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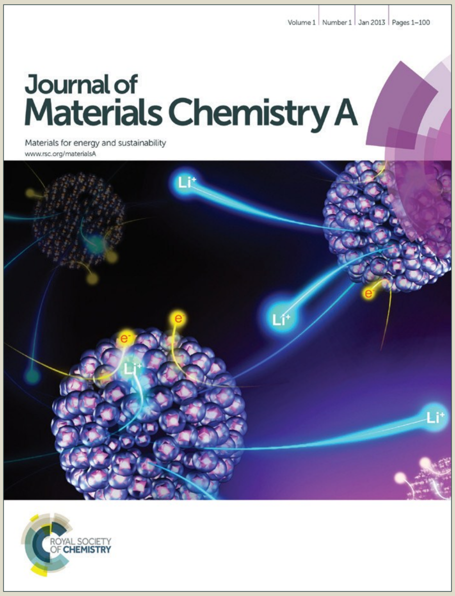

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ARTICLE

Preparation of Recyclable CdS Photocatalytic and Superhydrophobic Film with Photostability by Screen-printing Technique

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PDMS-coated CdS film has been fabricated using screen-printing technique on common glass without calcination. The as-synthesised CdS film exhibits excellent photostability and enhanced photocatalytic activity for the degradation of some organic pollutants, such as RhB, CB, MB, MO and 2,4-DNP. Above all, the CdS photocatalytic film displayed high stability, recyclability and practicability. In addition, the CdS film possess superhydrophobic and superoleophilic properties, and could effectively separate the mixture of water and oil. The coatings can clean themselves by the action of water, and have an property of chemically breaking down the complex dirt deposits by sunlight-assisted cleaning mechanism. The simple fabrication process and easy manipulation of the films cleverly combined superhydrophobic and photocatalysis, and promise practical applications and industrial use of these materials in environmental remediation.

Introduction

Along with the process of global industrialization, environment pollution from organic pollutants and toxic waste water has drawn increasing attention.[1-3] In recent years, semiconductor photocatalysis has been widely used as a potential route to solve the problem owing to its low energy input, easy operation, cleanness and high efficiency.[4-7] TiO_2 and ZnO have been investigated in photocatalysis intensively but they require UV radiation (<385 nm) for photoactivation.[8-9] It's well known that the amount of solar UV light reaching the earth is far less than visible-light (ca. 3–6%). Therefore, it is indispensable to develop novel photocatalysts with visible-light response.[10]

Among the reported photocatalysts with visible-light response, crystalline cadmium sulfide (CdS) has become one of the most widely studied nanocrystalline semiconductors in photosensitizers and photoelectrochemical cells due to the suitable band gap energy of 2.42 eV and important optical properties.[11-13] Owing to its unique properties, CdS is widely used in many fields such as photoelectric conversion,[14] light-emitting diodes,[15] biological labeling,[16] photocatalytic materials[17-18] and so on. However, there exist some problems over CdS nanoparticles including the photocorrosion and higher toxicity.[19-20] Therefore, it is absolutely vital to develop a suitable method to solve the above two problems of this material.

Recently, several attempts have been made to address the above two problems and this is a very active topic of research in CdS photocatalysis. For example, Hu et al. have reported a facile one-pot solvothermal method to synthesize carbon-coated CdS petalous nanostructures with enhanced photostability and photocatalytic activity.[20] Li et al. fabricated a CdS nanoparticle–carbon nanotube hybrid sponge which acted as an efficient porous photocatalyst with high stability and recyclability.[21] However, these synthetic processes require complicate equipment and high cost, and the products agglomerate easily in these processes. At the same time, the use up catalyst suspensions in slurry needs centrifugalization that is expensive in terms of time, reagents and manpower.[22] So it is still a great challenge to immobilize powdery photocatalist by a facile, economical and effective method. Currently, screen-printing method is widely used as a facile and efficient method for the synthesis of films with different photocatalists on different carriers owing to its recyclability, simple operation and low cost. Thus, a facile screen-printing method to obtain recyclable CdS film with high photocatalytic activity is highly desirable.

In this paper, CdS film has been deposited by screen-printing in glass and then used in the decolouration of organic pollutants (such as RhB, CB, MB, MO and 2,4-DNP) solutions under visible-light irradiation. As far as we know, this technique for the fabrication of CdS photocatalytic film on glass without calcination has never been reported, although TiO_2 and ZnO layers have been deposited by screen-printing in

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common ceramic tiles for photodegradation.[23-26] Meanwhile, the reports that the CdS photocatalytic degradates nitrophenols are few up to now. In addition, the CdS film possess superhydrophobic and superoleophilic properties, and could effectively separate the mixture of water and oil.

Experimental

Materials and synthesis

All reagents were of analytical grade, purchased from Shanghai Chemical Reagent Factory, and used without further purification. In a typical synthesis, $CdSO_4·8/3H_2O$ (0.02 mmol) and $Na_2S·9H_2O$ (0.022 mmol) were dissolved in anhydrous ethanol (40 mL) with stirring. This homogeneous mixture was then transferred into a Teflon-lined stainless-steel autoclave and kept at 160 ^{o}C for 12 h. After cooling down, the CdS products were collected by centrifugation, and then were washed with anhydrous ethanol and distilled water. Finally, the products were dried in air at 70 ^{o}C for 12 h. Active films of CdS were deposited on commercial glass (5 cm × 10 cm from local shop) using a well-established screen-printing technique. In order to deposit the photoactive film, the CdS powders were suspended (1:2 wt. %) in polydimethylsilicone (PDMS) and the films were printed through sieved screen on glass.

Characterization

The obtained samples were characterized by X-ray powder diffraction (XRD) using a MiniFlex ⅡX-ray diffractometer operated at 40 kV and 40 mA using Cu K radiation (λ=0.15406 nm). The scanning electron microscopy (SEM) images were obtained on JEOL JSM-6360. Fourier transform infrared spectra (FT-IR) were recorded on a Perkin-Elmer 1600 FT-IR spectrometer with a KBr disk. The content of Cd^{2+} in solution is determined by flame atomic absorption spectrometry (AA-7000, Shimadzu).

Photocatalytic activity under visible-light irradiation

The photocatalytic activity was evaluated using methyl orange (MO), rodamine B (RhB), cyanine blue (CB), methylene blue(MB), 2,4-dinitrophenol (2,4-DNP) aqueous solution (the initial concentration is 5 mg/L). The light source was a 300 W Xenon lamp, emitting UV and visible light simultaneously. For visible light measurements, a 420 nm cut-off glass filter was mounted before the output of light source to remove UV light and admit only visible light to enter into the reactor. The distance between the sample and lamp was 10 cm. Before irradiation, all the samples were immersed in the dyes solution and kept in the dark for 30 min to ensure that an adsorption/desorption equilibrium was established. In order to compare the photocatalytic activity of as-synthesized photocatalysts, the irradiation areas for all the samples were kept the same. The absorbance changes of dyes were monitored by measuring the maximal absorption using a UV–vis spectrophotometer (Cary 100, Agilent). In the durability test of the catalyst for the photodegradation of dyes under visible light, five consecutive cycles were tested. The samples were washed thoroughly with water and dried after each cycle. In order to study the effects of relevant reactive species, 1 mM different radical scavengers ($EDTA-Na_2$, potassium iodide, isopropanol and ethanol) were added into RhB solutions in a manner similar to the photocatalytic degradation experiment.

Results and discussion

Figure 1 shows the XRD patterns of the as-prepared CdS and CdS/PDMS samples. All of the diffraction peaks can be indexed to standard pattern of hexagonal phase structure of CdS (JCPDS No. 65-3414).[27] Obviously, no impurity peaks are observed, which indicates the high purity of products. No peaks assigned to PDMS are observed because PDMS is amorphous. After adding PDMS, no obvious changes occurred in the XRD patterns of CdS, which may be because the PDMS layer formed on the surface of CdS was too thin. These results show that the introduction of PDMS does not affect the phase of CdS.

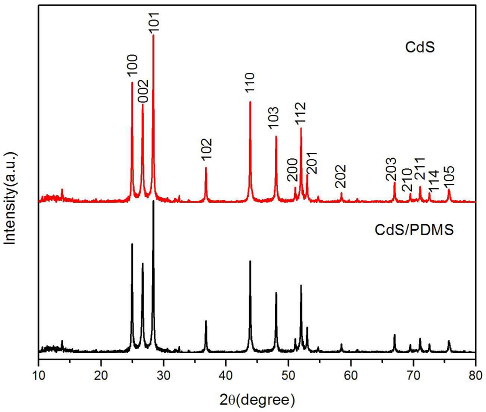

Figure 1 XRD patterns of CdS and CdS/PDMS samples.

Surface morphology, complexity and nanostructure are the most crucial factors for photochemically induced processes especially efficient thin film photocalyst. As shown in Figure 2a, there are uniform CdS nanoparticles with 100 nm diameter. Analysis performed by means of scanning electron microscopy (SEM) (Figure 2b) reveals that the surface of the CdS film possess a sponge like structure with extended roughness and complex characteristics. The average diameter of the nanocrystallites increased to 10 um with aggregation. And the thickness of the screen-printed CdS film reaches 30 um (Figure 2c). The SEM study shows that the CdS particles surface is coated with a layer of PDMS, and this layer may serve as a protective coating for the CdS particles and inhibit the photocorrosion process effectively. On the other hand, the appearance of the film corresponds to a porous network with extended surface area, are ideal for heterogeneous energy conversion processes, such as the photocatalytic procedure. Moreover, the surface coated PDMS shows a good affinity for organic molecules. The enhancement of adsorption and condensation of organic molecules may be realized by the hydropholic CdS film, leading to the efficient degradation of organic molecules.

In order to better understand the the screen-printed CdS film morphology and nanostructure, we have undertaken their characterization by atomic force microscopy (AFM). As shown from the Fig. 2d , we estimated roughness values of about 83.2 nm for the CdS film. Interconnected grain particles with an average diameter

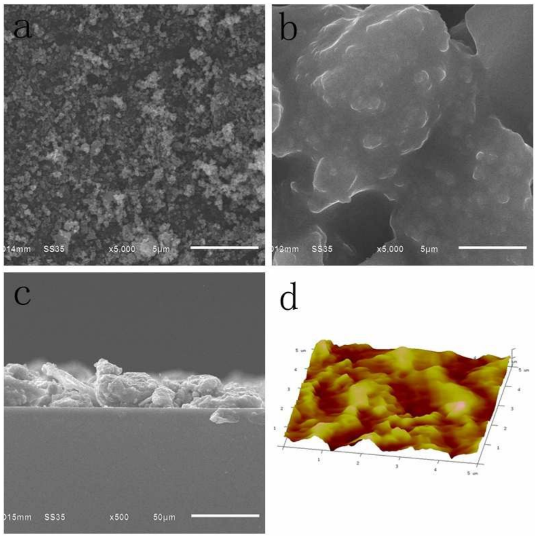

Figure 2. SEM micrographs as-prepared (a) CdS powder, (b) and (c) CdS film, (d) AFM images of the screen-printed film.

of about 100 nm are fused together to form the three-dimensional network of high mountains and big valleys of the thin film semiconducting material.[24] These results are coherent with SEM measurements. The higher roughness of CdS film might contribute to improve their photocatalytic performance, since it might enhance the number of active sites per unit area contacting with the degrading species.

As shown in Figure 3, typical FT-IR is used to testify the PDMS coating on CdS surface. Figure 3a shows the IR spectrum of as-prepared CdS mulled in a KBr wafer. The absorption peaks at 620 nm^{-1}, 1120 nm^{-1} and 1190 nm^{-1} are assigned to CdS.[28] The broad absorption bands from about 3600 nm^{-1} to 3000 nm^{-1} corresponding to O-H stretch vibration may be due to the water absorbed in the sample. For native PDMS molecules (Figure 3c), the peaks centering at 2962nm^{-1} and 1460 nm^{-1} are attributed to the stretching and bending vibration of C−H. The absorption peaks at 1260 nm^{-1} and 1110 nm^{-1} to 1020 nm^{-1} correspond to the vibration of the CH_3 and the Si−O−Si, respectively. The peaks at about 800 nm^{-1} are originated from the stretching vibration of CH_3−Si.[29] In Figure 3 (b), the characteristic peaks of PDMS molecules and CdS are all observed. Thus, we conclude CdS nanoparticles are coated with a thin layer of PDMS.

To demonstrate the potential applicability of the present CdS film in photocatalysis, we investigated its photocatalytic activity under visible-light by the photodegradation of RhB. The characteristic absorption of RhB at 554 nm was chosen as the monitored parameter for the process of photocatalytic degradation. As shown in Figure 4(a), when CdS film is used as the catalyst, the degradation efficiency reaches 34.14 % in 3 h. To improve the efficiency, 1 mL H_2O_2 (30%) is added into the RhB solution. When the mixed solution containing CdS film is kept in dark place, 7.13 % RhB molecules disappear because of the adsorption of CdS film. Under the irradiation of the xenon lamp, the degradation efficiency increases to 99.19 % after 3 h (Figure 4(b)), while the mixed solution without CdS only reaches 10.01 %. Obviously, CdS film shows higher photocatalytic activity in the presence of H_2O_2. The increased activity when assisting photocatalysis with H_2O_2, can be explained by: First, it takes surface-trapped electrons, and hence limits the electron-hole recombination rate and makes more holes available for reactions producing $\cdot$ OH radicals (e.g.,$\cdot$ OH$^-$ +h$^+$ → $\cdot$ OH).[29] Second, H_2O_2 plays a role of electron acceptor in CdS photocatalytic systems under the irradiation of visible light to lower the electron–hole recombination.[30] Hence H_2O_2 can increase the efficiency of holes utilization for photocatalytic oxidation of the RhB molecules.[31]

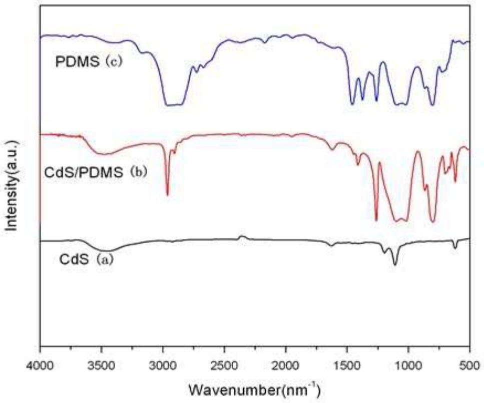

Figure 3. FT-IR spectra as-prepared (a) CdS, (b) CdS/PDMS and (c) PDMS

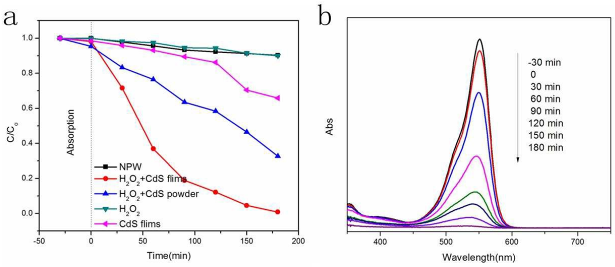

Figure 4. (a) The photodegradation of RhB in the presence of different photocatalysts under visible-light irradiation; (b) The temporal evolution of the spectra during the photodegradation of RhB mediated by CdS film in the presence of H_2O_2.

The obtained CdS film shows high efficiency in the removal of dye by photocatalysis because of the uniform nanoparticle distribution and more efficient photocatalytic process. For comparison with the CdS film that contained 20 mg CdS nanoparticles, we dispersed the as-prepared CdS powders (20 mg) and H_2O_2 (1 mL, 30%) into a dye solution (5 mg/L). (The average value of ten measurements obtained at differents films was used as the final mass. (Table S1)). After 3 h, the degradation efficiency of RhB reaches 99.19 % with the CdS film in the presence of H_2O_2, and 77.28 % of RhB is

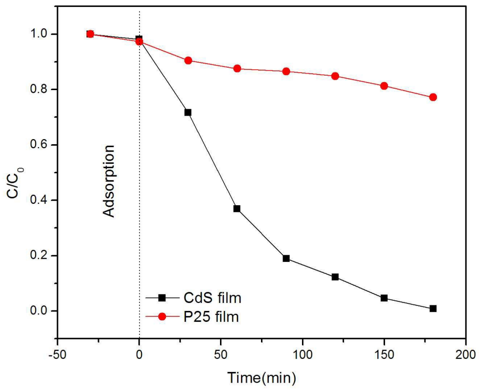

Figure 5. Performances of the CdS film and the commercial TiO_2 P25 film for photocatalytic degradation of RhB under the irradiation of visible light.

photodegraded by the CdS powders in the presence of H_2O_2 (Figure 4(a)).Compared with CdS powders, the nanocomposite film can maintain a relatively large surface area due to the immobilization of the porous structures. At the same time, CdS nanoparticles usually tend to agglomeration, and lead to a lower photodegradation efficiency. As for the P25 film (Figure 5), the degradation efficiency of RhB was 22.85 % within the same time. It indicates that the CdS film exhibits higher photocatalytic efficiency than the P25 film under visible light. Therefore, the as-synthesized CdS film could act as an efficient photocatalyst.

The experiments were then expanded to use other organic compounds of different types as model pollutants. Figure 6 displays the concentration changes of CB, MO, RhB, MB and 2, 4-DNP (C/C_0) as a function of irradiation time in the presence of H_2O_2. With regard to CB, the color of the dispersion solution completely disappears after 3 h of irradiation, and 99.12 % of CB is degraded. For RhB, 2,4-NP and MB, the degradation efficiency reaches 99.19 %, 99.53 %, 91.17 %, respectively. However, less than 71.47 % of MO is degraded within the same time (Figure S1). The above results can be attributed to the different adsorption characteristics of different dye molecules on CdS films.[32] Hence, the CdS film exhibits a high photocatalytic activity on many organic pollutants, such as RhB, CB, MB and 2,4-DNP except MO.

The recovery and reuse of catalysts are important factors in terms of the stringent ecological and economic demands for sustainability in “green chemistry”. Besides the higher photoactivity, the CdS film also showed photostability and reusability during the experiments of visible-light-driven photodegradation of organic pollutants. As shown in Figure 7, after five cycles of photocatalysis reactions, the sample is capable of degrading RhB dye up to 90.61 %, and it can decompose the RhB solution completely under 3 h irradiation of visible light. The results show that there is only an insignificant loss in the photocatalytic activity, which might be partly caused by loss of the photocatalyst during each round of collection and rinsing. The XRD pattern image of the film after five cycles of photodegradation testing also demonstrated the intact structure and morphology (Figure S2). We also use MO, CB and 2,4-DNP aqueous solution to do the cycle experiments (Figure S3). The CdS film exhibited a high photocatalytic stability and reusability on RhB, CB, 2,4-DNP except MO. Therefore, it illustrates that the CdS film not only enhances the visible light photocatalytic performance of CdS, but also inhibits the photocorrosion, which makes the CdS film has a stable durability of photoactivity. It should be noted that the photocatalytic film can be reused easily just after rinsing the glass in distilled water without affecting the photocatalytic activity.

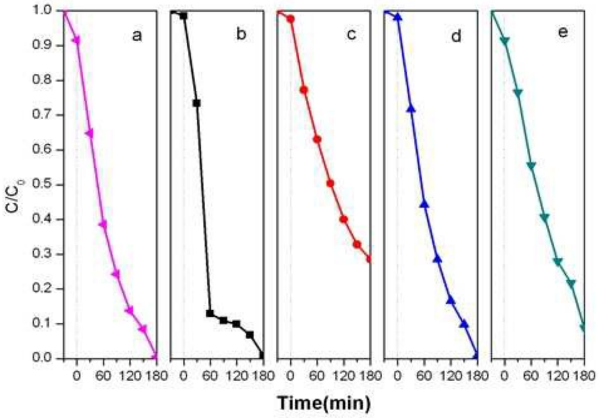

Figure 6. The photodegradation of different organic pollutants in the presence of CdS film and H_2O_2 under visible-light irradiation. (a) 2, 4-DNP, (b) CB, (c) MO, (d) RhB, (e) MB.

In order to investigate the degradation mechanism of RhB over the samples, various substances were introduced to react with active species for evaluating their contribution to the photodegradation of RhB by the change of degradation efficiency. In this work, we adopted isopropanol and ethanol for scavenging ·OH,[34-35] and EDTA-Na_2 and potassium iodide (KI) as h^+ scavenger.[36-37] The above scavengers were added to the pollutant solutions together with CdS films before irradiation. The photocatalytic degradation curves of RhB over CdS film with various scavengers are shown in Figure 8.

Without any scavenger, the degradation rate of RhB was 99.12% after irradiation for 3 h. When isopropanol and ethanol were added, the degradation rate changed largely, indicating that ·OH was important in the photocatalytic process. With the addition of EDTA-Na_2 and KI, the photocatalytic degradation rates of RhB decrease to 17.70 % and 10.65 %, respectively, indicating that h^+ is also an important active species besides ·OH.

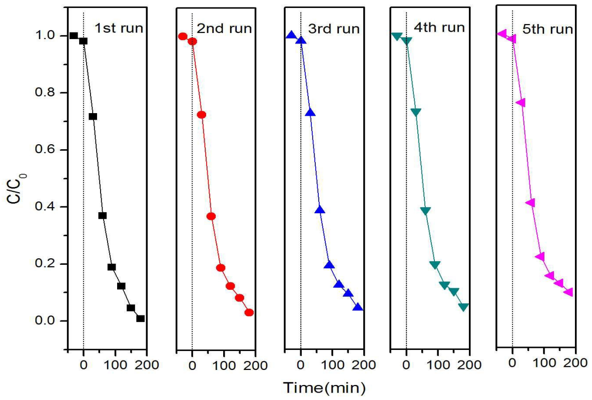

Figure 7. Cycle experiments of RhB degradation over CdS film.

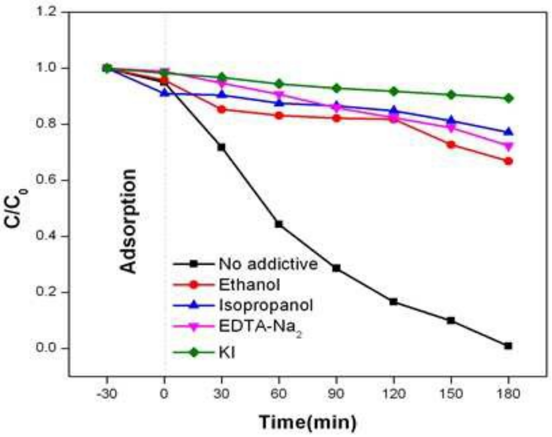

Figure 8. Degradation of RhB over CdS film in the presence of different radical scavengers under visible-light irradiation

In conclusion, ·OH and h^+ are the main active species for the degradation of RhB over the CdS film under visible-light irradiation, which is consistent with the result that H_2O_2 can improve the degradation efficiency.

To understand the enhancement of the photocatalytic activity of the CdS film, several factors such as morphology, coated layer, H_2O_2 and the electron transport have to be addressed. Firstly, it is well known that the photocatalytic activity is closely related to the adsorption ability and surface area of the catalyst. The appearance of the CdS film corresponds to a porous network with extended surface area, are ideal for heterogeneous energy conversion processes. At the same time, the large surface area is helpful to increase the photocatalytic reaction sites. Secondly, the CdS particles surface is coated with a layer of PDMS, which may serve as a protective coating for the CdS particles and inhibit the photocorrosion process effectively. Meanwhile, the coated layer shows a good affinity for organic molecules. The enhancement of adsorption and condensation of organic molecules may be realized by the hydrophilic CdS film, leading to the efficient degradation of organic molecules. Thirdly, H_2O_2 greatly increased activity rate. On one hand, H_2O_2 limits the electron-hole recombination rate and makes more holes available for reactions producing · OH radicals. On the other hand, H_2O_2 plays a role of electron acceptor under the irradiation of visible light to lower the electron–hole recombination. Hence H_2O_2 can increase the efficiency of holes utilization for photocatalytic oxidation of the RhB molecules. On the basis of the above analysis, it is concluded that the CdS film has an obvious advantage in the photocatalytic degradation of the RhB, CB, MB, MO and 2, 4-DNP pollutants.

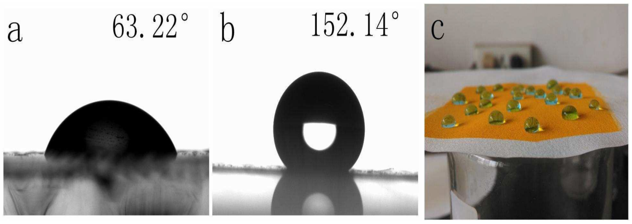

Figure 9. CAs of water droplets on the surfaces (a) none (b)CdS film, (c) photo of water droplet on the CdS film.

Figure 9 shows the CAs of water droplets on the surfaces of glass coated by CdS film. As shown in Fig. 8b, the CA values of 152.14°. Meanwhile, we measured the CAs of water droplets on the surfaces of glass, and the CA values of 63.22°. Therefore, the wettability of glass modified by CdS/PDMS samples improved largely, and achieved superhydrophobic without the use of fluorinated materials and stearic acid. This result is possibly because that random distribution of CdS particles formed to the nano/micro-structure, and combined with PDMS motified, lead to their surface with superhydrophobicity. And we also measured the CAs of droplets on the surfaces of fliter paper coated by CdS films, the CA value is more than 150°. That to say, this method applied to other substrates, such as filterstuff, copper mesh and stainless steel mesh, to fabricate superhydrophobic surfaces.

Additionally, the oil wettability of the as-prepared CdS film was also examined through the contact angle measurement. Figure 10a-c shows a drop of octane absorbed by the asprepared CdS film. As we expected that the octane spread quickly on the coating and absorbed thoroughly within about 1.5 s, which shows that the as-prepared CdS film absorbed oil quickly, but repelled water completely. This indicate that as-prepared CdS film has excellent superhydrophobic and superoleophilic, and has a good potential in the lives and production applications, such as such as oil mixture separation, self-cleaning materials and drag-reducing materials, and so on.[38-40]

The oil-water separation experiment was performed as shown in Fig. 10d When the mixture of water ((dyed with methyl blue)) and hexadecane was poured onto the fliter paper, the hexadecane spread out rapidly and then penetrated the fliter paper and flowed down the beaker below, whereas the water retained on the surface of the fliter paper, showing its excellent ability for oil-water separation. Moreover, the obtained functional CdS flim can retain their high oil-water separation efficiency even after five cycles of oil-water separation.

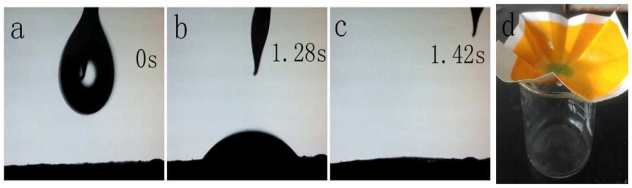

Figure 10. (a), (b), (c) video snapshots of a drop of octane absorbed by the as-prepared CdS/PDMA film, (d) the superhydrophobic fliter paper used for oil-water separation.

These results indicated that the as-prepared superhydrophobic CdS film is promising candidates for oil-spill cleanup.

Conclusions

In summary, we report the fabrication of CdS photocatalytic film on common glass without calcination using screen-printing technique for the first time. The as-prepared CdS film not only exhibit enhanced photocatalytic activity for the degradation of RhB, CB, MB and 2,4-DNP, but also show excellent photostability due to the efficient inhibition of the PDMS layer from the photocorrosion. The recyclability of the CdS film is worthy to be noted, because the films can be reused just after rinsing the glass in distilled water without affecting the photocatalytic activity. This fabrication process is a general approach, and it can be extended to print different photoactive nanoparticles on a variety of carriers (such as glass, ceramic tile, polymer materials and metal). In addition, the CdS film possess superhydrophobic and superoleophilic properties, and could effectively separate the mixture of water and oil. The coatings can clean themselves by the action of water, and have an property of chemically breaking down the complex dirt deposits by sunlight-assisted cleaning mechanism. The simple fabrication process and easy manipulation of the films cleverly combined superhydrophobic and photocatalysis, and promise practical applications and industrial use of these materials in environmental remediation.

Acknowledgements

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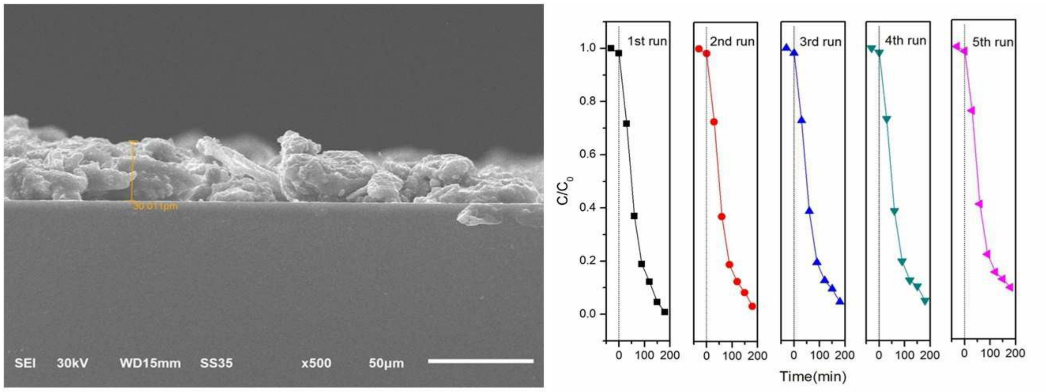

PDMS-coated CdS film has been deposited by screen-printing in glass and then used in the decolouration of organic pollutants and possesses superhydrophobic and superoleophilic properties.