

# Ultrasonic cavitation for obtainment of nanometric sized particles

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**Abstract:** This project aims to determine the possibility of obtaining nanometric size particles of aluminium oxide ( $\text{Al}_2\text{O}_3$ ) and titanium dioxide ( $\text{TiO}_2$ ) from commercial micron-sized powders, through the physical principle of ultrasonic cavitation, in order to be used as supply material in coatings made through a process of thermal spray by flame. The tests are performed on a Hielscher UIP 1000hd Ultrasonics equipment, in a 20 micron wave amplitude and in times of 6, 8, 12, 18 and 24 hours. The determination of the particle size is done through image processing using ImageJ software, obtained by the technique of scanning electron microscopy (SEM); while the elemental composition of the processed samples is analyzed through the technique of energy dispersing spectroscopy (EDS). The results show that  $\text{Al}_2\text{O}_3$  and  $\text{TiO}_2$  have a reduction behaviour of the particles size after being subjected to ultrasonic cavitation, however is only reached the nanometric size in the  $\text{TiO}_2$  samples.

## 1. Introduction

In recent years have been developed a series of methods for obtaining of nanoparticles, which are usually grouped into two categories, the approaches "from top to bottom" which consists of the division of solid mass into smaller portions [1]. Recently, it has been reported results of the use of a method based on the use of ultrasound for the reduction of size from micrometer-scale particles to Nanometric dimensions, mainly in elements such as carbon, gallium, zinc, gold, etc. [2-4]. The principle searches through the application of ultrasonic waves in a medium, usually water, generate a phenomenon known as cavitation, which can locally cause temperatures of up to 5000K, pressures up to 2000MPa and speeds up to 280m/s.

The size of the particles used on the accomplishment of coatings, significantly influences the mechanical properties related to its behaviour to the wear phenomenon; it has been shown that the hardness and the yield strength of a material increases with decreasing grain size of the crystal until a critical value of 20nm; from this value, these properties decrease with decreasing grain size [5].

This project wants to apply the technique of ultrasonic cavitation for the processing of  $\text{Al}_2\text{O}_3$  particles and  $\text{TiO}_2$  micron-sized, contained in water, to determine the behaviour of size depending on the time of exposure to the phenomenon and determine whether it is possible or not to reach nanometric sizes of these oxides from this principle, for its use in obtaining coatings through thermal spray by flame process.

## 2. Conceptual framework

### 2.1. Synthesis of nanoparticles

Various physical methods are currently used for synthesis and commercial production of nanostructured materials. The first and most widely used technique involves the synthesis of single-phase metals and ceramic oxides by the technique of inert gas evaporation [6]; the cathodic pulverization is another



technique used to produce groupings of nanostructured materials, as well as a variety of thin layers. The third physical method involves the generation of nanostructured materials through a severe mechanical deformation [7].

Chemistry has also played an important role in the development of new materials with innovative and technologically important properties [8]. The advantage of chemical synthesis is its versatility in designing and synthesizing new materials that can be refined in the final product. The main advantage offered by chemical processes on the other methods is its good chemical homogeneity, since it enables the mixture at the molecular level [9]. The general procedure involves reactions in aqueous or non-aqueous solutions containing soluble or suspended salts. Once the solution is super saturated with product, the precipitate is formed both by homogeneous or heterogeneous nucleation [10]. Nanostructured materials are also prepared by chemical vapour deposition (CVD) or chemical vapour condensation (CVC) [11]. CVC method is used to produce a variety of powders, metal fibres and compounds, whereas the CVD method has been employed in the synthesis of ceramic metals, intermetallic and composites [12].

## 2.2. Ultrasonic cavitation

Ultrasound has evolved in the last ten years and has become a processing technology completely commercial of high reliability, scalability, and energy efficiency. Ultrasonic processor generates cavitation, ultrasonic basic effect that allows new results on physical and chemical processes.

While low intensity or high frequency ultrasound is mainly used for the analysis of non-destructive testing and imaging; high intensity ultrasound is used for the treatment of liquids, in other words what is meant to achieve is shearing, in order to cause cracks in the surface of the particles.

Recent researches have used ultrasonic cavitation method for obtaining nanometric size particles of metals of low melting point and alloys [2]; ferrite of zinc [3] and gallium, among others, showing promising results that allow to expect positive results with other materials.

## 3. Methodology

### 3.1. Preparation of $Al_2O_3$ and $TiO_2$ samples

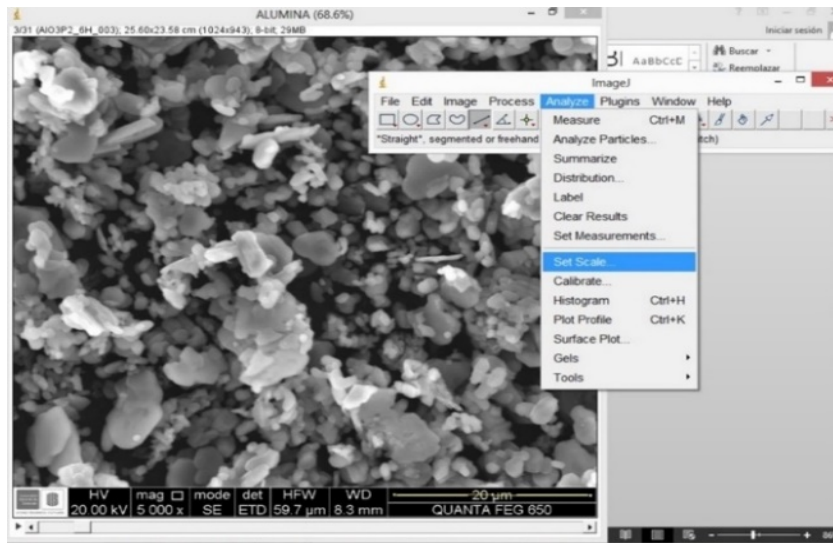
The weight of the material used for the tests was 5 grams, which are diluted in 500mL of sterile water; the homogenization of the mixture was made through a process of stirring with a glass rod. Finally the sample enters the flow cell of the system UIP 1000hd used.

### 3.2. Testing

The equipment used for testing was a processor of ultrasound UIP 100hd, which gives a power of 1000W at a frequency of 20 kHz; amplitude used was 20 microns, which is equivalent to 80% of the maximum allowed by the equipment. The mixing process was carried out continuously and processing time of them was 6, 8, 12, 18 and 24 hours. After the test time, the mixture is brought to an electric oven, which is set at a constant temperature of 250°C for a period of 30 minutes, causing evaporation of the water.

### 3.3. Morphological characterization of processed materials

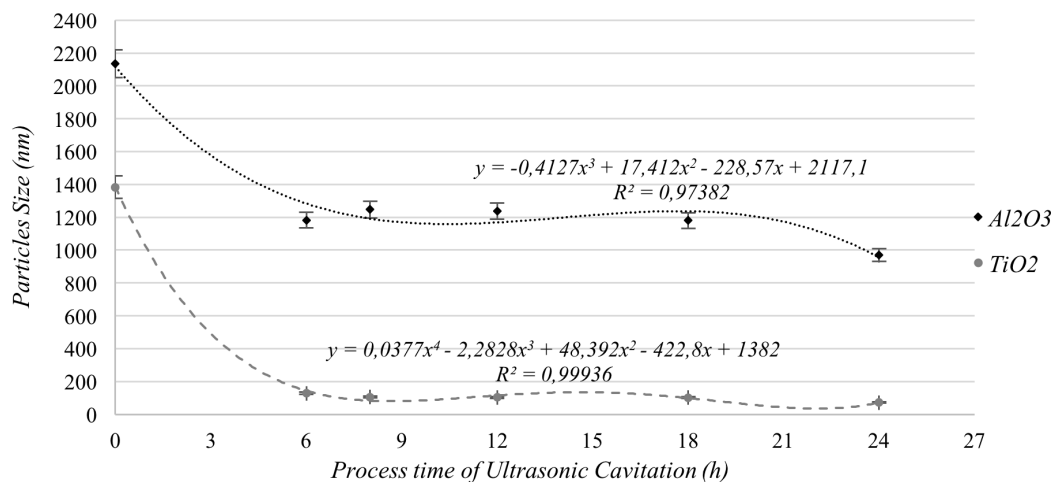
In order to obtain a value for the size of the particles with a reliability greater than 95%, a statistician analysis is made with a pre-sample of 10 magnitudes, for both the  $Al_2O_3$  as for the  $TiO_2$ . From images obtained of SEM and a public domain digital image processing software called ImageJ, it was proceeded to dimension 10 particles randomly, always taking the lower dimension of them. The procedure consisted of the following steps: load the image into ImageJ; scale the image in order to obtain a constant dimension during all performed measurements, as shown in Figure 1; perform the conversion of the value of the indicated scale on the label of the image, to obtain its corresponding value in nanometers; check the scaling and last perform the measurement of the size of the particles, through dimensioning of the lower section of the same.



**Figure 1.** Image of scanning electron microscopy of  $\text{Al}_2\text{O}_3$  used to obtain the size of the particles.

#### 4. Results

The total number of required measurements to achieve a reliability of 95%, with a 5% error rate was 115 for the alumina and 100 for titanium dioxide; the results of the average values for each material in each of the defined procedures, are listed in Figure 2.



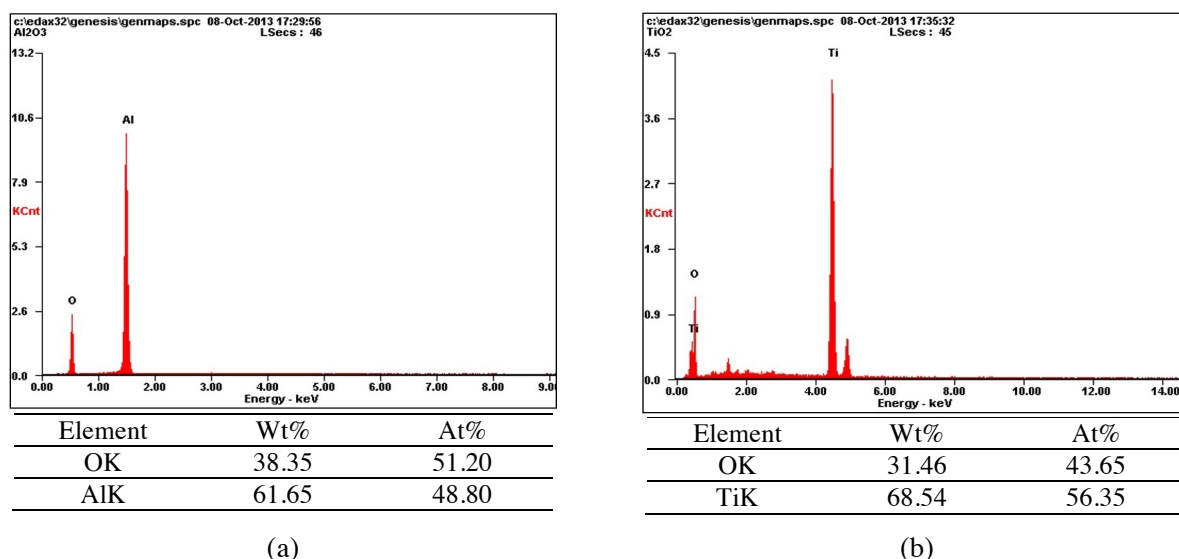
**Figure 2.** Behaviour of the particle size of  $\text{Al}_2\text{O}_3$  and  $\text{TiO}_2$  in time function of process.

The results obtained of the grinding process through ultrasonic cavitation embodied in Figure 2, show a concordance with those reported by Vargas-Hernandez et al [13], where is evidence of an effect of acoustic cavitation in the grinding of particles. The curve of the behaviour of the particle size of  $\text{Al}_2\text{O}_3$  depending on the time of ultrasonic cavitation process shows a 4 degree polynomial relationship. There is evidence of an approximate decrease of 45% of the size in the first 6 hours of process; in processing times of 8, 12, 18 and 24 hours, no significant change is seen in the average particle size. The average size of the particles obtained by ultrasonic cavitation process is 969.64nm, which represents a decrease of the 55.4% of the initial value; however nanometric sizes (< 100 nm) are not reached.

The curve of the behaviour of  $\text{TiO}_2$  particles size on the basis of the time of ultrasonic cavitation process also shows a 3 degree polynomial relationship. There is evidence of an approximate decrease of 92.76% of the size in the first 6 hours of process; processing time of 8, 12 and 18 hours does not show a significant difference with the results obtained in a time of 6 hours; there is again a tendency to reduce

size for 24 hour processing time. The average size of the smallest particles of  $\text{TiO}_2$  obtained through the process of ultrasonic cavitation is 70.8nm, which represents a decrease of the 94.9% of the initial value.

**4.1. Chemical characterization of the materials processed through the process of ultrasonic cavitation**  
Processed powders chemical analysis was carried out through the obtained values of microanalysis by energy dispersion spectroscopy of x-rays (EDS), which are listed in Figure 3.



**Figure 3.** Spectrum obtained in EDS analysis. (a) Alumina powder; (b) titanium dioxide powder.

The percentage of error that was obtained between the calculated theoretical atomic weight and the experimental retrieved by EDS analysis, for  $\text{Al}_2\text{O}_3$  and  $\text{TiO}_2$  was 3.3% and 9% respectively; which are consistent with the values obtained from the material without processing through ultrasonic cavitation.

## 5. Conclusions

The ultrasonic cavitation process originates in the tested materials an average decrease of the size of the particles that comprise it; reaching nanometric values on the titanium dioxide.

The ultrasonic cavitation process does not affect the chemical composition of the materials processed when the medium used to dissolve the material and propagate sound waves is distilled water.

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