

STATISTICS-BASED EXPERIMENTAL DESIGN TO STUDY THE FORMATION OF CARBON-COATED MAGNETIC NANOPARTICLES BY PLASMA ARC

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Statistics-based experimental designs have been usually exploited in fields related to biology such as agriculture, biochemistry or medicine, where a great number of parameters are involved in the processes under study [1]. In contrast, fields where physical factors are implicated, the systematic studies are carried out varying one of the process parameter and maintaining the rest of them constant.

Carbon-coated nanoparticles have been a focus of interest due to their advantageous properties over other coatings, such as their higher chemical and thermal stability and compatibility, very suitable for biological applications [2]. However, the understanding of the formation mechanism of this kind of structures is still unknown and remarkable field of research.

In an attempt to use the potential of statistics tools above mentioned, here we report, for the first time, the Plackett-Burman (PB) design was applied in order to study the formation process of carbon-coated magnetic nanoparticles by using a plasma arc technique, which is the most common technique to produce this kind of nanostructures.

The plasma arc reactor used is described elsewhere in the literature [3]. A schematic picture of the experimental apparatus is shown in Fig. 1. The parameters employed for the PB study are the following ones: pressure (evaluated from 500 mbar to 200 mbar), current applied (from 5 A to 55 A), amount of iron material (from 0.05 g to 0.17 g), helium flow (from 800 sccm to 1800 sccm), electrode diameters (from 0.9 mm to 7 mm), plasma time duration (from 180 s to 420 s) and flow geometry (gas flow coaxial nozzle or surrounding gas flow). All parameters were varied within the scope to our system and taking into account the values used in the literature [4-6].

In particular, there are three common problems in carbon-coated magnetic nanostructured products. On one hand, the poor yield achieved. A great quantity of nanoparticles is required for the commercialization and to produce enough signals for some measurements, such as, magnetization responses. The second problem is the fabrication of smaller nanoparticles. Ultrafine nanoparticles are very suitable for some activities, for example, in biomedical applications where superparamagnetic behaviour is requested. The last problem is about the polydispersity of the samples, which is one of the challenges in this field.

Pareto charts in PB design show the most influential control parameters in each case. The main factor to control the yield of nanoparticles is the flow geometry; gas flow coaxial nozzle allows obtaining higher yields. Smaller diameters are obtained using higher intensities and a surrounding gas flow. In addition, lower dispersions are also achieved with surrounding gas flow.

Fig. 2 shows TEM images of some samples obtained in PB experimental designs. Nanoparticles from 3 to 30 nm core diameters with dispersions from 20 % to 50 % were obtained.

The screening Plackett-Burman experimental design in 12 runs has been used to identify the most influent control parameters in the plasma arc method. As it is shown, geometry flow is a crucial parameter to control diameters and dispersions of nanoparticles. We conclude that this fact is related with the residence time of the nanoparticles in the plasma region. Surrounding

gas flow implies lower residence time, which involves minor diameters and dispersions. On the other hand, higher concentration of nanoparticles in the plasma region causes higher yields, but a gas flow coaxial nozzle is required. It means that the quality and quantity of nanoparticles are in competition. It is essential to find an intermediate point where yield, small diameters and minor dispersion are optimized.

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Figures:

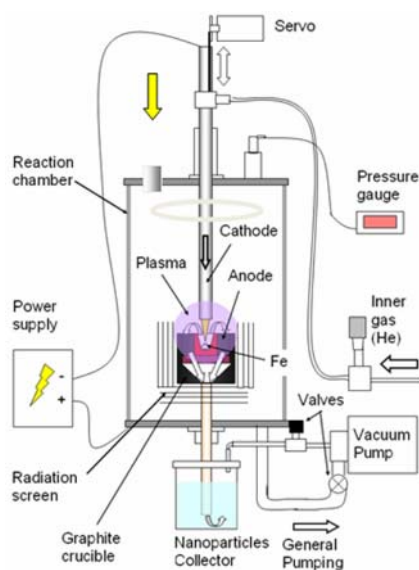


Fig. 1 Schematic picture of the experimental setup.

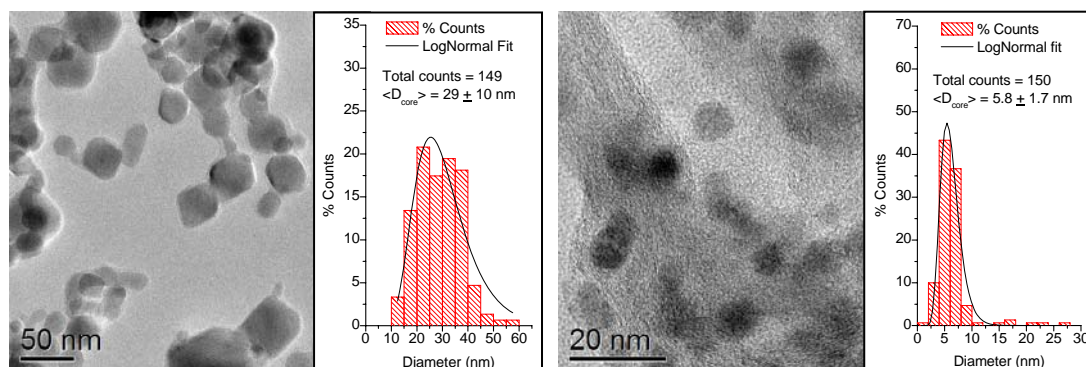


Fig. 2 TEM images of carbon-coated iron nanoparticles obtained in Plackett-Burman experimental design. Histograms are represented in both cases.