



## Research article

# Nano-alumina powders/ceramics derived from aluminum foil waste at low temperature for various industrial applications



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## ABSTRACT

In this work, nanoscale single crystalline  $\gamma$ - and  $\alpha$ -alumina powders have been successfully prepared from aluminum foil waste precursor via co-precipitation method using  $\text{NH}_4\text{OH}$  as a precipitant. The obtained gel after co-precipitation treatment, was calcined at different temperatures (500, 700, 900, 1050, 1100, 1300 and 1500 °C) and the products were characterized by XRD, FTIR and HRTEM. The results revealed that nano- $\gamma$ - $\text{Al}_2\text{O}_3$  was fully transformed to nanometer-sized  $\alpha$ - $\text{Al}_2\text{O}_3$  (36–200 nm) after annealing at temperatures as low as 1100 °C. The thermally preheated powder at 500 °C was further pressed under 95 MPa by the uniaxial press and the obtained bodies were found to have 98.82% of the theoretical density, 1.18% porosity and 708 MPa compressive strength, when sintered at temperatures as low as 1600 °C without using any sintering aid. These excellent results proved that this work will contribute to finding a commercial source for preparing sub 100 nm  $\alpha$ -alumina through the secondary resources management and even more so to synthesizing strong  $\alpha$ - $\text{Al}_2\text{O}_3$  bodies which are promising in terms of their structure and compression. The  $\alpha$ - $\text{Al}_2\text{O}_3$  bodies synthesized by the present work could be used as a feedstock for fabrication of various kinds of functional and structural materials that are extensively used in high tech.

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## 1. Introduction

Corundum ( $\alpha$ -alumina) is the most thermodynamically stable phases of alumina that forms above 1200 °C as a result of thermal conversion of other alumina phases ( $\beta$ ,  $\gamma$ ,  $\theta$ , and  $\delta$ ) (Sharma et al., 2003). In recent years, nano-corundum has recently attracted considerable attention in fabrication of various kinds of functional and structural nanoceramics that are extensively used in high tech due to its superior characteristics such as high elastic modulus, good electrical resistivity, perfect strength and toughness, superb thermal and chemical stability as well as excellent catalytic and dielectric properties (Hariharan et al., 2014; Varghese et al., 2014). Sadly, despite having huge potential, there is no any commercial sources for preparing sub 100 nm  $\alpha$ -alumina until now (Laine et al., 2006).

Numerous methods have been employed to synthesize  $\alpha$ -alumina such as mechanical milling (Panchula and Ying, 1997; Wu

et al., 1991), vapor phase reaction (Chou and Nieh, 1991; Varma et al., 1994), sol-gel (Dilsiz and Akoval, 2002; Ibrahim and Abu-Ayana, 2008; Jiang et al., 2005; Mirjalili et al., 2010; Nan et al., 2001; Varghese et al., 2014; Zeng et al., 1998), co-precipitation (Wu et al., 1991), hydrothermal (Gao-feng et al., 2008), thermal decomposition (Bhattacharya et al., 2004; Ma et al., 2001; Park et al., 2002) and combustion synthesis (Bhaduri et al., 1996; Geik et al., 2007; Toniolo et al., 2005). To the authors' knowledge, it is difficult to process sub 100 nm  $\alpha$ -aluminapowder using all these aforementioned methods due to the high transformation temperature of transitional alumina phases into  $\alpha$ -alumina that leads to a particle size coarsening and surface area decreasing (Sharma et al., 2003). Research has been continued in regard to control of the particle size of  $\alpha$ -aluminaby means of reducing the temperature of phase transformation (Wu et al., 1991). In this context, dopants, surface modifiers and  $\alpha$ -aluminaseeds have been employed during the sol-gel process so that temperature for formation of  $\alpha$ -aluminacan be lowered from 1200 to 1000 °C and nano- $\alpha$ -alumina could be obtained. However, surface modifiers are expensive organic compounds that cause a higher harm to health and the environment by emitting large amounts of corrosive gases during their

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calcinations (Thiruchitrabalam et al., 2004). Moreover, dopants are impurities that cause a higher level of pollution to the product. Also, the sol-gel on the other hand is tedious and expensive because of the high cost, toxicity and flammability of the starting chemicals and the moisture sensitive nature of alkoxides. Therefore, development of cost-effective green routes for the synthesis of nano- $\alpha$ -alumina precursors at relatively low temperature using inexpensive feedstock is now the area of interest.

More interestingly and surprisingly, recycling of municipal solid waste (MSW) is now recognized as the most environmentally sound strategy that generates a host of benefits at every level: environmental, financial and social through reducing greenhouse gas emissions and turning materials that would otherwise become waste into a valuable resource.

Accordingly, in the present work, we report the green synthesis of a commercial nano- $\alpha$ -alumina powder at relatively low temperature, 1100 °C, by the simple and cost-effective co-precipitation method using  $\text{NH}_4\text{OH}$  as precipitant and aluminum foil waste as raw material, in the absence of any seeding material or surface modifier. Processing of nano- $\alpha$ -alumina from aluminum foil waste occurs at low temperature 1100 °C and it hasn't been reported elsewhere. Here we also report the influence of sintering at 1600 °C for 3 h on the densification, morphological and mechanical properties of the alumina bodies processed from the calcined powder at 500 °C by the uniaxial press under 95 MPa. The results clearly show an improvement in the densification and compression characteristics of the obtained  $\alpha$ -alumina ceramics. This enhancement in the physico-mechanical properties of the synthesized  $\alpha$ -alumina bodies would open the door wide for their usage in various applications such as refractory materials, abrasives, electronic packaging materials, composites and corrosion resistant ceramics.

## 2. Materials and methods

### 2.1. Experimental procedures

Aluminum foil waste emerging from food packaging in take away restaurants was collected and utilized as a feedstock for nano- $\alpha$ - $\text{Al}_2\text{O}_3$  synthesis using precipitation method. In a typical procedure,  $\text{Al}(\text{NO}_3)_3$  stock solution was foremost prepared by dissolving 27 g aluminum foil in 300 ml diluted aqua regia (1 vol 20%  $\text{HNO}_3$ : 3 vol 20%  $\text{HCl}$ ). A requisite amount of the industrial grade  $\text{NH}_3 \cdot \text{H}_2\text{O}$  solution was added drop wise to the vigorously stirred stock solution until its pH reached 10 and a gelatinous precipitate of  $\text{Al}(\text{OH})_3$  was formed. After precipitation reaction, the obtained precipitate was stirred mechanically for 1 h to achieve good homogeneity. The resulting homogenous suspension was filtrated, washed with distilled water, dried overnight at 105 °C and thereafter calcined at temperatures between 500 and 1500 °C in a muffle furnace at a constant heating rate of 5 °C/min and maintained at this temperature for 2 h. Finally, fluffy powders of nano- $\alpha$ - $\text{Al}_2\text{O}_3$  were obtained. The resultant powders were ground with a mortar and then characterized by XRD, FTIR and HRTEM. The fired powder at 500 °C was thoroughly mixed with 0.4 wt% tri ammonium citrate and 40 wt% distilled water in the planetary ball mill for 1 h. The dried powder mixture at 80 °C for 6 h in a controlled oven was uni-axially pressed at 95 MPa to form cylindrical shaped objects of 1.28 cm diameter. The obtained discs were dried, then sintered at 1600 °C for 3 h in a tube furnace. The densification properties, microstructure and compressive strength of the sintered specimens have been investigated. A flow chart of the process was given in Fig. 1.

### 2.2. Characterization and testing

The mineral composition of the fired powders was identified by

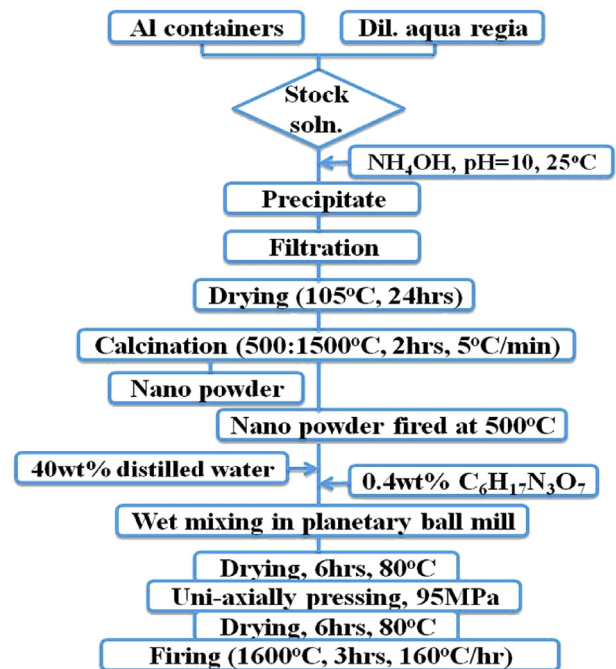


Fig. 1. Flow chart for synthesizing powder.

a Bruker advanced x-ray diffractometer model D8 Kristalloflex (Ni-filtered  $\text{CuK}_\alpha$  radiation;  $\lambda = 1.544 \text{ \AA}$ ). The crystallite size of the produced nano- $\alpha$ -alumina powders was determined from the most intense peak (104) using the Debye-Scherrer formula:  $d_{\text{RX}} = K\lambda/\beta \cos\theta$ , where  $d_{\text{RX}}$  is the crystallite size,  $K$  is a correction factor related to crystallite shape and it's equals 0.9,  $\lambda = 1.544 \text{ \AA}$  and it's the wavelength of Cu X-ray,  $\beta$  is the full width at half maximum (FWHM) of the most intense diffraction peak (104) of nano- $\alpha$ -alumina, and  $\theta$  is the Bragg angle.

Fourier transform infrared spectra (FTIR) spectroscopy (Model, Jasco-6300 type A, Japan spectrometer) was used at room temperature in the range of 400–4000  $\text{cm}^{-1}$  with a resolution of 4  $\text{cm}^{-1}$  to identify functional groups in the fired powders.

The microstructure of the sintered discs was investigated using backscattered electron (BSE) in the field emission scanning electron microscopy (FESEM: Quanta FEG 250, Holland), however the texture and crystallinity of the produced nano- $\alpha$ -alumina powders were examined by high resolution transmission electron microscope (HRTEM: JEM- 2100, Japan). TEM samples were prepared by dispersing the powders in ethanol. Ultrasonic oscillation for 30 min was introduced to decrease the aggregation followed by placing a drop of the suspension on holey carbon film supported on copper grids.

Densification parameters in term of apparent porosity and relative density of sintered materials were determined by Archimedes immersion technique in ethanol using vacuum pressure according to ASTM C 830-00, 2000.

The cold crushing strength of the sintered objects was also determined at a rate of 1.3 mm/min by compression universal testing machine (Shimadzu, UH-F 1000 KN, Japan).

## 3. Results and discussion

### 3.1. Aluminum foil waste analysis

At first, the foil waste was elementally analyzed using the Spectro analysis instrument and the results are shown in Table 1.

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