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Short communication

# A novel microwave combustion approach for single step synthesis of α-$Al_2O_3$ nanopowders

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**Abstract**

A novel method for synthesis of nano-sized α-$Al_2O_3$ particles in a single step using microwave is being reported for the first time. The sol of aluminum nitrate with urea mixed in the stoichiometric ratios in accordance with jet propellant chemistry, when combusted in a microwave oven gave fine single phase α-$Al_2O_3$ nanoparticles. The resultant oxide powder was characterized by TGA (Thermo-Gravimetric Analysis), FTIR (Fourier Transform Infra-Red Spectroscopy), XRD (X-ray Diffraction) and TEM (Transmission Electron Microscopy). The XRD analysis of the microwave combusted powder showed complete formation single phase α-$Al_2O_3$ without contamination of other phases of alumina. In comparison to the well known furnace combustion method for the direct synthesis of α-$Al_2O_3$, microwave combustion gave finer particles with very small agglomerate size as revealed by TEM analysis.


*Keywords:* Single phase α-$Al_2O_3$ nanopowder; Direct synthesis; Microwave combustion; Deagglomeration

## 1. Introduction

The nanometric α-$Al_2O_3$ powders are advanced materials for their varied applications in microelectronics, refractories, catalysis and technical ceramics [1–3]. Polycrystalline transparent alumina has optical applications since 1961 [4]. Single phase α-$Al_2O_3$ nanopowders are also important component for solid state fabrication of YAG (yttrium aluminum garnet) transparent laser ceramics [5,6]. There are various techniques used for the synthesis of α-$Al_2O_3$ nanopowders such as reverse micelle [7], sol–gel processing [8], flame spray pyrolysis [9] which require calcinations at 1000–1100 °C to obtain completely phase pure α-$Al_2O_3$. A low-temperature combustion synthesis (LCS) of α-$Al_2O_3$ without calcination has been reported in which a mixture of aluminum nitrate and urea was treated in a furnace at 700 °C resulting into the formation of α-$Al_2O_3$ [10,11]. But this technique also requires critical handling precautions while putting the mixture inside the furnace which is already pre-heated at 700 °C. In the recent trend of research in materials synthesis microwave energy is being increasingly used by many researchers for synthesis of phase pure oxide materials [12–15].

We are reporting a rapid and novel single step synthesis of α-$Al_2O_3$ by microwave combustion method for the first time. Since urea has been proven to be the best fuel for combustion of aluminum nitrate [10,11,16,17] aluminum nitrate and urea were used as the starting materials.

## 2. Experimental

$Al(NO_3)_3{\cdot}9H_2O$ (98–102%, Alfa Aesar) and urea (99–100.5%, Merck) were weighed to obtain their molar stoichiometric ratio of 1:2.5 to fulfil the equivalence theory where ratio of oxidizing valency to reducing valency is equal to unity [18]. The mixture was dissolved in D/W to make a clear solution. One part of the sol was treated in a domestic microwave working at 900 W, 2.45 GHz for 3–5 min and another part was treated in a muffle furnace [Make Nebertherm, Model LH 60/13] pre-heated at 700 °C for 5 min using alumina crucible as described in a flow chart detailed below.

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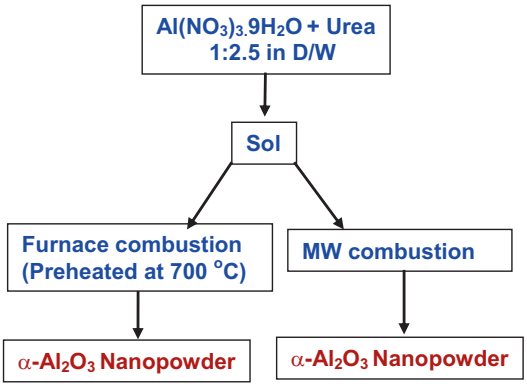


The combusted powders were characterized by TGA (Thermo Gravimetric Analysis) on Perkin Elmer Diamond Simultaneous TGA/DTA in air at the heating rate of 10 °C/min from room temperature to 1000 °C. FTIR (Fourier Transform Infra-Red Spectroscopy) of powders was carried out on Bruker, Vector 22 Spectrophotometer. Phase purity and primary particle size were monitored by XRD (X-Ray Diffraction) on X'PERT PRO PANalytical PW 3050/60 Standard Resolution Goniometer, $2\theta$ range from 15° to 75°. The crystallite size was determined by using Scherrer's equation [19]:

$$t = \frac{0.9\lambda}{(\beta^2_{\text{sample}} - \beta^2_{\text{inst}})^{1/2} \cos\theta}$$

where $t$ is the crystallite diameter, $\lambda = 1.54056$ Å, $\theta$ is the diffraction angle, $\beta_{\text{sample}}$ is the FWHM of the diffraction peak and $\beta_{\text{inst}}$ is characteristic of the instrument. TEM (Transmission Electron Microscopy) was done on FEI Philips Morgagni-268 by preparing powder samples on copper grids.

## 3. Results and discussion

TGA (Fig. 1) of the microwave combusted powder showed only ~5% total wt loss up to 1000 °C, which indicates that maximum gaseous products have been evolved with the formation of final product during combustion in microwave. This is in good agreement with only 3% total wt loss for preheated furnace combusted product which results in formation of phase pure α-$Al_2O_3$ nanopowders [10,11].

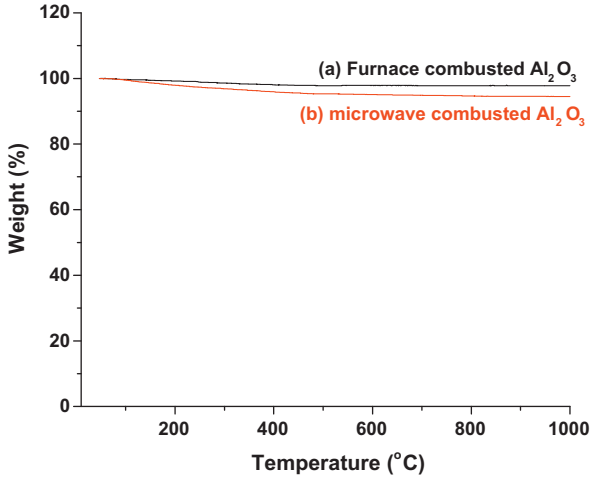


Fig. 1. TGA of powders combusted in (a) furnace at 700 °C and (b) microwave oven.

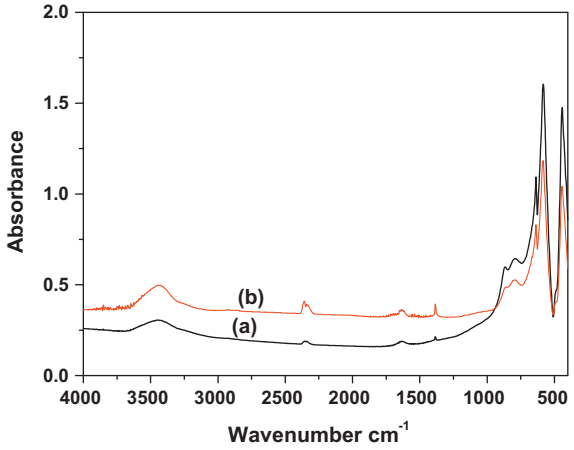


Fig. 2. FTIR of powders combusted in (a) microwave oven and (b) furnace at 700 °C.

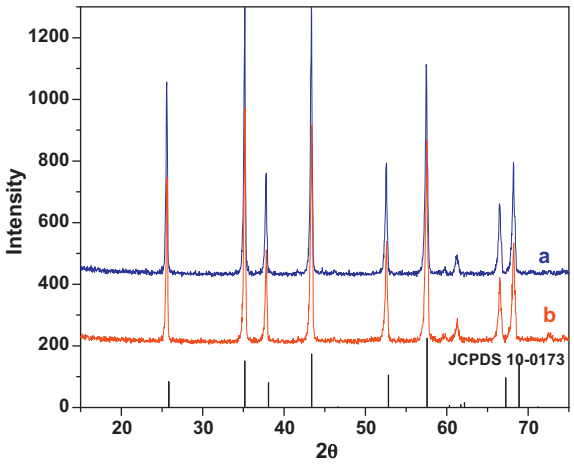


Fig. 3. XRD showing phase purity in powders combusted in (a) microwave oven and (b) furnace at 700 °C.

FTIR (Fig. 2) of the microwave combusted powder showed characteristic peaks of O–Al–O bonds [10,11] at 865, 784, 635, 581 and 440 $cm^{-1}$ indicating that the combustion in the microwave oven resulted into the formation of α-$Al_2O_3$ directly as also observed for preheated furnace combustion product.

Peak indexing was done using least square method [20] of the XRD peaks (Fig. 3) of both microwaves as well as furnace combusted powders using JCPDS 10-0173. It confirmed that complete formation of single phase α-$Al_2O_3$ took place in both the cases. However particle size using Scherrer's equation was found to be 20 nm for microwave combusted powder and 40 nm for furnace combusted powder.

TEM (Fig. 4) of the microwave combusted powder showed very fine particles in the range of 18–20 nm with close to spherical and uniform morphology. Further the particles form

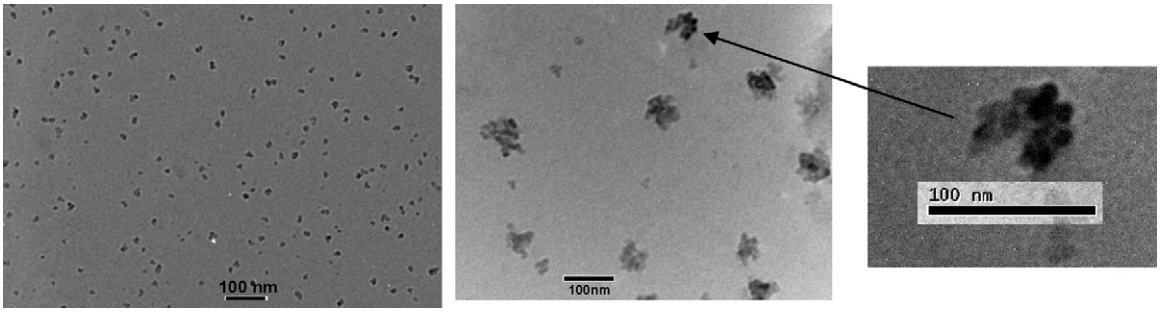


Fig. 4. TEM showing $\alpha$-$Al_2O_3$ nanopowder prepared in microwave.

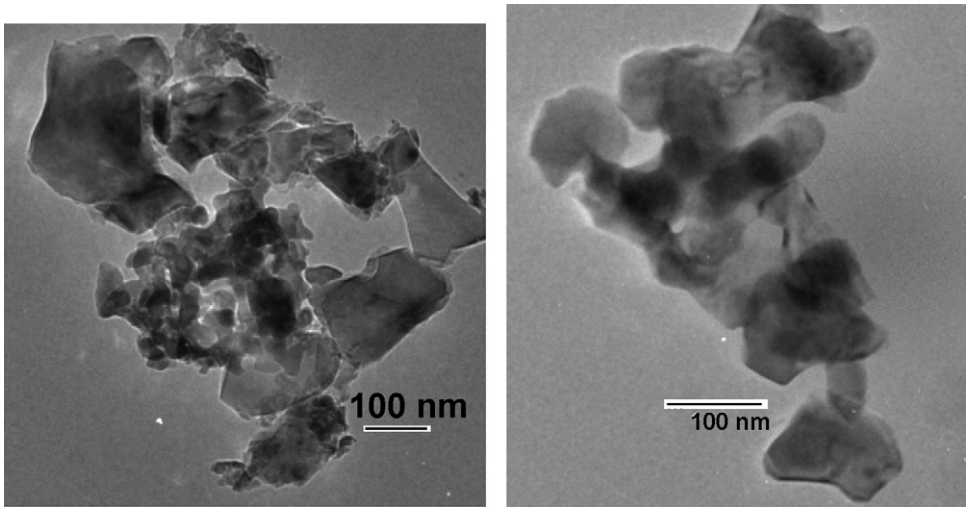


Fig. 5. TEM showing $\alpha$-$Al_2O_3$ nanopowder prepared in furnace at 700 °C.

very small agglomerates of ∼100 nm whereas particles obtained by LCS method were in the range of 50–90 nm with polyhedral morphology and quite big agglomerates (Fig. 5). Further discrepancy in particle sizes are seen for particles with LCS method because a large amount of heat is produced due to sudden combustion of urea nitrate mixture [21] in conventional LCS method. However, local uniform heating of reaction mixture takes place in microwave compared to inhomogeneous heating in preheated furnace which results in finer, uniform and smaller agglomerates formation.

## 4. Conclusions

Microwave gel combustion resulted in formation of single phase $\alpha$-$Al_2O_3$ nanopowders in a single step. Fine particles with size range 18–20 nm and close to spherical morphology were obtained by microwave gel combustion compared to large particles of 50–90 nm size range and polyhedral morphology obtained from the well known LCS method for direct synthesis of $\alpha$-$Al_2O_3$. Thus microwave gel combustion method in addition to its energy and time efficiency, is easy for handling and scalable compared to LCS method for synthesis of single phase $\alpha$-$Al_2O_3$ nanopowders.

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