

Characterization and photocatalytic activities of nanosized titanium dioxide thin films

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ABSTRACT: Thin films of titanium dioxide with high surface area are prepared by sol-gel dip-coating technique. In this regards, Titania nano sols with high photocatalytic activity were prepared by dissolving titanium alkoxide in alcohol and water under acidic conditions. Photocatalytic activities of titanium dioxide thin films were measured in the presence of methylene blue. Microstructure and photocatalytic activity of the films, nanopowders and titanium dioxide sols were investigated using X-ray diffraction, scanning electron microscopy, specific surface area, zeta sizer and ultraviolet-visible spectrometry techniques. Particle size analysis of sols showed that the mean particle sizes were 15 to 128 nm. X-ray diffraction analyses revealed that anatase crystal structure was produced with crystallite size below 11 nm. Increasing mass percent of anatase phase and specific surface area, enhance the photocatalytic activity. Scanning electron microscopy images showed that the addition of methylcellulose as a dispersant, not only produced rough texture in the thin film, but also enhanced photocatalytic activity. The thin films prepared by using nitric acid as a stabilizer, revealed higher photocatalytic activity, surface area and sol stability and these data were more than those prepared with acetic acid.

Keywords: Dip-coating; Environmental purification; Morphology; Stabilizer

INTRODUCTION

Titanium dioxide (TiO₂) is the most promising semiconductor photocatalyst, which has been extensively studied for environmental purification applications, due to its good characteristic of powerful oxidation strength, chemical stability and nontoxicity. It is especially interesting to support TiO₂ on inert substrates for waste water and indoor atmosphere remediation (Negishi and Takeuchi, 2001; Hidalgo *et al.*, 2004; Jing-Sheng *et al.*, 2006; Wang *et al.*, 2007). Recently, Titania nano particles proved to be very effective in degrading organic pollutants in environment and petrochemical industry (keshmiri *et al.*, 2004; Sankapal *et al.*, 2005; Babel and Opiso, 2007; Samarghandi *et al.*, 2007; Gan *et al.*, 2008; Giri *et al.*, 2010; Hussain and Siddaqa, 2011). Volatile organic compounds (VOCs) are triggering serious

environmental problems such as alcohols, mercaptans, ketones, ethers and dye dissolved in the solution, among which Methylene blue (MB) has been used as much as dye (Wang *et al.*, 1998; Mills *et al.*, 2003). The effectiveness of TiO₂ photocatalyst in aqueous system is evaluated by degradation rate of an organic model molecule such as MB because it has less absorption at the absorption edge (~380 nm) of anatase phase and proved to be a relatively strong and stable molecule under UV irradiation (Wold, 1993; Habibi *et al.*, 2007; Medina-Valtierra *et al.*, 2007; Yogi *et al.*, 2008). TiO₂ exists in three principal crystallographic forms: anatase, brookite and rutile, however only the anatase form is photocatalytic active (Yang *et al.*, 2004; Collins-Martinez *et al.*, 2007). Most of the photocatalysts have been used as a fine powder form which has strong oxidative power of photogenerated holes on the surface. However, the fine powder form has several problems as follows:

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- 1) Separation of the catalyst from the suspension after the reaction is difficult.
- 2) The suspended particles tend to aggregate at high concentrations.
- 3) The amount applied is small and limited.

When the catalyst is coated on some supports, detachment from the supports easily occurs in the liquid phase (Verma *et al.*, 2005; Cernigoj *et al.*, 2006b; Ming-Fang *et al.*, 2006; Zhu *et al.*, 2008; 2009). Many techniques have been developed for immobilizing TiO₂ catalyst on solid substrates, for example, dip coating from suspension and spray coating, sputtering, sol-gel-related methods, electrophoretic deposition and atomic layer deposition of TiO₂ thin films. Among them the relatively simple sol-gel method is the most widely used (Guo *et al.*, 2005; Cernigoj *et al.*, 2006a; Mahshid *et al.*, 2007; Vilhunen *et al.*, 2008; Vilhunen and Sillanpaa, 2009). The sol-gel technique has attracted more attention due to low process cost, low temperature of heat treatment and wide possibility to vary film properties by changing the composition of the solution. The morphology, particle size and crystalline phase of the films, as well as their porosity and surface area, are important factors to influence catalytic activity (Verma *et al.*, 2005; Cernigoj *et al.*, 2006b; Gan *et al.*, 2008).

Photocatalytic activity of titanium is strongly dependent on its phase structure, crystallite size, specific surface areas, and pore structure. It seems of the high crystallinity of anatase phase extends the recombination rate of the electron and hole, hence, strong reducing and oxidizing power of the photocatalyst. In addition, the high surface area facilitates adsorption of the target molecules on to the surface of the catalyst and accelerates the photocatalytic reaction (Kana and Wongnawa, 2008).

The objective of this study is the investigations on processing and structural characteristics of high photocatalytic activity TiO₂ thin films with high surface area deposited on glass substrates through the dip-coating method. The photocatalytic activities of TiO₂

thin films have been studied by addition two different kinds of stabilizers in the presence of dispersant.

This research was carried out in Research Institute of Petroleum Industry in 2010.

MATERIALS AND METHODS

Experimental procedure

The thin films of TiO₂ were deposited by dip-coating sols which were prepared through a sol-gel route. The substrate used were pieces of soda lime glass plates (76 mm × 26 mm × 1 mm) purchased from Corning Inc. The substrates were cleaned for 2 h in an ultrasound bath (Model Starsonic 60) with ethanol and then dried in an oven at 80 °C for 15 min. In order to coat supports with titanium dioxide, titania sol prepared as follows: 30 mL of titanium (IV) iso-propoxide (TTIP) (Merck, 98 %) was dissolved in 10 ml of iso-propanol (Merck, 99.5 %) and proper amount of doubled distilled water. Then the prepared solution stirred by mechanical impeller (800 rpm) for 8 h.

After complete mixing of the solution, nitric acid (HNO₃) (Merck, 65 %) or acetic acid (CH₃COOH) (Merck, 95 %) were added as stabilizers and Methylcellulose (MC) (Merck, 2 Wt %) was used as a dispersant in preparation of samples.

The sols were deposited on glass plates while the temperature was kept at 25 °C during the deposition and the operation was repeated several times with content of 0.3 mg TiO₂ per g of plates. Then, the dip-coated plates were calcined at 350 °C for 1h with a heating rate of 2 °C/min. Table 1 shows the synthesis conditions of prepared samples. It is worth mentioning that the sols which named Sol.1, Sol.2, Sol.3 and Sol.4 after calcination process are called CAT.1, CAT.2, CAT.3 and CAT.4, respectively in Table 2.

Evaluation of photocatalytic activity

The photocatalytic activity of the thin films was evaluated by decomposition of Methylene blue (20 ppm) versus 50 ppm of TiO₂ photocatalyst. The photoreactor consisted of a cylindrical glass reactor with a high pressure mercury 400 w lamp located in front of vessel.

The variation of MB absorbance through 1h was measured at $\lambda_{\text{max}} = 665 \text{ nm}$ using a Du series 500 UV-Vis spectrophotometer. The degradation of MB values were calculated as follows:

$$\text{Degradation (\%)} = [(C_0 - C)/C_0] \times 100 \quad (1)$$

Table 1: Synthesis condition of Sols

Sol sample	CH ₃ COOH (Mol)	HNO ₃ (Mol)	MC (Wt %)
Sol.1	0.0068	-	2
Sol.2	-	0.0068	2
Sol.3	0.0068	-	-
Sol.4	-	0.0068	-



Where C_0 and C are initial and ultimate concentrations of MB respectively (Akyol *et al.*, 2004; Tobaldi *et al.*, 2008).

Characterization techniques

The crystal structure of TiO_2 coatings was identified by XRD (XRD, Philips PW 1840; $\text{CuK}\alpha$ radiation at 40kV and 200 mA). Preferred orientations were determined by comparing their relative intensities with those reported in the Joint committee on powder diffraction standards (JCPDS) cards (Ming-Fang *et al.*, 2006). The phase content of samples can be calculated from the integrated intensities of anatase (101) peak as:

$$W_A = \frac{K_A A_A}{K_A A_A + A_R + K_B A_B} \quad (2)$$

Where W_A represents the mass fraction of anatase and A_A , A_B , A_R are the integrated intensities of the anatase (101), brookite (121) and rutile (110) peaks and K_A and K_B are two coefficients and their values are 0.886 and 2.721, respectively. With Eq. 3, the phase contents in any TiO_2 samples can be calculated (JCPDS Powder Diffraction File, Card 21-1272, 21-1276, 29-1360).

The average crystallite size of anatase was determined according to the Scherrer equation using a FWHM data of each phase (Keshmiri *et al.*, 2004; Sankapal *et al.*, 2005; Hosseini *et al.*, 2007; Sreemany and Sen, 2007). The Brunaur-Emmett-Teller (BET) surface of the powders was measured by N_2 gas adsorptions-desorption at 77K using a Quantasorb BET surface area analyzer.

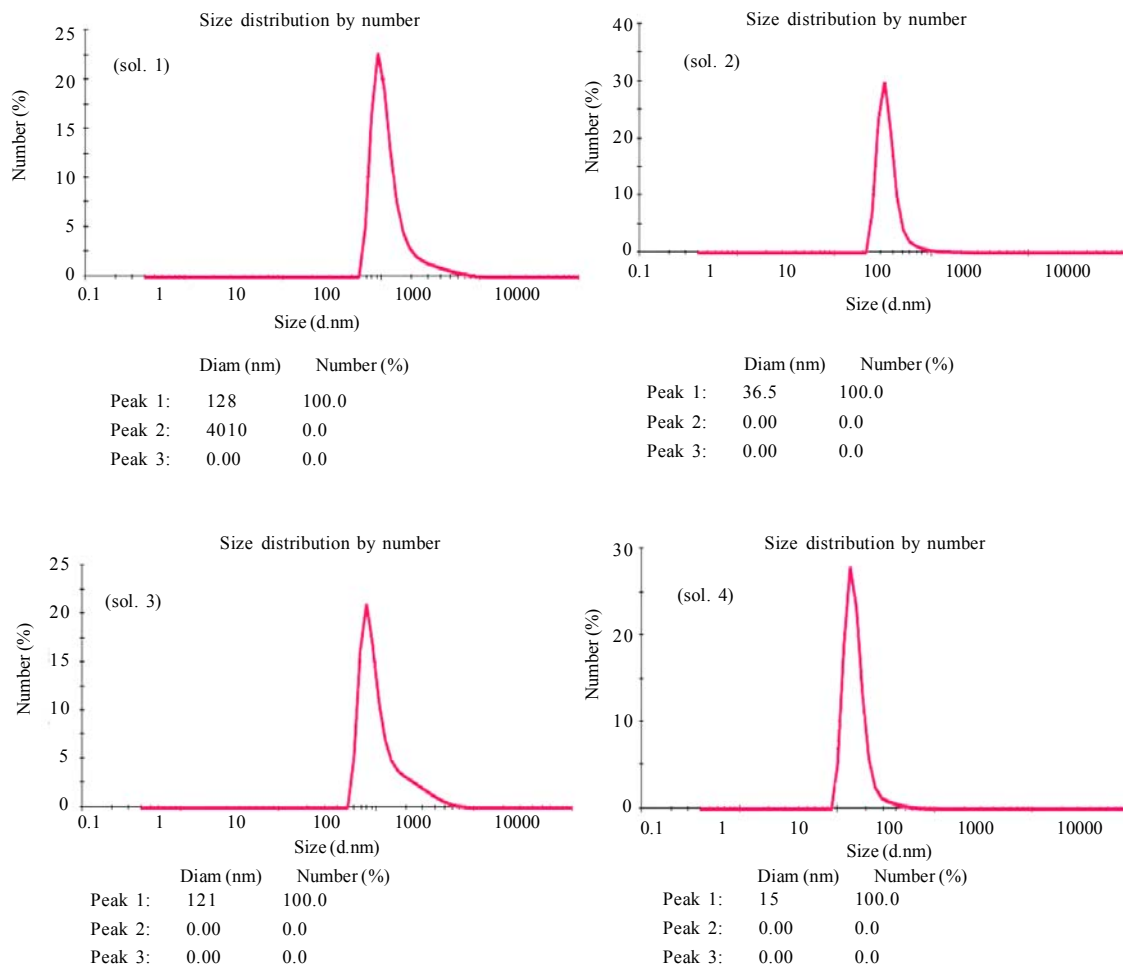
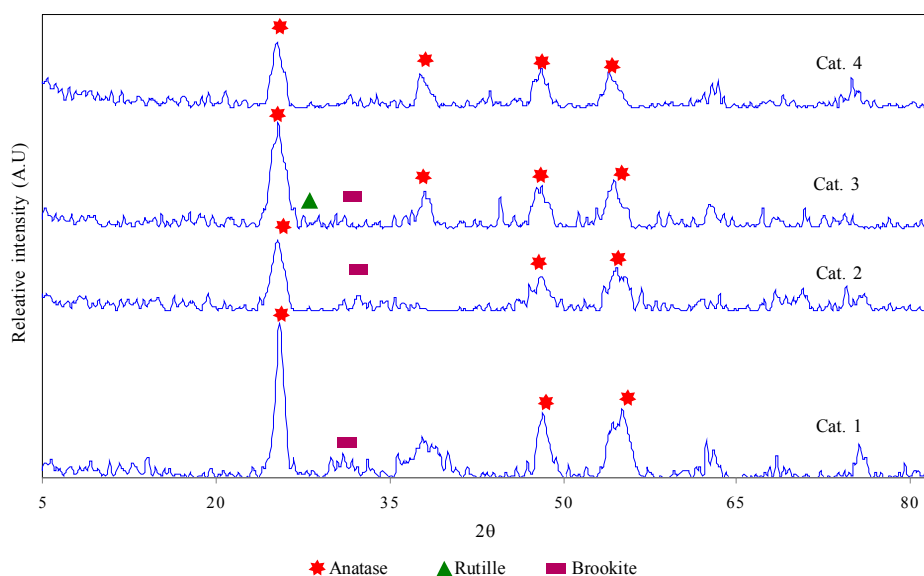


Fig.1: Size distribution and mean particle size of Sol.1-Sol.4



Fig. 2: XRD patterns of prepared TiO₂ nano filmsTable 2: Characterization data of the TiO₂ thin films

Sample	CAT.1	CAT.2	CAT.3	CAT.4
Analysis				
Anatase phase (%)	69	80	74	89
*d (nm)	11	7.1	7.4	6

*Calculated from Scherrer equation for anatase phase

The particle sizes of prepared sols were analyzed using a Malvern particle size analyzer, 24 h after preparation. The samples must be diluted in water and they were also sonicated for 2 min. Microstructure of the thin films was observed by Scanning electron microscope (SEM) Philips XLC30 at a voltage of 30 kV (sputtered by gold).

RESULTS AND DISCUSSION

Stability of TiO₂ suspension

Fig. 1 shows that type of acid and presence of MC had a huge impact on stability of the sols after some months and it implied that Sol.2 with nucleophilic group such as NO₃⁻ was more stable than Sol.3. However, CH₃COO⁻ group cannot counteracts the particle agglomeration induces by natural attractive Vander Waals forces and after passing time the whole sol was precipitated. Therefore, Sol.2 and Sol.4 which contained HNO₃ as an acid catalyst demonstrate diminutive size distributions (Fig. 1).

On the other hand, addition of massive component such as MC enhanced the sol density, so, Sol.1 and Sol.3 with poorly defined size distributions precipitated after short time. That explains that HNO₃ prevents aggregation strongly (Yu *et al.*, 2003).

Physical properties of synthesized TiO₂ powders

Fig. 2 shows the XRD patterns of the CAT.1-4 of thin films after calcination at 350°C. Table 2 shows the effects of HNO₃, CH₃COOH as hydrolysis catalysts on phase structure and average crystalline size of TiO₂ thin films. As it is cleared, mass percent of anatase phase of theses catalysts has altered in order of CAT.4 > CAT.2 > CAT.3 > CAT.1 which has implied the role of acids. It is obvious that HNO₃ is a much stronger acid catalyst than CH₃COOH to crystallize anatase phase. In order to literature (Yu *et al.*, 2003) acid concentration is a dominant factor in formation of anatase phase, because H⁺ ions rearrange the amorphous aggregates to form a crystalline anatase.

These results are in good agreement with that of other researches (Terabe *et al.*, 1994) who claimed that the anatase phase is stable in acidic solutions. Furthermore, the calculated average crystallite size of prepared sols proved the fact that the presence of H⁺ can affect the average crystallite size. According to the order of pH value of the sols CAT.1 > CAT.3 >



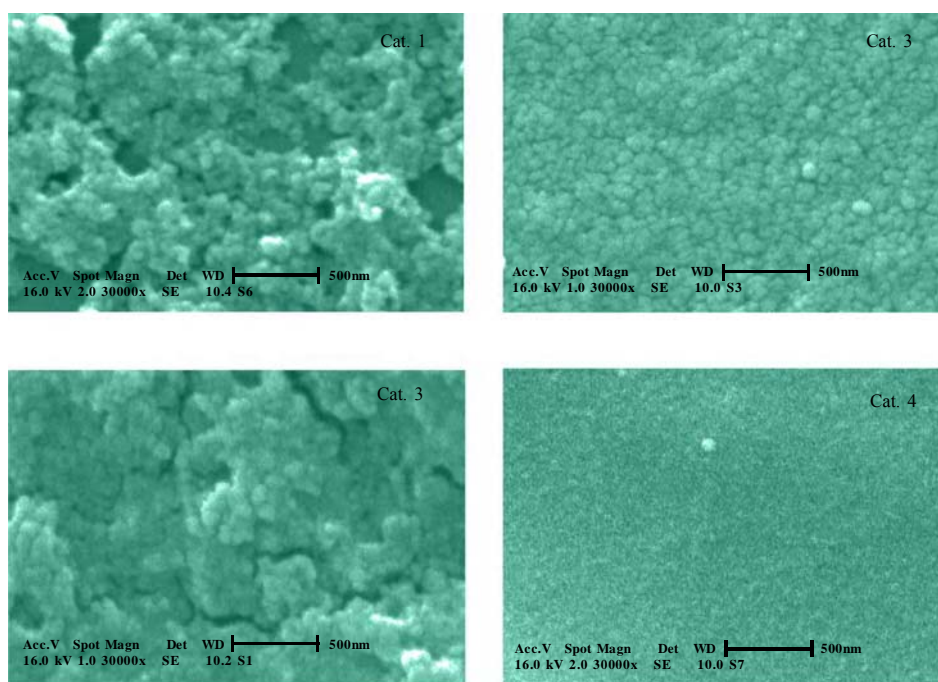


Fig.3: SEM images of the TiO_2 thin films deposited from sol on glass substrates with 2 dipping-heating cycles

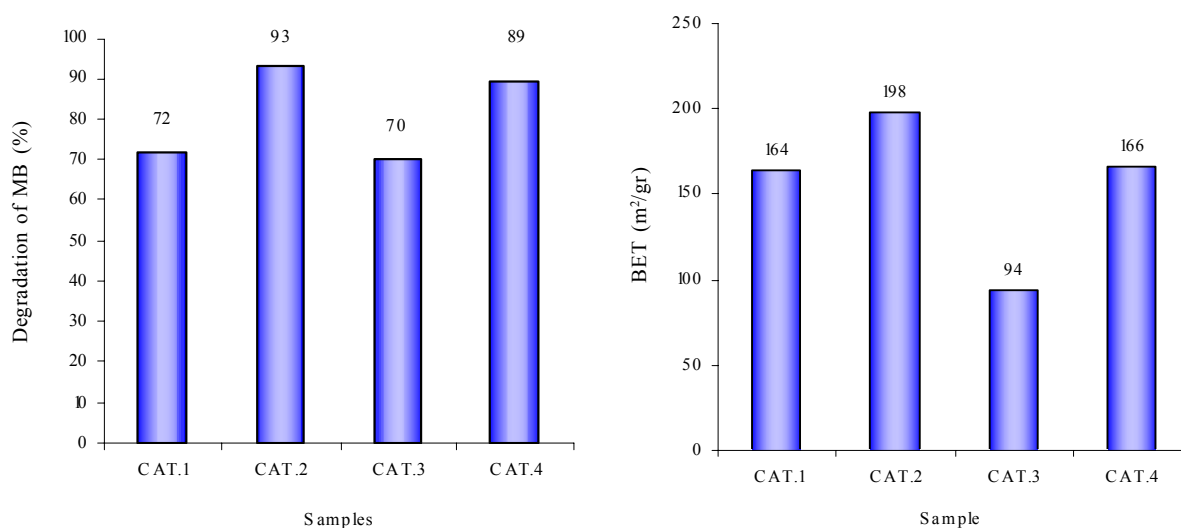


Fig. 4: a) Degradation of MB after 60 min, (b) Specific Surface area of CAT.1-CAT.4

CAT.2 > CAT.4, it is obvious that TiO_2 particle sizes growing with enhancing of pH value. These results are similar to those observed by other researches (Hu and Yuan, 2005) who pointed out that a single crystallite of TiO_2 is appeared since the acidity of the solution was

high enough (pH=1.2) and agglomeration will increase as acidity of the solutions decreased.

SEM studies

Fig. 3 Shows the SEM images of nano TiO_2 films of



CAT.1-4. As it can be seen, CAT.4 contains non-agglomerated spherical morphology, however the rest of the samples have incoherent surfaces which is due to their synthesis conditions.

CAT.4 among all the samples has the best quality as a coated film because HNO_3 is strong enough to reduce hydrolysis and condensation speed of the sols. Hence, CAT.4 appears as a unity structure but in CAT.3, CH_3COOH caused phase separation in the sols and produced cracked and rough texture in films. Although these results are in good agreement with Bu *et al.* (2005) who found that CH_3COOH origins phase separation but in CAT.3 it hasn't been marked any isolated islands similar to their results. This may be attributed to the different preparation route of the sol especially the presence of iso-propanol as a solvent. In fact, side branches in iso-propanol provide the ability to adjust opposite charges efficiently, in a way that isolated islands have not appeared in CAT.3. The presence of MC displays rough texture and granular microstructure in CAT.4 and CAT.2, respectively (Fig. 3).

This may be ascribed by vanishing MC after calcinations. Moreover, Cernigoj (Cernigoj *et al.*, 2006b) reported alike results and pointed out that using surfactant has a huge impact on morphology of the films by producing coarser but well structured granular surface. In conformity with the SEM results, CAT.1 has higher porosity and roughness than CAT.3 remarkably. It can be assigned to the fact that using CH_3COOH and MC in a sol solution simultaneously caused massive lost of material in ultimate films after calcinations and most of the porosities correspond to the vaporized water and carbon dioxide.

Photocatalytic properties of TiO_2 thin film

Fig. 4a and b demonstrates the comparison between photocatalytic activity and specific surface area of CAT.1-4 respectively. Evaluation of the photocatalytic properties of TiO_2 nano films during the degradation of MB reveals that photocatalytic activity of the catalyst increased with mass percent of anatase phase and BET. In fact, anatase phase with a band gap of 3.23 eV diminishes recombination process in the transformation of electrons-holes to the adsorbed species on the surface of the films. Furthermore, MC generates vast porosity and large number of active surface sites whereas photocatalysts reaction takes place. Therefore, among all the samples

CAT.2 which contains MC and has the most mass percent of anatase phase has the utmost photocatalytic activity.

CONCLUSION

Titania thin films with anatase crystallite sizes of as small as 11 nm were deposited on soda lime glass substrates in order to enhance their photocatalytic activity.

The phase structure, photocatalytic activity and texture of Titania thin films prepared by sol-gel route were remarkably influenced by the presence of stabilizer and solvent. Since HNO_3 is used as a stabilizer, the crystallization of anatase phase and photocatalytic activity was enhanced considerably and the prepared samples showed high surface area of $198 \text{ m}^2/\text{g}$. In contrast, the CH_3COOH not only prevents phase transformation of the TiO_2 powders from amorphous to anatase but also decreased sol stability and photocatalytic activity as well and devastated the surface morphology of the thin films. Worth mentioning, HNO_3 has much more effect on sol structure than CH_3COOH . In addition, results revealed that although addition of MC in the sols increased the porosity of the prepared films and consequently photocatalytic activity but ultimate thin films had rougher surface morphologies. Thus, MC showed binary effect on physico-chemical properties of Titania thin films.

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