Influence of the external heating type in the morphological and structural characteristics of alumina powder prepared by combustion reaction

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Abstract: The aim of this work is to evaluate the influence of the external heating in the morphological and structural characteristics of the alumina powder prepared by combustion reaction. It was evaluated different types of external heating: muffle oven, microwave oven and ceramic plate with electrical spiral resistance. The powders were prepared according to the propellants and explosives theory, using urea in the stoichiometric proportion ($\Phi e = 1$). During the synthesis parameters such as flame combustion time and temperature were measured. The structural and morphological characteristics of the powders were evaluate by XRD, particle size distribution, SEM and nitrogen adsorption (BET). The results showed the production of α-alumina as unique phase and formed by agglomerates with irregular plate shape of thin particles for all studied conditions. The powders prepared by electrical oven presented small particle size, with narrow agglomerates size distribution.

Introduction

Recently, a great deal of research effort is in synthesizing nanocrystalline ceramics powders, including alumina. The interest in the synthesis of nanocrystalline materials is from the belief that the mechanical properties are greatly affected by the presence of extremely fine crystallite size, typically in the order of 100 nanometers or less [1].

Alumina powders are extensively used in the production of ceramics, abrasives, medications, membranes, chromatographic column support materials, adsorbents and catalysts [2].

Various chemical methods such as, sol–gel [3,4], hydrothermal [5], precipitation [6] and combustion synthesis [7,8] have been employed to synthesize α-Al_2O_3 powders. These methods are used to produce powder with high purity, chemical homogeneity and controlled particle size. The combustion method, which is characterized by its an inexpensive process and significant savings in time and energy consumption compared to some other wet methods, also offers the advantage of high chemical homogeneity, small crystallite size and purity [9].

Combustion synthesis is particularly an easy, safe and rapid production process wherein the main advantages are energy and time savings. This method is versatile to

synthesize a broad range of particle sizes, including alumina nano-sized powders as related by Mimani and Patil [10].

The final characteristics of the powder depends on the synthesis conditions, that is, type and amount of fuel (citric acid, aniline, carbohydrazide, glycine, urea, etc.), container type (beaker, silica vitreous crucible, porcelane crucible, stainless steel crucible, etc.) and external heating source (muffle oven, hot plate, heating mantle, microwave oven, etc.).

Therefore, the aim of this work is to make the morphological and structural characterization of alumina powders prepared by combustion reaction using different external heating systems and urea as fuel.

Experimental

Al_2O_3 samples were produced using the following materials: aluminum nitrate and urea, with purity of 98% . The proportion of each reagent was calculated based on the theory of propellants and explosives [11]. The batches were placed in a vitreous silica basin, homogenized and submmited to a different heating conditions, i) ceramic plate with electrical spiral resistance (maximum temperature close to 600^{o}C); ii) microwave oven, with previous heating in ceramic plate until solubilization and after submmited to a microwave, Eletrolux model, pre-setting in output power of 500 W for a period of 5 minutes; iii) muffle oven. The samples were designated as: MI, MU and RE, for the powder prepared in microwave oven, mufle oven and ceramic plate, respectively.

These sample were characterized by X-ray diffraction (Shimadzu diffractometer, model LAB 6000, CuKα radiation), and scanning rate of 2◦ 2θ/min, in a 2θ range of 20–80°). The crystallite size was calculated from the basal reflection peaks using Scherrer's equation [12]. Surface area was measured in N_2 adsorption using an NOVA 3200 particle size analyzer (Quantachrome) apparatus. The average particle size was calculated from BET data using the equation $D_{BET} = 6/(D_t S_{BET})$ [13], where D_{BET} = equivalent spherical diameter (nm); D_t = theoretical density (g/cm3) and S_{BET} = surface area (m^2/g). The agglomerates size distribution was analyzed by a sedimentation method (Particle Size Distribution Analyzer, Cilas 1064L). The morphological characteristics were analyzed by SEM (Philips, XL30 FEG).

Results

Fig. 1 shows the X-ray patterns of the powders MI, MU and RE prepared by combustion reaction.

The X-ray diffraction patterns of powders produced by the different heating condition showed the formation of single phase, crystalline α-alumina (α-Al_2O_3) [JCPDF 89-3072] for all samples.

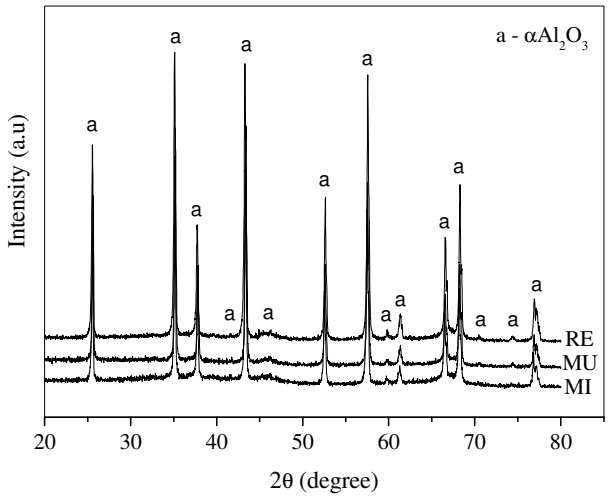


Figure 1- X-ray powder diffraction patterns of the α-Al_2O_3 powders prepared by combustion reaction.

Table 1 shows the powders characteristics obtained by combustion reaction for the aforementioned compositions (MI, MU and RE).

Table 1 – Characteristics of the α-Al_2O_3 powders prepared by combustion reaction

Composition	MI	MU	RE
Crystallite size (nm)*	64	63	57
Medium agglomerated size d50% [μm]	12.51	10.31	10.79
Specific surface area (BET) [m^2/g]	52	61	35
Particle size (D_{BET})** [nm]	29	25	43

*calculated by Sherrer equation [12] **Calculated from specific surface area (BET) Theoretical density = 3.98 g/cm^3

From Table 1, it can be observed that the heating conditions affect the strucutural and morphological characteristics of the alumina powder. The microwave oven present crystallite size slightly higher when compared with the powder produced by ceramic plate and muffle oven, indicating that the way of heating in the macrowave oven provide higher combustion temperature and give great growth of crystals.

Kiminami and coworkers [14] synthesized alumina powders in microwaves oven and obtained specific surface area of 25.5 m^2/g and particle size of 0.06 μm.

Fig. 2 shows the values of agglomerated size as a function of cumulative mass of powders produced by different heating conditions.

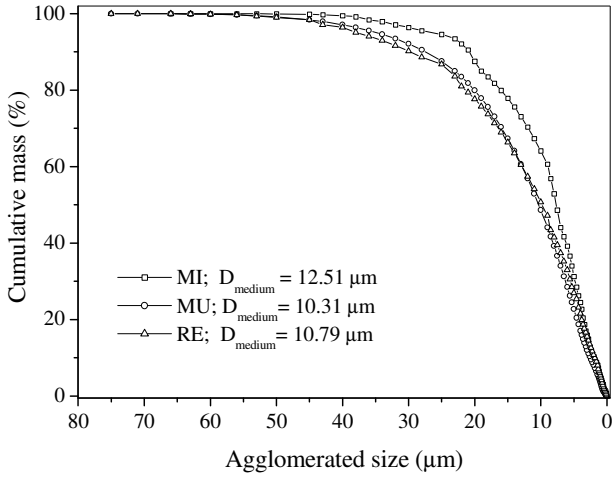
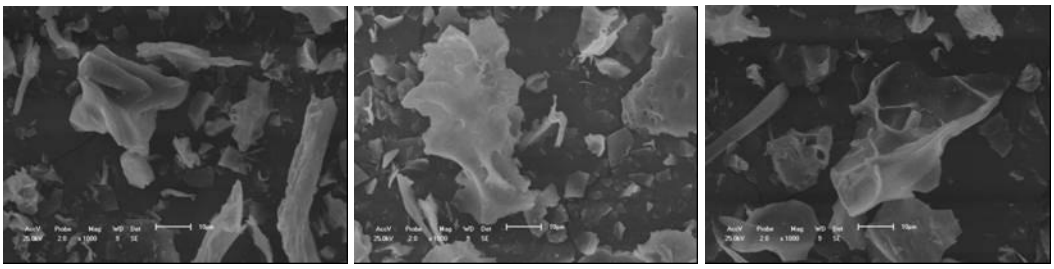


Figure 2 – Agglomerates size distribution of the α-Al_2O_3 powders prepared by combustion reaction**.**

The powders prepared by the MI, MU and RE compositions resulted in agglomerates with average diameter of 12.51, 10.31 and 10.79 μm respectively. According to Lange et al [15] agglomerates with size greater than 5μm indicate hard characteristics, however, in this work the size of the agglomerates are greater than 5μm and they are ease to de-agglomerate, presenting Van der Waals forces and "soft" agglomerates.

Fig. 3 shows morphology aspects of the agglomerated powders obtained from SEM of MI, MU and RE compositions.



(a) (b) (c)

Figure 3 - Micrographs obtained by SEM showing the morphology of the α-Al_2O_3 powders prepared by combustion reaction: (a) MI (microwave oven); (b) MU (mufle oven) and (c) RE (ceramic plate).

The micrographs show powders formed by agglomerates with sharp and irregular shape with different size and some porous plates. Also it can be observed agglomerates easily broken, indicating the existence of Van der Waals forces and the formation of soft agglomerates of fine particles.

Conclusion

According to the results from this study it was possible to conclude that the combustion reaction method with different heating systems is viable and recommended to produce fine and crystalline particles in a short period of time. The heating system have influency in the structural and morphological characteristics of the powders. The

powders prepared in the microwave oven present greatest crystallite size and agglomerates, due to the high temperature reached during the combustion reaction. The sample MU showed the highest surface area when compared with MI and RE. The micrographs show the formation of agglomerates with sharp irregular, with different size and some porous plates.

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