TiO₂/SiO₂ composite for efficient protection of UVA and UVB rays through of a Solvent-less synthesis

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TiO₂/SiO₂ composite for efficient protection of UVA and UVB rays through of a Solvent-less synthesis

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Abstract

In an effort to discover new inorganic UV absorbers, titania included into silica was prepared using a solvent-less solid state method involving the pyrolysis of the as prepared precursor Chitosan•(TiOSO₄)/SiO₂, as an alternative and versatile way to using these compounds for practical applications. The new TiO₂/SiO₂ composite was characterized by PXRD, SEM-EDS, TEM and UV-vis absorption analysis. The SEM-EDS mapping images show a uniform distribution of TiO₂ into the silica matrix. The optical properties of the composite have shown an interesting result related to high absorption of UVB rays and an improved absorption of UVA rays than pure TiO₂. Efficient suppression of photocatalytic behavior of TiO₂, when is incorporated into silica, was evidenced from 85% to 31%, suggesting it material as alternative inorganic UV absorber to remains the properties of the methylene blue dye. These results reveal their potential use in practical textile industry and UV protection agent to avoid human damage.

Keywords: TiO₂/SiO₂ composite, UV absorber, UV blocking property, photocatalysis

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Introduction

Among all the semiconductors, titanium dioxide (TiO₂) presents high photochemical and physical stability, an increased number of available sites for oxygen to generate reactive oxygen species (ROS), and lower toxicity and cost in comparison with other materials [1-3]. As a consequence, TiO₂ is employed in different fields such as anti-bacterial agent [4], solar energy conversion [5], water splitting to produce hydrogen fuel, decomposing [6-9] and removal of organic or inorganic pollutants [10-12], among other practical applications. TiO₂ is a semiconductor presenting a wide energy gap (3.0-3.4 eV), and then a wide absorption of UV rays [13]. Due to its good capability to absorption and scattering of UV photons, it could be used as an interesting alternative to organic UV absorbers [14]. Another important aspect related to this challenge is the photocatalytic properties of many UV absorbers because can lead to the degradation of organic materials, which act as matrices to host these protection agents. [15]. The UV light is composed in three areas: UVA (315–400 nm), UVB (280–315 nm and UVC (100– 280 nm). In this respect, UVA rays are few absorbed by the atmosphere, leading to erythema if the exposure is excessive. However, UVB rays are not entirely absorbed by the atmosphere, generating cellular DNA damage, which is responsible of skin cancer, erythema and cataracts, among others diseases. [16] UVB rays not only cause damage to humans but also to textile materials causing degradation and yellowing of clothes. [17] One of the most common dyes widely used in cotton, wood and silk industry is Methylene Blue (MB), and their photodegradation by ROS is a great challenge. For example, Montazer et al [18] reported TiO₂ nanostructures as an alternative UV absorber to care for wool. In this trend, silica could be used as a surface coating to protect the textile, presenting high stability, low cost, and very efficient behavior [19-22]. However,

it is still a challenge to suppress the photochemical reactions generated by TiO_2 and their synthesis, and mainly to maintain the absorption and dispersion of the UV photons, together with the transparency of the material after the treatment with silica.

Although there are several methods in the literature to prepare TiO_2 nanoparticles (TiO_2NPs), [23,24] solid-state methods have scarcely reported in comparison to solution methods [23,24]. For example, a solid-state metathesis reaction of TiCl_3 and Na_2O_2 in presence of NaCl to give mixtures of anatase and rutile has been achieved [26]. The incorporation of metal oxide nanoparticles into solid devices is not straightforward [28-31] when they are produced via a solution method, as the solid-state isolation of these nanoparticles produces their agglomeration [32-35]. If they could be generated directly from a solid-state approach will be of great interest and their development is a continuous challenge [35]. An interesting method to obtain TiO_2NPs in solid anatase and rutile phases was developed for Park et al. through thermal plasma treatment, achieving a remarkable control of particles sizes [36]. In this regard, many solid state methods have some disadvantages, such as sophisticated and expensive equipment, high dispersion of sizes, mixtures of phases, agglomeration, among others and the chemistry implicated in this kind of synthetic processes is restricted [37,38]. Additional difficulties include the incorporation of nanostructured metal-oxides into solid matrices [39] and the use of high working temperatures on several practical applications [28-31]. Silica is possibly the most suitable material to be used as a solid state matrix [34]. Several methods to prepare nanoparticles into silica matrix, mainly sol-gel based, have been reported, [40,41] but to the best of our knowledge, alternative solid state methods have not been reported.

In this work, a solid state method to prepare efficient $\text{TiO}_2/\text{SiO}_2$ absorbers (UVA and UVB rays) from pyrolysis of $\text{Chitosan} \cdot (\text{TiOSO}_4)/\text{SiO}_2$ mixtures is presented. Thermal treatment of

Chitosan•(TiOSO₄) affords pure anatase TiO₂ [42-44]. A comparison between commercial TiO₂ and the obtained materials in terms of their optical properties will be discussed.

Experimental

Materials

Chitosan, TiOSO₄, hydrochloric acid (HCl, 37 wt%), tetraethylorthosilicate (TEOS), dichloromethane, acetic acid (99%) and ethanol were purchased from Sigma-Aldrich company. All reactants were analytical grade and used without further purification.

Synthesis of Chitosan•(TiOSO₄)/SiO₂

Stoichiometric amounts of chitosan (1 g) and TiOSO₄ (1 g) were stirred at room temperature in dichloromethane (60 mL) during two weeks. After this, solid Chitosan•(TiOSO₄) was added over the intermediate preparation of a SiO₂ colloid (sol-gel method was conducted by stirring TEOS, ethanol and acetic acid in molar ratios 1:4:4 and deionized water for 5 h) and it was stirred for 3 additional days.

Fabrication of TiO₂/SiO₂ composite

The formed Chitosan•(TiOSO₄)/SiO₂ wet gel was stirred at room temperature and dried at 100 °C under reduced pressure in a vacuum furnace, and the solid was finally calcined at 800 °C for 2 h. Pyrolysis experiments were conducted by pouring a weighed portion (0.05–0.15 g) of the as prepared precursor into alumina boats and calcined in a furnace (Labtech.LEF-10 oven) under air atmosphere, using a heating rate of 10 °C min⁻¹ up to 800 °C and annealed for 2-4 h.

118 **Characterization**

119 The SEM images and elemental analysis of the pyrolyzed product were measured using a
120 JSM-6380LV JEOL microscope, with an acceleration voltage of 20 keV equipped with an
121 Energy Dispersive X-ray Spectroscopy (EDS). Transmission Electron Microscopy (TEM)
122 was performed using a JEOL 2000FX TEM microscope at 200 kV to characterize the average
123 particle size, distribution and elemental and crystal composition. The powder X-ray
124 diffraction (XRD) was conducted at room temperature on a Siemens D-5000 diffractometer
125 with θ - 2θ geometry. The XRD patterns were collected using Cu-K α radiation (40 kV, 30
126 mA) at a scanning rate of 0.02 s^{-1} in the 2θ range from 5° to 40° . Raman scattering
127 measurements were performed using a DILOR XY spectrometer with a CCD detector and 2
128 cm^{-1} of spectral resolution. The 514.5 nm line of an Ar⁺-ion laser was used as excitation
129 source. The optical properties of TiO₂/SiO₂ systems and the photodegradation of MB were
130 characterized by UV-Vis absorption spectroscopy performed on a Shimadzu UV-2600
131 spectrophotometer. The diffuse reflectance data of solid samples were transformed to
132 absorbance using the Kubelka-Munk equation.

134 **Photocatalytic behavior of the TiO₂/SiO₂**

135 The photocatalytic activity was evaluated by measuring the bleaching rate of MB under UV-
136 Vis illumination in a range of 330-700 nm at room temperature using a CuSO₄ filter (0.1
137 mol/L), to avoid the self-degradation and thermal catalytic effects of this cationic organic
138 dye. A quartz tube of 25 mL was used as a photoreactor vessel. The optical system used was
139 a xenon lamp (150W) which was positioned 20 cm away from the photoreactor. 25 mg of the
140 photocatalyst and 25 mL of MB aqueous solution (1×10^{-5} mol/L) were placed into the reactor
141 with continuous magnetic stirring. Prior to irradiation, the suspension was stirred in the

darkness for 30 min to establish an adsorption/desorption equilibrium, after which the photocatalytic degradation of MB was initiated. Samples were withdrawn from the reactor at certain time intervals and centrifuged to remove the particles. Photodegradation was monitored by measuring the absorbance of the solution at 655 nm under different time intervals.

Results and Discussion

XRD and Raman analysis

TiO₂/SiO₂ was prepared by hydrolysis of TEOS according to the sol-gel method reported by Liu and Han [39]. Once the gel was formed, the sample was dried under vacuum and pyrolyzed at 800 °C yielding a white solid, as shown in Fig. 1. XRD pattern (Fig. 2) exhibits a broad signal between 15° and 25° of 2θ value, due to the amorphous silica. The intense sharp peak at 2θ ≈ 25° is characteristic of the anatase crystal phase. Due to the high content of silica, the diffraction peaks from crystalline SiO₂ are masked by the broad peak of amorphous silica. Thus, the XRD pattern shown is characteristic of TiO₂/SiO₂ materials with low TiO₂/SiO₂ ratio [42,43].

Additional evidence of the presence of TiO₂ in the silica matrix is given by the Raman spectrum of the TiO₂/SiO₂ composite (see Fig. S1 in suppl. material). The Raman spectrum exhibits a relatively intense peak at 148 cm⁻¹, that can be ascribed to the E_g Ti-O bond stretching vibration mode of the anatase phase.

165 **Morphological and structure analysis**

166 SEM images of the $\text{TiO}_2/\text{SiO}_2$ composite are shown in Fig. 3. showing the variable
167 morphology typically obtained from solid-state high-temperature methods [35]. Spheres of
168 about 20 μm are observed in Fig. 3(a) and Fig. 3(b) together with other more irregular shapes.
169 In general, the observed morphologies are similar to those of $\text{TiO}_2/\text{SiO}_2$ obtained by other
170 solution methods [44-48].

171 EDS analysis was performed in different regions, confirming the presence of the TiO_2
172 embedded into the SiO_2 matrix (Fig. 3(c)). As shown in Fig. 3(d), it could be confirmed that
173 TiO_2 is distributed uniformly over the SiO_2 matrix. The different analysis in other areas of
174 the $\text{TiO}_2/\text{SiO}_2$ particles showed a similar distribution.

175 Further analysis of the $\text{SiO}_2/\text{TiO}_2$ was performed by TEM. A representative distribution of
176 particles is shown in Fig. 4(a), where TiO_2 nanoparticles are embedded into the SiO_2 matrix.
177 Detailed images (Fig. 4(b) and 4(c)) show that the TiO_2 nanoparticles (as confirmed by EDS)
178 are in the range of 10 to 20 nm in size. However, due to the presence of amorphous silica, it
179 was not possible to obtain high resolution images.

181 **Optical Properties and Photocatalytic behavior of the $\text{TiO}_2/\text{SiO}_2$**

182 The material was studied in terms of UV light absorption and their photocatalytic behaviour.
183 The UV/Vis light absorption for commercial TiO_2 (Degussa P25) and $\text{TiO}_2/\text{SiO}_2$ and their
184 photocatalytic performance to degrade MB dye are shown in Fig. 5(a). The characteristic
185 broad absorption maximum was found between 395-250 nm for pure TiO_2 in UV region,
186 typical of the electronic transition associated with the energy gap of this semiconductor.
187 $\text{TiO}_2/\text{SiO}_2$ is evidencing a slight red shift (from 363 to 404 nm) to the absorption edge of
188 TiO_2 , which can be attributed to the interaction to a SiO_2 material. This finding indicates that

the visible transparency remains practically unchanged and higher absorption of the UVA rays was observed for the $\text{TiO}_2/\text{SiO}_2$ (a blue shifting of absorption maxima of $\text{TiO}_2/\text{SiO}_2$ was evidenced in UV region at 260 nm), compared to pure TiO_2 which has a less absorption in this region and shows very high transparency. These results suggest that this kind of material is a good candidate for UV absorber because the key parameters such as transparency in the visible region and a high UV absorption in the UV range of 290-400nm are in good agreement with an efficient inorganic UV absorber, mainly UVA and UVB rays.

Fig. 5(b) shows the absorbance spectra over the entire MB degradation for $\text{TiO}_2/\text{SiO}_2$ at certain irradiation time, which represents a remarkable suppression effect of the dye degradation with respect to commercial TiO_2 . Fig.5(c) shows a comparative photocatalytic behavior between pristine TiO_2 and this semiconductor embedded into SiO_2 , achieving a reduction of the MB photodegradation from 86% to 31% after 25min. In Fig. 5(d) the photocatalytic reaction of TiO_2 materials can be followed for the apparent rate constant of pseudo-first-order for this process, as the concentration of UV absorber is kept constant. K value is evidencing a slower degradation process for $\text{TiO}_2/\text{SiO}_2$ composite, attributed to the SiO_2 blocking effect, in comparison to commercial TiO_2 .

Conclusions

An alternative and versatile solvent-less synthetic method has been achieved to get a uniform TiO_2 dispersion embedded into SiO_2 matrix, as potential inorganic UV absorber. TiO_2 included into the SiO_2 was prepared by pyrolysis of the precursor Chitosan•(TiOSO_4)/ SiO_2 . As a perspective, this method has shown to be a simple and useful solid-state way to prepare $\text{M}_x\text{O}_y/\text{SiO}_2$ materials that can be easily incorporated in solid-state devices for practical applications. The optical properties of the $\text{TiO}_2/\text{SiO}_2$ composite have shown an effective

blocking effect to suppress dye degradation by the UV light. The key parameters, such as visible transparency and high UV absorption remains high after the incorporation of TiO₂ in silica, suggesting an attractive alternative for conventional inorganic UV absorbers.

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Figures Caption

Fig. 1 Schematic illustration of the Synthetic route of $\text{TiO}_2/\text{SiO}_2$ composite.

Fig. 2 XRD patterns of $\text{TiO}_2/\text{SiO}_2$ composite.

Fig. 3 SEM images of $\text{TiO}_2/\text{SiO}_2$ composite **(a)** showing different morphologies and **(b)** a high magnification of two spherical particles, **(c)** and **(d)** are evidencing the homogeneous TiO_2 distribution on SiO_2 matrix by EDS analysis.

Fig. 4 TEM image of $\text{TiO}_2/\text{SiO}_2$ composite **(a)** in a low magnification, **(b)** and **(c)** are showing the TiO_2 particles into amorphous silica (red circles).

Fig. 5 UV-Vis absorption spectra of **(a)** commercial TiO_2 (Degussa P25) and $\text{TiO}_2/\text{SiO}_2$ composite recorded by Diffuse Reflectance Spectroscopy, **(b)** photodegradation curves of MB in presence of obtained UV absorber. Normalized concentration of MB as function of irradiation time of **(c)** pure TiO_2 and $\text{TiO}_2/\text{SiO}_2$ composite, showing the suppressed photocatalytic activity and **(d)** pseudo-first order kinetic of photocatalytic reactions for this degradation process.

Fig. 1 Schematic illustration of the Synthetic route of $\text{TiO}_2/\text{SiO}_2$ composite.

257x144mm (96 x 96 DPI)

Fig. 2 XRD patterns of TiO₂/SiO₂ composite.

170x126mm (96 x 96 DPI)

Fig. 3 SEM images of TiO₂/SiO₂ composite (a) showing different morphologies and (b) a high magnification of two spherical particles, (c) and (d) are evidencing the homogeneous TiO₂ distribution on SiO₂ matrix by EDS analysis.

224x165mm (96 x 96 DPI)

Fig. 4 TEM image of $\text{TiO}_2/\text{SiO}_2$ composite (a) in a low magnification, (b) and c are showing the TiO_2 particles into amorphous silica (red circles).

198x144mm (96 x 96 DPI)

Fig. 5 UV-Vis absorption spectra of (a) commercial TiO_2 (Degussa P25) and $\text{TiO}_2/\text{SiO}_2$ composite recorded by Diffuse Reflectance Spectroscopy, (b) photodegradation curves of MB in presence of obtained UV absorber. Normalized concentration of MB as function of irradiation time of (c) pure TiO_2 and $\text{TiO}_2/\text{SiO}_2$ composite, showing the suppressed photocatalytic activity and (d) pseudo-first order kinetic of photocatalytic reactions for this degradation process.

223x181mm (96 x 96 DPI)