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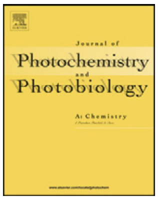

Photocatalytic coatings of silver–TiO_2 nanocomposites on foamed waste-glass prepared by sonochemical process

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ABSTRACT

Silver–TiO_2 nanocomposite was prepared by photodeposition of silver nanoparticles on the surface of titanium dioxide. The sonochemical method was used for the deposition of silver–TiO_2 powder on commercial foamed waste-glass strips (FWGS). The silver–TiO_2 and the coated FWGS was characterized by XRD, DRS, SEM, TEM and nitrogen adsorption. In order to enhance silver–TiO_2 deposition, different parameters were evaluated such as the solvent effect and use of stabilizing agents. The best deposition was obtained with an aqueous solution of polyvinyl alcohol (PVA) and an ultrasound irradiation source of 23.3 kHz. The photocatalytic activity of the silver–TiO_2 coated FWGS was evaluated in the UV photo-assisted destruction of the noxious microalgae, *Tetraselmis suecica*. It was found that after the photocatalytic irradiation, for 180 min, the algae cells were deformed, fragmented and annihilated, thereby avoiding its regeneration.

1. Introduction

Titanium dioxide is a potent photocatalyst that can breakdown almost any organic compound, upon exposure to UV light in aqueous medium. During the process, two highly reactive species are formed, hydroxyl radicals $OH^\bullet$, and superoxide ions $O_2^{\bullet -}$ [1–3]. Hydroxyl radicals are very strong oxidizing agents and can destroy organic materials including, those that make up living cells. Titanium dioxide and TiO_2-composites can achieve a complete bacterial killing process within a few hours. It has been reported that microorganisms such as *Staphylococcus*, *Bacillus subtilis*, *Escherichia coli* [4–8], algae marine [9,10], fungi [11], and virus [12] are inactivated using irradiated TiO_2. Silver–TiO_2 nanocomposites destroy or deactivate pathogenic microorganisms resulting in the inhibition of their growth and reproduction [3–13]. This antimicrobial silver–TiO_2 inorganic material offers several advantages such as high chemical stability, re-usage, and in particular the use of expensive oxidizing agents is not necessary to carry out the photodegradation. Moreover, the silver particles can maintain its metallic state under continuous UV irradiation [7,8]. The immobilization of these antimicrobial materials, on polymers, glass, membranes and foams has been reported previously [5–9]. Foamed waste-glass is a recycled material made of waste bottles and others glasses with good absorption capacity for water that makes it a good support for photocatalytic materials, with a surface roughness. It also has a low density (approximately 0.41–0.44 $g\,cm^{-3}$) and thus will float on water (light-weighted material). A possible solution for the immobilization will be the use of a low density porous support [14]. However, the deposition of silver–TiO_2 as a film coating is an important issue to be satisfactory resolved.

High concentrations of harmful algae microorganisms at the interface of seawater and air, blocking the sunlight illumination have been retentively reported [13–15]. Harmful algae bloom (HAB) species segregate potent toxins that damage the living species in sea water and coastal organisms. In the present work, UV irradiated silver–TiO_2, deposited by sonochemical procedures on commercial foamed waste-glass strips (FWGS) was proposed as an effective photocatalytic material for the inactivation of the noxious marine microalgae *Tetraselmis suecica* (green tide), which was used as a model toxin in the work described herein.

2. Experimental

2.1. Preparation of silver–TiO_2 sample

The silver–TiO_2 nanocomposite was prepared by photodeposition of silver nanoparticles on the surface of commercial TiO_2

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Degussa P25 (TiO_2-P25). First, 0.472 g of $AgNO_3$ (Samchun, 99.8%) were added to 1 L of distilled water and placed in an ultrasound bath for 5 min to assure its complete disaggregation. Then, 29.7 g of TiO_2 were added to the mixture and placed under vigorous stirring for 1 h in order to achieve the adsorption–desorption equilibrium of Ag^+ ions on the TiO_2 surface. Afterwards, the slurry was irradiated with two 20 W UV C-type lamps (Lamp, Sankyo Denky, Japan) for 2 h. The resulting mixture was dried in a vacuum rotary evaporator at 80 °C for 4 h. The solid obtained was dried at 100 °C for 12 h and stored in the dark.

2.2. Characterization of the sample

The structural characterization of the nanocomposite was made by X-ray powder diffraction using a Bruker D8 Advanced diffractometer with CuKα radiation. X-ray diffraction data of samples were recorded between 10° and 70° (2θ). The morphology of the powder was analyzed by scanning electron microscopy (SEM) using a Scanning Electron Microscope JEOL JSM 6700F with an accelerating voltage of 5.0 kV and by transmission electron microscopy (TEM) using a JEOL 2200 FS TEM microscope. The diffuse reflectance spectra of TiO_2-P25 and silver–TiO_2 samples were obtained by using an UV-Vis spectrophotometer Perkin-Elmer Lambda 35 equipped with an integrating sphere, using $BaSO_4$ as coated standard pattern. The energy band gap (E_g) values were calculated with the equation $\alpha(h\nu) = A(h\nu - E_g)^{m/2}$, where α is the absorption coefficient, $h\nu$ is the photon energy, A is a constant and $m = 2$ for a direct transition between the valence band and the conduction band. For the estimation of the E_g from the UV–vis spectra, a straight line was extrapolated from the absorption curve to the abscissa axis. When α is zero, $E_g = h\nu$. The (BET) specific surface area was determined from the nitrogen adsorption isotherm using an Autosorb 3B Quantachrome apparatus. The samples were degassed for 12 h at 300 °C and then the N_2 adsorption–desorption isotherms were recorder at −196 °C. Silver content was determinated by inductively coupled plasma-optical emission spectrometry (ICP-OES) analysis. Samples were first dissolved in acid digestion (HNO_3 solution) prior to analysis with a Varian 700 ICP-OES Atomic emission spectrometer at wavelength of the emission line of 328.068.

2.3. Sonochemical deposition of silver–TiO_2 sample over foamed waste-glass strips

The foamed waste-glass (FWGS) was provided by IPP Ltd., of South Korea. The chemical composition of the FWGS was approximately 74% SiO_2, 15% Al_2O_3, and 11% alkaline oxides and alkaline earth oxides, such as K_2O, Na_2O, MgO, and CaO. The dimension of the foamed waste glass strips was 0.1 m × 0.1 m × 0.01 m. This size was designed in accordance with the dimensions of the reactor system. First, the FWGS was washing with bidistilled water and in an ultrasound of 23.3 kHz for 2 h to remove impurities. Then, the FWGS was coated with TiO_2-P25 or silver–TiO_2 sample using ultrasound irradiation. For this purpose, in 1 L of bidistilled water was added 0.01 g of the stabilizing agent, Polyvinyl alcohol 500 (Junsei Chemical Co., Ltd., chemical pure grade) or citric acid (Tokyo Chemical Industry Co., Ltd., chemical pure grade). The mixture was placed in a homemade ultrasound bath for 0.25 h to promote the dissolution of the stabilizing agent. Then, 1.0 g of silver–TiO_2 or commercial TiO_2-P25 sample and one FWGS was added to the aqueous solution. Thereafter, the samples were placed under ultrasound irradiation for 2 h at 23.3 kHz, 800 W and a pressure of 8×10^{-2} MPa. The temperature was maintained at 20 °C with a cooling system. Subsequently, the FWGS was dried at 60 °C under vacuum overnight.

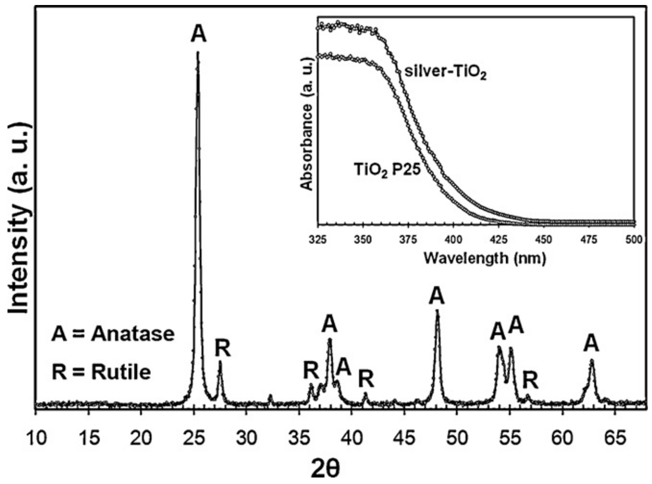

Fig. 1. XRD pattern of powder silver–TiO_2; on insert diffuse reflectance spectra of TiO_2-P25 and silver–TiO_2 powders.

2.4. Photoinactivation of harmful algae

The photocatalytic tests were carried out in a batch reactor similar to that previously reported [16]. The coated FWGS was put into the reactor containing 3 L of a dispersion of the noxious microalgae *T. suecica* in sea water whose initial concentration was $250 \pm 20 \times 10^3$ cells mL^{-1} (NLP Co., Ltd., 99.9%). Then, the dispersion was exposed to UV irradiation (315–400 nm) with three 30 W A-type UV lamps (Black light lamp-BL, Sankyo Denky, Japan). The experiments were performed under magnetic stirring at room temperature (25 °C). At given irradiation time intervals, 100 μL of the suspension were collected and analyzed for the counting of cell concentration with a haemacytometer using an optical microscope Motic-BA200 [10]. The haemacytometer also known as Neubauer chamber consists of a ruled glass slide containing a central square area of 0.1 mm in depth. The haemacytometer is filled by the sample and sealed with a coverslip. About 30–100 living cells were counted and the counting was repeated three times for each sample. The living algae cells show a random movement, while the dead algae do not move and their cell morphology changed to a deformed round shape. The pH of the dispersion was monitored during the photocatalytic experiments, although significant changes were not observed.

3. Results and discussions

3.1. Preparation of silver–TiO_2 sample

The X-ray diffraction pattern of the silver–TiO_2 nanocomposite is shown in Fig. 1. An intense peak at 25.4° (2θ), which corresponds to the anatase phase (JCPDS 21-1272), as well as the diffraction peaks, which correspond to the rutile phase (JCPDS 70-7343) can be seen. The detection of peaks identifying Ag° are not observed, possibly due to its low concentration, 1 wt%. The diffuse reflectance spectra of silver–TiO_2 and TiO_2-P25 samples are shown in inset of Fig. 1. A shift to wavelengths closer to visible light was observed for the silver–TiO_2 sample. The energy band gap values (E_g) for silver–TiO_2 and TiO_2-P25 were 3.0 and 3.2 eV, respectively. The absence of surface plasmon resonance band in the silver impregnated sample may be explained by the possible presence of highly dispersed silver particles on the surface of the TiO_2. On the other hand, the BET specific areas were almost similar (~54 m^2/g) on both samples. Thus, no textural effects were observed due to the deposit of silver nanoparticles on the TiO_2-P25 semiconductor. Previous XPS studies of these samples recently reported showed that

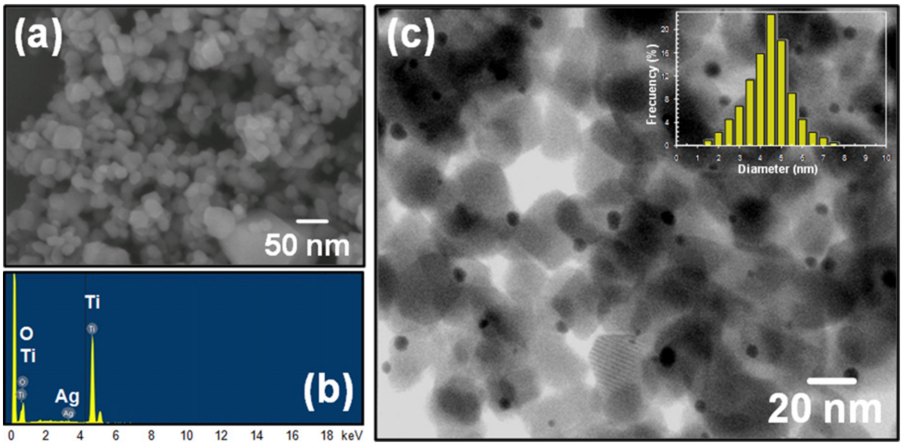

Fig. 2. Electron microscopy images of silver–TiO_2 powder by: (a) SEM; (b) EDS analysis; (c) particle size distribution from TEM micrographs.

the silver nanoparticles are found scattered on the titanium dioxide surface, since there was no evidence of anchorage or chemical bonds of Ag° with Ti^{4+} or oxygen [10]. The morphology of the silver–TiO_2 solid was analyzed by scanning electron microscopy and the SEM picture is presented in Fig. 2(a). The image shows nanoparticles of TiO_2 of 20–30 nm as well as some isolated sintered particles (>50 nm). The presence of silver nanoparticles was not observed by this technique. However, the EDS analysis of the sample confirmed the presence of Ag° on the surface of titanium dioxide, Fig. 2(b). To quantify the amount of silver in the silver–TiO_2 solid nanocomposites, we performed an acid digestion of the sample and analyzed the amount of dissolved silver with an inductively coupled plasma atomic emission spectrometer (Varian 700 ICP-OES). Silver levels of 0.95–0.99% have been obtained.

The TEM image of the sample is shown in Fig. 2(c), where quasi-spherical silver nanoparticles deposited over the TiO_2 surface can be seen. The Ag° particle size observed by TEM micrographs is about of 4.5 nm (inset of Fig. 2(c)).

3.2. Ultrasound-assisted deposition of silver–TiO_2

The deposition of the silver–TiO_2 nanocomposite was carried out through an ultrasound-assisted process. Fig. 3(a) shows the morphology of the uncoated FWGS, where the irregular surface of this material is observed, a waste material as highly porous glass ceramic. When the deposition of silver–TiO_2 was performed in aqueous media under ultrasound irradiation, several interconnected aggregates of quasi-spheres were observed attached to the FWGS surface, as it can be seen in Fig. 3(b). Kurikka et al. [17] reported a mechanism for the ultrasound deposition showing that the ultrasound process does not alter the TiO_2 structure. In brief, when the aqueous media contain the powder, the ultrasound irradiation seems to cause cavitations phenomenon, providing the formation, growth and collapse of bubbles. It is proposed that the cavity collapse drives high-speed microjets of water contained the powder to the FWGS surface. Then, these microjets hit the FWGS surface and can interact with free Si–OH, Si–O, or Al–OH and Al–O bonds. This may account for the adhesion of the nanocomposite to the surface, forming Ti–O–Si or Ti–O–Al [17–19].

The sonochemical deposition of silver–TiO_2 was also performed in ethanol as solvent under the same conditions of ultrasound irradiation and amount of sample. The results showed low deposition, as is indicated in Fig. 3(c). One explanation for this remarkable difference between both solvents (water and ethanol) can be due to the different dielectric constants, which is a property of a solvent to separate electrically charged particles. Solid oxide powders can be stable in suspension because of their surface-electrical charges. However, the particles lean to agglomerate according to the ceramic nature of the powder causing sedimentation. Electrostatic stabilization is thought to be more effective in high-dielectric-constant media because the particles are surrounded by concentric layers of solvent molecules, the well-known process of solvation. Thus, water has a dielectric constant of 80 (at 20 °C), while ethanol has only a value of 25 at the same temperature. This is explain that in aqueous media the nanoparticles are not agglomerated and can be launched in the high-speed microjets during the cavity collapse which is caused by the ultrasound irradiation.

On the other hand, two additional experiments were conducted using citric acid and polyvinyl alcohol (PVA) as stabilizing agents of the silver–TiO_2 powder in aqueous suspensions. Fig. 4(a) shows the SEM micrographs for the deposition process using citric acid at a concentration of 0.01 g/L. The SEM results showed that the deposition under these conditions was similar that the obtained with ethanol. However, when PVA (0.01 g/L) was used, a significant increase in the deposition on the FWGS was observed, Fig. 4(b). This result suggests that polyvinyl alcohol stabilizes the nanoparticles in aqueous media by the formation of adsorbed polymer layers avoiding the particles agglomeration. In fact, the PVA is often used as a stabilizer due to its optical clarity, which enables the investigation of nanoparticles formation [20]. Because the PVA favored the deposition process, only this sample was used for the evaluation of the photocatalytic activity.

After the deposition using PVA as stabilizer, the slurry was separated and dried in a vacuum rotary evaporator at 80 °C for 4 h. The resultant powder was dried at 100 °C for 12 h in an oven and then analyzed by scanning electron microscopy. Several aggregates with dendritic-like structures were observed. These aggregates possess several trunks between 0.5 and 1.5 μm, where each trunk contains secondary and tertiary branches with lengths less than 100 nm, Fig. 5(a) and (b). Each branch seems to be built by silver–TiO_2 crystallites of 25 nm in average [10]. A similar morphology was observed by Koltypin and co-workers in the preparation of WO_3 by ultrasound irradiation [21]. It is possible that the formation of the dendritic-like structures involves the spontaneous self-organization of the particles. In a surface a raising coat of silver–TiO_2 is formed, Figs. 2–5. Many models and explanations have been proposed to account for this phenomenon, most of which were focused on non-equilibrium growth and molecular anisotropy [22]. Based on our experimental conditions, the formation of these dendritic-like structures may be due to the use of ultrasound irradiation. When the ultrasound irradiation is applied to the system, the shock waves hit the surface of the particles, causes local heating of several hundred degrees in its surface. However, the cooling

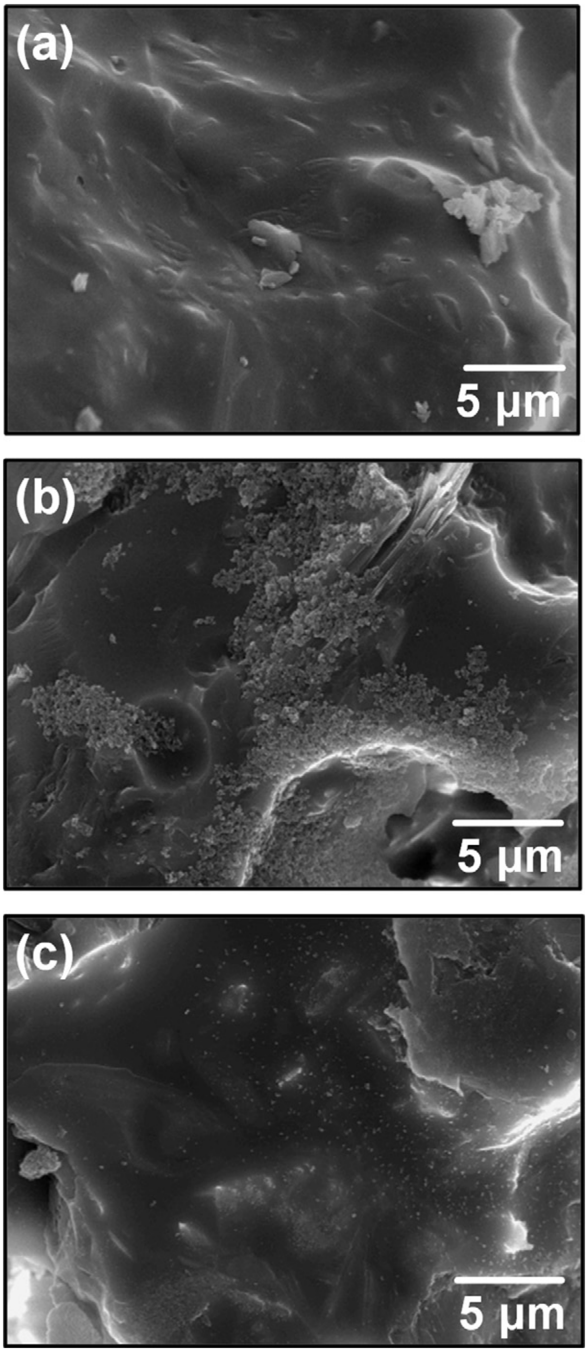

Fig. 3. SEM micrographs of: (a) uncoated foamed waste glass; (b) foamed waste glass deposited with silver–TiO_2 in aqueous media; (c) foamed waste glass deposited with silver–TiO_2 in ethanol media.

system of the ultrasound bath system may give fast cooling to the surface of the particles. According to non-equilibrium growth theory, dendritic pattern growth from solutions depends on the applied supercooling conditions, so the external conditions may significantly affect the morphological growth of the crystals [22]. Alternatively, previous studies have shown that PVA can act also as a structure-directing agent, resulting from the number of polar hydroxyl groups which are present in the PVA molecule. They can strongly interact with a variety of organic and inorganic materials [23].

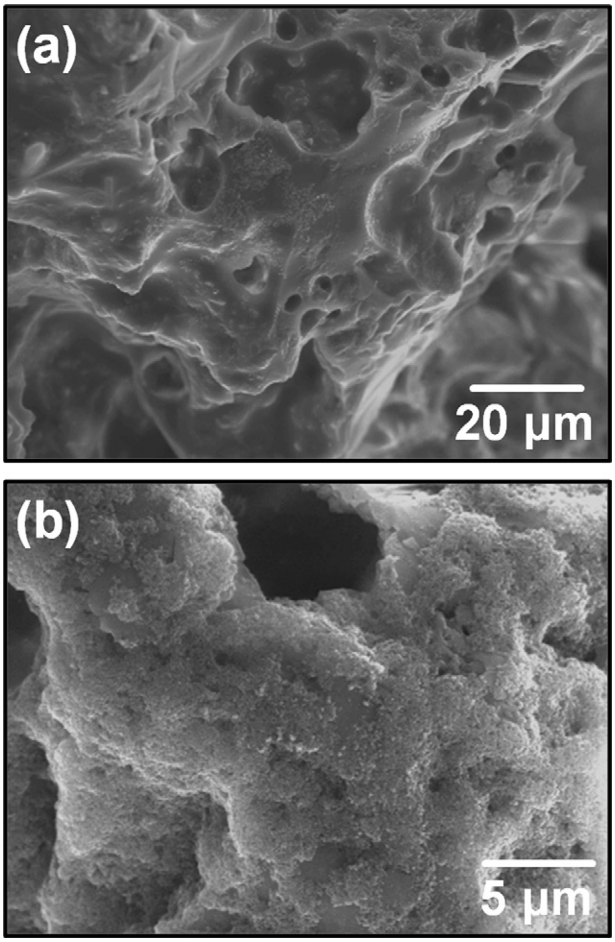

Fig. 4. SEM micrographs of the foamed waste glass deposited with silver–TiO_2 using: (a) citric acid; (b) polyvinyl alcohol (PVA) as stabilizing agent in aqueous media.

3.3. *Photoinactivation of harmful algae*

The photocatalytic activity of the silver–TiO_2 coated FWGS was evaluated by the elimination of the harmful algae *T. suecica* under UV irradiation. For comparative purposes, TiO_2-P25 coated FWGS were evaluated under the same photocatalytic conditions. The TiO_2-P25 coated strips were prepared under identical conditions to that used for the silver coated FWGS. Fig. 6 shows the survival fraction of the *T. suecica* microalgae irradiated with UV light (315–400 nm) in the presence or absence of photocatalysts. In absence of photocatalyst about 6.4% of the cells stopped moving and drop to in the bottom of the reactor after 180 min of irradiation. However, the algae regained movement when the light was turn-off suggesting that the UV irradiation does not induce the fatal damage of marine algae, Fig. 6(a). A second experiment was performed with the uncoated FWGS and the algae dispersion under UV light; see Fig. 6(b). The results show a decrease of 13% of living cells after 3 h of irradiation. The silver–TiO_2 coated strip was examined for the removal of the microalgae in dark conditions in order to check the capability of silver biocide in absence of UV light. Under these conditions, the biocide ability of the silver nanoparticles was close to 26% after 180 min, Fig. 6(c). On the other hand, the TiO_2-P25 coated sample showed a remarkable increase in the photokilling properties under UV irradiation up to the conditions of points (a) to (c), Fig. 6(d). However, the sample with the best photocatalytic activity was the silver–TiO_2 coated FWGS, Fig. 6(e). This nanocomposite shows an elimination of 99% of the algae cells after 180 min

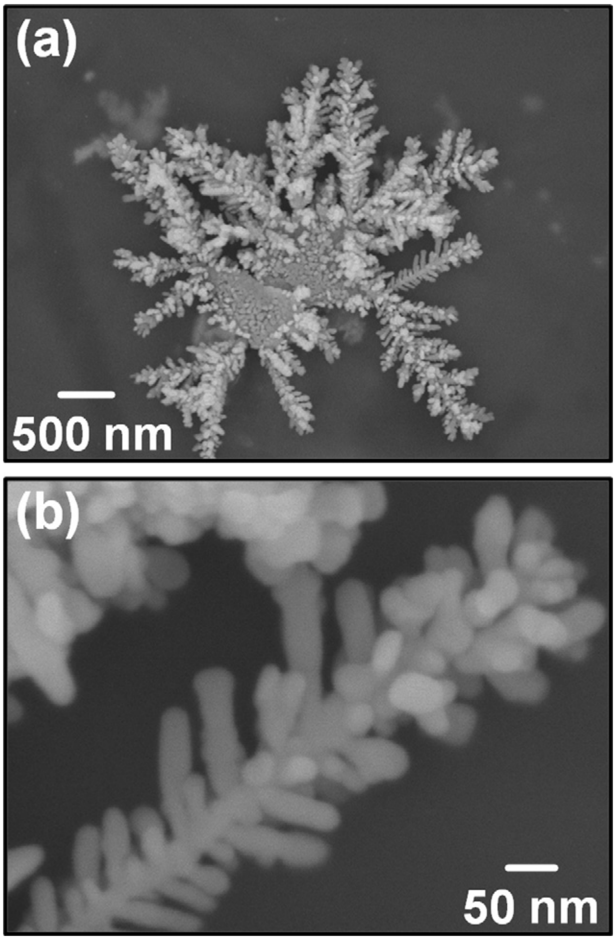

Fig. 5. SEM images showing the formation of silver–TiO_2 dendritic-like structures after ultrasound irradiation.

under UV irradiation. From to observations made with the optical microscope, it can be seen that the algae cells adsorbed over the coated FWGS were deformed in appearance from round to semi-ovoid forms and then fragmented until they are totally destroyed. After the UV irradiation of silver–TiO_2 coated FWGS, a sample of 1 L of irradiated solution was added to 2 L of nutritive water (Natural Live Plankton Co., Ltd.,) and incubated at 25 °C for 72 h. Then this suspension was analyzed for the counting of cell concentration; no living cells were found. These results imply that the combination of photocatalytic coated FWGS and UV irradiation are needed to inactivate the marine algae from the dispersion.

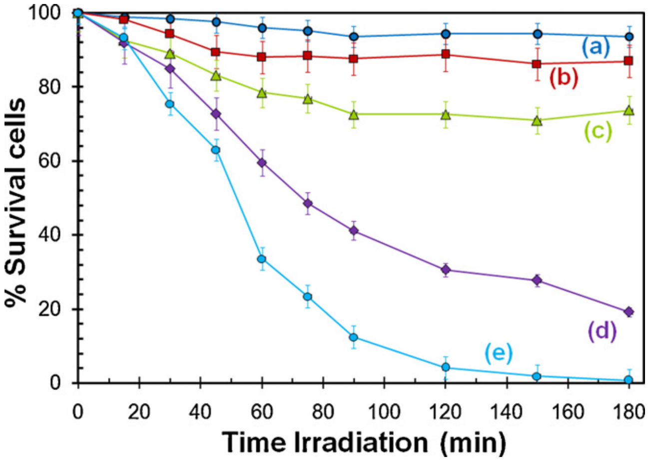

Fig. 6. Survival fraction of *Tetraselmis suecica* under different photocatalytic conditions: (a) only UV-irradiation without photocatalyst; (b) uncoated foamed waste glass and UV-light; (c) silver–TiO_2 coated strip in dark conditions; (d) TiO_2-P25 coated strip and UV-light; (e) silver–TiO_2 coated strip under UV irradiation.

The mechanistic insight of the photokilling action of the silver–TiO_2 nanocomposite to the algae remains unclear. Several studies have proposed that the silver nanoparticles may attach to the surface of the cell membrane disturbing permeability and respiration functions of the cells [24]. On the other hand, TiO_2 particles generate strong oxidizing radicals when they are illuminated with UV light. The oxidizing agents, superoxide ions and hydroxyl radicals, cause irreversible damage to the living algae cells. The synergistic effect of the silver and TiO_2 help to the fragmentation of the cytoplasm, because the biocide properties of silver inhibit the growth of unicellular algae. Silver nanoparticles, deposited on a TiO_2 surface also diminish the electron–hole recombination, leading to the destruction of marine algae. Under UV irradiation the lixiviation of silver to the solution may become negligible. The ICP analysis detected 0.0727 ppm of silver after 1 h of stirring a piece of FWGS under UV light. FWGS preparation results in better stability than coated photodeposited material, which may improve the photochemical process. Lixiviation of the coating during the reaction is negligible. One of the main contribution is the formation of an oxide composite coating on the surface of a low-dense material (FWGS); this process seems to be inexpensive and practical compared to aqueous suspensions systems for decontamination of water that contains pathogenic microorganisms.

4. Conclusions

The silver–TiO_2 nanocomposite was immobilized on a low-density glass material (FWG) by a novel sonochemical process in the presence of polyvinyl alcohol (PVA) which is used as a stabilizing agent. The low-density glass material permits to float on water and therefore the photocatalytic material can react under UV-A light. It is shown that the sonochemistry is a technique which can be used for the coating of nanoparticles, such as it was achieved with silver–TiO_2. The immobilized silver–TiO_2 coat shows significant photocatalytic activity for the elimination of the microalgae *T. suecica* under UV-A irradiation. The synergic effect of silver nanoparticles and TiO_2 allows the deactivation of the pathogenic *T. suecica*, resulting in inhibition of its growth and reproduction. This immobilized system may be practical for microorganism elimination in seawater.

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