Surface Morphology of Nanostructured TiO₂@Graphene Composites Obtained by Lyophilization

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 TiO_2 is a very important photocatalyst that can decompose organic pollutants and is therefore increasingly attractive for application in today's environmental industry because it is non-toxic, harmless, chemically stable, and readily available [1]. To make the properties more efficient, there is the possibility of doping titanium dioxide with metallic and non-metallic elements and introducing them into the TiO₂ crystal lattice. TiO₂ based composites with graphene are known to improve the resulting photocatalytic efficiency due to the synergistic effect.

In this work, we deal with the synthesis and analysis of 2D nanostructured material based on TiO_2 and graphene (Gra). Reaction solutions were prepared by hydrolysis of aqueous titanyl sulfate ($TiOSO_4$) solutions, to which graphene was then added. Part of the sample was supplemented with Urea (U). This was followed by a lyophilization [2, 3] and an annealing process at 150, 300, 400, and 500 °C. Then Ti_Gra and To_Gra_U nanocomposites were formed. Morphology and microstructural changes with SEM and their PEC activity for water splitting have been evaluated.

Scanning electron microscope images of Ti_Gra nanocomposites confirmed the formation of 2D nanosheet morphology. For the Ti_Gra_500 (Figure 1a), the nanosheet size is in the range of $20 - 50 \mu m$. The nanosheets of the nanocomposite Ti_Gra_U_500 (Figure 1b) with the urea addition show the same size, but compared to the sample without the urea, the nanosheets appear finer and do not tend to clump together.

Performed PEC experiments suggested, that Ti_Gra_500 and Ti_Gra_U_500 nanocomposites annealed at 500 C have had the best performance among the whole set. The induced current achieved was similar for both materials. The Ti_Gra_500 first reached maximum current (Figure 2a), after that then began to degrade. In contrast, Ti_Gra_U_500 has similar photoactivity for PEC water splitting and at the same time higher stability with increasing of the current (Figure 2b) [4, 5].





Figure 1. SEM images of (a) Ti_Gra_500 and (b) Ti_Gra_U_500.



Figure 2. PEC experiment of (a) Ti_Gra_500 and (b) Ti_Gra_U_500.

References:

[1] A Fujishima, K Honda, Nature 238 (1972), p. 37.

[2] S Bakardjieva et. al., RSC Advances. 9 (2019), p. 22988-23003.

[3] S Bakardjieva et. al., Cat. Today 328 (2019), p. 189-201.

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