Final Report

Mineralization of organic compounds over 1D nanostructure Ti-O photocatalyst

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King Mongkut's Institute of Technology Ladkrabang

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Abstract

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Project Title : Mineralization of organic compounds over 1D nanostructure Ti-O photocatalyst

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Abstract:

The aim of the present work is to enhance the photocatalytic antibacterial performance of plastics according to JIS Z 2801:2010 standard and their mechanical properties by studying: (i) the influence of calcination for TiO_2; (ii) modified with different TiO_2 concentrations; (iii) effect of silane as a coupling agent. Acrylonitrile-Butadiene-Styrene plastics (ABS) and *Escherichia coli* (*E. coli*) were chosen as a model of plastic and bacteria, respectively. The 500°C calcined TiO_2 had successfully provided the best photoantibacterial activity with an approximately 62% decrease of *E. coli* colonies upon 30 minutes of exposure. Heat treatment could improve the crystallinity of anatase TiO_2, resulting in low electron-hole recombination while effectively adsorbing reactants on a surface. However, the aggregation of TiO_2 particles could occur at a high temperature (800°C) leading to the recombination and subsequently, decreasing the efficiency. ABS with 500°C calcined TiO_2 at the concentration of 1%wt had given rise to the highest performance due to their proper distribution. At this point, blending silane as a coupling agent with TiO_2 could improve efficacy of photoantibacterial activity of material up to 75% (remaining bacteria survivor 25%) due to greater interactions with polymer matrix. Moreover, mixing TiO_2 in ABS was found to influence yield strength enhancement of workpiece simultaneously due to its benefits in creating temporary crosslinks between the polymer chains during the deformation process. Treating with silane could promote a 1.5 times increase of yield strength via more adherent bonding between TiO_2 and ABS matrix. Excellent photocatalytic and material stability can be achieved as constant photocatalytic efficiency up to 5^{th} reuse cycle without loss in the yield strength.

Keywords: Titania photocatalyst, Photocatalytic degradation, Disinfection, APTES treatment, Photocatalytic inactivation, Photokilling

Executive summary

The thermoplastic polymers is widely used with a board area in everyday life for appliances such as sanitary ware, medical appliance, furniture and children toys due to favorable properties such as excellent impact resistance, good machinability, excellent aesthetic qualities and good machinability (Forrest, 2004). The durable plastics with a useful life of three years or more such as ABS could be potentially colonized by myriad microorganisms. Therefore, one of the biggest problems is microorganism contamination such as viruses, fungi and bacteria which are harmful to the environment, hazardous to humans and difficult to be disinfected.

Photocatalysis has attracted considerable attention in solving the bacterial contamination as a clean, energy incentive, low-cost and environmentally friendly technology. The photocatalytic bacterial inactivation has been proposed that upon the generation of highly reactive short-lived hydroxyl radicals and oxygen reactive to efficiently damage cell membrane of microorganisms. The TiO_2, is widely used for photocatalytic applications due to its high stability, low cost and non-toxicity (Gaya and Abdullah, 2008; Hashimoto et al., 2005). For example, Ti-O nanostructures synthesize from TiO_2 alkaline hydrothermal have the advantages in enhancing photocatalytic reaction and sedimentability (Kiatkittipong and Assabumrungrat, 2017). TiO_2 coated mortars had a much elevated removal robust weathering resistance (Guo et al., 2016). Coating TiO_2 on limestones yielded photocatalytic surface with high self-cleaning (Calia et al., 2017).

Coating TiO_2 on polymers such as polypropylene (Chawengkijwanich and Hayata, 2008; Maneerat and Hayata, 2006) , polyethylene terephthalate (Kanazawa and Ohmori, 2005), polycarbonate (Ratova et al., 2014), and polystyrene (Loddo et al., 2012) had been proposed to reduce the colony of *E. coli*. The properties of TiO_2 such as crystallinity, surface defect, and particle size may affect to the photocatalytic activities. However, flake off of TiO_2 is a limited issue when the polymer is used in many times. Apart from microorganism inhabitant, awareness of the mechanical properties of the thermoplastic polymers is another concern. Many studies had reported that the filler adhesion and dispersion on the matrix of polymers were the main factors. (Selvin et al., 2004)

Although many studies had demonstrated the development of thermoplastic polymers on either restraining microorganisms or mechanical properties, there is limited assessment regarding the role of TiO_2 on the antibacteria and the strength of plastic. In particular, there is no previous study available on ABS plastic which provides effective and quality products for various applications. Moreover, the impact of calcination and fillers on TiO_2 properties and its potential photoantibacteria have not been investigated off late. This study focuses on improving photoantibacterial performance following the standard JIS Z 2801 presenting the ability of plastic surfaces to inhibit the growth of microorganisms, and strength properties (ASTM D639 Type I) on ABS plastic with modified TiO_2. The role of calcination temperature, concentration and silane- coupling agent is as well assessed.

Objective

1. Study the role of TiO_2 on the antibacteria and the strength of plastic.
2. Study the impact of calcination and fillers on TiO_2 properties and its potential photoantibacteria

Research methodology

1. Reagents

TiO_2 (anatase 100%), Acrylonitrile-Butadiene-Styrene (ABS Resin, PA - 717C Grade), 3-Aminopropyltriethoxysilane (APTES) (Sigma-Aldrich), stock Phosphate Buffer Solution (PBS) (Sigma-Aldrich), Plate Count Agar (PCA), Tryptic Soy Broth (TSB), Peptone (J.T.Baker) were used.

2. Calcination of TiO_2 powder

Anatase TiO_2 particles was preheated in an oven at 90°C for 2 h, and then calcined at temperatures of 300, 500 and 800 °C for 2 h with a ramping rate of 5°C/min

3. Surface modification of TiO_2 with silane

A 0.5 g of calcined TiO_2 was dispersed in 50 ml of 2.5%v/v ethanol. Then, the silane coupling agents (APTES) were added in the dispersion, and stirred for 45 min. The resulting slurry was centrifuged and dried in an oven for 24 h at 80 °C

4. Preparation of TiO_2/ABS compositions

Prior to blending polymers, ABS plastic was dried in an oven at 90 °C for 2 h to remove moisture. The TiO_2 or calcined TiO_2 particles (concentration of 0.5, 1 and 2 wt%) or calcined TiO_2 treated with silane and ABS plastic was loaded into a twin-screw extruder, and then mixed well in an internal mixer at 250 °C with speed round mixing 60 rpm for 6 min. The melting material was put in the mold (5×5 cm^2) by using a compression molding at temperature of 250°C and pressure of 125 kg cm^{-3} for 5 min, and rapidly cooled for 5 min. The resulting TiO_2/ABS compositions were air-cooled at room temperature before being tested.

5. *E. coli* bacteria preparation and photoantibacterial activity

Photoantibacterial activity and efficacy assessments were following the standard JIS Z 2801 (The test for antibacterial activity and efficacy to bacteria on surface of antibacterial products). A colony of *E. coli* bacteria was transferred into TSB solution, and then incubated at (32 ± 0.5) °C for 24 h. The bacterial suspensions were diluted into 2.5×10^8 cfu/ml, dropped on TiO_2/ABS workpieces, and covered with a polyethylene (PE) film size 4×4 cm^2. The sample was illuminated by UVC light (15 W) for 30 min. The bacteria population was determined by plated serial dilution, and incubated at 32 ± 0.5 °C for 24 h

6. Sample Characterization

Crystal and structural characteristics of the products and crystallinity were investigated by powder X-ray diffraction (XRD) system with monochromatized $Cu_{k\alpha}$ radiation (λ = 1.5406 Å). Full width at half maximum (FWHM) derived from XRD pattern at 2θ =25° presents the degree of crystallinity. Sample morphology was investigated by a scanning electron microscope, and the surface area and pore size distribution was determined by N_2 adsorption.

7. Mechanical tensile strength

The workpieces were tested by an universal testing machine according to the guidelines set in ASTM D639 Type I (Standard Test Method for Tensile Properties of Plastics).

Results and discussion

1. Effect of Calcination Temperatures on TiO_2

The crystal structure of TiO_2 was investigated by XRD analysis as shown in Fig. 1. The XRD pattern demonstrated the structures of TiO_2 were influenced by calcination temperatures. The starting TiO_2 confirmed the presence of peaks at 2θ = 25.4°, 37.8°, 48°, 54.5° and 62.8°, corresponding to the anatase phase of TiO_2 with the FWHM value of 0.5. There was no peaks of impurities was observed. TiO_2 calcined at temperature of 300°C showed that the intensity of crystallinity of anatase increased with a lower FWHM value of 0.41. A further increase in temperature to 500°C and 800°C had promoted anatase with FWHM value of 0.27 and 0.08, respectively. The decrease in the FWHM value indicated an increase of the degree of crystallinity. Therefore, crystalline structure was slightly developed by increasing calcination temperatures.

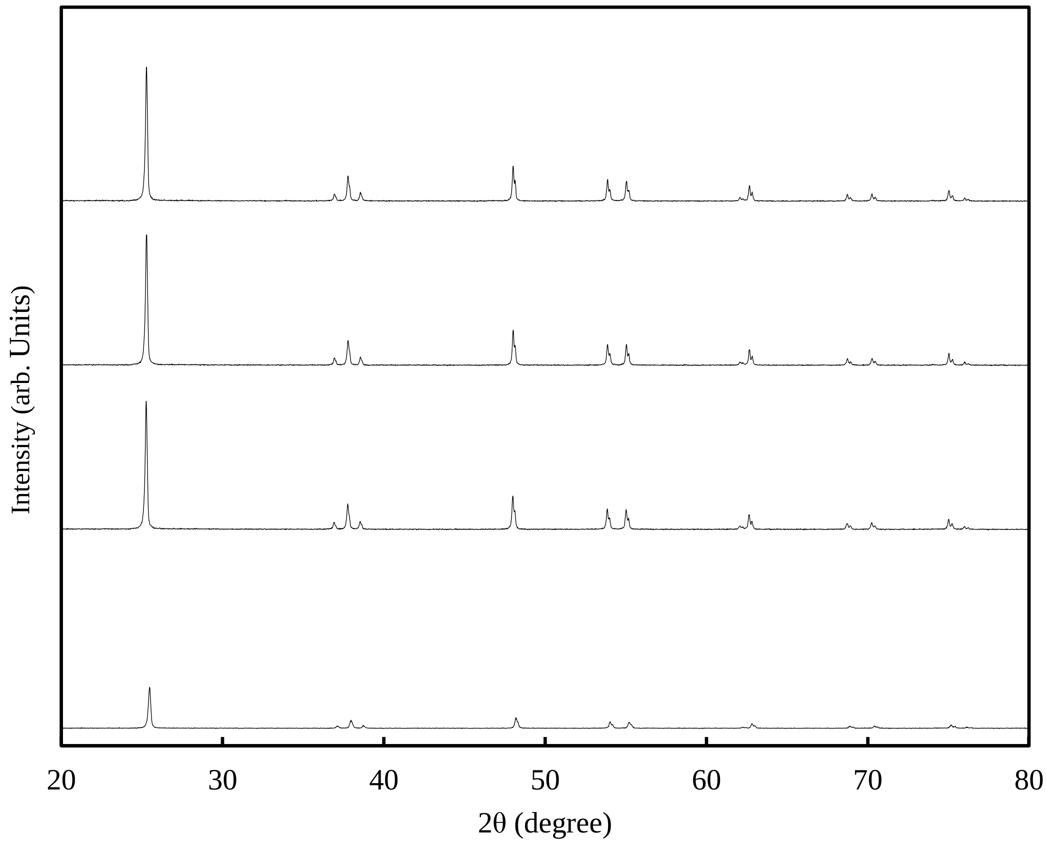

Fig. 1 XRD pattern of uncalcined TiO_2 and TiO_2 calcined at different temperatures

The initial surface area of TiO_2 was 10 m^2/g. At 300°C, the specific surface area was reduced to 9 m^2/g. A continuous decrease in surface area with increasing calcination temperature was observed at 500°C and 800°C calcined TiO_2 with the respective surface area of 7 and 4 m^2/g. The influence of calcination temperature on morphology was investigated by SEM images as presented in Fig. 2. The starting TiO_2 had a diameter of approximately 200 nm as shown in Fig. 2(a). When mixing TiO_2 particles with ABS plastic, the particles appeared to be on the ABS plastic, resulting in the rough surface as observed in the SEM image (Fig. 2(b)). Calcining at 300°C had led to structural aggregation of TiO_2 particles (Fig. 2(c)), whereby the surface of ABS plastic was observed to be assembled in some area (Fig. 2(d)). At 500°C, it was also observed that a continuous aggregation in TiO_2 particles occurred (Fig. 2(e)) and assembled on the ABS surface (Fig. 2(f)). As the temperature was increased to 800°C the TiO_2 particles became obviously aggregating as seen in Fig. 2(g). This had resulted in poor distribution of TiO_2 particles on ABS plastic as shown in Fig. 2(h).

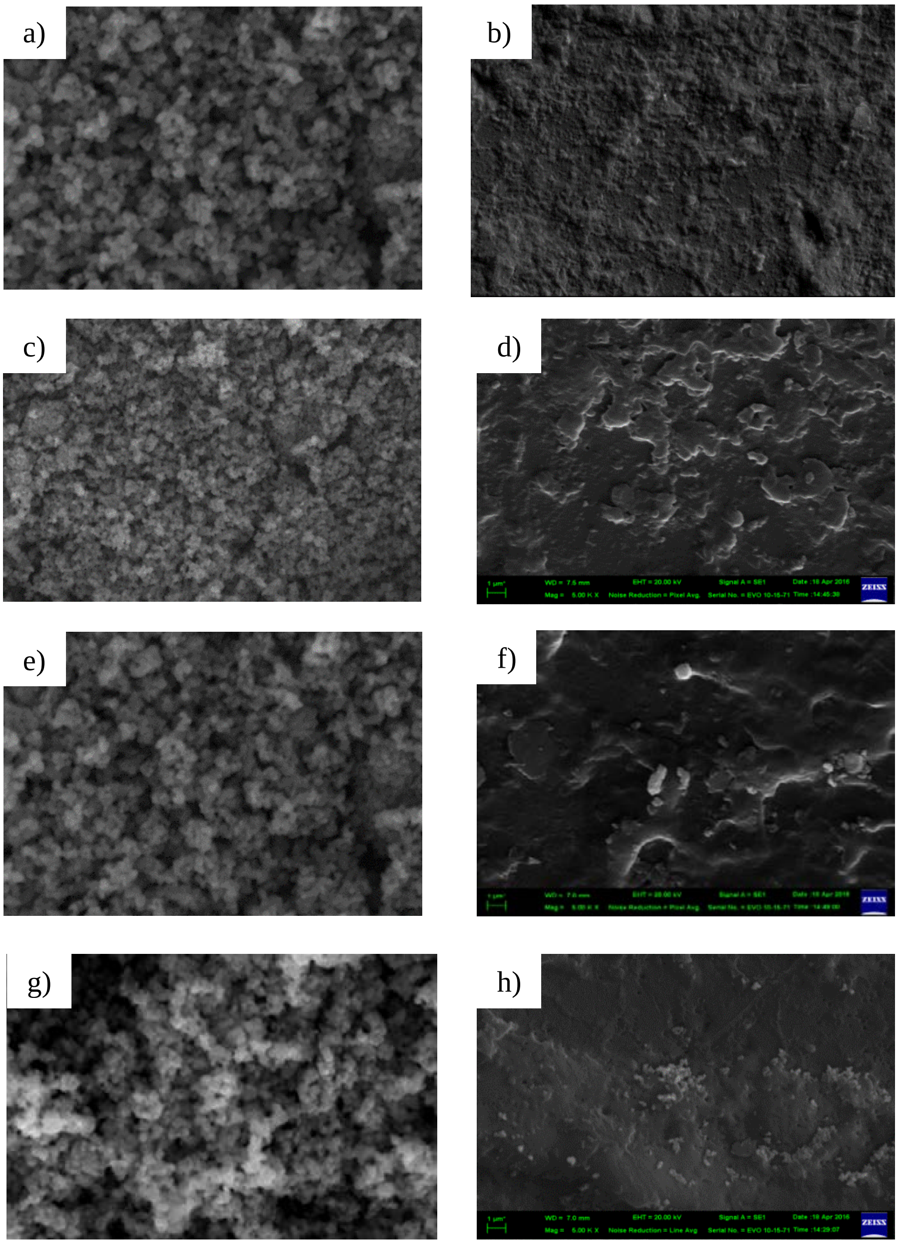

Fig. 2 SEM images of (a) TiO_2, (b) TiO_2/ABS, (c) 300 °C calcined TiO_2, (d) 300 °C calcined TiO_2/ABS, (e) 500 °C calcined TiO_2, (f) 500 °C calcined TiO_2/ABS, (g) 800 °C calcined TiO_2, (h) 800 °C calcined TiO_2/ABS.

2. Effect of TiO_2 with and without calcination on photoantibacterial activity and yield strength of ABS

The influence of TiO_2 and calcined TiO_2 mixed with ABS on photoantibacterial *Escherichia coli* (*E. coli*) by JIS Z 2801: 2010 standard test are illustrated in Fig. 3 Photoantibacterial for *E. coli* was presented in a form of bacterial survival. The result for a plain ABS under UV illumination for 30 min showed little antibacterial activity, while the *E. coli* survival of 60% was observed in ABS with TiO_2, highlighting the dominant impact of TiO_2 in photocatalytic activity. The mechanism of photocatalytic antibacterial proposed that the OH radicals and reactive oxygen species (ROS) generated by TiO_2 would damage the cell membrane, resulting in the leakage of bacterial cytoplasm that leading to death (Fagan et al., 2016; Podporska-Carroll et al., 2015; Ratova and Mills, 2015). Moreover, the calcined TiO_2 mixed with ABS provided a greater reduction of *E. coli* as compared with uncalcined TiO_2/ABS. Calcining TiO_2 at 300°C could decrease *E. coli* on ABS by 45% (remaining bacteria survival 55%), and further decreasing to 60% at 500 °C (remaining bacteria survival 40%), despite a decrease in surface area. The photocatalytic improvement could be plausibly explained by a higher degree of crystallinity as mentioned earlier. That is, more active crystal phase was improved and surface defects were reduced as documented by several studies (An et al., 2008; Yang et al., 2009; Yu et al., 2006; Zhou et al., 2010). However, when increasing calcination temperature up to 800°C, the performances of photoantibacterial decreased. This may derive from sintering and agglomeration effects during calcination at high temperature as presenting in SEM (Fig. 2).

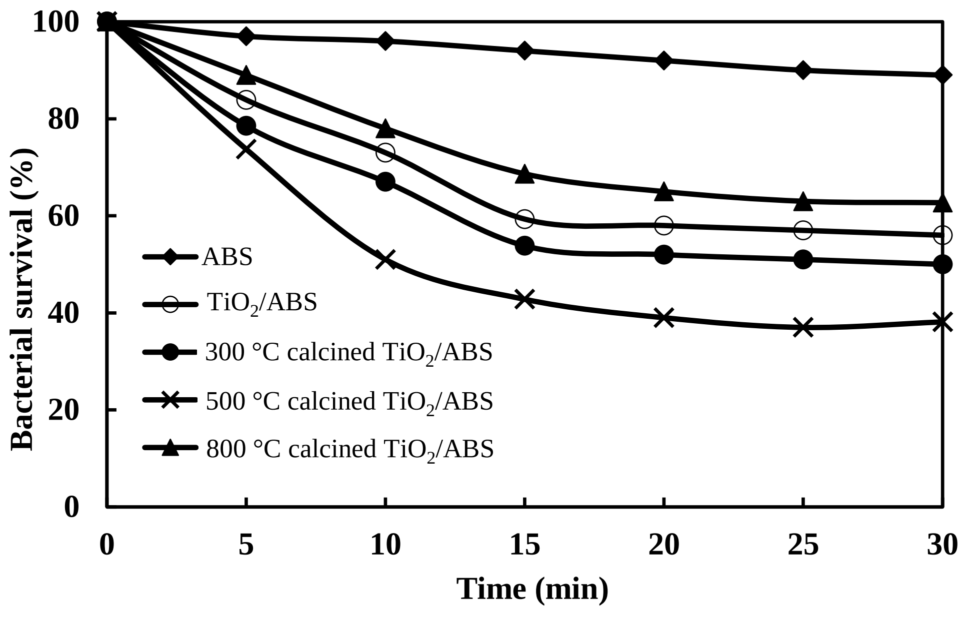

Fig. 3 Bacterial survival by photocatalytic reaction of ABS, TiO_2/ABS and 300 °C -800°C calcined TiO_2/ABS.

Figure 4 shows that the yield strength of plain ABS was 16.7 MPa, which was in a standard range of mechanical property of ABS. ABS containing TiO_2 could improve the yield strength of the workpiece. The enhancement of strength could be explained by TiO_2 creating temporary crosslinks among the polymer chains during the deformation process. In particular, uncalcined TiO_2/ABS showed the highest values of yield strength at yield point 17.2 MPa. A slight decrease of yield strength was observed when increasing calcination temperature of TiO_2. The yield strength of 300 °C TiO_2/ABS, 500 °C TiO_2/ABS and 800 °C TiO_2/ABS was 17.1, 17.0 and 16.9, respectively. The decrease of yield strength may attribute to a more agglomeration and reduced surface area of TiO_2 with increasing calcination temperature as seen from SEM (Fig. 2), which affected to less crosslinks among the polymer chains.

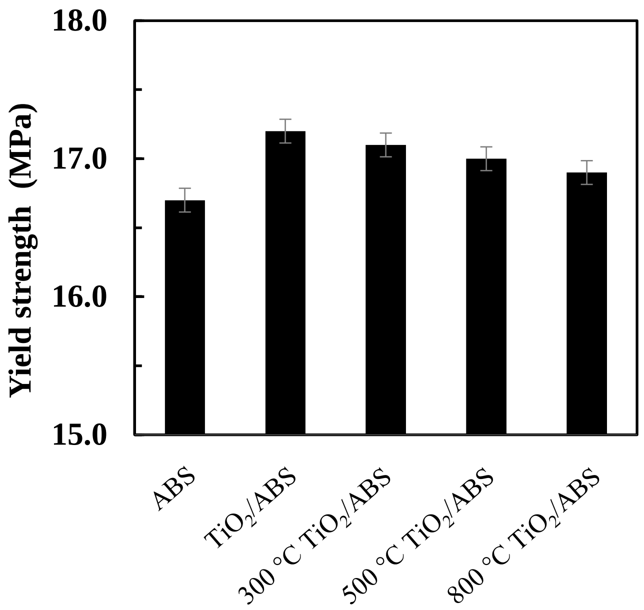

Fig. 4 Yield strength of ABS, TiO_2/ABS and 300 °C -800°C calcined TiO_2/ABS.

3. Effect of concentration of calcined TiO_2 on photoantibacterial activity and yield strength of ABS

The previous section demonstrates that optimum performance on photocatalytic performance occurred at 500°C for calcined TiO_2/ABS. In this part, the influence of 500°C calcined TiO_2 concentration in ABS on photocatalytic performance was considered. From the 40% bacterial survival by 1%wt calcined TiO_2/ABS, changing of TiO_2 concentration had been considered as shown in Fig. 5. The reduced concentration of TiO_2 had resulted in the increase of bacterial survival by 50%. This indicated that the less amount of TiO_2 was not enough to produce OH radicals and reactive oxygen species (ROS) to inactivate the bacteria. However, with the increasing concentration to 2%wt, the bacterial survival increases to 70%. This showed that high amount of TiO_2 did not always lead to the high photocatalytic activity, but may suppress the activity due to their aggregation instead.

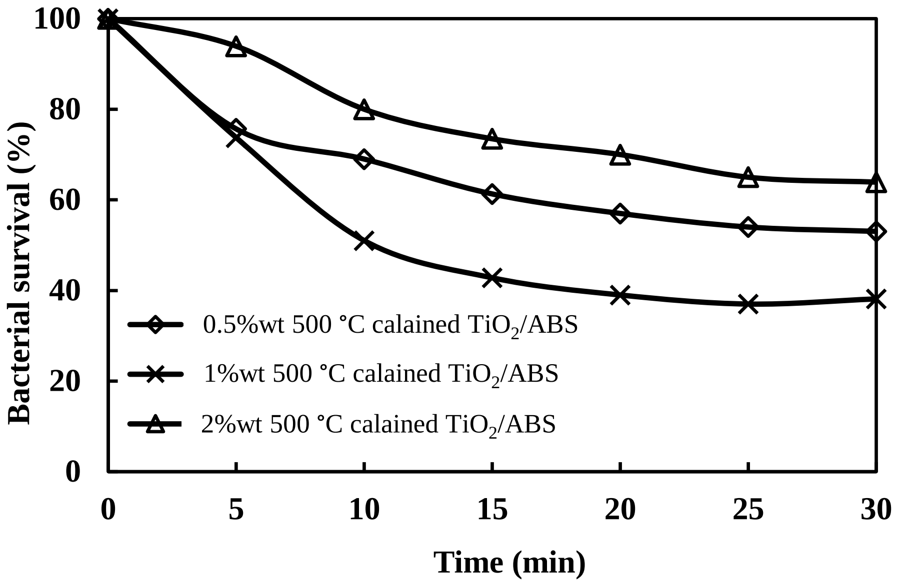

Fig. 5 Bacterial survival by photocatalytic reaction of ABS with 500°C calcined TiO_2 at different concentration.

Figure. 6 shows that yield strength decreased with increasing of TiO_2 loading. A 0.5 %wt TiO_2/ABS had the highest yield strength of 18.0 MPa. This was to be expected given the higher TiO_2 inducing the effective matrix reduction.

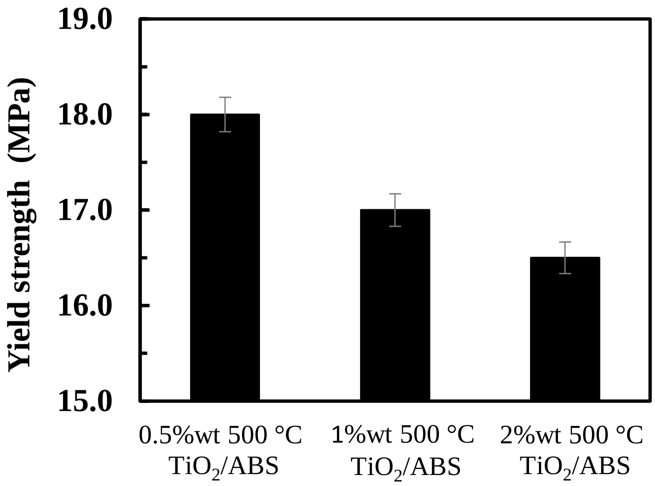

Fig. 6 Yield strength of ABS with 500°C calcined TiO_2 at different concentration

4. Effect of silane on photoantibacterial activity and yield strength of ABS

The influence of silane on TiO_2/ABS was observed in SEM images in Fig.7, in which the 500°C calcined TiO_2/ABS without silane possessed rough surface and small particles on ABS surface as illustrated in Fig. 7(a). After mixing silane, the morphology of blended polymer in Fig. 7(b) shows the presence of a smooth surface and better dispersion, rather than TiO_2/ABS. The difference was attributed to a greater interactions with polymer matrix which silane- coupling agent showed the swelling behaviour of ABS/TiO_2 by creating more adherent bonding between TiO_2 and ABS matrix, as similarly explained for the modified SiO_2 with silane (Li et al., 2007; Li et al., 2006; Xu et al., 2007).

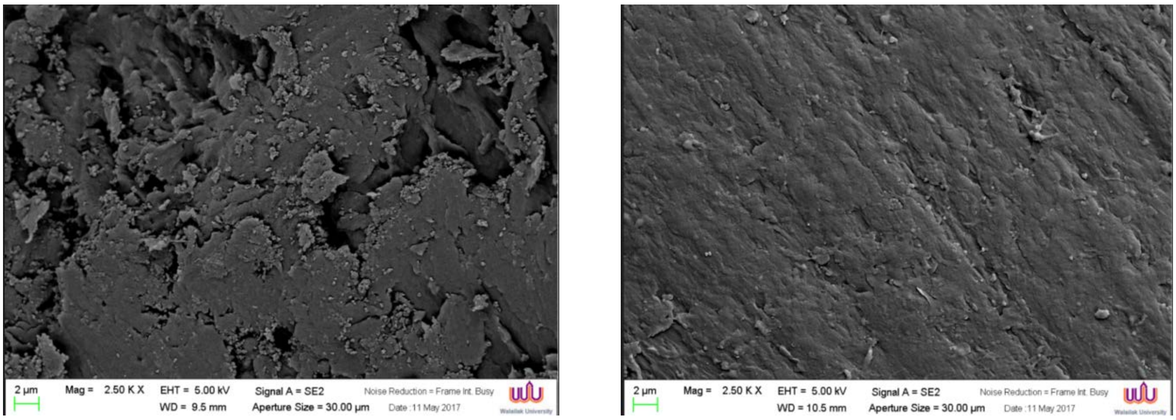

Fig. 7 SEM of (a) 500°C calcined TiO_2/ABS and (b) 500°C calcined TiO_2/ABS with silane.

Figure 8 reveals that 500 °C calcined TiO_2/ABS/silane displays a better photobacterial activity as compared with 500 °C calcined TiO_2/ABS without silane. The 500°C calcined TiO_2/ABS/silane could reduce 75% of *E. coli* (remaining bacteria survival 25%). The higher activity of TiO_2/ABS/silane on photobacterial activity attributed to the comparatively greater distribution of TiO_2 on ABS which promoted good UV absorption, however it was inconformity with the finding by Pazokifard, Sh. et al. (2012) (Pazokifard et al., 2012) reporting that in a case of degradation of Rhodamine TiO_2 P25 nanoparticle showed a better activity than silane-treated particles due to a reduced surface area of TiO_2 affecting poor photon absorption. The differences between the photocatalytic activity findings for bacteria and Rhodamine likely originate from the operating condition under blending composite and particle itself. The inset of Fig. 8 shows that the yield strength of TiO_2/ABS with silane was 1.5 times higher than TiO_2/ABS without silane. The yield strength improvement was ascribed by the better dispersion and adhesion of TiO_2 in ABS matrix which arising from silane coupling agent, making interfacial bonding between the TiO_2 and the matrix. We could highlight one key finding here that addition of silane results in improvement of TiO_2 particles dispersion on matrix (as higher photocatlytic activity) and increasing possible interactions between TiO_2 particles and ABS polymeric matrix (as higher yield strength).

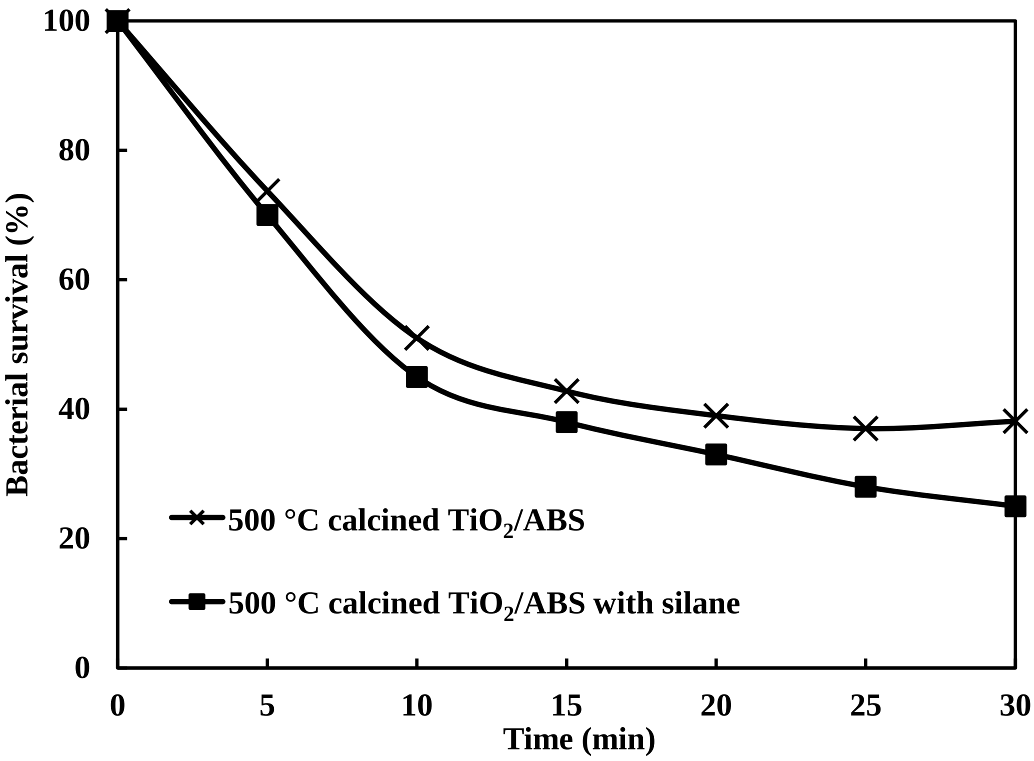

Fig. 8 Bacterial survival by photocatalytic reaction of ABS with 500°C calcined TiO_2 (1%wt loading) with and without silane and corresponding their yield strength (inset)

5. Reusability and robustness

The stability and reusability in term of photocatalytic activity and the robustness of the composite material are very crucial in practical application. Therefore, the antibacterial experiments were repeated without any treatment on the specimen between the cycle runs. Constant of photocatalytic efficiency was observed after 5^{th} reuse cycles without loss in the yield strength of material as shown in Fig.9. This presume that blending calcined TiO_2 into ABS preventing the loss of TiO_2 photocatalyst particles from the surface which usually observed when coating TiO_2 on the polymer surface.

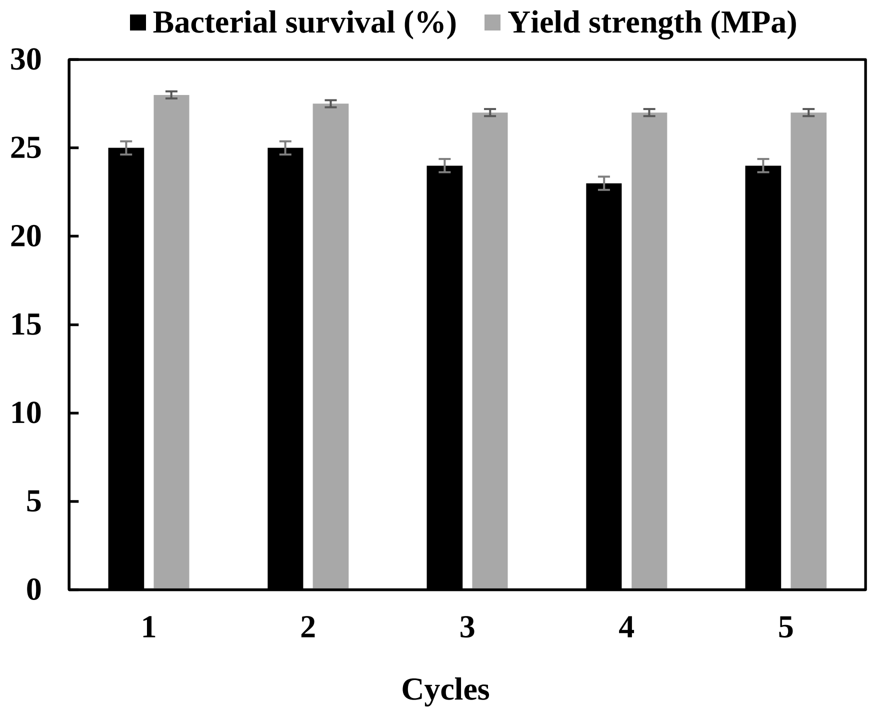

Fig. 9 Reusability of ABS with 500°C calcined TiO_2 (1%wt loading) with silane grafting upto 5th cycle in term of bacterial survival by photocatalytic reaction and yield strength of material (photo irradiation time of 30 min for each cycle)

Conclusion

Titanium dioxide (TiO_2) was found influencing antibacterial performance and yield strength enhancement when subject to Acrylonitrile-Butadiene-Styrene plastics (ABS). The optimum photoantibacterial activity occurred for ABS with the 500°C calcined TiO_2 at concentration of 1%wt. At this temperature and concentration, high degree of crystallinity and proper amount of TiO_2 was sufficient to produce OH radicals and reactive oxygen species (ROS), resulting in the damage of bacterial cell membrane. The photoantibacterial performance for 500°C calcined TiO_2 at 1%wt in ABS was more efficient than plain ABS over 62%. With an optimal condition, silane addition could further improve TiO_2 dispersion on ABS. This resulted in the decrease of bacterial survival by 75%. Moreover, the benefit of TiO_2 embedded ABS plastic could improve yield strength than plain ABS. The yield strength of TiO_2/ABS with silane was 67.7% higher than of plain ABS. The efficiency of TiO_2/ABS with silane photocatalyst showed an excellent photocatalytic antibacterial stability after five reuses without loss in the yield strength.

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Output

8.1 International Journal Publication

Kunlanan Kiatkittipong, Jun Wei Lim, Chin Kui Cheng, Worapon Kiatkittipong, Suttichai Assabumrungrat. Simultaneous enhancement of photocatalytic bactericidal activity and strength property of Acrylonitrile-Butadiene-Styrene plastic: Journal of Hazardous Materials (Impact factor 7.65), Under review

8.2 Application

For appliances restraining microorganisms or mechanical properties such as sanitary ware, medical appliance, furniture and children toys

8.3 International conference

Photocatalytic degradation of nitrate in water using TiO_2 and ZnO, TiChE Conference, 2020