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TiO_2 Nanocrystals: Synthesis Parameters and Photocatalysis

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Abstract – Anatase TiO_2 have been synthesized by hydrothermal treatment of amorphous TiO_2 obtained by decomposition of peroxo complex titanium (PCT) and hydrothermal treatment at different pH values (Fig. 1). The samples obtained at pH 6 and 8 show very similar specific surface area (SA), but less than that obtained at pH 4 (Table 1). Photodegradations of Rhodamine B were performed to study the relationship between synthesis pH and degradation rates (Fig 2). The higher pH synthesis, the better is photocatalytic performance. It may be related to recombination centers generated by oxygen vacancy in acidic environment, which inhibit the photocatalysis process.

Semiconductor nanocrystals have been widely used in photocatalysis due their high surface area[1]. It is consensual that the most used is the TiO_2, especially in anatase form. The synthesis method has an important role in the photocatalytic process, because it can generate lattice and surface defects, which are important features in the system. This work aimed to study the influence of pH during the hydrothermal treatment of amorphous precursor to crystallization of nanoparticles in the TiO_2 photoactivity. The PCT has been synthesized by dissolution of metallic Ti in H_2O_2/NH_3 solution (3:1). After heating and cooling in an ice bath, the amorphous precursor is formed. To the crystallization, this precursor was dispersed in water and pH value adjusted with HNO_3 [2]. X-Ray Diffraction (XRD) analyses were carried out with radiation in 1.5456 Å, corresponding to the Cu Kα emission and scanning at 1,2°/min. Surface areas were determined by N_2 adsorption at 77 K and evaluated by the standard BET procedure. The photodegradation experiments were done under UV illumination and the concentration of residual Rhodamine B was measured at the time through its UV-vis spectra. The sample obtained at pH 8 showed the best photocatalytic performance, followed by those obtained at pH 6 and pH 4. The surface area play an important role in photocatalysis, but in this case, the difference can not be attributed at this parameter, because the sample obtained at pH 4 showed higher SA and worse photocatalytic performance. The difference between the samples may be in an amount of oxygen vacancy from the synthesis environment, since acid environment has less oxygen than basics. This is evidenced by the formation of rutile structures in acidic environments. Such structures tolerate these conditions. The formation of vacancies may act like recombination centers, where electrons in the conduction band and roles in the valence band generated during excitation are destroyed. Thus, recombination prevents the formation of radicals and electronic transference between nanocrystals and dye, responsible by degradation process.

Table 1: Specific surface area of the as-synthesized nanoparticles and its average diameter

Sample	SA / ($m^2.g^{-1}$)	Average diameter / (nm)
pH 4	93,3	15,2
pH 6	68,4	20,7
pH 8	66,9	20,4

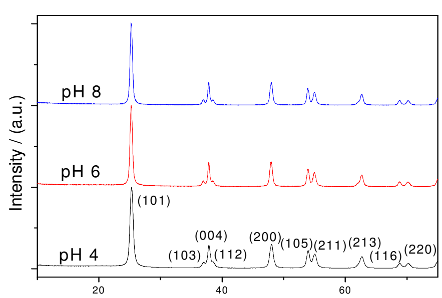

Figure 1: XRD patterns of the as-synthesized samples. (Anatase)

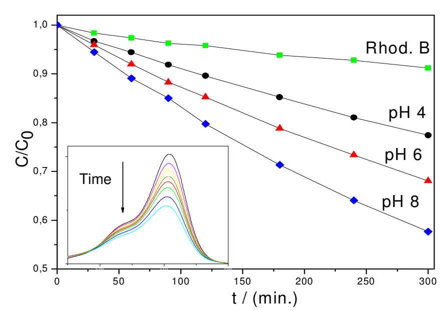

Figure 2: Photocatalytic kinetics of Rhod. B degradation

References

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