Effect of PEG addition on the microstructure of TiO_2 coating on unglazed ceramic tile

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ABSTRACT – The effect of PEG on the microstructure of TiO_2 coating prepared by sol-gel dip-coating on unglazed ceramic tile was investigated. TiO_2 films, without and with PEG were deposited on unglazed ceramic tiles. The crystalline phase of the coating was characterized using Glancing X-Ray Diffraction (GAXRD), while the coating morphologies and thickness were analyzed using Scanning Electron Microscope (SEM). Results showed a thicker film with presence of anatase and rutile phase with the addition of PEG. It also reduces cracks but induces pores. Furthermore, anatase to rutile transformation was suppressed, maximizing the formation of anatase.

1. INTRODUCTION

Among different photocatalysts, TiO_2 has received special attention due to the stability of its chemical structure, biocompatibility, and optical properties as stated by Lončarević et. al [1]. TiO_2 can exist in three crystallographic forms; anatase, rutile and brookite. Anastasescu et. al [2] and Guo et. al [3] found that the most used among TiO_2 crystalline phases is the anatase phase due to its high photocatalytic activity.

The sol-gel method is a well-known and suitable method for the preparation of crystalline TiO_2 thin film. The advantages of this method are; good homogeneity, strict composition control, and low equipment cost [2]. Moreover, in depositing TiO_2 coating on a substrate, dip-coating is a preferable method as it is time saving, low cost, and produces high film uniformity [4-5]

Ceramic tiles are commonly used as building materials, it is an advantage should they contain parasitic and bacterial inhibitors. Thus, TiO_2 coating has the potential for this purpose. However, previous study by Lopez et. al [6] has indicate poor adhesion of the TiO_2 coating on ceramic tile where the coating peels off and cracks were observed. Therefore, adding additives to act as binders might be a promising solution in producing a microstructure that is crack free, henceforth improving its adhesion.

In this work, the effect of PEG addition acting as a binder on the microstructure of TiO_2 coating was investigated.

2. METHODOLOGY

TiO_2 coating were prepared by using titanium(IV) isopropoxide (TTiP) (Sigma Aldrich Co.), ethanol, hydrochloric acid (37% HCl) and deionized water as titanium precursor, solvent, catalyst and hydrolysis medium respectively. The preparation of the sol-gel was exactly as in previous study [7]. Then, 2.5g of Degussa P25 was added as the anchor for the growth of TiO_2. To study the effect of PEG, PEG2000 was added prior ageing process as previous study [2]. Then the sol was aged for 48 hours.

For coating deposition, the unglazed ceramic tiles were prepared inhouse similar to the study by Ayoob et. al [8], (20mm x 10mm x 5mm) with average surface roughness of 3.72µm were utilized. The dip-coating procedure was carried out via mechanical dip coater set at 0.5mm/s dipping speed. Heat treatment applied on the films was at 500'C for 1 hour.

The coating crystallinity was recorded by Glancing Angle X-ray Diffusion (GAXRD) in the 2θ range of 20˚-75˚ at a grazing angle of 4˚ Co (λ = 1.79026 A) and radiation operated at 160 mA and 45 kV. Coating morphology was examined by using Scanning Electron Microscope (SEM) (Hitachi S-4500).

3. RESULTS AND DISCUSSION

Figure 1 shows the XRD patterns of TiO_2-PEG films deposited on unglazed ceramic tiles. The patterns show all samples contain both anatase and rutile crystalline phase. Although both were subjected to the same thermal treatment, the obtained results revealed that they showed different phase intensity at 2θ of 25.4˚. TiO_2 coating with PEG2000 showed higher anatase intensity than without PEG. As stated in previous study [1,3], addition of PEG inhibits the formation of rutile phase and maximizes the formation of anatase.

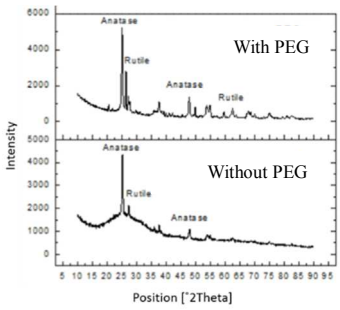

Figure 1: XRD analysis of TiO_2 coating on unglazed tile.

Figure 2 shows the surface morphology of TiO_2 coating without and with PEG2000 on unglazed tile. As shown in Figure 2(a), the film formed without PEG2000 showed cracking whereas film with PEG2000 showed no cracks (Figure 2(b)).

The cracking of the coating without PEG is mainly caused by thermal stress generated by capillary pressure of the films during heating stage of the firing process. Moreover, the cracking is due to the thermal stress between substrate and coating which contributes to formation and propagation of microcrack [6].

On the other hand, coating with PEG showed no cracks despite the presence of pores. The decomposition of PEG when calcined at 500˚C causes CO_2 trapped within the film, thus creating pores. The similar result are reported as previous study [2,3,9] which the decomposition of PEG had led to creation of pores. Anastasescu et. al [2] reported that the porosity of the film will contribute to a larger surface area needed for photocatalytic activity purposes. However, the influence of pores towards the adhesion is yet to be studied.

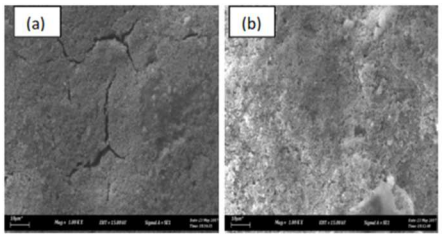

Figure 2: SEM image of TiO_2 coating on unglazed tile (a) without PEG2000, (b) with PEG2000

Figure 3 shows the cross-section of TiO_2 coating without and with PEG2000. The surface of the film with PEG is smoother whereas without PEG the film is rougher with jagged microstructure. This is due to the delamination and cracking of the coating creating uneven surface of the coating.

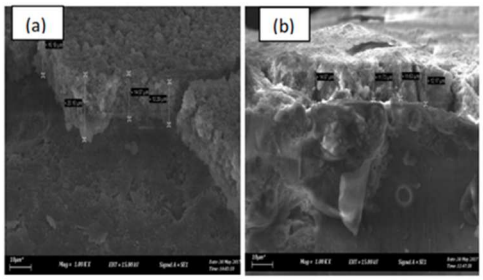

Figure 3: SEM image of cross-section of unglazed tile (a) without PEG2000, (b) with PEG2000

4. CONCLUSION

Cracks on TiO_2 coating were not seen when PEG was added. Decomposition of PEG during calcination led to generation of pores, thus improving the textural properties compared to TiO_2 coating without PEG, where no cracks and smooth surface of the coating were produced. Also, the addition of PEG had suppressed the transformation of anatase to rutile thus maximized anatase formation. Further study is to be conducted on evaluating the adhesion properties of TiO2 coating with PEG addition.

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