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Deactivation and regeneration of Pt-Titania-reduced graphene oxide nanocomposites as catalysts in the photochemical generation of Hydrogen

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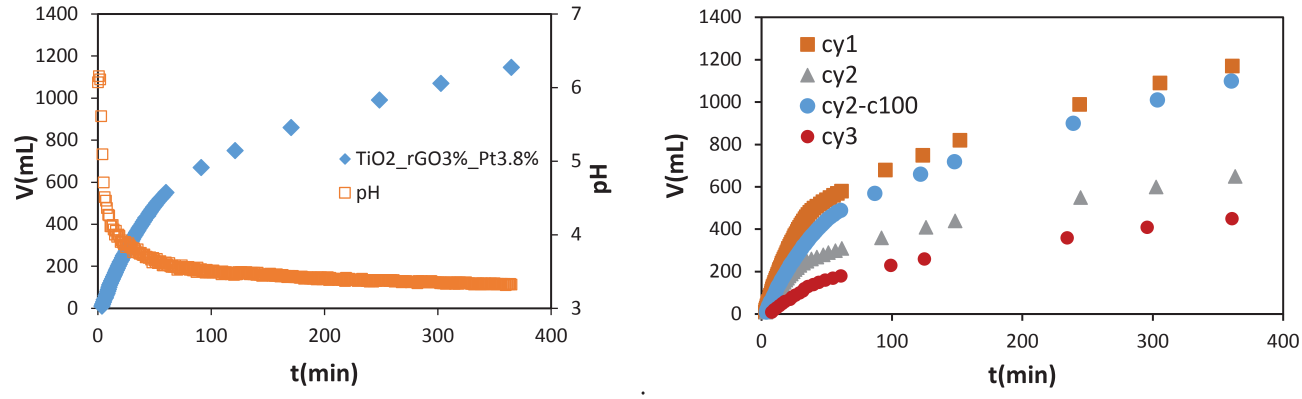
Abstract

A series of catalysts TiO_2_rGO(x)_Pt(y) (x=0-5 wt%; y=0-3.8 wt%) have been synthesized, characterized and used, mainly under UV light, in the photogeneration of hydrogen using various sacrificial agents amongst others methanol, ethanol and glycerol. Selected optimal operational conditions for TiO_2_rGO3%_Pt3.8% have given place to very high initial rates of hydrogen production with values of ~560 and 70 mmolh^{-1}g$_{cat}^{-1}$ for ethanol and glycerol, respectively, which are found within the highest in the literature [1]. The catalysts were synthesized by a two step procedure that uses the hydrothermal method to obtain the TiO_2_rGO composites and subsequent photodeposition of platinum. Characterization has shown platinum nanoparticles in the composite catalysts to be well distributed (TEM) and evidenced the reduction of graphene oxide in the process (FTIR) as well as band gap narrowing of TiO_2 (UV-vis diffuse reflectance spectroscopy). In the case of ethanol as sacrificial agent, the produced gases, analyzed by gas chromatography, showed hydrogen as the main product (above 95%) with remaining components being CO and CH_4 and minor amounts of CO_2, C_2H_6 and C_2H_4. Analysis of the liquid phase points towards the presence of acetaldehyde as the main reaction product. Obtained results regarding evolved gas volume *vs* UV irradiation time, recorded simultaneously with solution pH, showed drastic changes associated to variations in the rate of hydrogen production as seen in figure 1a), which exhibits two slopes for data acquired along 6 hours irradiation time. Re-using of the catalyst evidenced a loss of its photoactivity from cycle to cycle (see figure 1b, cy1 to cy3), suggesting deactivation by increasing poisoning by the reaction products. Regeneration of the catalyst was firstly approached by using heat treatment at 100 ^{o}C for 3 h with excellent results, see figure 1b). Heat treatment up to 300 °C followed with slightly better results. The spent catalyst was also treated chemically in an attempt to remove adsorbed reaction products on the catalyst. Chemical treatment with a strong oxidizing agent such as H_2O_2 (a 30 % solution, 20 mLg$_{cat}^{-1}$) proved to be very effective in the regeneration of the catalyst, giving results closed to those obtained by thermal treatment. A fuller characterization of the spent and regenerated catalyst is in progress in order to ascertain the species involved in the deactivation process and the more effective treatment for regeneration.



(a) (b)

Fig. 1. Hydrogen evolved and pH *versus* irradiation time for TiO_2_rGO3%_Pt3.8% (a). Deactivation and regeneration of catalyst photoactivity after heat treatment (b). Experimental conditions: Initial pH=7; 5M ethanol; 0.5 g L^{-1} , T=40 ^{o}C, UV lamp 450 W.

Acknowledgements

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References

[1]Paula Ribao, M.Alexandra Esteves, Vitor R. Fernandes, Maria J. Rivero, Carmen M. Rangel, Inmaculada Ortiz, Int. J. Hydrogen Energy, 2018. DOI: 10.1016/j.ijhydene.2018.09.148