ELECTROPHORETIC DEPOSITION OF TiO_2 NANO-PARTICLES: A COMPARISONBETWEEN THE PATTERNS OBTAINED IN METHANOL AND PENTANOL

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ABSTRACT

In the present study, the effect of two organic solvents, metanol and pentanol, on the deposition pattern of electrophoretically deposited TiO_2 nano-particles was investigated. Characterization of the obtained layer by scanning electron microscope (SEM) and optical microscope (OM) showed that a non-uniform porous layer was obtained when using methanol, however, a deposition from the TiO_2/pentanol cell resulted in the formation of a relatively uniform microstructure. The difference between deposited layers was attributed to considerably high deposition rate of TiO_2 nano-particles in methanol as well as the formation of large aggregates within the medium over time.

Key words: electrophoretic deposition, uniform layer, methanol, pentanol

INTRODUCTION

The electrophoretic deposition has recently become one of the most promising techniques for producing thin and thick ceramic layers for both experimental and industrial applications. In EPD process, charged ceramic particles dispersed in a liquid medium migrate towards an electrode of the opposite charge and consequently get deposited there under the influence of a DC electric field. The process has advantages such as simple apparatus, low process costs, uniformity of the deposited layer, short formation time and etc [1,2]. In general, non-aqueous liquids are preferred to water as the suspending medium, because it eliminates the electrode reaction and gas evolution commonly encountered due to electrolysis of water on application of electric field [3]. However, as previously reported by Panigrahi et al. [3], different deposition microstructures are obtained from different organic media. In this paper, considering the numerous applications of TiO_2 films in photocatalysis, gas sensors, organic light emitting diodes and dye sensitized solar cells, the effect of two organic solvents, methanol and pentanol, onthe microstructure of the electrophoretically deposited TiO_2 films has been investigated.

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METHODS OF SAMPLE MANUFACTURING AND ANALYSIS

The organic solvents used in this study were purchased from Merck and used without further purification. Two suspensions of TiO_2nano-particles (Degussa P25) in methanol and pentanol with a concentration of 2 and 4 g/lit,repectivley, were prepared. Each suspension was first magnetically stirred for 24 h at 25 °C at the rate of 400 rpm and then sonicated for 15 min. The EPD set-up consisted of two copper electrode (2.5 × 1.5 cm) positioned at a distance of about 1cm and connected to a DC power supply. In order to evaluate the rate of deposition in each medium, deposition was carried out in different time intervals of 30, 60, 90, 120, 150 and 180 s atconstant applied voltage of 50 V.

In order to analyze the deposition pattern of the obtained layers, scanning electron microscope (Hitachi.S4160) was used. Also, the quantitative analysis of the thickness of the obtained deposit was explored using optical microscope (OM) BX61.

RESULTS AND DISCUSSION

In order to investigate the kinetic of deposition within the two organic media, the variation of deposition yield as a function of time was studied.The obtained results which are illustrated in *Fig. 1*, basically support Hamaker's linear model of the EPD described by the following equation [4]:

$$M = EtSC \tag{1}$$

The quantitative analysis over the thickness was carried out by optical microscope (cross section). The deposition time needed to obtain a thickness of about 7 μm is 20 and 160 secondsin methanol and pentanol suspention , respectively. It indicates that the electrophoretic deposition rate in methanol suspention is eight times higher than that in pentanol suspention.

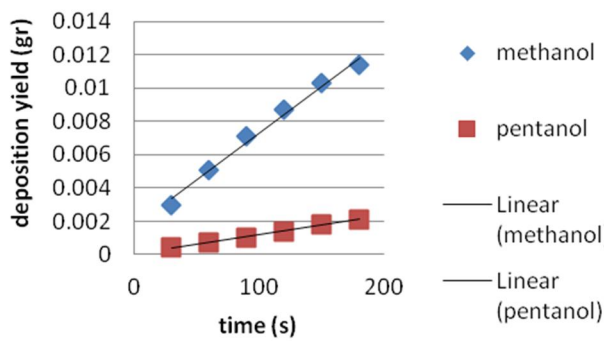

Fig. 1 – Deposition weight as a function of deposition time

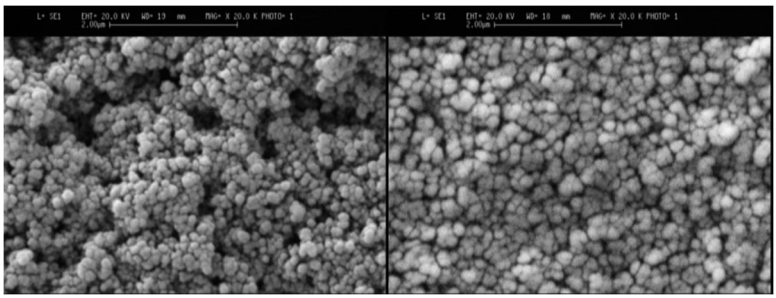

Fig. 2 – SEM images of nanocrystalline TiO_2 films formed in methanol (left) & pentanol (right).

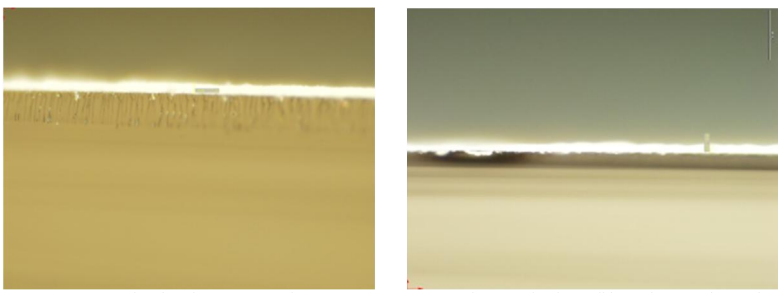

Fig. 3 – Optical microscope images (cross section) of TiO_2 films in methanol suspention (left, 7.9 μm thickness) and pentanol suspention (right, 7.05 μm thickness)

According to the Smoluchowski's equation [2], the electrophoretic mobility of the suspended particles depends linearly on the dielectric constant of the fluid and the zeta potential and is inversely proportional to the fluid viscosity. According to the presented data in *Table 1*, methanol is a less viscous medium with relatively higher dissociation power than pentanol, hence, the particles mobility in methanol is expected to be greatert than in pentanol which would result in a higher deposition rate.

Table 1 –Properties of the organic solvents [6].

Property	Methanol	Pentanol
Dielectric constant	32.6	13.9
Viscosity (mPa.s)	o.6	4
Conductivity (μS/cm)	3.4	0.2

From *Fig. 1*, the average deposition rates for methanol and pentanol were derived to be 78 and 12 micrograms per second, respectively.

Although the effect of electrophoresis parameters such as voltage on deposition pattern has been studied by many researchers, few papers have focused

on different microstructures and deposition rates obtained at different organic media. In present study, we did not measure the mobility, however, the significant difference observed in deposition rates and suspension conductivities obviously indicate a higher electrophoretic mobility for TiO_2nano-particles in methanol. At high deposition rates and low viscosities, the rapid attraction of suspended particles towards the oppositely charged electrode gives them little chance to be assembled in a close-packed structure. Panigrahi et al.reported high deposition rate and an uneven microstructure for doped ceria micropowder deposits formed in ethanol. In contrast, the deposition rate in butanol was small and smooth deposits were obtained. High rates of particles deposition result in the random assembly and consequently the formation of nonuniform films. However, at lower deposition rates, which is a result of lower electrophoretic mobility and high viscosity, the particles have enough time to deposit on uncovered areas next to previously deposited particles to form a uniform structure. In addition, at low viscosities, large aggregates are formed over time due to frequent particles collisions and the inherent tendency of nanoparticles to reduce their surface energy through agglomeration. Thus, it is difficult for large aggregates to deposit in a closepacked structure and a nonuniform microstructure would be obtained.

CONCLUSIONS

Electrophoretic deposition of TiO_2nano-particles from methanol and pentanol-based suspensionsresulted in the formation of different patterns. The layer obtained in methanol was characterized to be porous and non-uniform, however, deposition from the TiO_2/pentanol cell resulted in the formation of arelatively uniform microstructure. The difference is attributed to much higher deposition rate of TiO_2nanoparticles in methanol as well as the formation of large aggregates within the medium over time.

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