



Experimental investigation of photocatalytic and photovoltaic activity of titania/rice husk crystalline nano-silica hybrid composite



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ABSTRACT

As a kind of highly operative, stable photocatalysts and low-cost, TiO_2 has received significant scientific attention. However, it can only be initiated under ultraviolet light irradiation owing to its wide bandgap, weak separation efficiency of carriers and high recombination. Doping or making composites is an active method to outspread the light absorption to the visible light region. This article describes the extraction procedure of nano-silica from rice husk through a dissolution-precipitation process and producing titania-nanosilica composites by sol-gel method for photocatalytic and photovoltaic application where extracted nanosilica act as green filler to enhance the surface area of titania photocatalyst and an inhibitor of recombination. Our general plans are anticipated by passivated composite to enhance the activity of TiO_2 semiconductors. This results show that the TiO_2 /rice husk extracted crystalline nanosilica (RHCNS) has formed an active hybrid photocatalyst which is significant for photovoltaics application also. Large enhancement of photovoltaic parameters and rate of dye degradation of the TiO_2 /RHCNS composite observed which is stepping path to further development in adsorption-assisted photocatalysts under both UV and visible lights.

1. Introduction

Titanium oxide is a transition metal functional oxide that engrossed wide studies as a photocatalyst. This material has the larger chemical and physical properties in terms of stability, low cost and non-toxics. These properties make titanium oxide as an environmental friendly photocatalysts [1]. The photoactivity of pure titanium oxide can degrade dye like methyl orange (MO) using sunlight radiation as photon energy source [2]. The MO concentration was significantly decreased in one hour irradiation and titanium oxide produces various compound such as NO^{-3} , CO_2 , SO_4^{-2} , NH^{+4} [3]. To improve the photocatalytic effect in the visible light region, several processing methods were suggested to dope impurity in TiO_2 . Recent literature showed the development in enhancing photocatalytic activity if other oxides were combined with TiO_2 [4]. Essentially in titania-silica nanocomposites, the photocatalytic activity increased compared to pure TiO_2 . In this case, silica plays an essential role to enhance photon absorption activity. Rice husk, a waste product of rice industry is a unique source of this high-grade amorphous silica. Literature reports that rice husk comprises active silica in high content around 87–97 wt% [5] which is smooth grain, and reactive. In addition, the availability of rice husk in our region was also the reason as a background to conduct this investigation. Extraction of this silica through a cost effective process can

also minimize the environmental impact caused by improper disposal of rice husk. Several researchers have accompanied various studies related to combustion of rice husk to utilize energy maximum and to produce improved grade residue [5]. Several reports also available on the designing of silicon solar cells using of $\text{SiO}_2/\text{Si}_3\text{N}_4$, $\text{TiO}_2/\text{SiO}_2$, a-Si/ SiO_2 , c-Si/ SiO_2 , porous silicon layers etc. [5]. At higher temperatures, it undergoes a phase change resulting into crystalline forms of silica/titania composites which enhance the surface area with enough pore size. Several issues can affect the photocatalytic property of titania-silica such as its particle size, crystalline, surface area, and dislocations on the surface [6]. The decreasing in particle size inhibited the recombination of electron photoexcitation, and hole so that it photocatalytic property will increase [7].

In the present study, first we report an innovative approach of extraction nanosilica from rice husk and then we highlight the emerging applications of this extracted nanosilica doped TiO_2 hybrid thin films as well as powder composites in the areas of photovoltaics and photocatalysis. Rice husk was chosen as a nanosilica source because it is much cheaper than other oxides since this material is naturally available in large quantity. First, the rice husk has been burnt at a controlled temperature to get pure white rice husk ash. In Dissolution-precipitation technique which contains alkali treatment, concentration of sodium silicate, gelation, variation of pH, aging time and aging

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temperature were optimized to prepare pure nano-silica particles. The silica present in rice husk is inherently amorphous and biogenic. Normally, controlled combustion below 800 °C yields white ash with amorphous silica [8]. Obtained amorphous silica from rice husk is a very useful product as it is chemically active.

The mixing of extracted nanosilica powder with titania sol was conducted where titanium butoxide was used as a source of titanium oxide. Various instrumental tools like X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Fourier Transform Infrared Spectrometer (FTIR), Energy Dispersive Spectroscopy (EDS) have been used to know the grade of nanosilica. Our attempt was to overcome the limitation of TiO₂ thin films which induce great influence for a wide practical application. That is, an increased amount of energy from the solar light spectrum is well utilized.

2. Experimental

2.1. Materials and chemicals

Titanium isopropoxide (TTIP)(99.99%, Sigma-Aldrich), is used as a raw materials for titania. The used Minikit type of rice husk samples were obtained from a rice husk mill of Burdwan district, West-Bengal, India. The samples were washed with distilled water to remove adhering soil and dust. An acid leaching step using hot hydrochloric acid (HCl) solution (Merck) removed metallic impurities prior to extracting silica from the rice husk. Analytically pure NaOH was used in the process.

2.2. Synthesis of pure silica from rice husk ash (RHA)

Rice husk was dried in an oven at 120 °C for 24 h to remove water content. The dried rice husk was then taken in a crucible and placed it in an electrical furnace for 1 h at 500 °C to get a black form ash. The ash was then ground into powder and later heated again at 800 °C for 2 h for complete combustion to obtain white rice husk ash (RHA) powder.

The selection of ash is important as the quality of ash determines the total amount as well as the quality of silica recoverable. The initial step is the extraction of silica from ash which form as sodium silicate after adding caustic soda. 10 g of RHA samples were added with stirring in 80 ml 2.5(N) Sodium Hydroxide solution. This solution was boiled in a covered 250 ml Erlenmeyer flask for 3hr. After that the solution was filtered and the residue was washed with 20 ml boiling water. After washing with boiling water the filtrate was again allowed to cool down at room temperature (25 °C) and added 5(N) H₂SO₄ until pH reach 2 and then added NH₄OH until pH 8.5, allowed for 3.5 h. The filtrate was then dried at 120 °C for 12 h in an oven. Ultimate received product is pure amorphous silica (as confirmed by XRD, FTIR). Formation of Nano crystalline silica from amorphous silica was carried out by refluxing with 6(N) HCl for 4 h and then washed repeatedly using de-ionized water to make it acid-free. It was then dissolved in 2.5(N) NaOH by continuous stirring for 10hr and then concentrated H₂SO₄ was added to adjust the pH range from 7.5 to 8.5. The precipitated silica was washed repeatedly with warm de-ionized water (60 °C) until the filtrate became completely alkali free. The washing process continued by deionized water repeatedly, and finally calcined at 800 °C for 2 h. in the muffle furnace. Fig. 1 illustrate the elemental analyses using Energy Dispersive Spectroscopy (EDS) of synthesized rice husk ash after calcined at 800 °C and extracted pure crystalline nano-silica (calcined at 1000 °C).

2.3. Preparation of Titanium(IV) isopropoxide (TTIP) sol and titania-silica composites

For preparing 20 mmol TTIP solution 5 ml ethanol and 0.5 ml acetic acid were taken in a conical flask. Stirring was conducted for 30 min. Further dropwise 0.6 ml TTIP was added under constant stirring for another 30 min. The transparent solution was kept overnight in a

desiccator. After that 0.1 mg rice husk nano silica power (amorphous) and 0.1 mg rice husk nano silica powder (crystalline) was added separately to the diluted solution of 10 ml 20 mmol TTIP under the constant stirring for 2 h and then preserved overnight into a desiccator. Each of these two solution was used for synthesis powder for photocatalysis experiment (450 °C at the furnace) and as well as preparation of thin films for photovoltaic application.

2.4. Sample characterization

Morphological study of the RHA and RHNS were observed by Scanning Electron Microscope [SEM] (ZEISS EVO-MA10, Germany) and EDS (Oxford Instruments, UK). The major chemical groups present in the samples have been identified by the FTIR spectra (Alpha FTIR, Bruker, Germany). X-Ray diffractometer was used to analyses the phases of samples keeping the scanning rate 10/min in the 2θ diffraction angle between 10° and 70°. XRD diffractograms (PANalytical PW3040/60, Netherlands). The microstructural properties and crystal structures of the samples were characterized using a transmission electron microscope (TEM) system (HRTEM: JEM-2100, JEOL, Japan) operated at 200 kV.

3. Result and discussion

3.1. Rice husk extracted silica characterization

The phase analysis of the extracted rice husk silica (RHS) were carried out using a rapid analytical technique, X-Ray Diffraction (XRD) tool with Cu Kα (λ = 1.54 Å). The objective of the analysis was to reveal the sample purity of the materials and crystallinity. The XRD patterns of the obtained amorphous silica powder shown in Fig. 2(a). The characteristic broad silica peak indicates the presence of amorphous silica. In contrast, to achieve the crystalline phase and to measure the crystallite size of the pure nano silica, the product is calcined at 800 °C after refluxing. This crystalline nanosilica RHCNS (Fig. 2(b)) showed strong sharp crystalline peak between 20° and 30° (2θ). (22.27°) in X-ray spectra. This sharp peaks also suggest the formation of cristobalite structures [9]. The average crystallite size calculated from the broadening of the corresponding peaks by the Scherrer formula is 25 nm in case of crystalline silica.

In order to get an idea about the shape, size, textures, composition and features present of the rice husk ash (RHA) and crystalline nanosilica (RHCNS), Scanning Electron Microscope (SEM) analysis was carried out. The obtained morphological structure (see in Fig. 3(a)) RHA reveals that the structures are loosely agglomerated, canals, pores, and striations existing with regular smaller grains. EDS profile of RHA contain predominantly the elements of Si, O and minor percentage of K, Ca, Mg and Pt (Due to Pt coating of the sample) (see in Fig. 1(a)). Both Si and O peaks correspond to the existence of the silica. Hence the composition analysis through EDS peaks is the evidence that rice husk ash (burnt at high temperature) can be a potential source of nano-silica. Fig. 3(b) shows that morphology of extracted RHCNS calcined at 800 °C. Small sphere-like particles with average 20–30 nm diameter observed. Presence agglomeration was due to the hydrogen bonding between silanol groups on the surface of RHCNS [10]. Also, EDS peaks of this extracted nanosilica confirmed the elemental compositions and it is also evidence of pure silica with a higher percentage from raw rice husk.

In addition, TEM studies have been carried out on the extracted silica from rice husk samples to correlate the particle size of the obtained in amorphous (Fig. 4(a)) and crystalline nano silica powder (Fig. 4(b)). The obtained amorphous silica powder is formed agglomerated but crystalline nanosilica has uniform spherical shape of the silica grains with an average homogeneous particle size distribution of about 25 nm, which is in good agreement with the average crystal size estimated from XRD pattern.

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