Facile preparation of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites as an effective photocatalyst via a new route

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Abstract

$\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites were prepared via a facile process with propylene glycol as novel connecting agent and benzene tricarboxylic acid as a new complexing agent. The as-obtained $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites were characterized by transmission electron microscopy (TEM), UV–vis diffuse reflectance spectroscopy, energy dispersive X-ray microanalysis (EDX), Fourier transform infrared (FT-IR) spectroscopy,

field emission scanning electron microscopy (FESEM), and X-ray diffraction (XRD). According to the morphological studies of the as-synthesized nanocomposites, it was found that the shape and particle size of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites depended on the space-filling template type, dosage of space-filling template and tricarboxylic acid as complexing agent. $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites with different shapes and grain sizes have been synthesized. The photocatalytic behavior of as-produced $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites was also investigated through photodegradation of methylene blue dye and 2-naphthol as water pollutants.

Keywords: Photocatalyst; Nanomaterials; Photodegradation; Nanocomposites; Electron microscopy

1. Introduction

The nanoscale materials have various characteristics with bulk materials owing to their characteristics depends on their shape and grain size. In recent years, the investigation of synthesis and characteristics of several nanoscale materials has received intense attention [1,4]. One of the considerable and attractive nanoscale materials is $\text{Ln}_2\text{Zr}_2\text{O}_7$ -kind ternary oxides (Ln is a lanthanide ion) that they find applications in high-temperature fuel cell, photocatalyst, oxide conductor, fluorescence centers, oxygen monitoring sensors, gas turbines, actinide transmutation, oxidation catalysts and nuclear waste form owing to their excellent and remarkable characteristics [5–10]. To date, different ways, such as hydrothermal, sol–gel combined electrospinning, complex precipitation, Pechini polymeric route, solid-state reaction and sol–gel have been presented for the production of $\text{Ln}_2\text{Zr}_2\text{O}_7$ [11–18]. The synthesis of $\text{Ln}_2\text{Zr}_2\text{O}_7$ and its nanocomposites through Pechini polymeric approach as friendly to the environment way provides a reliable, simple and cost effective process to control their grain size, shape and purity. Great attentions have been devoted to the size and shape controlled fabrication of nanoscale materials owing to their characteristics and ultimate applications depends on their size and shape [19–22].

Herein, $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites are prepared through a simple process by utilizing propylene glycol as novel connecting agent and benzene tricarboxylic acid as a new complexing agent. To our knowledge, it is the first time that benzene tricarboxylic acid is employed as a complexing agent in the presence of propylene glycol as connecting agent for the production of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites and the effects of factors, including space-filling template type, dosage of space-filling template and tricarboxylic acid as complexing agent on the shape and particle size of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites through a simple process are investigated.

2. Experimental

2.1. Materials and characterization

Neodymium nitrate, poly ethylene glycol 6000 (PEG 6000), propylene glycol, zirconyl nitrate, poly ethylene glycol 600 (PEG 600), benzene tricarboxylic acid, poly ethylene glycol 400 (PEG 400) and liquor ammonia solution containing 25% ammonia with analytical grade were bought from Merck Company and were utilized as received. Powder X-ray diffraction (XRD) pattern of the as-obtained nanocomposites were recorded by applying a diffractometer of the Philips Company with X'Pert Promonochromatized Cu $K\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$). The EDS analysis of the as-produced $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites was done with a Tescan mira3 microscope. FESEM images of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites were visualized by a Tescan mira3 field emission scanning electron microscope (FESEM). Transmission electron microscope (TEM) images of the as-synthesized $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites were taken on a JEM-2100 with an accelerating voltage of 200 kV. Fourier transform infrared spectra of the as-formed samples were obtained on a Shimadzu Varian 4300 spectrophotometer in KBr pellets in the $400\text{--}4000 \text{ cm}^{-1}$ range. GC-2550TG (Teif Gostar Faraz Company, Iran) were used for all chemical analyses. The UV-vis diffuse reflectance spectra of the as-produced nanostructured ZrO_2 and $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites were obtained on a UV-vis spectrophotometer (Shimadzu, UV-2550, Japan). Thermogravimetric-differential thermal analysis (TG-DTA) of the as-produced product was done through a thermal gravimetric analysis instrument (Shimadzu TGA-50H) with a flow rate of 20.0 mL/min and a heating rate of $10 \text{ }^\circ\text{C/min}$.

2.2. Synthesis of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites

$\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites were prepared through the modified Pechini process. First, 1 mmol of neodymium nitrate and 2 mmol of zirconyl nitrate were dissolved in distilled water. After adding 2 mmol of benzene tricarboxylic acid as new complexing agent to the mixed solution as well as adjusting the pH of the resultant solution to 6 by adding ammonia solution drop-wise, 1.2 mmol of propylene glycol as novel cross-linker was added the solution. Evaporation of the gel-like solution results in the formation of porous solid mass which was ground utilizing acetone in the agate mortar. After heating the produced powders at $350 \text{ }^\circ\text{C}$ for 3 h and then at $800 \text{ }^\circ\text{C}$ for 5 h, the $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites were synthesized (sample no. 2). [Fig. 1](#) illustrates a schematic

diagram of the preparation of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites. The effects of space-filling template type, dosage of space-filling template and tricarboxylic acid as complexing agent on the shape and particle size of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites were investigated (Table 1). To examine the space-filling template influence, a stoichiometric quantity of the space-filling template added after adding cross-linker (propylene glycol) to the solution.

Fig. 1. Schematic diagram of the preparation of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites.

2.3. Photocatalytic measurements

The photocatalytic behavior of as-obtained nanostructured ZrO_2 and $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites was investigated by utilizing of the cationic dye (methylene blue) and 2-naphthol as water pollutants. The reaction solution comprising the 40 g of the nanostructures and 1 mg of the water pollutant in the quartz reactor was employed to examine the photocatalytic behavior. After aerating for 1/2 h, the mixture was subjected to the

illumination of the UV light from the 400 W mercury lamps. The water pollutant photodegradation percentage was determined as follows:

$$\text{D.P. } (t) = (A_0 - A_t/A_0) \times 100 \quad (1)$$

where A_0 and A_t are the absorbance value of water pollutant solution at 0 and t min by a UV-vis spectrometer, respectively.

Table 1. The preparation conditions of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites produced in this investigation.

Sample no.	Complexing agent	Space-filling template	Dosage of space-filling template (mmol)	Nd:Zr	Figure of FESEM images
1	-	-	-	1:2	Fig. 6(a) and (b)
2	Benzene tricarboxylic acid	-	-	1:2	Fig. 6(c) and (d)
3	Benzene tricarboxylic acid	PEG 400	0.015	1:2	Fig. 7(a) and (b)
4	Benzene tricarboxylic acid	PEG 600	0.015	1:2	Fig. 7(c) and (d)
5	Benzene tricarboxylic acid	PEG 6000	0.015	1:2	Fig. 7(e) and (f)
6	Benzene tricarboxylic acid	PEG 600	0.0075	1:2	Fig. 8(a) and (b)
7	Benzene tricarboxylic acid	PEG 600	0.03	1:2	Fig. 8(c) and (d)
8	Benzene tricarboxylic acid	PEG 600	0.045	1:2	Fig. 8(e) and (f)
9	Benzene tricarboxylic acid	PEG 600	0.015	0:2	-

3. Results and discussion

Fig. 2 presents the infrared spectra of the as-obtained powders (sample no. 4 before calcination stages) and as-produced $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4). The peaks located at 1701 and 1617 cm^{-1} in the Fig. 2(1) may correspond to C=O stretching vibration of benzene tricarboxylic acid and O–N=O asymmetric stretching vibration of the nitrates [23]. In the Fig. 2(1), the peaks appear at 1549, 1392 and 1043 cm^{-1}

Fig. 2. FT-IR spectra of the as-obtained powders (sample no. 4 before calcination stages) (1) and as-produced $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4) (2).

belong to the (C=O) vibration as well as the COO^- and (C–O) stretching vibration, respectively [24]. In Fig. 2(1), the peaks located at 1132, 933 and 827 cm^{-1} may correspond to the C–C–O stretching vibration of PEG and propylene glycol [23]. The peaks appear at 760, 731 and 555 cm^{-1} in the Fig. 2(1) belong to the N–O stretching vibration of the nitrates and C–O vibration of the PEG and propylene glycol [23]. The peak centered at 3422 cm^{-1} in the FT-IR spectrum of the as-obtained $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4) may correspond to the $\nu(\text{OH})$ stretching vibration of the surface adsorbed H_2O molecules (Fig. 2(2)) [3]. In Fig. 2(2), the characteristic peak of $\text{Nd}_2\text{Zr}_2\text{O}_7$ appears at 420 cm^{-1} [25].

Fig. 3 depicts thermogravimetry (TG) of as-obtained powders (sample no. 4 before calcination stages). The TG curve demonstrated that as-produced powders (sample no. 4 before calcination stages) undergo two mass loss steps. The first mass loss occurs in the 25–425 °C and displays 52.1% mass loss, which corresponds to the dehydration and depolymerisation of the polymeric residual in the as-formed powders as well as complex

Fig. 3. TGA curve of the as-obtained powders (sample no. 4 before calcination stages).

decomposition and polymeric resin combustion. The occurred step at the 425–800 °C (illustrating 22.34% weight loss) may be related to the removal of the nitrates and residual carbonate intermediate and indicate the preparation of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites.

The composition and crystalline structure of the as-obtained $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4) were characterized by XRD pattern, as depicted in Fig. 4. The XRD pattern of the sample no. 4 illustrates the formation of cubic phase $\text{Nd}_2\text{Zr}_2\text{O}_7$ (JCPDS No. 78-1618) along with tetragonal phase ZrO_2 (JCPDS No. 80-0965). The mean crystallite diameter of the as-synthesized $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4) determined by the Scherrer formula [3] was about 14 nm.

Fig. 4. XRD pattern of the as-produced $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4).

The chemical purity level of the as-synthesized nanostructured $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ (sample no. 4) was evaluated by EDS pattern, as illustrated in Fig. 5. This EDS pattern reveals the presence of Nd, Zr and O in samples no. 4. Hence, the synthesized product may be called $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites.

Fig. 5. EDS pattern of the as-produced $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4).

As mentioned before, in this investigation $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites were prepared via a facile modified Pechini process with benzene tricarboxylic acid as a new complexing agent in presence of propylene glycol as cross-linker. To investigate the influence of the benzene tricarboxylic acid as new complexing agent on the shape and particle size of $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites, sample nos. 1 and 2 were synthesized in absence and presence of the benzene tricarboxylic acid, respectively. The sample no.1 prepared in absence of the benzene tricarboxylic acid show not uniform nanobundles with large particle size (Fig. 6a and b). By applying the benzene tricarboxylic acid, less uniform sphere-like nanoparticles with fine particle size are prepared (Fig. 6c and d). It

seems that the benzene tricarboxylic acid as organic ligand with chelating effect acts as complexing agent and capes the external surface of nanocrystals, and hence controls growth of formed particles.

Fig. 6. FESEM images of the sample nos. 1 and 2 were synthesized in absence (a and b) and presence (c and d) of the benzene tricarboxylic acid, respectively.

To study the effect of the different space-filling templates on the shape and particle size of the $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites, sample nos. 3, 4 and 5 were synthesized by 0.015 mmol of PEG 400, PEG 600 and PEG 6000 in

presence of benzene tricarboxylic acid. The FESEM images reveal that the high agglomerated particle-like

Fig. 7. FESEM images of the sample nos. 3, 4 and 5 were synthesized by 0.015 mmol of PEG 400 (a and b), PEG 600 (c and d) and PEG 6000 (e and f) in presence of benzene tricarboxylic acid, respectively.

structures (Fig. 7a and b), very uniform nanoparticles with spherical shape (Fig. 7c and d) and irregular nanobundles (Fig. 7e and f) are prepared in the presence of PEG 400, PEG 600 and PEG 6000, respectively. It seems that among these space-filling templates with different molecular weights, PEG 600 can act as the best space-filling template because of the suitable quantity of carbon source and leads nanoparticles embedded in polymer matrix to become crystalline without interacting with other prepared nanoparticles. It can be deduced that PEG 600 in presence of benzene tricarboxylic acid was the best space-filling template type for very uniform spherical $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanoparticles with fine particle size (Fig. 7c and d).

To examine the effect of space-filling template dosage on the shape and particle size of the $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites, FESEM images of sample nos. 6, 7 and 8 with 0.0075, 0.03 and 0.045 mmol of PEG 600 were taken and depicted in Fig. 8(a-f), respectively. The FESEM images demonstrate that the less uniform nanostructures with string-like (Fig. 8a and b), high agglomerated particle-like structures/irregular micro-bundles (Fig. 8c and d) and high agglomerated particle-like structures (Fig. 8e and f) are synthesized in the presence of 0.0075, 0.03 and 0.045 mmol of PEG 600, respectively. By changing the PEG 600 dosage from 0.0075 to 0.015 mmol, the amount of the uniform $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanoparticles enhanced and the particle size becomes finer (Fig. 7c and d). It seems that the more dosage of PEG 600 causes the formed particles to be agglomerated (Fig. 8c-f). It can be deduced that 0.015 mmol of PEG 600 is the optimum dosage for very uniform spherical $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanoparticles.

According to the FESEM studies, utilizing propylene glycol as cross-linker and benzene tricarboxylic acid as new complexing agent in presence of 0.015 mmol of PEG 600 as space-filling template is the optimum condition for very homogeneous spherical $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanoparticles with fine particle size (Fig. 7c and d). To elucidate

the detailed morphological characteristics of the prepared $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites in the presence of the benzene tricarboxylic acid and 0.015 mmol of PEG 600 (sample no. 4), TEM analysis was

Fig. 8. FESEM images of the sample nos. 6, 7 and 8 with 0.0075 (a and b), 0.03 (c and d) and 0.045 (e and f) mmol of PEG 600.

performed. The TEM image (Fig 9a) demonstrates that quasi-spherical $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanoparticles with diameter in the range of 6–10 nm are agglomerated. The high-resolution TEM (HRTEM) images of a single nanoparticle (sample no. 4) in Fig. 9(b–d) demonstrate that the nanoparticle is highly crystalline. The marked lattice fringes (white and yellow color) correspond to (102) plane in tetragonal phase of ZrO_2 with a d-spacing of 0.21 nm and (331) plane in the cubic phase of $\text{Nd}_2\text{Zr}_2\text{O}_7$ with a d-spacing of 0.244 nm (Fig. 9d).

Fig. 9. TEM (a) and HRTEM (c-d) images of the as-produced $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4).

To examine the optical characteristics of the as-synthesized $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4) and nanostructured ZrO_2 (sample no. 9), UV-vis diffuse reflectance spectroscopy was utilized. Fig. 10(a and b)

Fig. 10. UV-vis diffuse reflectance spectra (a) of the as-synthesized $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites (sample no. 4) and nanostructured ZrO_2 (sample no. 9) and the curves of $(\alpha h\nu)^2$ against $h\nu$ (b).

presents the UV–vis diffuse reflectance spectra of the sample nos. 4 and 9. The absorption bands located at 360 and 356 nm in the spectrum of Nd₂Zr₂O₇-ZrO₂ nanocomposites and nanostructured ZrO₂, respectively. The energy gap (E_g) as considerable parameter in the photocatalytic performance of the nano-sized materials may be estimated based on the UV–vis diffuse reflectance data utilizing Tauc's formula [3]. The E_g of Nd₂Zr₂O₇-ZrO₂ nanocomposites and nanostructured ZrO₂ was calculated through extrapolating the linear section of the plot of $(\alpha h\nu)^2$ against $h\nu$ to the energy axis (Fig. 10b). The E_g amount of Nd₂Zr₂O₇-ZrO₂ nanocomposites and nanostructured ZrO₂ were estimated to be 3.28 and 3.375 eV, respectively. From the determined E_g values, it is found that the as-synthesized Nd₂Zr₂O₇-ZrO₂ nanocomposites and nanostructured ZrO₂ (sample nos. 4 and 9) may be applied as the photocatalyst.

In this study, the photooxidation of methylene blue dye experiments under UV light were carried out to investigate the photocatalytic behavior of the as-obtained Nd₂Zr₂O₇-ZrO₂ nanocomposites (sample no. 4) and nanostructured ZrO₂ (sample no. 9). Fig. 11 presents the results of the photocatalytic tests. No methylene blue dye was practically decomposed after 50 min in the absence of the as-produced Nd₂Zr₂O₇-ZrO₂ nanocomposites and nanostructured ZrO₂ (sample nos. 4 and 9) or the UV light illumination. From the obtained results, it was found that the contribution of self-decomposition was negligible. The proposed mechanism of the decomposition of methylene blue may be assumed as:

Fig. 11. Photocatalytic methylene blue dye degradation of the as-synthesized Nd₂Zr₂O₇-ZrO₂ nanocomposites (sample no. 4) and nanostructured ZrO₂ (sample no. 9).

Applying the photocatalytic calculations by Eq. (1), the methylene blue dye decomposition was about 85% and 42% after 50 min irradiation of UV light and the as-synthesized Nd₂Zr₂O₇-ZrO₂ nanocomposites (sample no. 4) as a new type of photocatalyst under illumination of UV light illustrated better photocatalytic performance than the nanostructured ZrO₂ (sample no. 9). In this study, Nd₂Zr₂O₇-ZrO₂ as new type of nanocomposites was produced. The photocatalytic activity of the synthesized Nd₂Zr₂O₇-ZrO₂ nanocomposites illustrates an improvement under illumination of UV light compared with the nanostructured ZrO₂ (Fig. 11). It seems that the better photocatalytic behaviour of the as-obtained Nd₂Zr₂O₇-ZrO₂ nanocomposites than the nanostructured ZrO₂ may be attributed to its lower E_g value compared with nanostructured ZrO₂ (Fig. 10b). In the case of Nd₂Zr₂O₇-ZrO₂ nanocomposites

with lower E_g value, the recombination rate of charges (e^- and h^+) is lower and thus the photocatalytic efficiency is better [26]. The photocatalytic activity of the as-obtained $Nd_2Zr_2O_7-ZrO_2$ nanocomposites (sample no. 4) and nanostructured ZrO_2 (sample no. 9) was further studied by monitoring the degradation of 2-naphthol as water pollutant in an aqueous solution, under UV light irradiation (Fig. 12). According to photocatalytic calculations by Eq. (1), the 2-naphthol degradation was about 92% and 48% after 20 min irradiation of UV light and the as-synthesized $Nd_2Zr_2O_7-ZrO_2$ nanocomposites (sample no. 4) as a new type of photocatalyst under illumination of UV light illustrated better photocatalytic performance than the nanostructured ZrO_2 (sample no. 9). These results suggest as-produced $Nd_2Zr_2O_7-ZrO_2$ nanocomposites as an interesting candidate for photocatalytic applications under UV light in pollutant elimination process from waste-water.

Fig. 12. Photocatalytic 2-naphthol degradation of the as-synthesized $Nd_2Zr_2O_7-ZrO_2$ nanocomposites (sample no. 4) and nanostructured ZrO_2 (sample no. 9).

4. Conclusions

This work describes an eco-friendly, simple, reliable and cost-effective process to produce $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites with the aid of propylene glycol as novel connecting agent and benzene tricarboxylic acid as a new complexing agent. By changing the space-filling template type and dosage of space-filling template, we could prepare $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites with different shapes and grain sizes. Photooxidation of methylene blue and 2-naphthol tests were done to evaluate the behavior of the produced $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ as new type of nanocomposites. It was illustrated that the photocatalytic behavior of the obtained $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites was better than that for nanostructured ZrO_2 under irradiation of UV light. This result suggests that as-produced $\text{Nd}_2\text{Zr}_2\text{O}_7\text{-ZrO}_2$ nanocomposites as new, desirable and promising material has fine potential to be employed for photocatalytic uses under UV light.

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