed to the burning of residual organic. From TG–DTG results, it may be concurred that the majority of the decomposition processes were completed below 600 °C. Hence, the calcination temperature and time of as-synthesized powder were taken as 600 °C and 2 h, respectively.

Fig. 2a shows the XRD pattern of the powder (calcined at 600 °C). The diffraction peaks suggest that the calcined powder is polycrystalline in nature. The diffraction peaks can be indexed according to the JCPDS number (04-008-1061) of cubic spinel $MgAl_2O_4$ structure [22]. No impurity peak observed in the XRD pattern indicates the phase purity of powder. The particle size of the calcined powder was calculated using Scherrer's formula. Here, the crystallite size of the powder was determined by considering the characteristic (311) peak of spinel phase. The crystallite size of the powder was found to be 15 nm.

The infrared spectrum of $MgAl_2O_4$ powder, which is calcinated at 600 °C, is shown in Fig. 2b. From the IR spectra, a broad peak at

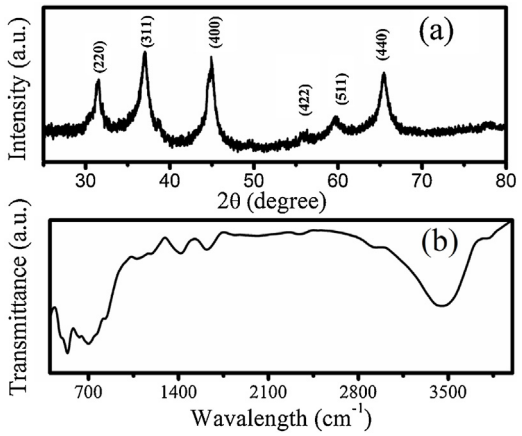

Fig. 2. (a) X-ray diffraction patterns of calcined $MgAl_2O_4$ powder and (b) its corresponding FTIR spectra.