

# SYNTHESIS OF TiO<sub>2</sub>-SiO<sub>2</sub> POWDER PHOTOCATALYST VIA SOL-GEL METHOD: EFFECT OF TITANIUM PRECURSOR TYPE ON POWDER PROPERTIES

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## ABSTRACT

TiO<sub>2</sub>-SiO<sub>2</sub> powder photocatalysts with varied SiO<sub>2</sub> portion were prepared through sol-gel technique using two types of metal alkoxides: titanium tetraisopropoxide (TPT) and titanium diisopropoxide (PTP). Tetraethoxysilane (TEOS) as the silicon precursor was partially hydrolysed at room temperature, followed by the dropwise addition of these two metal alkoxides to produce polytitanosiloxane (PTS) solution. Hexane filtration of the PTS solution followed by drying and calcination gave TiO<sub>2</sub>-SiO<sub>2</sub> powder. The physico-chemical characteristics of the obtained powders were investigated using TG-DTA, FTIR, XRD, and FESEM. Both types of powder particles were in nano-sized with spherical shape. The powder derived from PTP showed particle size smaller than that derived from TPT. From TGA analysis, it can be observed that the PTP derived powder start to crystallise at lower temperature compared to the TPT derived powder. However, TPT derived powder showed a stability of anatase crystal structure even after high temperature calcination.

**Keywords:** Sol-gel Method, Synthesis, TiO<sub>2</sub>-SiO<sub>2</sub> Photocatalyst, Titanium Precursor

## 1.0 INTRODUCTION

It is well established that particle size is one of the most important parameters to be optimised for improving the catalytic or photocatalytic properties of a semiconductor [1]. Small particles affect photocatalytic properties by increasing the number of active surface sites in contact with the organic pollutant molecules to be photocatalytically degraded. It also can lead to the less risk of charge carrier recombination [2; 3].

Nowadays, a number of promising synthesis routes available that can influence the physico-chemical properties of the TiO<sub>2</sub> photocatalyst (e.g., sol-gel [4; 5], chemical vapour deposition [6], liquid phase deposition [7]). Such physico-structural parameters include crystallisation degree, crystal shape, surface area, as well as the eventual presence of a secondary rutile phase [1].

The sol-gel process is one of the most potential technologies for the preparation of TiO<sub>2</sub> photocatalyst. It can be classified as a wet-chemical technique as it used chemical solutions (*sol*) as a precursor for an integrated network (*gel*) of either discrete particles or network polymer in order to fabricate materials (*typically metal oxide*). The particle size, morphology and porosity of poly-ceramic based materials are generally considered as governed by the kinetic factors of hydrolysis-polycondensation process [8].

Typical precursors used in forming sol are metal alkoxides and metal chlorides, which undergo various forms of hydrolysis and polycondensation reactions. The hydrolysis process occurs when the metal alkoxide react with water molecules as the hydroxyl ion becomes attached to the metal atom. The process may go to completion and may stop while the metal is partially hydrolyzed depending on the amount of water and catalyst

present. The rate of hydrolysis can be accelerated by employing acid or base catalysts. Polycondensation begins before hydrolysis completes. It happens when the partially hydrolysed molecules link together and liberates a small molecule, such as water or alcohol. Via this method, the microstructure of the gels (e.g. pore volume, pore size, and surface area) can be easily controlled by changing the reaction parameters such as the H<sub>2</sub>O to alkoxide ratio, hydrolysis procedure, *etc* [9]. The dopant can be easily inserted into the TiO<sub>2</sub> matrix via this method [10]. Besides, low cost uncomplicated equipment is used and no special precautions are needed to be taken in order to protect the starting products and precursor solutions since insensitivity of the reagents to air and humidity.

The physical and chemical properties TiO<sub>2</sub> crystallite size can be controlled by adding second semiconductor into the TiO<sub>2</sub> matrix. Silicon dioxide (SiO<sub>2</sub>) has been incorporated into the TiO<sub>2</sub> matrix to enhance the photocatalytic process [11-14]. Addition of SiO<sub>2</sub> content can suppress the crystal growth of TiO<sub>2</sub> grains [11; 13]. SiO<sub>2</sub> has high thermal stability, excellent mechanical strength and helps to create new catalytic active sites due to interaction between TiO<sub>2</sub> and SiO<sub>2</sub> [15]. A mixed metal oxide (TiO<sub>2</sub>-SiO<sub>2</sub>) can enhance the photocatalytic performance due to improving the ability of the surface adsorption and increasing surface hydroxyl group of the photocatalyst [14]. And at the same time, SiO<sub>2</sub> acts as the carrier of TiO<sub>2</sub> and helps to obtain a large surface area as well as suitable porous structure [5].

It was well proven that physico-chemical properties of TiO<sub>2</sub> photocatalyst varied with the type of metal alkoxide used as it shows different hydrolysis rate and polycondensation process. Several types of metal alkoxides have been used to produce

TiO<sub>2</sub> photocatalyst; titanium diisopropoxide [16; 17], titanium tetraisopropoxide [18-20], titanium tetra-*n*-butoxide [21-23], ammonium hexafluorotitanate [24], titanium tetrachloride [25-27], titanil sulphate [28-31], titanium oxyhydrate [14].

Peroxo titanic acid derived from TiCl<sub>4</sub> needs a lower temperature operation such as 0-5°C in the ice-water bath which is difficult to fit in the industrial process [25]. Titanium tetraisopropoxide which are mostly used as a precursor material for sol-gel process is generally highly reactive species [32]. To control the hydrolysis and polycondensation process in sol-gel route, it may be achieved through the addition of “modifiers”, such as β-diketones (*e.g.*, acetylacetonate), carboxylic acids (*e.g.*, acetic acid) or other complex ligands [32]. Titanium diisopropoxide is categorised as modified metal alkoxide due to the presence of acetylacetonate (acac) ligand; where the acac ligand is attached to the Ti element. It is known that the acac ligand structure can increase the stability of the metal alkoxide by functioning as a chelating agent [33]. Besides, the chemical modification of titanium alkoxide would lead to the production of well-defined powders at lower reaction temperature. This chelating ligand is used to control the condensation and the evolution of the polymer [34] and prevents the precipitation of undesired phase [32]. The acac group bonded to titanium prevents rapid hydrolysis by altering the condensation pathway [32; 35].

In this present study, we have synthesised the TiO<sub>2</sub>-SiO<sub>2</sub> powder via sol gel method using two different metal alkoxide: titanium diisopropoxide (PTP) and titanium tetraisopropoxide (TPT). The physico-chemical property of the TiO<sub>2</sub>-SiO<sub>2</sub> powder obtained has been also carried out to investigate the effect of different metal alkoxide use in synthesing the TiO<sub>2</sub>-SiO<sub>2</sub> powder.

## 2.0 EXPERIMENTAL

### 2.1 Materials

For preparation of PTS as precursor solution, titanium (di-isopropoxide) bis(acetyl-acetonate) (PTP, Strem Chemicals, 75% in isopropanol), titanium (IV) isopropoxide (TPT, Sigma-Aldrich), tetraethoxysilane (TEOS, Alfa Aesar, 98%), methanol and hydrochloric acid (System, 36%) were used as raw materials.

### 2.2 Preparation of TiO<sub>2</sub>-SiO<sub>2</sub> precursor solution

The PTS precursor solution was produced by mixing TEOS and 30ml of methanol into the round-bottomed flask with stirring condition. Into this solution, a mixture of 6N of HCl, distilled water and 30ml of methanol was added dropwise with 10minutes stirring process to partially hydrolyse TEOS. Then, into the partially hydrolysed of TEOS solution, PTP or TPT was

added dropwise. The molar ratio of HCl/TEOS, H<sub>2</sub>O/TEOS and PTP (or TPT)/TEOS were summarised in Table 1. The heating process was started and continued until the reflux temperature reached about 74°C. The heating was stopped after 10 minutes and PTS solution was finally obtained. This PTS solution was used to produce TiO<sub>2</sub>-SiO<sub>2</sub> powder via acetone dissolution and hexane filtration process. The procedures were summarised in the flowchart (Figure 1).

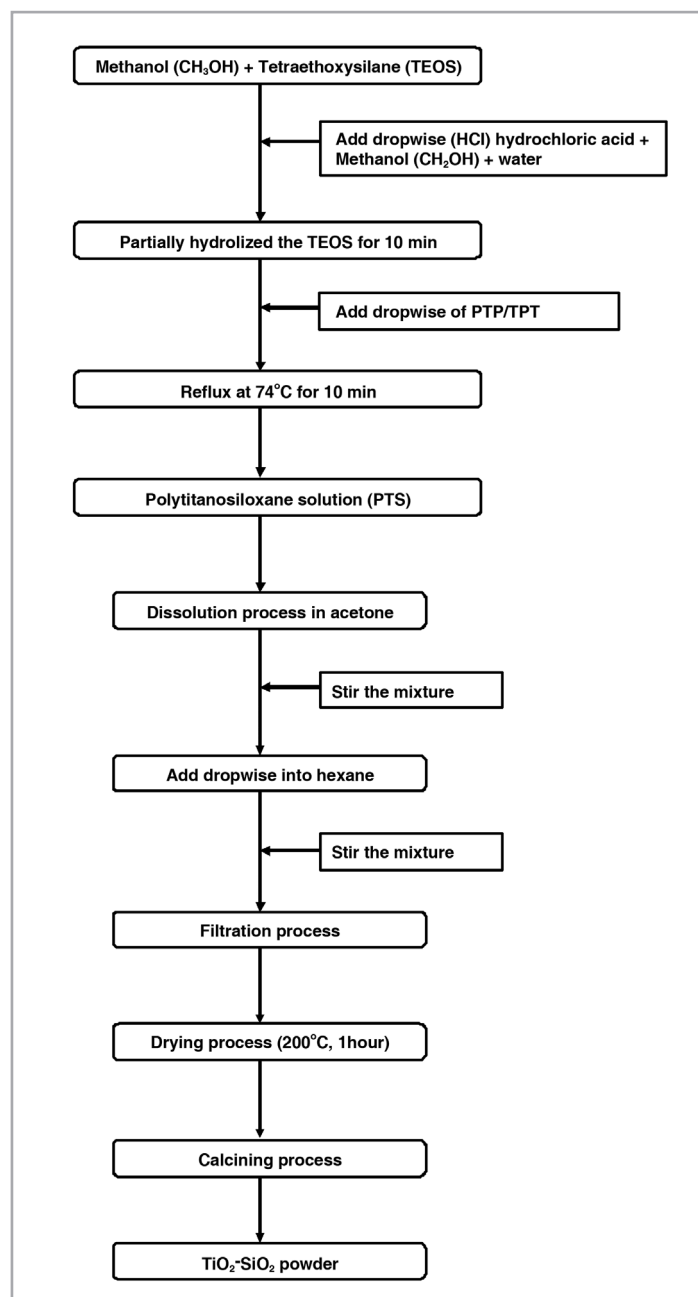


Figure 1: Scheme of TiO<sub>2</sub>-SiO<sub>2</sub> preparation via sol-gel method

Table 1: Weight composition of the raw materials used

Sample	TiO <sub>2</sub>	SiO <sub>2</sub>	PTP or TPT (mol)	TEOS (mol)	H <sub>2</sub> O/TEOS mol ratio	HCl/TEOS mol ratio	Methanol (ml)
P95/5	95	5	0.095	0.005	2	0.1	30
P90/10	90	10	0.09	0.01	2	0.1	30
P80/20	80	20	0.08	0.02	2	0.1	30
P70/30	70	30	0.07	0.03	2	0.1	30

### 2.3 Characterisation

Crystal structure of sol-gel  $\text{TiO}_2\text{-SiO}_2$  powder was characterized using Shimadzu Diffractometer, XRD 6000 model with copper  $K\alpha$  radiation. All samples were scanned between  $20^\circ$  and  $50^\circ$  with  $2.00^\circ/\text{min}$  scan rate. Physical properties such as size and shape of particle and surface morphology were characterised by using Field Emission Scanning Electron Microscope (FESEM) (JSM 6700F, JEOL). Differential and thermogravimetric analysis was carried out on the amorphous  $\text{TiO}_2\text{-SiO}_2$  powder in ambient air using Thermogravimetric and Differential Thermal Analyser (TG/DTA) (Pyris Diamond, Perkin Elmer) with a  $2^\circ\text{C}/\text{min}$  heating rate to determine the thermal properties of the sample. To examine the chemical bond that appeared in the sample, the IR spectra were recorded using Fourier Transmission Infra-Red (FT-IR) (Spectrum 100, Perkin Elmer).

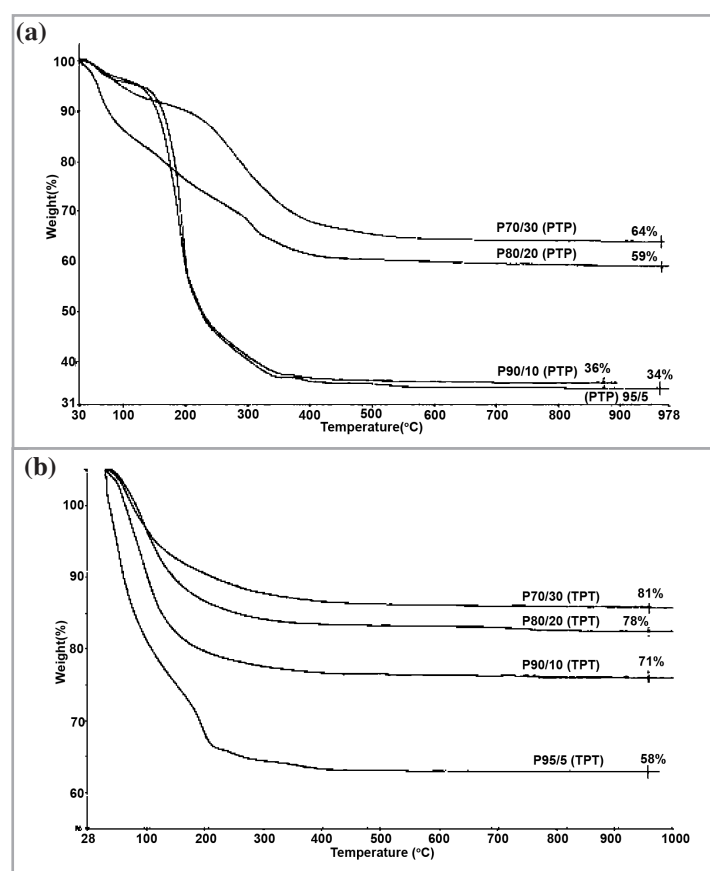


Figure 2: TG curve of  $\text{TiO}_2\text{-SiO}_2$  powder derived by (a) PTP and (b) TPT with different content of  $\text{SiO}_2$

### 3.0 RESULTS AND DISCUSSION

Thermal property of the mixed  $\text{TiO}_2\text{-SiO}_2$  powder was characterized by TG-DTA analyser. This characterisation step was done to determine the crystallisation temperature of the powder and to investigate the effect of addition second semiconductor into the  $\text{TiO}_2$  matrix towards its thermal property. Besides, the effects of different metal alkoxide on powder's thermal property were also studied. The amorphous powder was heated from room temperature until  $1100^\circ\text{C}$  with the rate of  $2^\circ/\text{min}$ . From the TG curve in Figure 2, it can be seen that the first weight loss occurs at temperature  $\sim 100^\circ\text{C}$  is attributed to the evaporation of water molecules. This weight loss continued with the second stage of weight loss at temperature of  $100\text{-}320^\circ\text{C}$ . It can be ascribed as

the decomposition of residual organic compounds contained in the metal alkoxide (acetylacetonate group) and organic solvent used (methanol). A further weight loss after  $350^\circ\text{C}$  is probably due to the densification process of amorphous  $\text{TiO}_2$  powder until it crystallises to anatase crystal phase.

From the Figures 2(a) and 2(b), it can be observed that different weight loss occurred for different  $\text{SiO}_2$  level.  $\text{SiO}_2$  has high thermal stability and excellent mechanical strength [15]. Besides, it can help to create new catalytic active sites due to interaction between  $\text{TiO}_2$  and  $\text{SiO}_2$  [15]. It showed that high composition of  $\text{SiO}_2$  leads to the less weight loss of sample during heating process. This observation may be attributed to the thermal stability of sample with high  $\text{SiO}_2$  composition (e.g., P70/30) compared to that of sample with low  $\text{SiO}_2$  composition (e.g., P95/5). The Si-O-Si bond is very bendable and flexible. When adding bulky group into the structure, the rigidity of the structure will increase. The bulky group also hindered the flexibility and raised the transition temperature. Since the portion of Ti in P70/30 is high comparable with the Si portion for polycondensation reaction, so that, more Si-O-Ti bonds are formed. This assumption is in good agreement with FT-IR result. Then, this would lead to the decrease in flexibility of structure and small weight loss occurred during heating process. Compared to the P95/5, less of Ti composition could not hinder the flexibility of the structure, so that higher weight loss occurred.

From the TG curve, it was observed that ca. 66 % weight of P95/5-PTP powder and ca. 42% weight of P95/5-TPT diminished after  $1100^\circ\text{C}$  heating. Figure 3 shows the predicted molecular structure of the polytitanosiloxane for both samples which is ladder type. The theoretical weight losses according to the molecular structure calculation are 69% and 34% for P95/5-PTP and P95/5-TPT powders, respectively. It can be therefore deduced that the polytitanosiloxane assume the ladder structures as shown in the figure.

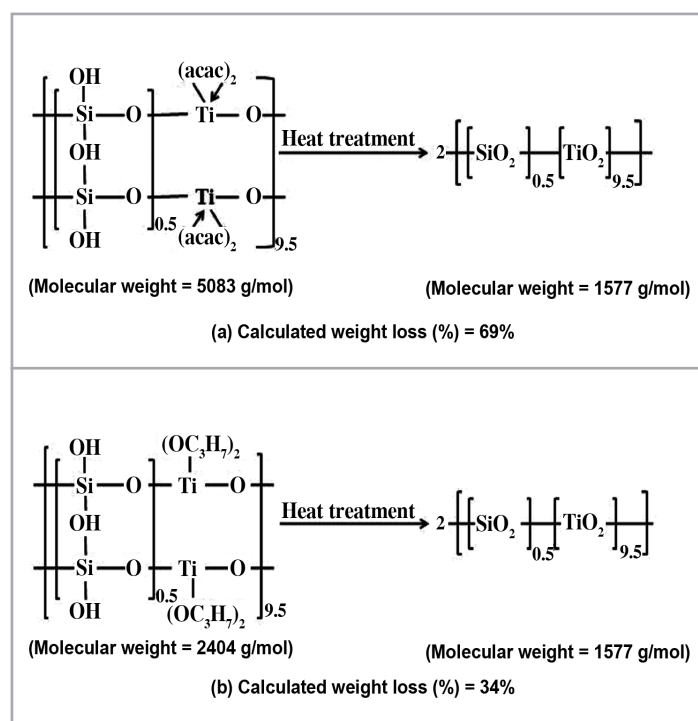


Figure 3: Possible molecular structure of polytitanosiloxane solution derived by (a) PTP and (b) TPT

As mentioned previously, the function of acetylacetonate as a chelating agent is to increase the stability of the metal alkoxide [32; 33]. As compared to the weight loss occurred in this two different types of powder (P95/5-PTP and P95/5-TPT), it can be observed that the P95/5 (PTP) showed higher weight loss during heating process, which is attributed to the disappearance of acetylacetonate ligands in the metal alkoxide.

From the DTA curve (Figure 4), the crystallisation temperature of all samples can be determined by indicating the exothermic peak appeared. The system released heat to allow the transition phase from amorphous to anatase crystalline phase. The effect of prolongation crystallisation temperature towards high temperature can be seen clearly when the SiO<sub>2</sub> level increased [13]. It showed that P95/5 (PTP) started to crystallise at lower temperature (378°C) than that of P70/30(PTP) (650°C). Similar phenomenon occurred on the TPT derived powder. The P95/5 (TPT) started to crystallise at lower temperature (428°C) than that of P70/30 (TPT) (604°C). It is confirmed that the blocking effect of crystallisation process existed on the powder which has high SiO<sub>2</sub> content.

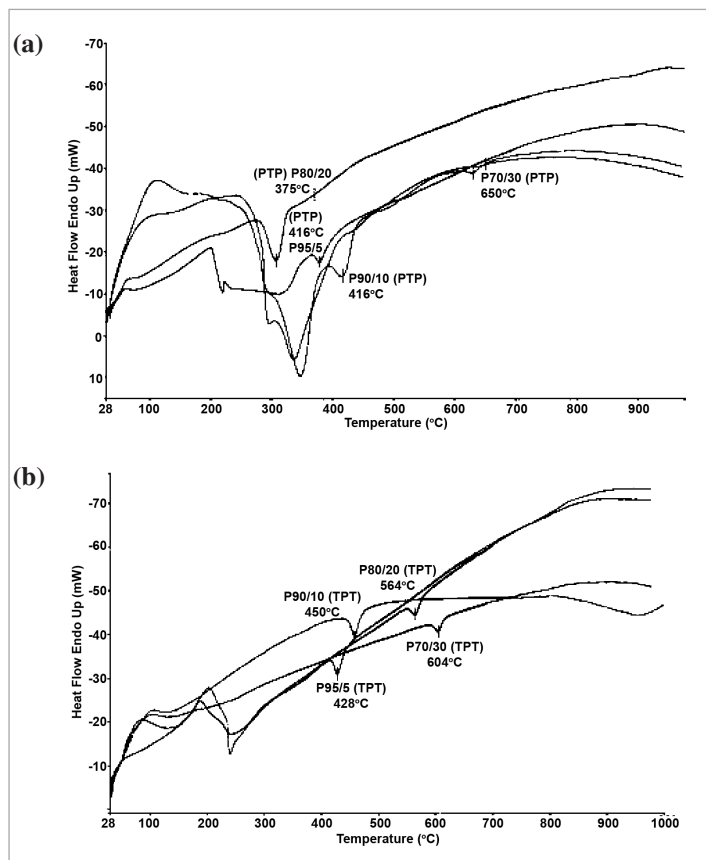


Figure 4: DTA curve of TiO<sub>2</sub>-SiO<sub>2</sub> powder derived by (a) PTP and (b) TPT with different content of SiO<sub>2</sub>

Another aspect which can be clearly observed on the thermal property of the powder is the difference in crystallisation temperature as the different metal alkoxide was used. Referring to the DTA curve in the Figure 4, it showed that the modified metal alkoxide solution (PTP) has lower crystallisation temperature compared to that of unmodified metal alkoxide solution (TPT). It may be attributed to the Si-O-Ti linkage in gel powder derived by PTP much higher than that of in TPT. This can be explained by the difference of hydrolysis rate between PTP and TPT. Since the PTP is the most difficult to be hydrolysed, the polycondensation reaction

continued faster during synthesis of the PTS solution using PTP instead of using TPT. This would lead to the more Si-O-Ti and Si-O-Si linkage in the polytitanosiloxane precursor solution that was derived by PTP as confirmed by FT-IR analysis. Therefore, the retardation effect was obvious in the PTP derived powder during calcination. This result is in good agreement with the XRD analysis.

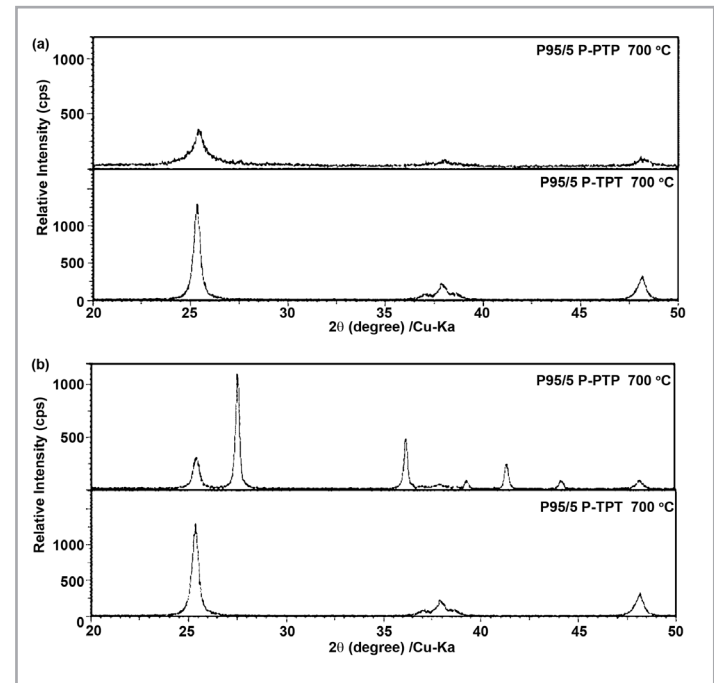


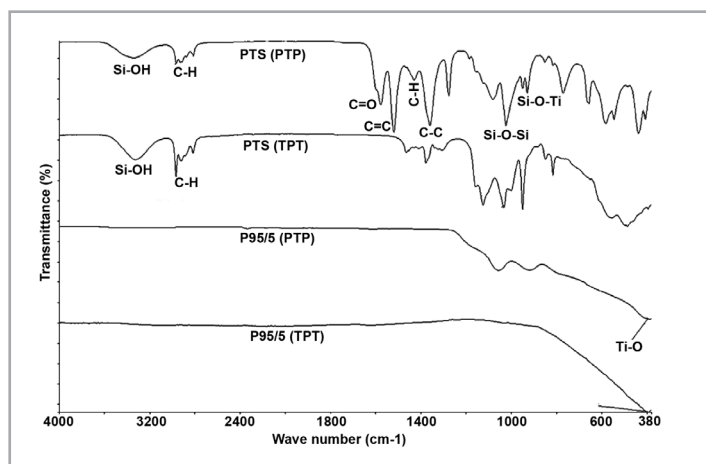
Figure 5: XRD pattern for TiO<sub>2</sub>-SiO<sub>2</sub> powder derived by different metal alkoxide after calcined at (a) 700°C and (b) 900°C

Figures 5(a) and 5(b) showed XRD pattern of TiO<sub>2</sub>-SiO<sub>2</sub> powder derived from different type of metal alkoxide calcined at 700°C and 900°C, respectively. As shown in Figure 5(a), it can be inferred that the smaller particle was obtained in P95/5 (PTP) compared to that of P95/5 (TPT). The appearance of smaller particle size of TiO<sub>2</sub> may be due to the suppressive effect of SiO<sub>2</sub> on the crystal growth of TiO<sub>2</sub>. However, P95/5 (TPT) powder has higher crystallinity.

From the XRD analysis, it was observed that the crystal structure of anatase is more stable at high temperature for TPT derived powder. No secondary phase appeared in the XRD pattern of P95/5 (TPT) sample after 900°C calcination [Figure 5(b)]. The P95/5 (PTP) powder shows higher crystallinity of rutile phase. It is due to the crystallization temperature of P95/5 (PTP) powder from amorphous to anatase has been lowered, leading to the lower temperature of anatase-rutile transformation. This results are in good conformity with the above explained TG-DTA analysis.

The FT-IR spectra of two types of different precursor solutions (PTS (PTP) and PTS (TPT)) along with the TiO<sub>2</sub>-SiO<sub>2</sub> powder (P95 (PTP) and P95 (TPT)) spectra are showed in Figure 6. The spectra were recorded at the wave number range from 4000 to 380cm<sup>-1</sup>. The PTS (PTP) showed the fundamental vibrational modes of acetylacetonate group at 1580cm<sup>-1</sup> (C=O), 1520cm<sup>-1</sup> (C=C) and 1340cm<sup>-1</sup> (C-C). However, none of these vibrational modes appeared in the spectra of PTS-TPT due to the absence of acetylacetonate ligand group. The absorption bands at 3500-3000cm<sup>-1</sup> and 2960cm<sup>-1</sup> in the spectra is attributed to the stretching vibration of Si-OH and aliphatic group (C-H), respectively. It showed that the absorption band of Si-OH at

3400 $\text{cm}^{-1}$  is sharper in PTS (TPT) showing the faster hydrolysis rate of this solution than that of PTS (PTP). The absence of acetylacetonate ligand in the mixture of titanium alkoxide and silicon alkoxide contributes to the fast hydrolysis rate. It is worthy to note that the reaction times for hydrolysis of titanium alkoxides have order of magnitude of milliseconds [8]. All these vibration bands disappeared as the sample underwent calcination process. And after calcination, the residual peak located at the 380 $\text{cm}^{-1}$  indicates the existence of Ti-O-Ti bond as its structure transformed to the anatase phase as shown in spectra P95/5 (PTP) and P95/5 (TPT).



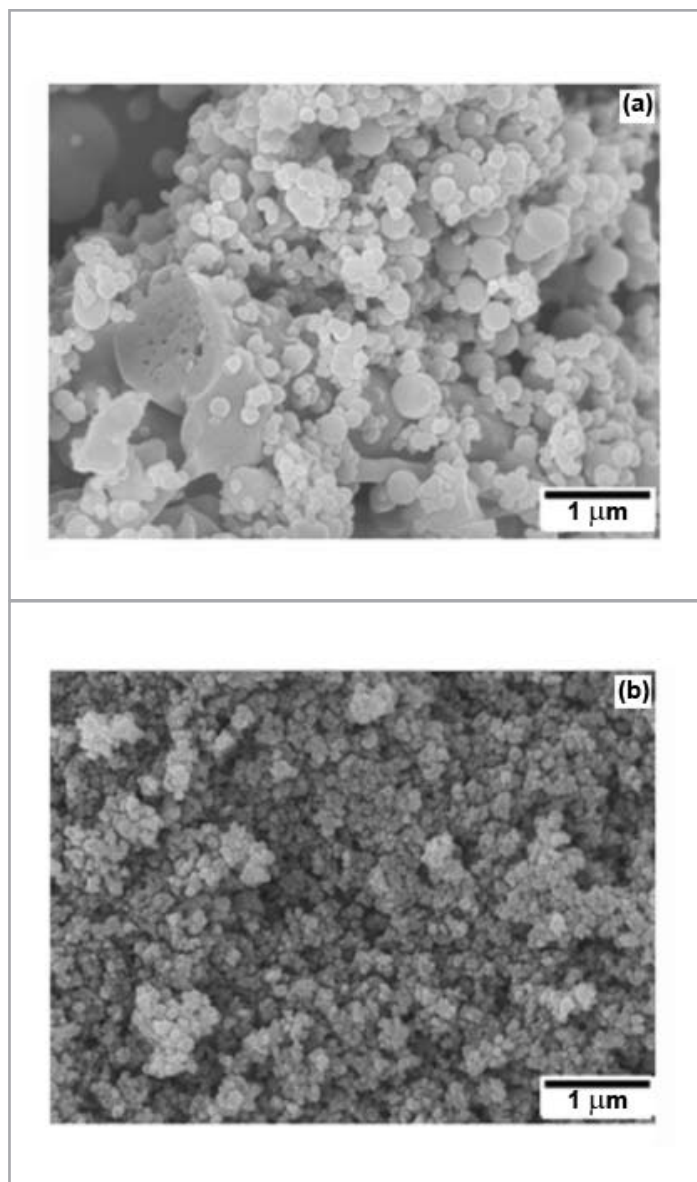
**Figure 6:** FT-IR spectra of polytitanosiloxane solution and calcined powder derived by different metal alkoxide

From this analysis, it shows that PTS (PTP) has sharper absorption band at 1090 $\text{cm}^{-1}$  and 1010 $\text{cm}^{-1}$  than the PTS (TPT). These absorption bands were attributed to the Si-O-Si linkage. The absorption peak of Si-O-Ti linkage was observed at 925 $\text{cm}^{-1}$ . Besides, the absorption band of Si-O-Ti linkage was not observed in the spectra of P95/5 (TPT) powder. However, the appearance of this band still can be seen in the P95/5 (PTP) due to the polycondensation reaction takes place faster in synthesizing the PTS precursor solution.

Figure 7 showed FESEM picture of the  $\text{TiO}_2$ - $\text{SiO}_2$  powder derived by different metal alkoxide ((a) P95/5 (PTP) and (b) P95/5 (TPT)) has been calcined at 700 $^\circ\text{C}$ . From the picture, it can be observed that the P95/5 (TPT) powder is tightly agglomerated and some are non-spherical.

P95/5 (PTP) powder meanwhile has more highly dispersed with well-defined spherical shape of  $\text{TiO}_2$  particles. This can be ascribed to the presence of the nucleophilic ligands that can produce fine and discrete sphere particles [32]. However, the particle size of P95/5 (PTP) seems bigger compared to the P95/5 (TPT) and this result are not corroborate with XRD pattern shown in Figure 5. This can be explained by the difference of measurement principle in XRD and FESEM analysis. The XRD analysis determines the particle size of the individual particle. However, the FESEM observation is based on the primary particle. Since the individual particles of P95/5 (PTP) is too small, the particles started to agglomerate and form bigger primary particles as can be seen in the Figure 7(a).

The different titanium precursor employed in this work gave rise to  $\text{TiO}_2$ - $\text{SiO}_2$  powder photocatalysts with different physical properties and morphology. Photocatalytic test is necessary for the future work to investigate the effect of different powder properties on the photocatalytic activity.



**Figure 7:** FESEM picture of  $\text{TiO}_2$ - $\text{SiO}_2$  powder derived by (a) P95/5 (PTP) and (b) P95/5 (TPT) and calcined at 700 $^\circ\text{C}$

## 4.0 CONCLUSIONS

$\text{TiO}_2$ - $\text{SiO}_2$  powders have been successfully synthesized through sol-gel method using two different types of titanium alkoxides: PTP and TPT, with TEOS was used as the silicon precursor. It was showed that  $\text{TiO}_2$ - $\text{SiO}_2$  powder derived by PTP has much smaller size of individual particles compared to the TPT derived  $\text{TiO}_2$ - $\text{SiO}_2$  powder. The finer particles, thus higher surface area, of the PTP derived powder gave rise to easier agglomeration of the particles and finally formed bigger agglomerate particles. In addition, as the result of the finer particles, the crystallisation temperature of the PTP derived powder become lower compared to the counterpart. It can be concluded that the use of PTP with controlled hydrolysis and polycondensation led to finer  $\text{TiO}_2$ - $\text{SiO}_2$  powder.

## 5.0 ACKNOWLEDGEMENT

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## PROFILES



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### DR IIS SOPYAN

Dr Iis Sopyan, born 13 June 1966 in West Java, Indonesia is presently working as an Associate Professor at the Department of Manufacturing and Materials Engineering, Faculty of Engineering, International Islamic University Malaysia (IIUM). He obtained his PhD in Applied Chemistry from The University of Tokyo, Japan in 1996. His B.Eng. (1991) and M.Eng. (1993) degrees were obtained from Tokyo University of Science in the area of Industrial Chemistry. Dr. Sopyan has published more than 100 scientific papers in local and international journals, mainly in the areas of biomaterials, advanced ceramics, photocatalysis, polymer science and electrochemistry. His three books on polymer chemistry, analytical chemistry, and scientific communication have also been published. He won more than 50 awards for his products showcased in various local and international exhibitions including MTE, ITEX, PECIPTA (Malaysia), iENA (Germany), EUREKA (Belgium), and BIS (UK). This year he was awarded "Highest Citation in Citation Index Journal Award" from IIUM. Eleven patents have been filed in Malaysia for his inventions in the areas of bioceramics and photocatalysis. His research interest is in development of nanomaterials, nanosized calcium phosphate for bone substitute materials, titanium dioxide nanotube, nanozeolite, photocatalytic deactivation of bacteria and degradation of environmental pollutants, conversion of organic solid waste to nanostructure functionalised materials, and development of high density cell culture techniques.