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Low-cost Functionalized Pseudoboehmite/Aluminum Substrates for the Analysis of Nanoparticles by SEM

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Aluminum is the most common metal element found in earth, one of its crystalline forms is the pseudoboehmite, used mainly as catalyst [1]. There are many chemical reactions to synthesize pseudoboehmite, most of them include heat treatment and result in a powder [2, 3]. While in other works high purity Al plates are necessary to growth pseudoboehmite on its surface; in this work, easily available aluminum alloy 6063 (Al-6063) plates were employed to synthesize pseudoboehmite/aluminum, resulting in a low-cost functionalized substrate for the analysis of nanoparticles by Scanning Electron Microscopy (SEM) [3].

Al-6063 is a very common and cheap alloy used widely in machine shops. The plates were worked in a grinding machine using 25µm alumina oxide, followed by Brasso metal polisher on a clean cotton fabric. The cotton fabric was replaced until a mirror like ending was obtained. To eliminate the residues of the metal polisher, they were rinsed with acetone and alcohol under sonication for 15min.

An electropolish solution was prepared by mixing perchloric acid (Aldrich, 70%) and ethanol (Aldrich, 99.8%) (4:1), this solution was kept at 4C by an ice bath. The Al-6063 plates were immersed in the perchloric acid solution at 25V and 2.5A for 40s. The plates were then rinsed with Milli-Q water, followed by immersion in sodium hydroxide (1.5M, Aldrich) and nitric acid (1.4M, Aldrich) solutions, rinsed again with water, immersed into boiling water for two minutes and finally blow dried with cleaned air.

The formation of a pseudoboehmite layer over the Al-6063 was confirmed by X-ray diffraction according to ICDD 00-021-1307 card (XRD, D2 Phaser Bruker, with a Bragg-Brentano geometry and Cu-kα radiation (λ=1.5418Å) and using the following scan: step size = 0.02°, t = 5s, $10° \leq 2\theta \leq 80°$) (Fig. 1A). The hydroxyl groups available in the surface allow the functionalization by immersing the plates in a 20mM (3-Mercaptopropyl)trimethoxysilane (MPTS, Aldrich) methanolic solution (Aldrich) for 15h. In order to eliminate the unbounded MPTS molecules, the plates were rinsed with deionized water and dried in an oven at 120C for 10 min.

These functionalized Al-6063 plates allowed a uniform deposit of gold nanoparticles by drop casting (Fig. 1B). As an example, concave gold nanocubes (CGNC) were synthesized in a seed mediated growth method [4,5] and concentrated 40x by centrifugation (1100g). Next, a 3µL nanocubes solution drop was placed on the functionalized Al-6063 plates and left to dry at room temperature. To ensure that the surfactant of the nanoparticles was removed, samples were plasma cleaned (Femto low-pressure plasma system, Diener) for 10min under nitrogen flow. SEM images (JEOL, JSM-7800F, operating in gentle beam mode at 15kV and 3mm working distance) show the mesoporous surface due to the pseudoboehmite and the attached CGNC (Fig. 1C). The functionalized surface of the Al plates allowed the homogeneous deposit of the CGNC (Fig. 1D).

Here we have demonstrated that by using a low-cost Al-6063 plates it was possible to synthesize a pseudoboehmite layer and functionalize it by silanization, choosing the functional groups at will.

References:

[1] A. Kocjan, *et al*, International Journal of Applied Ceramic Technology, **8** (2011), p. 848.
[2] T. Masuda, *et al*, Materials, **8** (2015), p. 1350.
[3] R. U. Din, *et al*, International Journal of Adhesion and Adhesives, **74** (2017), p. 167.
[4] J. Zhang, *et al*, Journal of the American Chemical Society, **132** (2010), p. 14012.
[5] M. M. Martinez-Garcia, *et al*, Colloids and Surfaces A: Physicochemical and Engineering Aspects, **493** (2016), p. 66.

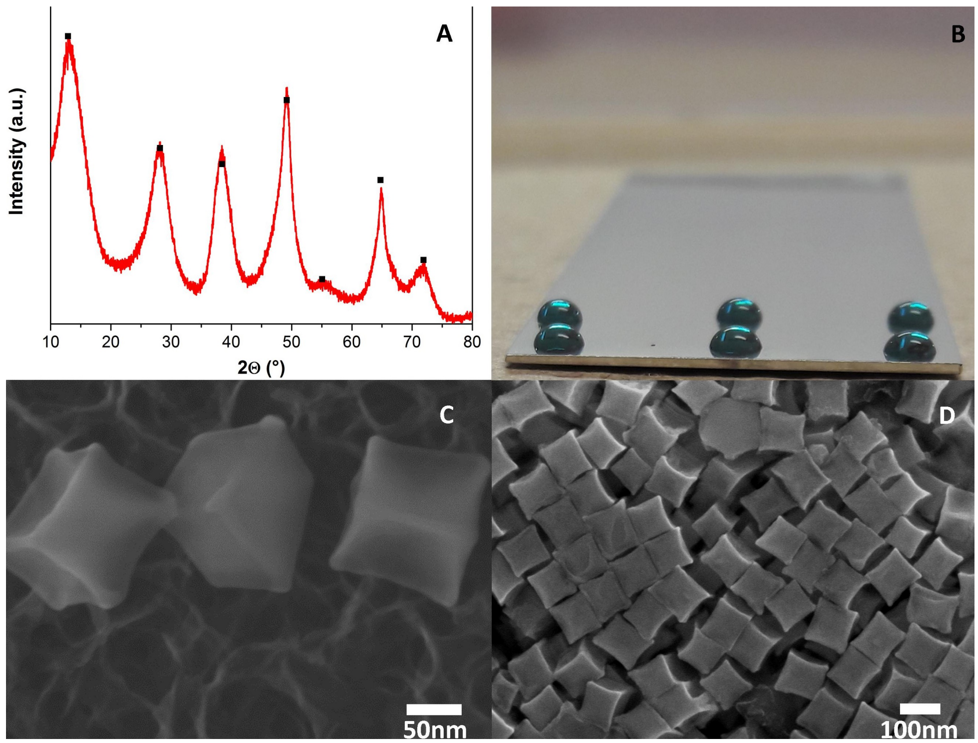

Figure 1. A XRD pattern of pseudoboehmite (AlO(OH)). **B** Optical image of CGNC on top of the functionalized Al substrates. **C** and **D** SEM images of the CGNC deposited on the pseudoboehmite/aluminum substrates.

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