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Electron Microscopy Characterization of Graphene/TiO_2 Nanocomposite for Application in Photocatalysis

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Titanium dioxide (TiO_2) has been investigated with special interest in recent decades because it is considered the most promising material among semiconductors applied in photocatalysis, due to its high photocatalytic performance, low cost, controllable structure and morphology, chemical stability, and its non-toxicity [1]. However, it has two main limitations: its high band gap that is approximately 3.05 eV and 3.20 eV for the rutile and anatase phases, respectively. When titania is combined with graphene, there is the possibility of improving absorption in the visible region, as has been confirmed in theoretical studies [2]. Therefore, multiple efforts have been made to obtain anatase-graphene nanocomposites with applications in photocatalysis [3].

This paper shows the characterization of a graphene/TiO_2 nanocomposite by Scanning Electron Microscopy (SEM), with a Jeol JSM 7600F Model field emission equipment, and Electron Microscopy of Transmission (TEM), using a Tecnai Supertwin Model equipment with field emission. SEM images of the synthetized precursors and the graphene/TiO_2 nanocomposite are shown in Figure 1. TiO_2 nanoparticles obtained by a low temperature sol-gel method and a subsequent hydrothermal treatment for 72 hours are mostly homogenous and form agglomerates, as shown in Figure 1(a). Graphene flakes obtained by electrochemical exfoliation of graphite (EEG) are appreciated in Figure 1(b), these flakes have different sizes and shapes and have a classic graphene morphology consistent with other work [4]. The EEG process consisted of an anodic exfoliation of a graphite rod, using a copper wire as a counter-electrode and 1 M sulfuric acid solution as electrolyte. With this arrangement, 12 V voltage was applied for 30 min to generate the separation of the graphite layers. Subsequently the exfoliation product was sonicated for 30 min. Observing the graphene/TiO_2 nanocomposite obtained by mixing and sonicating, it is clear that the graphene sheets and TiO_2 nanoparticles have been integrated by way of an intimate interfacial contact as shown in Figure 1(c). TEM images (Figure 2) allowed to measure the titania particles and identify the crystalline planes present in the samples. Titania ellipsoidal particles were measured to be approximately 17 nm in length and 10 nm in width, these particles form agglomerates as can be seen in Figure 2 (b). On the other hand**,** Figure 2(b) shows a folded graphene flake of only a few layers of thickness. In the image of the graphene/TiO_2 nanocomposite showed in Figure 2(c), the elliptic silhouette corresponding to anatase nanoparticles can be seen, in which the lattice plane (101) of the anatase predominates and forms a ring on the FFT. In contrast, few layer graphene is presented as ribbon-like structures and lattice plane (002) can be distinguished. The lattice spacings were measured to be 0.352 nm (101) for anatase and 0.346 nm (002) for graphene. TiO_2 particles tend to agglomerate at the edges of graphene flakes. These images are similar to what is found in other works of graphene/TiO_2 [5],[6]. TEM and SEM analysis allowed to confirm the successful synthesis of graphene/TiO_2 nanocomposite for possible application in photocatalysis.

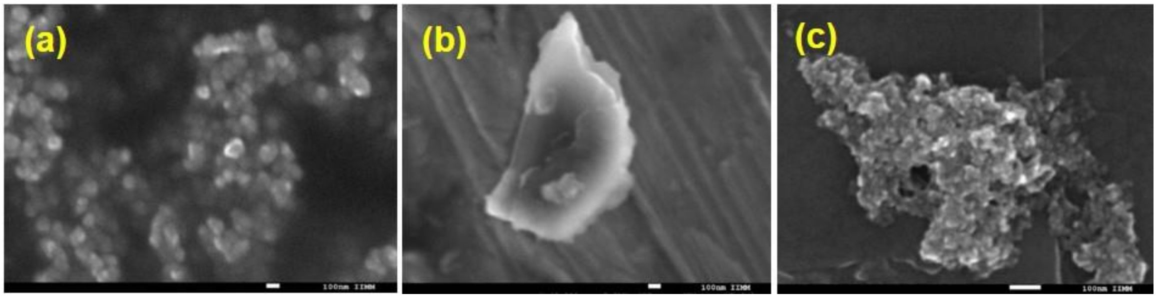

Figure 1. SEM images of (a) TiO2 synthetized by sol-gel method, (b) graphene obtained by electrochemical exfoliation of graphite and (c) graphene/TiO2 nanocomposite.

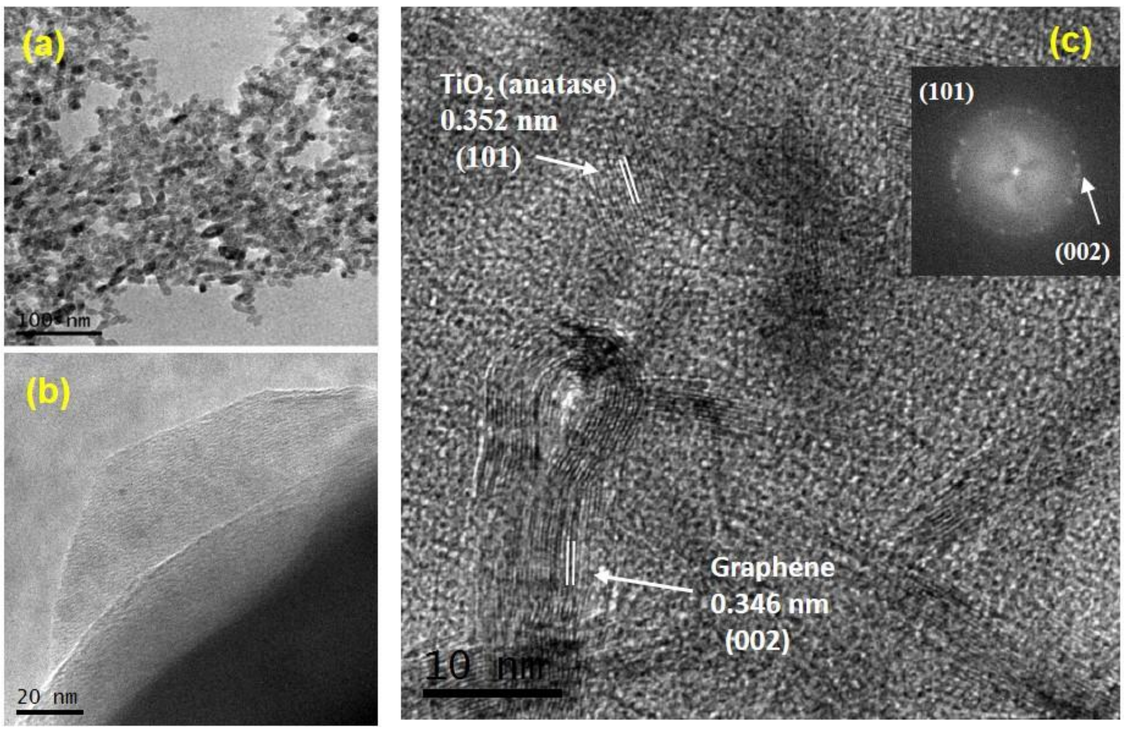

Figure 2. TEM images of (a) TiO2 nanoparticles with 17 nm in length and 10 nm in width, (b) folded graphene flake and (c) graphene/TiO2 nanocomposite and diffraction pattern FFT.

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