Nano cube of CaSnO_3 : Facile and green co-precipitation synthesis, characterization and photocatalytic degradation of dye

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Abstract

In this work, nanocubes of CaSnO_3 have been prepared by a simple and green co-precipitation method. In this technique, for preparation of calcium stannate, we have used from a complex structure of calcium as a new precursor and the synthesis of CaSnO_3 have been done in water as a green solvent. Using of complexing precursors were created a congestion in reaction medium. Different conditions have been studied in synthetic approaches and optimized the effect of different parameters on the morphology of product such as precipitation agent (alkaline), pH, temperature, the rate of stirrer, surfactants and the mole ratio of surfactants for preparation product and obtaine the best product in terms of quality and morphology. By using of this CaSnO_3 , two types of azo dyes (acid blue 92 and acid brown 14) have been degraded at presence of ultraviolet light from aqueous solution. Results display that the powder shows appropriate catalytic behavior for degradation of dyes (77% acid brown 14 and 67% acid black 92). Therefore these nano-cube structures have been used as photocatalysts in presence of UV light for degradation of azo dyes.

KEYWORDS: Ceramics; Nanostructures; Photocatalyst; Chemical synthesis; Electron microscopy

1. Introduction

Alkaline-earth stannates (which observed in the ASnO_3 formula and A is an alkaline earth element or IIB element) have been got a lot of consideration due to their potential applications in numerous fields, such as photocatalyst, lithium ion batteries as anode materials, ferromagnet, humidity sensors, thermally stable capacitors and electronic devices. [1-5].

According to obtained information, just two phases have been existed for CaSnO_3 and Ca_2SnO_4 in the binary systems of CaO and SnO_2 . CaSnO_3 has orthorhombic perovskite structure, which is made of octahedral SnO_6 that attach together with sharing pinnacles. Furthermore, the structure of Ca_2SnO_4 is comprised of SnO_6 octahedral that are connected via edges and the Ca^{2+} which bounded by seven ions of oxygen is arranged a structure with low symmetry [6]. The SnO_4 anions complicated in Ca_2SnO_4 have been being optically inert, and it might be a contender for host materials [7-8].

There are several synthesis methods for attaining the semiconductor materials, the peroxide precursors [9], the solvothermal and hydrothermal [10], the sol-gel [11], co-precipitation [12], the polymeric precursor method [13], and so on. In thermal treatment method, the temperature is a remarkable factor, and for achieving crystalline single phase materials should be used from different time for treating the precursors on a certain temperature [14]. In the case of peroxide precursor, the base of method is solid state reaction [15]. In polymeric precursor technique that has been happened a chelation of metallic cations in the process of formation of polymer chain from an esterification reaction among metallic citrate and ethylene glycol [16]. In this method, the precursors have an important role in the preparation of CaSnO_3 [17]. In the co-precipitin method, the preparation has been affected by the structures of precursors and another surfactant that solved in reaction solutions. Therefore in this work, we prepared CaSnO_3 in a co-precipitation method and improved the morphology with changing in reaction condition. The photocatalytic behavior of product have been investigated for degradation of azo dyes and observed suitable results.

2. Experimental

2.1. Materials and physical measurements

All the materials that used in our experiments are in analytical grade and purchased from Merck Company (pro-analysis) with no further purification. XRD patterns were recorded by a Philips, X-ray diffractometer using Ni-filtered Cu K α radiation. SEM images were taken by an LEO instrument model 1455VP. Prior to take images, the samples have been coated with a thin layer of Pt to create conductor the surface of samples and avoid charge accumulation, and procurement a better contrast. FT-IR spectra were recorded on Galaxy series FTIR5000 spectrophotometer. The beam splitter for the recording the FT-IR spectra is KBr and for detecting has been used from DTGS-KBr as detector and the range of 400-4000 cm⁻¹ have been scanned by the resolution that is better than 0.1 cm⁻¹. GC-2550TG (Teif Gostar Faraz Company, Iran) were used for all chemical analyses. Room temperature photoluminescence was studied by a Perkin Elmer fluorescence instrument. The electronic spectra of the complexes were taken on a JASCO UV–Visible scanning spectrometer (Model V-670).

2.2. Preparation of *[bis(salicylaldehydato)strontium (II)]; Ca(sal)₂*

According to previous description, in co-precipitation method for preparation of nanomaterial the structure of precursors is one of the main factor that affected on morphology and quality of product. Therefore Ca(sal)₂ as a precursor for preparation of CaSnO₃ have been synthesized.

For preparation of Ca(sal)₂, 4 g of Ca(NO₃)₂ dissolved in 40 mL distilled water at the temperature of 50 °C with magnetic stirring. On the other hand, on a separate vessel 3.55 mL of salicylaldehyde dissolved in 40 mL methanol. Then these 4.5 mL of the ethylene diamine adede to the mixture of Ca²⁺ solution and the solution of salicylaldehyde. The organometallic product separated as white powder and it recrystallized twice from alcohol. The compound are very stable at room temperature and it has been characterized via FT-IR spectroscopy.

2.3. Preparation of CaSnO_3

Calcium Stannate prepared via a co-precipitation method. Convenient amounts of $\text{Ca}(\text{sal})_2$ complex and SnCl_2 used as the Sr and Sn precursors. In a typical procedure $\text{Ca}(\text{sal})_2$ dissolved in methanol as solvent and SnCl_2 dissolved in distilled water separately. Then $\text{Ca}(\text{sal})_2$ heated to 60 °C for 10 min. Finally, the solution of SnCl_2 added drop to drop on this solution. Lastly, an alkaline agent (that listed in Table 1) added to this solution drop wise to reached pH = 13. As-obtained solutions heated at 60 °C for 1 h with stirring. The product centrifuged, washed with distilled water and ethanol and dried in the vacuum at 60 °C. Then dried powder has been calcinated at 900 °C for 5 h in air with the average temperature that increased the temperature by the rate of 30 °C every minute, before the temperature reached 900 °C, and after keeping the thermal treatment. It was approved to cool naturally to the room temperature. To extend the best conditions of synthesis, these procedures repeated on different experiment conditions that listed in Table 1.

Table 1. Different condition for preparation of CaSnO_3

Sample No	Amine	pH	Rate of Stirrer (rpm)	Temperature (°C)	Surfactant
1	NH_3	13	300	60	-
2	$\text{C}_2\text{H}_4(\text{NH}_2)_2$ (en)	13	300	60	-
3	$\text{C}_3\text{H}_6(\text{NH}_2)_2$ (pn)	13	300	60	-
4	$[\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2]_2$ (TETA)	13	300	60	-
5	$(\text{NH}_2\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2)_2\text{NH}$ (TEPA)	13	300	60	-
6	$(\text{NH}_2\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2)_2\text{NH}$ (TEPA)	9	300	60	-
7	$(\text{NH}_2\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2)_2\text{NH}$ (TEPA)	11	300	60	-
8	$[\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2]_2$ (TETA)	13	300	60	SDS
9	$[\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2]_2$ (TETA)	13	300	60	CTAB
10	$[\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2]_2$ (TETA)	13	300	60	PEG
11	$[\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2]_2$ (TETA)	13	100	60	CTAB
12	$[\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2]_2$ (TETA)	13	500	60	CTAB
13	$[\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2]_2$ (TETA)	13	300	25	CTAB
14	$[\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2]_2$ (TETA)	13	300	80	CTAB

2.4. Photo-degradation of azo dyes

The degradation of azo dyes executed in a home-made system of glass reactor enclosing 200 mL of dye solution with relatively high concentration (20 ppm) in pH = 3 and 0.005 g/L of CaSnO_3 nano-powder photo-catalyst. The suspension set sidewise via magnetic stirring (500 rpm) at room temperature and laid below dark conditions for 30 min. After that, the system is irradiated by UV lamps (Osram ULTRA-VITALUX 300 W).

3. Results and discussion

3.1. Characterization of *[bis(salicylaldehydato)calcium (II)]; Ca(sal)₂*

The XRD pattern of Ca(sal)_2 have six peaks and depicts in Figure 1a. The positions of these peaks are $2\theta = 25, 30, 36, 45, 52$ and 65 . The FT-IR spectrum of Ca(sal)_2 shows in Figure 1b. In this spectrum, the stretching vibrations of C-O located in 1430 and 1525 cm^{-1} . On the other hand, the peak at 1326 cm^{-1} have been attributed to C-C stretching vibrations of the salicylaldehyde. In free salicylaldehyde $\nu_{\text{C-O}}$ looks at 1680 and 1660 cm^{-1} , and $\nu_{\text{C-C}}$ looks at 1490 and 1380 cm^{-1} . So, The broad peak around 3450 cm^{-1} in Figure. 1b, has been assigned to the stretching vibrations of absorption water on the surface of product. Besides the peak on 1638 cm^{-1} have been distributed to bending vibration of water. Absorption peaks at 465 cm^{-1} have been caused by Ca – O bond, there aren't at all absorption bands nearby this range in salicylaldehyde [18-21].

Figure 1. (a) XRD pattern and (b) FTIR spectrum of Ca(sal)_2

3.2. Characterization of CaSnO_3

Figure 2a illustrates the XRD pattern of powder that achieved in co-precipitation method. The pattern has been assigned to CaSn(OH)_6 which indexed as a cubic phase (space group: Pn-3). As-achieved experimental pattern is very resembling to the literature values by JCPDS No. 74-1823, the small sharp peaks of CaSn(OH)_6 shows that this material has been crystallized very well. Figure 2b-e show the XRD patterns of different samples that synthesized in different calcination temperature. In low temperature about 300°C , just SnO_2 have been presented in the product (Figure 2b) by increasing the temperature to 500°C and 700°C the product (CaSnO_3) have been appeared in patterns but a few amount of impurities have been existed. The intensity of these impurities has been decreased by increasing the temperature of the reaction. The crystallite size has been also calculated by Scherrer equation, $D_c = K\lambda / \beta \cos\theta$, where β is the breadth of the observed diffraction line at its half intensity maximum, K is the shape factor, that takes a value of about 0.9, and λ is the wavelength of X-ray source used in XRD. The estimated crystallite size of this product is about 27 nm.

According to the XRD patterns of product in different temperature, it comes that the changing in temperature is the main factor on purity of product to produce CaSnO_3 . Without calcination process the product of reaction is CaSn(OH)_6 . By calcination of powder that made of from co-precipitation reaction the hydroxide of calcium and stanate have been changed and formed the oxide of metals but the structure of this oxide have been changed by changing the temperature. In the low temperature SnO_2 have been presented in product by increasing the temperature the CaSnO_3 have been appeared in product and the amount of SnO_2 have been decreased. The best temperature for preparation of CaSnO_3 is 900°C .

Figure 2. XRD pattern of as-prepared products (a) before calcination, after calcination (b) 300 °C (b) 500 °C (c) 700 °C (d) 900 °C

Figure 3a shows the FT-IR spectrum of as-prepared CaSnO_3 . The vibration bands that placed in the range of $3600 - 3000 \text{ cm}^{-1}$ and 1649 cm^{-1} have been assigned to different types of O–H stretching and bending vibrations of water molecules absorbed on the surfaces of CaSnO_3 particles, respectively. The vibration peaks in the range of $1350 - 850 \text{ cm}^{-1}$ are accountable to bending modes of different hydroxyl groups on the surface of the product that appears in the definitive product at 1350 cm^{-1} . The vibrations of the stannate group (SnO_2^{3-}) have been detected as high-intensity bands in the $300 - 400$ and $600 - 700 \text{ cm}^{-1}$ ranges, including the stretching vibration of the Sn–O bond at 530 cm^{-1} [22–24]. In this work, the calcined products have been detected at 670 cm^{-1} with high intensity. Low-intensity peaks that appeared in $2800 - 3000 \text{ cm}^{-1}$ have been related to C–H caused from a organic compound on the surface of the product. This organic compound has been eliminated by washing the product with water and drying at vacuum.

Figure 3b, c illustrates the optical absorption spectrum of Calcium Stannate nanocubes, the DRS spectrum has been applied to further access their quality and electronic structure information. It demonstrates three excitonic absorption peaks at 212, 264 and 366 nm (Figure 3b). The absorption that happened in 366 nm assigned to the essential absorption that caused because of the band gap of this material. The other absorption band in 240 nm attributed to the impurities or vacancies [25]. The optical band gap of calcium Stannate has been estimated by the optical density (OD) vs. energy (E) plot (Figure 3c). CaSnO_3 shows an optical absorption edge at $\sim 300 \text{ nm}$, as valued for direct band gap of simple cubic structures. Data in the range of $5.0 < h\nu < 5.7 \text{ eV}$ have been well fitted to a direct transition, according to these data and value of direct band gap is 4.15 eV. The direct band gap of this material is in good agreement with the other report in the literature [26].

Figure 3. (a) FTIR spectrum of SrSnO₃ (sample No. 4) (b) DRS spectrum of SrSnO₃ (c) Optical density plot SrSnO₃ (Sample No 11) (d) Photocatalytic degradation of Acid blue 92 and acid brown 14 in presence UV light by SrSnO₃ nanoparticles (Sample No 11)

The morphology and structure form of product has been characterized via scanning electron microscopy (SEM) images. For the formation of product, the reaction conditions have been optimized to achieve the best morphology. Figure 4 shows the SEM images of product in presence of different alkaline agent. When ammonia have been used as an alkaline agent for the preparation of CaSnO_3 , products in this condition have nanoparticles structures but these particles have been aggregated (Figure 4a).

Figure 4. SEM images of (a) Sample No 1 (b) Sample No 2 (c) Sample No 3 (d) Sample No 4 (e) Sample No 5

Via increasing the amine agent and using of ethylene diamine as alkaline agent, the size of product have been increased and shaped the semi-geometrical structures (Figure 4b). In attendance of propylene diamine for precipitation of CaSnO_3 , it seems that nanostructures have been aggregated and shaped compacted structures (Figure 4c). In presence of triethylene tetraamine (TETA), the morphology of product have been changed and shaped semi flowers structures but as-prepared product have been aggregated in this condition (Figure 4d). By increasing the cramming of alkaline agent and using of tetra ethylene pentaamine (TEPA) have been shaped ununiformed structures. In this condition, two types of materials have been existed in the as-prepared products (Figure 4e).

The effect of pH of solution has been studied on morphology of products. Figure 5a-b show the pH effect on presence of TETA. The pH of solution has been fixed on 11 and 9 instead of 13 (Figure 4b). In pH = 11 the products have been arrayed irregular nanostructures (Figure 5a), that involved from nanoparticles to hexagonal nanostructures. But when the pH of solution has been set on pH = 9, the products have been arrayed nanocube structures but these cubes have been aggregated and formed big structures (Figure 5b), therefore the best pH according to these SEM images is pH=13.

The effect of surfactant have been studied for preparation of CaSnO_3 nanostructures, three types of surfactants such as cationic, anionic and polymeric surfactant have been used in synthesis process [27-29]. In presence of CTAB as a cationic surfactant, according to SEM image have been specified the morphology of product is nanocubes with about 500 nm edges (Figure 5c). When used of SDS in synthesis procedure the morphology of product did not change and remained in nanocubes structure but the symmetry and similarity of product in size and morphology have not been being suitable (Figure 5d). In these conditions the size of nanocubes are not humdrum. Figure 5f shows the SEM image of CaSnO_3 that synthesized in presence of PEG600. The cubic structures that synthesized in this condition have been being very large size and the edges of the cubic structures are very bigger than 2 μm .

Figure 5. SEM images of (a) Sample No 6 (b) Sample No 7 (c) Sample No 8 (d) Sample No 9 (e) Sample No. 10 For preparation of calcium Stannate the rate of mixing solution by stirrer has been optimized in presence of TETA. At low speed for stirring (about 100 rpm) products have been formed big cube structures. The cause of forming microstructures could be the enough time for agglomeration (Figure 6a). By increasing the rate of stirrer (to 300

rpm), products have been gained appropriate similarity as shown in Figure 5c. The higher rate of stirring (about 500 rpm), have been led to prepare the best similar nanocubes that formed from small nanoparticles and some of these nanoparticles have been observed on the surface of nanocubes (Figure 6b). This result may appear because of the surface energy of intermediate in reaction. In the high rapid stirring of solution, the high surface energy may be provide for products and it may be cause to aggregate product with together. Therefore the size of the product increased by increasing the rate of stirring. In low rapid of stirring the product have enough time to compact together.

Figure 6. SEM images of (a) Sample No 10 (b) Sample No 12 (c) Sample No 13 (d) Sample No 14

The effect of temperature solution has been investigated to optimize the condition of preparation products. In low temperature (Figure 6c) product has cubic structures with about 1 μm edges, but by increasing the temperature from 25 $^{\circ}\text{C}$ to 60 $^{\circ}\text{C}$ the size of nanostructures decreased and at the higher temperature (80 $^{\circ}\text{C}$) the size of product want to

decrease. Therefore the cubic structures have been broken and formed different sizes of cubic structures and nanoparticles (Figure 6d). With increasing the reaction temperature, the morphology of products have been changed and the size of products has been decreased, therefore by increasing the temperature, surface energy of products increased therefore the collection for preparation of cubic structures have been decreased.

TEM images of CaSnO_3 that prepared in presence of TETA and CTAB shows in Figure 7. The TEM image has been confirmed that the product has been formed from nanocube structures. This is in agreement with the SEM image of this as-prepared CaSnO_3 .

Figure 7. TEM image of Sample No 11

3.3. Photocatalytic property

Two types of azo dyes (acid blue 92 and acid brown 14) have been eliminated by CaSnO_3 under irradiation of UV light. Acid blue 92 is a mono azo dye and Acid black 14 is a diazo dye. These dyes have been used in intinction and loom industrial. The known dyes on wastewater have been caused cancer on a human body. So removal of these contaminations from wastewater have been attracted a lot of consideration. The acidic solution of acid blue 92 and acid brown 14 have been prepared as wastewater for photo-degradation testing. Figure 3d exhibit the photocatalytic behaviours of CaSnO_3 powder for degradation of acid blue 92 and acid brown 14 as organic pollutions under UV

light. Results demonstrate that the absorption of acid blue 92 solution in beginning of test was 0.721 and this value for acid brown 14 was 0.913. At the end of reaction under UV light and after 120 min irradiation the absorptions were 0.235 and 0.203 respectively. The percentage of photodegradation have been calculated for Acid blue 92 and acid brown 14 on this time range under UV irradiation and presented that the degradation percentage were about 67 % and 77% respectively.

When the nanopowder exposed to UV light with the energy greater than or equal to the band gap, in the valence band of a semiconductor of nanopowder an electron excited to the conduction band and generated a hole in the valence band. These electrons and holes pairs interacted independently with other molecules in aqueous solution. The holes in the valence band reacted with the water molecules on the surface of CaSnO_3 or hydroxide ions and yielded extremely reactive hydroxyl radicals ($\cdot\text{OH}$) while electrons admitted oxygen by adsorption and formed superoxide radical anion (O_2^-). This radical might be shaped organic peroxides or H_2O_2 in attendance of organic adsorbent. The hydroxyl radical is a very strong oxidizing agent and aggress the dye molecule to provide the oxidized product [30-32]. According to this result, CaSnO_3 could be introduced as an effective photocatalytic agent in aqueous solution for degradation of organic pollution in presence of UV-light.

4. Conclusion

In this work Calcium Stannate have been prepared by a superficial co-precipitation method. Various parameters that affected on the morphology of product such as alkaline agent, temperature, rate of stirring and surfactant have been optimized to prepare the best product. Results indicate that the best morphology have been achieved at the presence of TETA as alkaline agent and the best solution temperature was about 60°C and the rate of stirrer was 300 rpm, on the other hand, the presence of surfactant are necessary. The edge of these products is about 500 nm. For the first time the photocatalytic properties of as-achieved CaSnO_3 under UV light have been investigated for degradation of two type azo dyes (acid blue 92 and acid brown 14). Results illustrate that the powders have suitable catalytic behavior for degradation of monoazo and diazo dyes from waste water. The degradation percentage of monoazo dye is about 67% in presence of UV light and this value for acid brown 14 is about 77%.

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