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Inkjetready Sol-gel Synthesis of Nanocrystalline Titania Colloids

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Materials based on titanium dioxide produced as a highly photoactive catalysts, attracted the attention of many researchers because of the environmentally friendly destruction of substantially all organic contaminants. In this study, the inkjet titania films with hierarchical structures were prepared by a combined sol–gel and inkjet method. The X-ray powder diffraction (XRD) result indicated that the synthesized titania nanoproducts were of pure anatase phase. The data of low-temperature nitrogen adsorption-desorption and TEM (SEM) images demonstrate that the formed nanocolloids had a narrow particle size distribution with average diameter of 15 nm and were composed of nanocrystals with about 7 nm in diameter. The Brunauer–Emmett–Teller (BET) results showed that the titania inkjet layers have high surface area (110-127 m^2/g) and lots of mesopores.

Keywords: Sol-gel, Titanium dioxide, Inkjet printing.

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1. INTRODUCTION

Due to its unique optical, electrical, chemical properties and nontoxicity, nanoscale titania (TiO_2) has been widely applied in the field of photovoltaic [1], solar energy conversion[1], water splitting[2], bioconjugates ect. In recent years, nanosized TiO_2 with hierarchical structures has attracted much attention due to its superior performance, such as simple crystallization in water media, low density, high crystallinity, high surface area, visible transparence and high stability. Consequently, great efforts have been devoted to creating the nanosized TiO_2 with hierarchical structures, and various morphologies and textures have also been synthesized [3]. Based on the reported morphologies, nanoporous TiO_2 with hierarchical structures have shown highly promising application in the photocatalysis and bioengineering. For example, when the degradation of aromatic compounds containing amino groups, which are widely used in industry as herbicides, explosives, solvents, chemicals and various precursors. The use of such material causes great damage to the ecological environment and leads to pollution of soil and water. It is believed the most important use of photocatalysts in the form of pellets for the decomposition of aromatic nitro compounds which are highly toxic and contribute to the development of mutagenicity in many living organisms. [4, 5]. For photocatalysis setups, a powder system is several times higher in efficiency then compared with a thin film type; unfortunately, the recovery of titania nanoparticles after the photocatalysis process is quite cumbersome. Thus, to create TiO_2 photocatalysts, inkjet thin film is the most suitable, due to one-step synthesis of colloids inks, have a high surface area and high stability.

As a result, we reported a simple preparation of highly photoactive TiO_2 inkjeatready of nanocrystalline titania colloids with mesoporous structures and well dispersity by using different sol-gel approaches. SEM, HRTEM, XRD, BET, were performed to confirm the synthesis and structure of TiO_2 samples and the photocatalytic decomposition study of methyl orange (MO) indicated that 2 of our samples exhibited high photocatalytic activity and had an important advantage over the commercial P25.

2. EXPERIMENTAL AND RESULTS

2.1 The Synthesis of TiO_2 nanoproducts

Synthesis of TiO_2 nanoparticles

TiO_2 nanoparticles were synthesized by hydrolysis of TIP in the presence of de-ionized water, under magnetic stirring and ultrasonic irradiation (24 KHz, 300 W/cm^2). In a typical synthesis, a TIP was injected dropwise into the aqueous solution in 3 min under intensive magnetic stirring (1000 rpm). The mixture was sonicated continuously under ambient air during for 1-3 hours. The sonication was conducted without cooling so that the temperature was raised from 25 to about 75 °C at the reaction end. Figure 1 shows the formation of a colloidal sol TiO_2 depending on the ratio of $[H_2O/TiO_2]$ and sonication time. The data obtained is easy to determine optimal conditions of formation of a stable nano-sized TiO_2 sol in terms of the highest values of the average crystallite size.

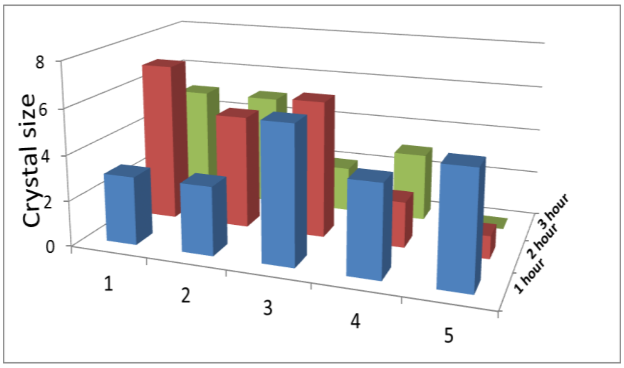

Fig.1 – 3D-plot of TiO_2 crystal Size vs. factors sonication time and $[H_2O/TiO_2]$ ratio.

As it has appeared at a ratio of $[H2O / TiO_2] = 3$ and 3-hour ultrasonic treatment formed the most stable colloidal system, Fig. 1. According to the survey, Figure

1, with a minimum ratio of H_2O/TIP = 1, we also observed the formation of the largest diameter of the particles. However, if this value increases to 100 again observed increase in the average particle size, especially after prolonged ultrasonic treatment. This can be explained by the fact that with increasing time of ultrasonic treatment is a reduction of vapor pressure at the interface between the reactants in the solution, resulting in intensive coagulated particles after their activation acoustic cavitation.

2.2 Results

The basis of challenges that were considered during the preparation this paper are 3 questions, the answers to which we have tried to reveal in this manuscript. 1) Can a stable crystalline TiO_2 sol to be stable during the inkjet printing 2) How do the external environment on the formation of the final material? 3) What are the future prospects of the obtained sols for bio-applications and electronics.

The developed approach for nanocrystalline TiO_2 sol in an aqueous solution under the action of ultrasonic cavitation provides the use of a pH close to neutral. Under these conditions, a stable complex is $Ti(OH)^{3+}$, which is under the influence of temperature dehydration becomes $[TiO(OH)_2(OH_2)_3]$, followed by coordination of hydroxyl group and successive dehydration between skew edged-water and hydroxyl group brings anatase TiO_2, Fig. 2.

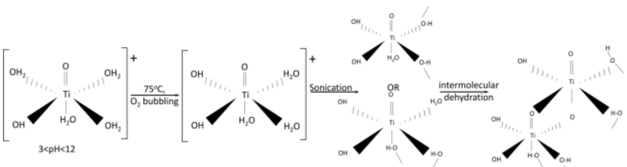

Fig. 2 – Plausible mechanism of TiO_2 crystallization in neutral pH under sonication.

Fig. 3 shows the typical SEM image of the as-prepared TiO_2 nanoproducts. It can be seen that the TiO_2 nanoproducts are uniform and have a very high homogenity. Achieving a high degree of purity of nanocrystalline TiO_2, prepared in an aqueous solution in the absence of the hydroxide and isopropanol amorphous phase makes this approach very promising, especially in electronics and biochemical engineering.

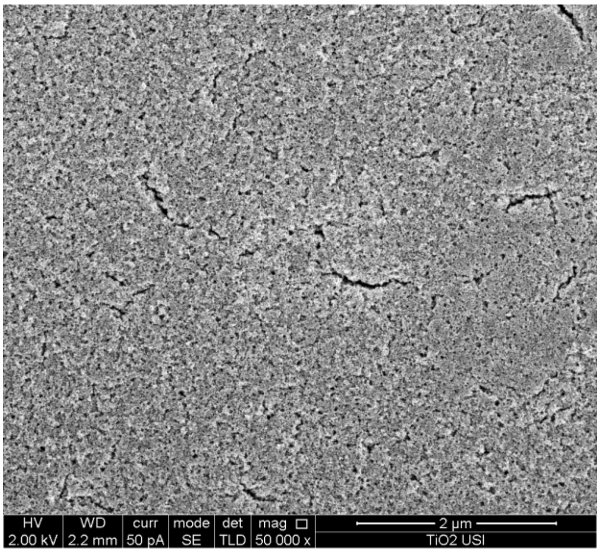

Fig. 3 – SEM image of inkjet titania film, prepared from TiO_2 colloids

3. CONCLUSION

In conclusion, we have developed a new method for synthesis of inkjet ready high purity titania sol, with controlled particle size and high degree of crystallinity. The ability to produce crystalline thin films of oxide materials like anatase TiO_2 at low temperatures has tremendous commercial advantage for thin film electronics and biochemical engineering.

We show that the proposed sonication-assisted process is highly versatile and can grow stable nanocrystalline anatase TiO_2 by inkjet printing on various substrates including plastic (PET).

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