REMOVAL OF DIRECT BLACK 38 DYE BY ADSORPTION AND PHOTOCATALYTIC DEGRADATION ON TiO_2 PREPARED AT LOW TEMPERATURE

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***Abstract*– TiO_2 powder was prepared by a hydrothermal process at 200ºC for 12 h. The material was characterized by X-ray diffraction and N_2 adsorption-desorption isotherm. The synthesized sample presented a pure phase anatase, with nanometric particle size. Its activity was tested for adsorption and degradation of the azo leather dye, Direct Black 38. The adsorption isotherm obtained followed the S-type in terms of the classification of Giles. Thermodynamic parameters were calculated, and the results revealed that the adsorption process is endothermic in nature. The material showed highly photocatalytic activity in the degradation of the dye, similar to that of the TiO_2 (P25 Degussa) photocatalyst.**

***Keywords*— TiO_2, photocatalysis, adsorption, degradation, dye.**

I. INTRODUCTION

Dye effluents may lead to certain environmental problems. Effluent treatment for dye-containing effluents from tanneries and textile and paper industries is currently capable of removing only about half of the dyes lost in residual liquors (Moreira *et al.*, 2005). Advanced oxidation processes (AOPs) have been proposed as an alternative method for water purification and an efficient wastewater treatment technique used for the total mineralization of organics (Akyol *et al.*, 2004). AOPs include ozonation, UV irradiation, photocatalysis, the Fenton and photo-Fenton reagent, and various combinations of these (Tarek *et al.*, 2009).

The photocatalytic degradation of toxic compounds in aqueous media provides a new method for wastewater treatment. The process couples low-energy ultraviolet light with semiconductors acting as photocatalysts. TiO_2 is generally used as a photocatalyst for the removal of organic pollutants (Tanaka *et al.*, 1997) and dye pollutants (So *et al.*, 2002) due to its high photocatalytic activity and stability, low cost, stability with respect to corrosion, and biological, nontoxic and chemical inertness (Ferguson *et al.*, 2005).

Different methods have been employed for the synthesis of titanium dioxide, as follow: chemical precipitation (Scolan and Sanchez, 1998), sol-gel method (Lin *et al.*, 2006a), hydrothermal (Wu *et al.*, 2002) and solvothermal processes (Yin *et al.*, 2003). Hydrothermal synthesis has become one of the notable methods employed in nano-material production and nanotechnology (Pavasupree *et al.*, 2008). The process includes a simple route and produces high crystallinity oxides under moderate conditions: low temperatures and short reaction times. It is one the most used methods to produce ceramic materials as it allows the control of particle size, morphology (Lin *et al.*, 2006a) and phase composition (Wang *et al.*, 2007).

In this study, TiO_2 was synthesized by the hydrothermal method at 200ºC for 12 h. Its characterization, adsorption capacity and photocatalytic activity in the degradation of azo leather dye, Direct Black 38, was investigated. Various thermodynamic parameters, such as entropy (ΔS^o), heat of adsorption (ΔH^o) and Gibbs free energy (ΔG^o) have been determined.

II. METHODS

A. Catalyst preparation

The synthesis of titanium dioxide was performed using hydrothermal treatment. The molar composition of the reaction mixture was $1TiO_2$:$10CH_3COOH$:$150H_2O$. Both glacial acetic acid (CH_3COOH) (Aldrich) and titanium (IV) isopropoxide ($C_{12}H_{28}O_4Ti$) (Aldrich) were of analytical-reagent grade and used as starting materials. Glacial acetic acid (36 mL) was slowly added to 20 mL of titanium (IV) isopropoxide in a water bath at 0ºC, under constant stirring. Afterwards, deionized water (170 mL) was gently added to the mixture under constant stirring. The solution was then mixed together under vigorous agitation for one hour and subsequently underwent ultrasonic treatment for 30 min. Once again, vigorous agitation was applied for 5 h. Afterwards, the solution was poured into teflon jars and placed in stainless steel autoclaves. The autoclaves were placed in an oven previously heated to 70°C in order to carry out the ageing process for 12 h. Following the ageing process, hydrothermal treatment was carried out 200°C for 12 h. Subsequently, the autoclaves were removed from the oven and cooled in running water. Eventually, the precipitate was washed with distilled water, centrifuged and dried in an oven at 100°C for 12 h. The solid sample obtained was finely ground using a mortar and pestle and stored in plastic containers.

B. Characterization methods

The powder was characterized by X-ray diffraction and nitrogen adsorption was determined by BET. X-ray diffraction (XRD) patterns were obtained using a Bruker D8 Advance diffractometer with a Cu-Kα radiation X-ray source (40 kV e 40 mA). Data were collected over the 2θ range 20–80º with a step size of 0.05º and a count time of 35 s. The average nanocrystal size was estimat-