New method to synthesize mesoporous titania by photodegradation of surfactant template

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Abstract
Mesoporous titania has been successfully synthesized by photodegradation removal of cetyltrimethylammonium bromide as the surfactant, after slow hydrolyzation of titanium(IV) isopropoxide. Fourier transform infrared spectra proved that photodegradation has successfully decreased the peak areas of the alkyl groups from the template. The nitrogen adsorption analysis showed that the pore size and the specific surface area of the mesoporous titania were 3.7 nm and 203 m² g⁻¹, respectively, proving the mesoporosity of the titania obtained with the existence of the interparticle mesoporosity which was confirmed by transmission electron microscopy. Based on X-ray diffraction results, the mesoporous titania obtained was in the form of crystalline anatase phase. Furthermore, results from the diffuse reflectance ultra-violet-visible spectra showed that the composition of tetrahedral titanium(IV) was more than the octahedral titanium(IV). When the mesoporous titania obtained was used as a catalyst in the oxidation of styrene, an improvement in the conversion of styrene (38%) was observed when compared to those obtained using Degusa P25 TiO₂ (14%) as the catalyst.

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1. Introduction

Mesoporous titania has been widely used in various chemical reactions as it provides high surface area and contains the necessary catalytic active sites [1]. Based on previous studies, the main pathway to synthesize mesoporous titania is by the soft-templating method [2]. The soft-templating method involves the co-assembly of the precursor and surfactant templates. This method follows the prior success in synthesizing ordered mesoporous silica [3,4] and it is extended to synthesize mesoporous titania [5–11]. In this method, the surfactant templates need to be removed or extracted to form the mesoporous materials. Surfactant removal methods used in previous researches are calcination, liquid solvent extraction, and supercritical fluid extraction (SCFE) [2]. Each of these methods has their own capabilities and limitations that are suitable for certain type of reactions.

Out of the methods listed above, calcination is the most popular approach taken. Although calcination is capable of eliminating the surfactant templates used in the materials and shows high efficiency, the mesoporous structure of the materials could be ruptured when exposed to high temperature for a period of time [2,12]. This will lead to the unsuccessful assembly of ordered mesoporous titania [13]. As for SCFE method, the procedures need to be repeated multiple times to ensure that most of the templates has been removed. These repetitions are often tedious, which consumes a lot of time and solvent. Due to these problems, it is ideal to develop a new, simple and environmentally-benign method which is capable of working at a lower temperature and can remove the surfactant with high efficiency without affecting the mesoporous structure.

It has been reported that UV irradiation is capable of forming many different radicals due to the breaking of all C–C, C–H, and
C–O bonds [11,14–16]. In line with that, a new surfactant removal method can be developed and initiated. Therefore, the ultimate goal of this study is to design a new surfactant removal method by using UV degradation to improve the titania’s mesoporous structure, increase the specific surface area and hence, increasing the photocatalytic activity. UV light is capable of degrading most of the alkyl groups in the surfactant, thus, the results may differ from the calcination method alone. A high power mercury arc lamp (1200 W) has been utilized to degrade the surfactants that are deeply embedded within the titania. The low current or flux of 16.73 mW cm$^{-2}$ used is predicted to be able to prevent rupturing of the mesoporous structure, which is usually caused by the sudden rise in pressure and release of huge amount of gasses.

The photocatalytic activity and selectivity of the synthesized materials have been examined in the oxidation of styrene by hydrogen peroxide (H$_2$O$_2$), in order to confirm the titania’s capability as a photocatalyst. The oxidation of styrene is an interesting process for both academic and industry fields, as the products can be used as an intermediate for a variety of fine chemicals, such as drugs and perfumes [17,18].

2. Experimental

2.1. Synthesis of titania with and without surfactant template

2.1.1. Materials

The chemicals used in this study were cetyltrimethylammonium bromide (CTABr, Merck, ≥98%), titanium(IV) isopropoxide (TTIP, Aldrich, 98%), Degussa P25 (Degussa, Germany), ethanol (HmbG Chemicals, 95%), styrene (Merck, Synthetic Grade), hydrogen peroxide (H$_2$O$_2$, Merck, 30%) and acetonitrile (QReC, HPLC Grade, 99.98%).

2.1.2. Synthesis of TiO$_2$(CTABr) and TiO$_2$(△)

All of the synthesis procedures were carried out in a fume cupboard. First, CTABr (10 mmol) and ethanol (10 mL) were mixed in a crucible. The mixture was stirred for a few seconds, followed by the addition of TTIP (10 mmol). Under continuous stirring, the sample was left to hydrolyze under atmospheric humidity (60%) to allow evaporation of the solvent, obtaining a white-colored sample. To ensure that the sample has fully dried, it was left in the fume cupboard overnight. Following that, the sample was washed with ethanol for three times to eliminate any impurities. The washed sample was filtered by gravitational filtration and the residue was left to dry overnight, before being ground to powder form. Till this stage, the sample is coded as TiO$_2$/CTABr. Half of the TiO$_2$/CTABr obtained was calcined in a furnace at 550 °C for 3 h, stored without further treatment and denoted as TiO$_2$(△), while the other half was used for the next step (Section 2.1.3).

2.1.3. Synthesis of TiO$_2$(‡) and TiO$_2$(‡)(△)

Another half of the TiO$_2$/CTABr sample prepared previously (Section 2.1.2) was used to proceed with the photodegradation method. For this step, a custom-made photoreactor was used and the design is shown in Fig. 1, where a mercury arc (1200 W, flux of 16.73 mW cm$^{-2}$) was used as the light source. The ground TiO$_2$/CTABr sample (1 g) was suspended in distilled water (12 mL) in a test tube and securely immersed in the water bath. The suspended sample was constantly stirred to ensure homogeneity of UV light exposure. During the photoirradiation, a thermostated water bath was used to maintain the temperature of the suspension. After irradiation for 12 h, the white suspension was washed with ethanol for three times, before being filtered by gravitational filtration. The sample was left in a fume cupboard overnight to allow self-drying, ground and denoted as TiO$_2$(‡). TiO$_2$(‡) was further calcined in a furnace at 550 °C for 3 h and denoted as TiO$_2$(‡)(△).

2.1.4. Synthesis of TiO$_2$(TTIP)

For comparison purpose, another TiO$_2$ sample was prepared from TTIP as a reference. 5 mmol of TTIP was left in the crucible for a day to allow hydrolyzation by air humidity and then self-dried at room temperature in a fume cupboard. The dried sample was ground and calcined in a furnace at 550 °C for 3 h. The sample were collected, stored without further treatment and denoted as TiO$_2$(TTIP). The samples prepared and the codes are listed in Table 1.

2.2. Characterizations

The prepared samples were characterized by Fourier transform infrared (FTIR) analysis, X-ray diffraction (XRD), diffuse reflectance ultraviolet–visible spectrometer (DR UV–Vis), nitrogen adsorption-desorption analysis and low vacuum scanning electron microscope (LVSEM) and transmission electron microscope (TEM). For FTIR analysis, the samples were ground to powder form with potassium bromide (KBr), with the weight ratio of 1:100 to form a pellet. The pellet was analyzed by Perkin Elmer Spectrum One spectrometer in the range of 4000–400 cm$^{-1}$, with the resolution of 4 cm$^{-1}$ for 10 scans. The FTIR analysis was used to detect the presence and disappearance of the alkyl group at the region of 3000–2900 cm$^{-1}$, as an early observation on the degradation of the surfactant template. A Bruker D8 Advance diffractometer with the wavelength of Cu Kα radiation = 1.54 Å was used to collect the XRD patterns of the samples and to identify the crystalline phase of the samples in the 20 range of 20–90°. The mesoporosity of the TiO$_2$ prepared can also be indicated by the presence of a small peak at around 20 of 1–10°. DR UV–Vis was conducted on a Lambda 900 UV/Vis/NIR model by Perkin Elmer with the wavelength range of 175–3300 nm. The reflectance and scattering were measured by using integrating sphere and barium sulphate (BaSO$_4$) acted as the blank for the solid sample. The specific surface area evaluation for the samples was assessed from Brunauer–Emmet–Teller (BET) analysis by measurement of nitrogen adsorption–desorption at multilevel as a function of relative pressure. Prior to measurement, the samples were outgassed at 110 °C for 7 h under vacuum before analysis by nitrogen adsorption surface area analyzer. This measurement produces crucial information in understanding the effects of particle size and surface porosity in the applications. LVSEM was used to observe the surface morphology of the solid particles of
the sample. A carbon tape was attached on a stub to secure the dispersion of powdered samples before being coated for 4–5 times with platinum using Auto-Fine Coater (JEOL JFC-1600). The morphology of the sample was observed under 15 kV of accelerating voltage. The transmission electron microscopy (TEM) study was conducted with JEOL JEM-2100 at 200 kV. The TEM samples were prepared by drying drops of dispersing samples in ethanol on copper grids coated with formvar/carbon. The chemical forms of the products were studied using X-ray photoelectron spectroscopy (XPS), AXIS Ultra DLD, Shimadzu, with monochromatic Al Kα radiation (1486.6 eV).

2.3. Photocatalytic testing by Oxidation of styrene using H2O2

The photocatalytic activity of the samples was tested out in the oxidation of styrene by H2O2. The reaction mixture of styrene (0.572 mL), powder photocatalyst (50 mg), aqueous H2O2 (0.817 mL) as the oxidizing agent and acetonitrile (5 mL) as the solvent was stirred with a 30 W xenon lamp at room temperature for 24 h. Throughout the irradiation, the mixture was magnetically stirred after the photoirradiation, the solid sample was removed by centrifugation prior to the measurement. The reactant and products of the reaction were analyzed by gas chromatography (Shimadzu Gas Chromatography model GC-2014) with a capillary column (BPX-5, I 25 m, φ 0.32 mm × 0.52 μm) using a flame ionization detector. The carrier gas was nitrogen with column flow of 3 mL min−1. The program of the temperature used was set at 80 °C for 1 min, followed by heating to 150 °C at 10 °C min−1. The final temperature (150 °C) was then kept for an additional 0.5 min. The percentages of styrene conversion, the selectivity of each oxidation product and turnover number were calculated from the GC results.

3. Results and discussion

3.1. Physicochemical properties

All the samples obtained from the syntheses are in the form of fine powder, light-weight, and white in color. Based on bare eyes observations, the particles size of TiO2(Δ) sample is smaller and finer compared to those of TiO2(Δ) sample. This observation is also supported by SEM and TEM images that will be discussed later.

3.1.1. Functional groups

The FTIR spectra for TiO2/CTABr, TiO2(Δ), TiO2(Δ) and TiO2(Δ) are shown in Fig. 2a–d, respectively. Initially, all of the titania samples contained CTABr in the mixture. At 640 cm−1, the peak observed in the spectra of TiO2(Δ), TiO2(Δ) and TiO2(Δ) corresponds to the Ti–O bonding. Meanwhile, in the same region for TiO2/CTABr’s spectrum, no significant peak can be detected. The fact that the Ti–O bonding cannot be detected when the surfactant was still present indicates that the active sites were covered as the surfactant has not been removed.

The main reason FTIR was employed is to investigate the effectiveness of alkyl group’s degradation by photoirradiation. The alkyl group can be detected at the region of 2950–2850 cm−1 for –CH2— stretching vibration [1]. In TiO2/CTABr’s spectrum (Fig. 2a), the intensity of C=CH2 peak is high because the sample has been incorporated with CTABr, which consists of a long chain alkyl. After calcination (TiO2(Δ)—Fig. 2b), these peaks were completely diminished, while UV irradiation (TiO2(Δ) — Fig. 2c) partially removed these, indicating the degradation of the alkyl groups. Based on these initial findings, UV irradiation can be considered as a new possible method for the removal of surfactant template.

After calcination of TiO2(Δ) sample TiO2(Δ), there was no significant difference between the spectra of TiO2(Δ) (Fig. 2c) and TiO2(Δ) (Fig. 2d), except for the increasing intensity for Ti–O’s peak. It is believed that photoirradiation has already degraded most, if not all, of the alkyl group and a stable TiO2 sample has been formed. The remaining alkyl group is assumed to be trapped and unable to dissipate from the sample, even when exposed to high temperature.

3.1.2. Crystal structure

The crystallinity and phase of the samples were determined by powder XRD. Fig. 3 shows the diffractograms for all of the samples prepared. The diffraction peaks can be categorized into two regions, which are small-angle (2θ = 20°–10°) and wide-angle (2θ = 20°–90°). At small-angle, a peak observed at 5° is attributed to the mesoporous structure of the sample [9]. All of the samples showed an appreciable peak at 5° except for TiO2(Δ). For TiO2(Δ), the lack of this peak shows that the mesoporous structure has not been fully formed. Mesoporosity is important in developing high surface area materials, which helps to provide a larger area for reactions to occur on the active sites [1].

By using Scherer equation, the crystalline size for TiO2(TTIP), TiO2(Δ), TiO2(Δ) and TiO2(Δ) are 23.37, 19.36, 3.82 dan 17.40 nm, respectively (Table 2). TiO2(Δ) has the smallest crystallite size compared to the others. It is observed that after calcination of TiO2(Δ) to form TiO2(Δ), the crystallite size increased from 3.82 to 17.40 nm and the mesopore structure has been formed. The increment of crystallite size is believed to be due to the agglomeration of TiO2(Δ) during calcination. The pressure released by the gasses from the inner part of the samples during the removal of the alkyl groups caused the formation of pores, which then increases the surface area. The combination of the porous structure and smaller particles size can enhance the surface area per mass [1,19]. The high surface area can enhance the diffusion of substrates to the active sites, thus increasing the photocatalytic activity. In the wide-angle XRD, major peaks were observed at 25, 37 and 48°, which correspond to the anatase phase of crystalline titania [21–23]. The diffractograms of all of the samples, except for TiO2(Δ), show similar pattern and intensities for the peaks, which indicate that the samples were of anatase titania. These samples were calcined at 550 °C, which will lead to the transformation to anatase phase. This is in agreement with the findings of Ovenstone’s [24] and Shao’s research group [25]. When titania is exposed to the temperature below 600 °C, between 600 and 800 °C, and above 800 °C, the phases that the titania will be in are anatase, anatase-rutile, and rutile, respectively [24,25]. As for TiO2(Δ), the peaks’ pattern resembled those of titania with anatase structure but with lower

<table>
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<th>Table 1</th>
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<td>The notation of sample materials.</td>
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<tr>
<td>Sample</td>
</tr>
<tr>
<td>TiO2(TTIP)</td>
</tr>
<tr>
<td>TiO2(Δ)</td>
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<tr>
<td>TiO2(Δ)</td>
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<tr>
<td>TiO2(Δ)</td>
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<tr>
<td>TiO2(Δ)</td>
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</tbody>
</table>
This indicates that although photoirradiation alone can degrade the surfactant template, calcination is still needed in order to form highly crystalline anatase titania. It has been well-established that highly crystalline anatase titania shows good photocatalytic activity in various photocatalytic reactions [1,20,26].

### 3.1.3. Local coordination and chemical form

Fig. 4 shows the deconvoluted DR UV–Vis spectra of the samples, which give an understanding for the local coordination of Ti. The deconvoluted bands at 220, 255 and 290 nm are assigned as tetrahedral Ti. Meanwhile, the bands at 325 and 355 nm are assigned as octahedral Ti [26]. The percentage of tetrahedral Ti for TiO$_2$(TTIP), TiO$_2$(Δ), TiO$_2$(+) and TiO$_2$(+) (Δ) are 35, 42, 61, and 45%, respectively. These figures indicate the occurrence of some transformation of octahedral Ti to tetrahedral Ti during the UV irradiation of the sample. TiO$_2$(+) spectra showed the highest composition of tetrahedral Ti amongst all the photocatalysts, but after the calcination, the percentage of tetrahedral Ti reduced while octahedral Ti increased.

Surface characteristics XPS wide scan spectrum of the TiO$_2$(+)(Δ) sample is shown in Fig. 5a. The spectrum shows two distinct peaks representing the major constituents, titanium...

### 3.1.4. Lattice spacing and crystallite size

The lattice spacing and crystallite size of the samples are shown in Table 2.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Crystallite size (nm)</th>
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<tbody>
<tr>
<td>TiO$_2$(TTIP)</td>
<td>8.43</td>
</tr>
<tr>
<td>TiO$_2$(Δ)</td>
<td>5.50</td>
</tr>
<tr>
<td>TiO$_2$(+)</td>
<td>3.95</td>
</tr>
<tr>
<td>TiO$_2$(+) (Δ)</td>
<td>5.58</td>
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</table>
(458.5 eV), carbon (284.5 eV) and oxygen (529.5 eV). The presence of carbon in the TiO_2(\Delta) sample is supported by the FTIR spectra (see Fig. 2). It has been reported the binding energy for Ti 2p/2 in stoichiometric TiO_2 and TiO_2 were in the range of 458.5–458.9 eV and 456.8–456.9 eV, respectively [27]. As shown in Fig. 5b, the binding energy of Ti 2p/2 in TiO_2(\Delta) sample was 458.5 eV. This energy is similar to that of TiO_2 and corresponds to Ti in TiO_2 anatase [27].

3.1.4. Surface area and pore distribution

Fig. 6 shows a pore size distribution of the samples TiO_2(T-TIP), TiO_2(\Delta) and TiO_2(\Delta). All isotherms are typical of Type IV which is characterized by its hysteresis loop and a steep increase at relative pressure P/P_0 of 0.8–1.0. This is associated with capillary condensation taking place in the interparticle mesoporosity [28,29]. The hysteresis behavior is clearly to be intermediate between Types H1 and H3. Type H1 hysteresis is often associated with porous materials exhibiting agglomerates of particles with a narrow distribution of relatively uniform cylindrical-like mesopores. Type H3 hysteresis is usually observed on many crystalline metal oxide aggregates of the sheet-like shape. Moreover, the Barrett–Joyner–Halenda (BJH) pore size distributions (Fig. 6) also confirm the presence of mesopores in TiO_2(T-TIP), TiO_2(\Delta) and TiO_2(\Delta) samples, and the mesopore size is mainly located at 8–12 nm. The existence of the interparticle mesoporosity was further confirmed by TEM (Fig. 8). This is a consequence of the agglomeration of particles as shown in TEM images described in Section 3.1.5. It is clearly observed that the mesopores are formed by interparticle voids.

The surface area of the synthesized samples is listed in Table 3. The surface area of TiO_2(T-TIP), TiO_2(\Delta) and TiO_2(\Delta) are 12 m² g⁻¹, 15 m² g⁻¹ and 202 m² g⁻¹, respectively. The surface area of TiO_2(\Delta) was the highest, which is around 13 times higher than that of TiO_2(\Delta), and 16 times higher than that of TiO_2(T-TIP). The pore size distribution of the samples are in the range of 2–50 nm, with the average pore size of TiO_2(T-TIP), TiO_2(\Delta), and TiO_2(\Delta) of 11.9, 12.5 and 9.6 nm, respectively (Table 3).

Based on the above findings, it is confirmed that TiO_2(\Delta) contains mesopores and possesses the highest surface area with the narrowest pore size distribution when compared to the other samples. These statements are further supported by the XRD...
results, where the mesoporosity of the sample has been proven. The high surface area can enhance the number of active sites on the surface to improve the photocatalytic reaction [30]. Thus, it is expected that TiO$_2$(\(\Delta\)) will have increased efficiency in the photocatalytic oxidation of styrene, that will be explained later.

3.1.5. Microscopy analysis

SEM was used to observe the morphology of the samples. In Fig. 7, in terms of shape, the images showed that TiO$_2$(TTIP) particles, which are synthesized without any surfactant, are smooth and spherical in shape (Fig. 7a). Meanwhile, TiO$_2$(\(\Delta\)), TiO$_2$(\(\nabla\)) and TiO$_2$(\(\nabla\)\(\Delta\)) samples are less spherical and have rough surfaces. It is hypothesized that the attachment of surfactants to TiO$_2$ caused the observed roughness. Relatively, the particles of TiO$_2$(\(\nabla\)\(\Delta\)) are smaller and finer in size compared to the other samples. These statements are in agreement with previous surface area analysis results (Table 3), where TiO$_2$(\(\nabla\)\(\Delta\)) has the highest surface area among the samples.

For the sample calcined directly at 550 °C (TiO$_2$(\(\Delta\)) - Fig. 7b), the pore size distribution is also not uniform, where uneven pores were formed. This is due to the rupturing of the structure caused by the gas pressure released during the decomposition of CTABr in high temperature, resulting in a low surface area (15 m$^2$ g$^{-1}$). On the other hand, the pore size distribution of TiO$_2$(\(\nabla\)\(\Delta\)) (Fig. 7d) was more uniform than that of TiO$_2$(\(\Delta\)) (Fig. 7b). A possible explanation is that the TiO$_2$ prepared by photoirradiation followed by calcination (TiO$_2$(\(\nabla\)\(\Delta\))) has no sudden rise in pressure and no release of huge amount of gasses, as photoirradiation allow gradual degradation of the template, without rupturing the structure of TiO$_2$. However, in order to transform the phase of the amorphous TiO$_2$ to crystalline anatase TiO$_2$, calcination is still needed. No further removal of CTABr took place after calcination for TiO$_2$(\(\nabla\)). This is shown by the FTIR spectra of TiO$_2$/CTABr that underwent

![Fig. 6. N$_2$-sorption isotherms and pore size distribution of the samples (a) TiO$_2$(TTIP), (b) TiO$_2$(\(\Delta\)) and (c) TiO$_2$(\(\nabla\)\(\Delta\)).](image)

![Fig. 7. SEM images of (a) TiO$_2$(TTIP), (b) TiO$_2$(\(\Delta\)), (c) TiO$_2$(\(\nabla\)), and (d) TiO$_2$(\(\nabla\)\(\Delta\)).](image)
photoirradiation (Fig. 2c) and TiO$_2$/CTABr that underwent photoirradiation followed by calcination (TiO$_2$(⊥)$\cdots$(Δ)) (Fig. 2d), that showed similar peaks with similar intensities.

TEM image of TiO$_2$(⊥)$\cdots$(Δ) is shown in Fig. 8. It is clearly observed that the existence of mesoporosity existing along intercrystalline boundaries in titania particles. The d-spacing of the resolved fringes is 0.250 nm, consistent with the lattice spacing {103} for anatase TiO$_2$. Fig. 8a shows the schematic illustration of the
aggregation of titania particles, where the pores located between the particles. The pore distribution shown in Fig 8a determined by TEM images is in agreement with the pore distribution obtained from N₂-sorption isotherm analysis (see Fig. 6). It is shown that the pore distribution is in the range of 5–35 nm.

3.2. Photocatalytic activity

The photocatalytic activity of the synthesized mesoporous TiO₂ was tested out in the photocatalytic oxidation of styrene by H₂O₂ as the oxidant. In order to properly evaluate and compare the photocatalysts’ photocatalytic performances, five different catalysts were used; (a) no photocatalyst, (b) TiO₂(TTIP), (c) TiO₂/CTABr, (d) TiO₂(Δ) and (e) TiO₂(Δ). The conversion percentage amongst all of the photocatalysts used, which was 38%. This is most probably due to the high surface area and more uniform pore size exhibited by the photocatalyst. These properties allow easier and better access of substrates to the active sites in the photocatalyst. The second highest conversion was shown by TiO₂(TTIP) with 21%, followed by TiO₂(Δ) photocatalyst with 17% of styrene conversion. The two lowest conversion of styrene were shown by TiO₂/CTABr and when no catalyst was used, which was 11% for both cases.

As explained earlier, the preparation methods for TiO₂(Δ) and TiO₂(Δ) were different, where TiO₂(Δ) was synthesized by using UV light to degrade the alky long chain of the surfactant before being calcined, while TiO₂(Δ) was calcined straightaway. Comparatively, there was a significant increment of percentage in the styrene conversion between both of these catalysts, most probably caused by the preparation methods. It is suggested that UV–photoinradiation broke the alky bonds in the surfactant gradually without rupturing the mesostructure of TiO₂. Generally, heat treatments (calcination) are the most effective in breaking the alky bonds in the surfactant template [1,13]. However, the process releases a large amount of gasses in high pressure, which might rupture the mesoporous structure of the titania [32]. This can cause the reduction in surface area and ununiformed pores. These statements are supported by the specific surface area of TiO₂(Δ), that is 13 times higher than the surface area of TiO₂(Δ), and a more uniform pore size distribution in the meso-range.

As for the comparison between TiO₂(Δ) and TiO₂(TTIP), the conversion of synthesized TiO₂(Δ) was still higher than that of TiO₂(TTIP), which is generally used as a benchmark. This significant increment reflects the efficient preparation of mesoporous TiO₂ by photodegradation of surfactant template, which is capable of increasing the photocatalytic activity of the resulting TiO₂

Fig. 9 also shows the selectivity percentage of the products in the photocatalytic oxidation of styrene by aqueous H₂O₂. For all of the photocatalysts, benzaldehyde was the main product, with phenyl acetyldehyde and styrene oxide as the side products. TiO₂(Δ) showed 100% selectivity towards benzaldehyde. On the other hand, TiO₂(TTIP) showed a slightly lower selectivity towards benzaldehyde (96%), with side products of phenyl acetyldehyde (3%) and styrene oxide (3%) being detected. For large scale use in industries, the existence of other products or byproducts will increase the operational cost as separation processes are needed. In the case of styrene oxidation, TiO₂(Δ) catalyst can fully convert styrene to benzaldehyde, without any impurities, which can prevent the extra cost to separate the byproducts. As shown in Fig. 9, the conversion of styrene over TiO₂(Δ) (38%) is higher than that of Degussa P25 TiO₂ (14%) suggesting that TiO₂(Δ) is a potential photocatalyst.

4. Conclusions

The physicochemical properties and photocatalytic activity of mesoporous titania synthesized by a new method of photodegradation to remove the organic surfactant template, have been studied. The results have proven that UV light is capable of degrading the surfactant template to form mesoporous titania with high surface area. Further calcination treatment was done to transform the amorphous TiO₂ to the crystalline anatase phase. The properties shown by the mesoporous titania with intercrystalline mesoporosity prepared by photodegradation can perform well in photocatalytic oxidation of styrene by aqueous H₂O₂ with 38% of styrene to benzaldehyde (100% selectivity).

Acknowledgments

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References


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Table 3

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<th>Sample</th>
<th>Specific surface area (m² g⁻¹)</th>
<th>Pore size (nm)</th>
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<tr>
<td>TiO₂(TTIP)</td>
<td>12</td>
<td>11.9</td>
</tr>
<tr>
<td>TiO₂/CTABr(Δ)</td>
<td>15</td>
<td>12.5</td>
</tr>
<tr>
<td>TiO₂/CTABr(1 × Δ)</td>
<td>202</td>
<td>9.6</td>
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