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Mechanochemical synthesis, characterization, and photocatalytic activity of CdS/TiO_2 composites in air purification

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Two samples of CdS/TiO_2 composites have been prepared by single-step solid-state mechanochemical route. Sample 1 was prepared in a ball mill by mixing commercial TiO_2 Degussa P25 with CdS mechanochemically synthesized in advance (cubic hawleyite phase). Sample 2 has been obtained by the mechanochemical synthesis from cadmium acetate and sodium sulphide in the presence of TiO_2 Degussa P25. Characterization of the synthesized composite materials by means of XRD, DRS, PL, SEM, and EDX showed that the CdS particles were attached to titania. XRD analysis proved that the rutile TiO_2 phase is prevailing in Sample 1, unlike the case of Sample 2, which contained mainly anatase TiO_2 phase. UV-vis diffuse reflectance spectroscopy was applied in order to evaluate the band gaps of the synthesized samples. Obtained values of the band gap for the composite CdS/TiO_2 samples are lying below the band gap value interval of pure anatase TiO_2 phase (3.2 eV) and that of pure CdS (2.42 eV) enabling visible light illumination. The photocatalytic oxidation of ethylene (model air contaminant) was studied both under the effect of UV- and under visible light irradiation. It was established that the mechanochemically synthesized CdS/TiO_2 shows a higher photocatalytic activity in the oxidation of ethylene under visible light irradiation in comparison with pure titania due to the effect of the CdS component.

Key words: mechanochemistry, photocatalysis, titania, CdS, ethylene, air pollution.

INTRODUCTION

Contaminated air exerts harmful effects on human health [1]. Volatile organic compounds are the basic pollutants in contaminated air [2]. A number of technologies have been elaborated to purify air, for instance passing it through a filter of activated carbon [3]. Processes of thermal oxidation or catalytic oxidation have also been applied [4]. However, these processes occur at high temperatures and pressures [5]. Heterogeneous photocatalytic oxidation is an alternative approach, which happens at room temperature and atmospheric pressure [6]. A great variety of photocatalytic materials enable efficient purification of polluted air [7]. Titania is the most widely applied photocatalyst to degrade organic contaminants in air and water [8]. But anatase TiO_2 absorbs only photons of the UV region, which comprises only 5% of the solar spectrum. Titania modifying with other semiconductors of narrow band gap and absorbing visible light is a popular approach to improving its light absorbance properties [9]. The present work is focused on TiO_2 modification with cadmium sulphide. Composite samples were prepared by the mechanochemical route. Mechanochemical synthesis produces surface clean semiconductor nanoparticles without any influence of organic ligands, applied in chemical process, and such nanoparticles can be tested as a photocatalyst [10].

Ethylene was selected as a model air contaminant [11]. Ethylene is causing air pollution due to its large-scale production in the petrochemical plants. The photocatalytic oxidation of ethylene is therefore a hot topic of the day [12, 13]. In the present work mechanochemically synthesized CdS/TiO_2 composites were tested and their photocatalytic activities were compared with data obtained beforehand by the traditionally used reference photocatalyst TiO_2 Degussa P25. The investigation of ethylene photocatalytic oxidation was carried out in a recently designed and constructed gas-phase flat-plate continuous flow photocatalytic reactor over a thin film coating of semiconductor material. The aim of the present paper was to investigate the influence of the synthesis conditions for preparing CdS/TiO_2 composites by monitoring their physicochemical properties and testing their efficiency as photocatalysts for complete oxidation of ethylene.

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EXPERIMENTAL

2.1 Materials

CdS/TiO_2 composites were synthesized from cadmium acetate $(CH_3COO)_2Cd.2H_2O$ (98%, ITES, Slovakia), sodium sulphide $Na_2S.9H_2O$ (98%, Aldrich, Germany), and TiO_2 Degussa P25.

2.2. Mechanochemical synthesis

The mechanochemical synthesis of the samples was carried out in a Pulverisette 6 planetary ball mill (Fritsch, Germany). The ball mill was charged with 50 balls made of tungsten carbide having a diameter of 10 mm and weighing 360 grams. The rate of rotation of the planetary carrier was 500 rpm. Experimental runs were conducted in argon at room temperature.

Pure cadmium sulphide was mechanochemically prepared by following equation (1). Further details are published elsewhere [14].

$$(CH_3COO)_2Cd.2H_2O + Na_2S.9H_2O \rightarrow CdS + 2CH_3COONa + 11H_2O \qquad (1)$$

One of the composite samples, denoted as CdS/TiO_2-1, was synthesized by milling in a planetary ball mill using pure cadmium sulphide prepared beforehand [14] and the commercially available TiO_2 at a ratio of 1:4. The milling time interval was 30 min.

For a second mechanochemically synthesized composite sample, CdS/TiO_2-2, a mixture of cadmium acetate, sodium sulphide, and TiO_2 P25 Degussa was used. The synthesis was carried out in the planetary ball mill in inert atmosphere under the same conditions as described above.

2.3. Characterization techniques

X-ray diffraction (XRD) patterns were recorded on a D8 Advance diffractometer (Brucker, Germany) using CuKα radiation.

Specific surface area (S_A) values were obtained by the low-temperature nitrogen adsorption method using a Gemini 2360 sorption apparatus (Micromeritics, USA).

Diffuse reflectance UV-vis spectra were measured on a Thermo Evolution 300 UV-Vis Spectrophotometer equipped with a Praying Mantis device with Spectralon as the reference. Spectralon is a fluoropolymer, which has the highest diffuse reflectance among all the known materials or coatings over the ultraviolet, visible, and near-infrared regions of the spectrum. Kubelka-Munk relationships were used to transform the reflectance data into absorption spectra.

Photoluminescence (PL) spectra at room temperature were acquired at right angle on a photon counting spectrofluorometer PC1 (ISS) with a photoexcitation wavelength of 325 nm. A 300-W xenon lamp was used as the excitation source. The emission was collected in a 25-cm monochromator with resolution of 0.1 nm equipped with a photomultiplier. The photoexcitation and photoemission slit widths were 1 and 2 mm, respectively. For measuring the PL intensity, the powders were suspended in absolute ethanol.

The photocatalytic activity of the samples in the reaction of ethylene complete oxidation was measured under UV-A polychromatic light illumination (the so called Black Light Blue BLB lamp with λ_{max} = 365 nm), UV-C monochromatic illumination (TUV lamp λ = 254 nm), and under visible light illumination. The standard reference experimental conditions and the reactor configuration have been described elsewhere [15], while the operational parameters are listed in Table 1.

Table 1. Comparison between CdS/TiO_2 composites and commercial TiO_2 Degussa P 25 in the photocatalytic conversion of ethylene (30% and O_2 excess) under illumination with different types of light

Photocatalyst	Ethylene conversion, %	Type of illumination	Intensity of illumination, $W.cm^{-2}$
CdS/TiO_2-1	0.7	UV-A	0.014
CdS/TiO_2-1	4.5	UV-C	0.014
CdS/TiO_2-1	11.5	visible	8.9
CdS/TiO_2-2	3.7	UV-A	0.014
CdS/TiO_2-2	5.1	UV-C	0.014
CdS/TiO_2-2	14.4	visible	8.9
TiO_2 Degussa P25	12.8	UV-A	0.014
TiO_2 Degussa P25	27.3	UV-C	0.014
TiO_2 Degussa P25	0	visible	8.9

The support bearing the photocatalytic layer of CdS/TiO_2 composites and TiO_2 was a commercially available Al foil, covered with silica gel (0.2 mm), manufactured by Merck for the purposes of thin layer chromatography (TLC Merck Art. 5554 Kieselgel 60 F_{254}). A suspension (56 mg CdS/TiO_2 in 5 ml H_2O) was ultrasonically treated as in Ref. [16].

RESULTS AND DISCUSSION

The XRD measurements were carried out to study the phase composition of the obtained samples. Figure 1 represents XRD patterns of the two mechanochemically synthesized CdS/TiO_2 composite materials. The X-ray patterns of the cadmium sulphide and of the commercial product

TiO_2 P25 Degussa are also given for comparison. The profile of TiO_2 P 25 Degussa shows that it is composed of crystallites of anatase and rutile at a ratio of 75:25. Some diffraction lines are observable in the XRD pattern of the CdS, which indicate the cubic structure of hawleyite CdS (JCPDS 00-010-044). All the diffraction lines are wide, which give evidence for the formation of fine-sized crystallites [17] as well as for structural disorder, which appears in the cadmium sulphide during the milling process [10].

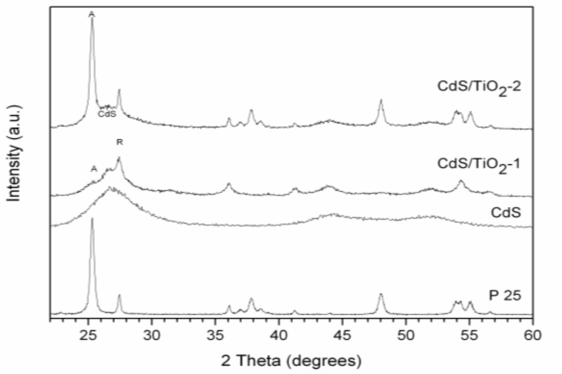

Fig. 1. XRD patterns of TiO_2 P25 Degussa and of mechanochemically synthesized CdS, CdS/TiO_2-1, and CdS/TiO_2-2 samples.

Substantial changes were registered in the XRD pattern of the CdS/TiO_2-1 sample. This sample, obtained by means of mechanochemical synthesis starting from a mixture of cadmium sulphide and TIO_2 P 25 in the ball mill, gives rise to a substantial decrease in intensity of the peak at $2\theta = 25.2°$ related to the plane (101) of the anatase. A significant widening was observed for all the registered diffraction lines. A considerable decrease of the crystallite size was registered with this sample. Some lines characteristic of CdS are also present. Rutile is the prevailing phase in this sample. Wide diffraction lines of the mechanochemically synthesized CdS clearly prove the nanosized nature of the sample [18]. The impact of energy during the milling process is manifested in the form of phase transformation [19].

Regarding the diffraction pattern of sample CdS/TiO_2-2, which was prepared by direct mechanochemical synthesis of CdS from cadmium acetate and sodium sulphide on the surface of TiO_2 P 25, all the lines characteristic of TiO_2 P 25 are present as well as some others, which are characteristic of CdS having a cubic structure of hawleyite. No phase transformation of anatase into rutile was registered in this case. Here, the high-energy milling was accompanied by generation of fresh surface, which is displayed in a considerable growing up of the specific surface area of the CdS/TiO_2-2 sample (72 m^2 g^{-1}) in comparison to that of P 25 (50 m^2 g^{-1}). The specific surface area of sample CdS/TiO_2-1 was measured to be 19 m^2 g^{-1}. Specific surface area values of the samples are one of the most important features of the milled samples [10]. High values of the specific surface area of mechanochemically synthesized samples suppose an effective application of the latter to surface-area dependent processes, such as sorption, heterogeneous catalysis (both dependent on the number of surface active sites) and others. The CdS/TiO_2-1 sample represents a less crystalline material, while sample CdS/TiO_2-2 is a more crystalline material (Fig. 1).

A SEM micrograph of the CdS/TiO_2-2 sample is shown in figure 2. It represents juxtaposition of back-scattered (left side) and secondary electron images (right side) of CdS deposited on TiO_2. There is better sharpness of secondary electron image (SEI), but the chemical contrast in the back scattered image (BEC) gives more information: bright area is CdS and dark area is TiO_2 as the atomic mass of Cd is higher than that of Ti; therefore, the electron yield (brightness) from Cd is also higher. Elemental composition of CdS/TiO_2-2 was determined by EDS (energy dispersive spectroscopy) and it is shown in Table 2.

Table 2. Composition of the CdS/TiO_2-2 sample supported on a commercially available Al foil covered by silica gel (0.2 mm)

Element	Weight, %	Content, at.%
O	41.91	71.09
Si	2.29	2.21
S	5.15	4.36
Ti	31.11	17.62
Cd	19.54	4.72
total	100.00	100.00

Figure 2 represents DRS spectra of the initial TiO_2 P25 Degussa sample and the mechanochemically synthesized sample CdS/TiO_2-2. The absorbance spectrum of TiO_2 P25 consists of intensive absorbance only in the ultraviolet region. The absorbance edge of the pure titania is about 390 nm. In contrast, the absorbance edge in the spectrum of the synthesized samples of CdS/TiO_2-2 was registered at 530 nm, i.e. already in the visible region (Fig. 3b). A red shift towards longer wavelengths is observed in the range of visible light. The band gaps E_g of the samples were calculated on the basis of Tauc equation [21]. Evaluated band gap values of the samples were 3.3, 2.46, 2.2, and 2.35 eV for TiO_2 P 25, CdS, CdS/TiO_2-1, and CdS/TiO_2-2, respectively. The band gap of the mechanochemically synthesized CdS/TiO_2 composites lies below that of CdS and thus allows visible light illumination.

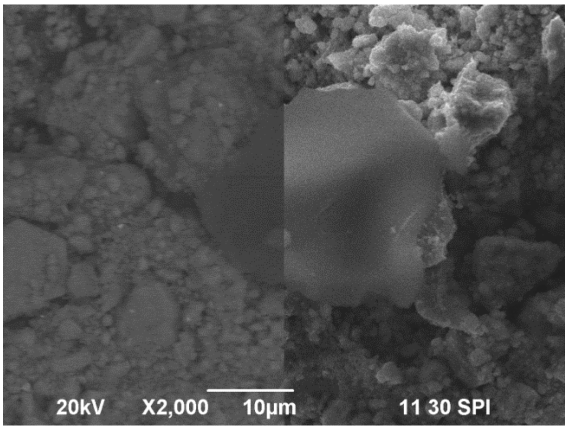

Fig. 2. SEM image of sample CdS/TiO_2-2 particles on the surface of SiO_2 coated by a TLC plate.

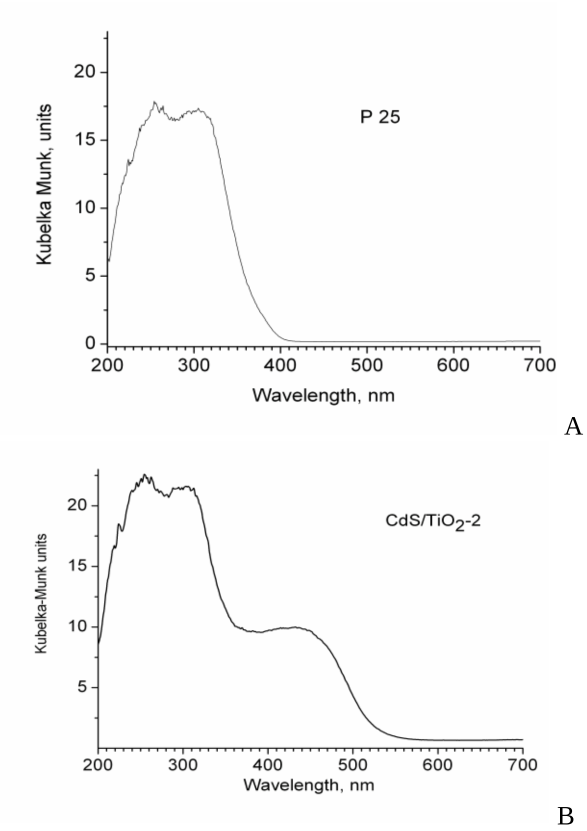

Fig. 3. A. UV-vis drs spectrum of TiO_2 P25 sample. B. UV-vis DR spectrum of mechanochemically synthesized CdS/TiO_2-2 sample.

The photoluminescence (PL) spectra of the initial TiO_2 P 25 Degussa and mechanochemically synthesized CdS, CdS/TiO_2-1, and CdS/TiO_2-2 samples, photoexcited at 325 nm at room temperature, are displayed in figures 4 and 5. The photoexcitation wavelength of 325 nm, corresponding to photon energy of 3.81 eV, is greater than the band gap energy of titania. This energy was an absorbed exciting transition of valence band electrons to the conduction band.

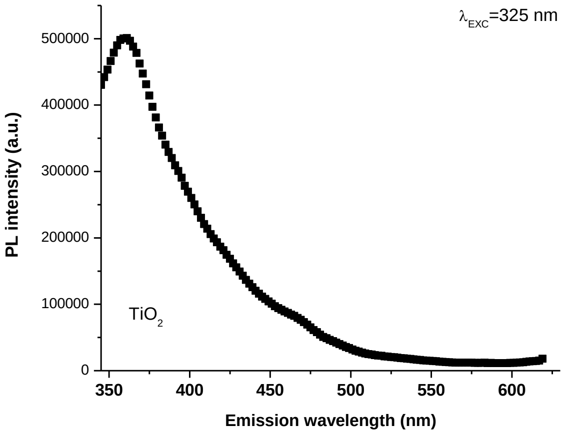

Fig. 4. Photoluminescence spectrum of TiO_2 P25 Degussa.

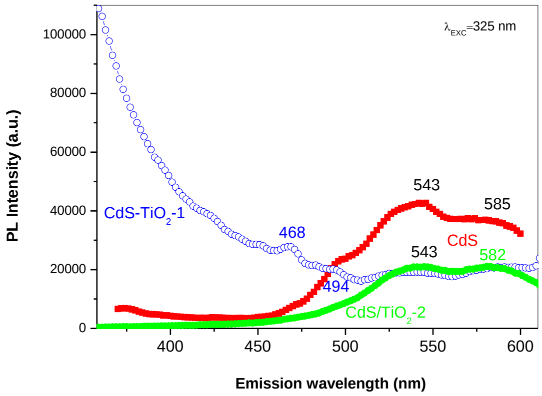

Fig. 5. Photoluminescence spectra of mechanochemically synthesized CdS, CdS/TiO_2-1, and CdS/TiO_2-2 samples.

The emission intensive peak in the spectrum of TiO_2 P 25 Degussa with a maximum in the near ultraviolet region at about 361 nm (3.43 eV) can be ascribed to emission of a photon having energy equal or slightly higher than the band gap width of the anatase phase of TiO_2 and it provides evidence for a direct recombination of a photoexcited electron and a positively charged hole [22, 23]. The photoluminescence emission is the result of two effects: part of the illuminating photons energy goes for a non-radiative transition (a transition between two vibrational energy levels of the crystal lattice nodes). Another part is emitted as a result of recombination of photoexcited electrons and holes (lower energy photons).

It is known that CdS nanoparticles manifest a light-emitting behaviour at a specific wavelength corresponding to the band gap width, which can be determined in this way. Thus, the formation of CdS nanoparticles can also be confirmed by photoluminescence spectroscopy [24]. The photoluminescence spectrum of the mechanochemically synthesized CdS shows a broad emission band with a maximum of the emission at 543 nm (2.3 eV) accompanied by a very slight emission peak at 585 nm (2.1 eV). The emission peak at 543 nm can be assigned to the surface-trap-induced photoluminescence, which involves the recombination of electrons with a trapped hole-sulphur vacancy inside the valence band of CdS nanoparticles [25]. A similar result has also been obtained [26].

The photoluminescence spectrum of the mechanochemically synthesized CdS/TiO$_2$-2 sample is similar to that of the pure cadmium sulphide, but having a substantially lower intensity. The lower PL intensity of the CdS/TiO$_2$-2 sample indicates a lower recombination rate of photoexcited electrons and holes [27]. No emission was observed in the case of TiO$_2$ P25 within the region of 475 up to 650 nm (Fig. 4). The presence of CdS in the composite CdS/TiO$_2$-1 sample is leading to substantial changes in their PL spectra (Fig. 5) indicating an efficient transfer of electrons from the CdS particles to the titania. This results in suppression of the recombination of charge carriers in CdS/TiO$_2$ compared to titania. A PL spectrum of this sample is complicated because there are many factors that can influence the fluorescence signals such as surface states and bulk defects. In the spectrum of mechanochemically synthesized CdS/TiO$_2$-1 sample, the minor emission peaks at 468 and 494 nm correspond to titania and only slightly pronounced wider emission peaks at 540 and 585 nm belong to CdS, accordingly. The emission at 468 nm (2.6 eV) proves the presence of oxygen vacancies, while that at about 494 nm (2.6 eV) could be ascribed to indirect recombination of defects involving interaction of phonons in the lattice oxygen vacancies in titania [20]. It is known that the electronic properties of CdS depend on the particle size [28]. Since the PL emission is the result of recombination of photoexcited electrons and holes, the lower photoluminescence intensity of synthesized composite CdS/TiO$_2$ samples proves a lower recombination rate of the photoexcited electrons and holes [29], which was aimed intentionally in this study by adding CdS.

It has been reported earlier that there is no PL peak emitted by the pure anatase phase, but a weak PL peak in the rutile phase of titania [30]. However, in our case we have observed fine PL peaks for rutile phase and enhanced PL entities for the anatase phase of the corresponding CdS/TiO$_2$ samples, which is in contrast with earlier reports.

The degree of ethylene conversion was taken as a measure of the photocatalytic activity. As it can be seen from the data presented in Table 1, the best photocatalytic efficiency in ethylene photocatalytic oxidation under both kinds of UV-light illumination is exhibited by the TiO$_2$ Degussa P25, where the UV-C light is more effective due to the higher energy of the photons with all the three studied samples.

However, anatase TiO$_2$ was inactive under visible light illumination, where both CdS/TiO$_2$ samples exhibited some activity (Fig. 6). The CdS/TiO$_2$-2 sample was superior to the CdS/TiO$_2$-1 sample under visible light owing to the higher specific surface area and higher quantum yield.

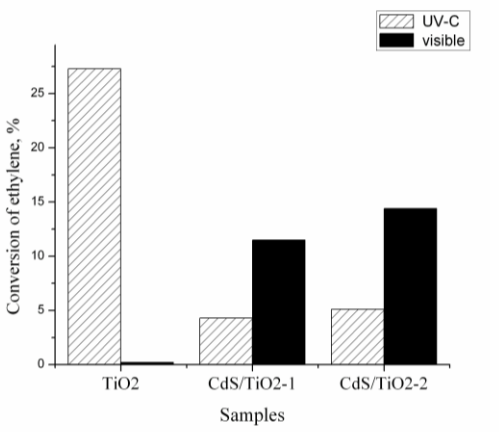

Fig. 6. Photocatalytic conversion of ethylene under illumination with different types of light over CdS/TiO$_2$ composites and commercial TiO$_2$ Degussa P25.

Composite CdS/TiO$_2$ materials could be a promising photocatalyst to utilize a wide share of solar light irradiation. Upon irradiation of the samples with visible light the electrons in the valence band of CdS are photoexcited and they are transferred to the conduction band and further to the TiO$_2$ conduction band as the two conduction bands are merging at the interphase boundary between the two attached particles. This is due to the fact that the oxidation potential of TiO$_2$ is superior to that of CdS, so the local electric field at the interphase boundary enables this transition of the photoexcited electrons from the conduction band of CdS to the conduction band of TiO$_2$ through the interphase boundary layer between the attached two particles. In this way the photoinduced charge carriers (e^- and h^+) in CdS appear to be effectively separated, which hinders their recombination prolonging the lifetime of the holes that are, in fact, the oxidation active sites of the catalyst in an oxidation reaction. Therefore, the result is that the mechanochemically synthesized CdS/TiO$_2$ samples manifest a higher photocatalytic activity in the oxidation of ethylene under visible light irradiation in comparison with pure titania.

CONCLUSION

The present paper represents a detailed study of the structural, optical, and photocatalytic properties of mechanochemically synthesized CdS/TiO_2 composite materials. The XRD pattern analysis of the CdS/TiO_2-1 sample, prepared by mechanochemical synthesis in a planetary ball mill of a mechanical mixture of CdS and TiO_2 P25, showed that a phase transition into rutile occurred in a larger fraction of anatase of the initial TiO_2 P 25 material. The phase composition of TiO_2 P 25 was preserved during the mechanochemical synthesis of the crystalline CdS/TiO_2-2 sample. The specific surface area of this sample was higher by 50% than that of the initial TiO_2 P 25. A DRS study revealed that all the mechanochemically synthesized samples demonstrated a strong absorbance in the visible light region. Evaluated band gap values of the samples were 3.3, 2.46, 2.2, and 2.35 eV for TiO_2 P 25, CdS, CdS/TiO_2-1, and CdS/TiO_2-2, respectively. An enhanced photocatalytic activity of the synthesized composite materials in the visible range of the spectrum for ethylene oxidation in polluted air is owing to an efficient separation of photoexcited electrons and holes, i.e. the charge carriers between attached CdS and TiO_2 phases.

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МЕХАНОХИМИЧЕН СИНТЕЗ, ОХАРАКТЕРИЗИРАНЕ И ФОТОКАТАЛИТИЧНА АКТИВНОСТ НА CdS/TiO_2 КОМПОЗИТИ ЗА ОЧИСТВАНЕ НА ЗАМЪРСЕН ВЪЗДУХ

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(Резюме)

CdS/TiO_2 композитни образци са получени чрез едноетапен твърдотелен механохимичен синтез. Образец CdS/TiO_2-1 е получен в топкова мелница чрез смесване на TiO_2 P25 Degussa с предварително получен CdS (кубична фаза hawleyite). Образец CdS/TiO_2-2 е получен чрез механохимичен синтез от кадмиев ацетат и натриев сулфид в присъствие на TiO_2 P25 Degussa. Охарактеризирането на синтезираните композитни материали с рентгенофазов анализ, дифузионно-отражателна спектроскопия, фотолуминесцентна спектроскопия, сканираща електронна микроскопия и енергийно дисперсионен анализ показа, че частици от CdS са закрепени в титановия диоксид. Рентгенофазов анализ показа, че в образец CdS/TiO_2-1 преобладава фазата рутил за разлика от образец CdS/TiO_2-2, който съдържа основно анатаз. Дифузионно-отражателна спектроскопия беше използвана за определяне на забранената зона на механохимично синтезираните образци. Получените стойности за забранените зони на композитните образци са между забранената зона на чистия TiO_2 (3.2 eV) и CdS (2.42 eV). Фотокаталитичното окисление на етилен (моделен замърсител на въздух) е изследвано при облъчване както с ултравиолетова, така също и с видима светлина. Установено е, че механохимично синтезираните CdS/TiO_2 композитни образци показват по-висока фотокаталитична активност в окислението на етилен при облъчване с видима светлина в сравнение с чист титанов диоксид.