Results in Physics 11 (2018) 72–78

Contents lists available at ScienceDirect

Results in Physics

journal homepage: www.elsevier.com/locate/rinp

Synthesis, characterization and investigation of photocatalytic activity of nano-titania from natural ilmenite with graphite for cigarette smoke degradation

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ARTICLE INFO

Keywords:
Titanium dioxide
Photocatalytic activity
Graphite
Nanocomposite
Smoke

ABSTRACT

Titanium dioxide (TiO_2) nanoparticles and TiO_2 with recycled graphite (TiO_2/G) nanocomposite have been successfully synthesized by alkaline fusion method using synthetic rutile for measuring the degradation time of the cigarette smoke under the visible light irradiation. In this work, the synthetic rutile was derived from natural Malaysian Ilmenite's waste to produce a low cost of TiO_2 nanoparticles via environmental friendly process. The prepared samples were then analyzed using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy and Energy Dispersive X-Ray Fluorescence (EDXRF) to study their structural phase composition, functional group, and elemental composition respectively. The surface morphology and the size of the particles were studied using Field Emission Scanning Electron Microscopy (FESEM) and Particle Size Analyzer (PSA), respectively. The functionality of the prepared nanoparticles in terms of photocatalyst activity was analyzed by degradation of cigarette smoke under the exposure to the visible light. The UV–Vis Spectroscopy (UV–VIS) results revealed that the energy band gap of modified TiO_2/G nanocomposite decreases to 2.90 eV compared with the commercial TiO_2, 3.06 eV. This is capable enough to TiO_2/G nanocomposite degrade the smoke under the visible light irradiation for 2 min faster compared to others types of TiO_2 nanoparticles. This indicated the material has the ability to purify the toxins in the air.

Introduction

Cigarette smoking can cause a various side effects not only for smokers and nonsmokers. For example, cancer, respiratory diseases and heart diseases. Moreover, the effect is intrinsically more toxic for nonsmoker than smoker who directly inhaled the smoke [18]. To date, TiO_2 in nano scales size offer many advantages to purify the environment from organic pollutants problem such as strong oxidation power, nontoxicity, long-term photo stability and also can act in the visible region [11]. Several studies have shown that TiO_2 has great potential application related to environment such as acting as photo induced removal of pollutant either from the air or wastewater [3]. Nikkanen et al. [16] also have been studied that TiO_2 is the most suitable material for the purification of contaminated water and air compared to others semiconductor material.

TiO_2 nanoparticles also called as Nano-Titania is an n-type semiconductor with specific functionalities especially for photocatalytic performance [1,14]. TiO_2 nanoparticles occurs naturally in nature as mineral which has several polymorphs with similar chemical formula but diverse in terms of crystalline structure. TiO_2 nanoparticles in anatase phase are considered to be the most thermodynamically stable for the nanoparticles with a size between 10 and 20 nm and widely used in the photacatalyst activities due to its lower energy band gap compared to the TiO_2 nanoparticles in rutile phase [24,12].

However, Gemmellaro et al. [2] previously reported that the undoped or pure TiO_2 nanoparticles (usually TiO_2 P25) does not show any reaction in the visible light. Another researcher proposed the modification on the surface properties of TiO_2 nanoparticles via doping or mixing with others element [21]. Owing to that, we modified the pure TiO_2 nanoparticles by simply added recycle graphites from pencil lead (TiO_2/G nanocomposite) in order to increase the effectiveness of photocatalytic capability to degrade the cigarette smoke in the visible light region.

Therefore, this paper focus on producing TiO_2 nanoparticles and

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https://doi.org/10.1016/j.rinp.2018.08.032
Received 19 May 2018; Received in revised form 16 August 2018; Accepted 16 August 2018
Available online 22 August 2018

TiO_2/G nanocomposite in anatase phase using low cost raw material and offers safer materials for the environment in contrast to the industrial and commercially available TiO_2 nanoparticles (TiO_2 P25). In our study, we used synthetic rutile derived from natural Malaysian Ilmenite's waste. Although this types of waste has low quality mineral but it has better potential to produce high quality metals and oxide [13]. So, TiO_2 nanoparticles and TiO_2/G nanocomposite can be produce through a simple and low cost technique.

In terms of preparation technique, there are several methods have been proposed to prepare nano-size TiO_2 such as sol-gel method, solvothermal methods and hydrothermal method [23]. However, the major bottle neck in synthesizing the TiO_2 consist of both high cost and hazardous chemicals used. Most of the methods involves the expensive precursor such as titanium butoxide, titanium alkoxide and titanium (IV) ethoxide which can be extremely harmful to human being [13]. Plus, Christie et al. (2006) have been proven titanium(IV) ethoxide show a most cytotoxic and inflammatory in a cell culture.

So here, in this project we used the simplest techniques Alkaline fusion method as it only involves a fusion process (using small amount of NaOH) and a leaching process (using small amount of H_2SO_4). In additions, this method can also be classified as an eco-friendly process due to the use of the non-toxic raw material such as natural Malaysian Ilmenite's waste .

Materials and experimental methods

Materials

Synthetic rutile derived from the mineral ilmenite was purchased from tin mining factory in Perak, Malaysia. Sodium Hydroxide (NaOH) supplied by R&M Chemical, 2 M of Sulfuric Acid (H_2SO_4) supplied by Emsure and recycled graphite from pencil lead as a dopant. Degussa P25 TiO_2 nanoparticles (P25) and Commercial rutile TiO_2 nanoparticles were used as a reference for photocatalytic experiment. Dionized water was used throughout in synthesis process.

Synthesis process

The TiO_2 nanoparticles and TiO_2/G nanocomposite was synthesized through a modified Alkaline Fusion method where these process consisted of fusion, washing, filtration and leaching process.

Firstly, for first sample (TiO_2 nanoparticles), 100 g of synthetic rutile was mixed with 200 g of NaOH at the stoichiometric ratio 2:1. Then followed by calcination in a furnace at 550 °C for 3 h. After fusion process, 2 M of H_2SO_4 was added to the product for leaching process. The solution was constantly stirred for 3 h and the temperature was maintained at 80 °C. This was the optimum concentration and temperature to ensure the final product completely forms as TiO_2 in anatase phase and to reduce the crystallite size [13]. Subsequently, the obtained product was washed with deionized water for three times. Then, the sample was dried in the oven at temperature range 70–80 °C for 24 h and was crushed using a mortar and pestle to produce a fine powder.

Second sample (TiO_2/G nanocomposite) was prepared in presence of graphite powder. The synthetic rutile was mixed with NaOH at similar to the first samples ratio before calcination in a furnace at 550 °C for 3 h. Then, during the leaching process, 2.58 g of recycled graphite from pencil lead and H_2SO_4 was added simultaneously. The solution was then constantly stirred for 3 h at 80 °C. After that, the obtained product was washed with deionized water for three times before dried in the oven at range 70–80 °C for 24 h. Finally, the final product was crushed using a mortar and pestle to produce a fine powder.

Structural and chemical characterization of the prepared sample

Phase composition of synthesized samples is analyzed by XRD analysis using a PANalytical PW3040/60 X'Pert PRO apparatus. The voltage and anode current used were 40 kV and 30 mA, respectively. The broad scanning analysis was typically conducted within the 2θ range between from 20° to 80° and the CuKα is 0.15406 nm. Next, the surface morphologies and size of particles of all the samples were determined by FESEM Carl Zeiss GeminiSEM500 and Honeywell Microtrac X100 PSA. For chemical characterization, Shimadzhu EDX7000 EDXRF spectrometers was used for the identification and quantification of elements in a substance. All measurements were carried out under air condition with the collimator was 10 mm, and equipped with silicon drift detector (SDD). The energy band gap of the prepared samples was determined and calculated using Perkin Elmer Lambda 35 UV–VIS. The frequency used is from 1100 nm to 2000 nm with the slit width is 4 cm and the ordinate mode is in terms of % Reflectance mode (%R). FTIR spectrum were obtained on a Perkin-Elmer (FT-IR Spectrum 200) spectrophotometer using KBr powder in the range of 4000–400 cm^{-1}.

Photocatalytic activity of the prepared sample

To investigate the photocatalytic capability of each type of TiO_2 nanoparticles, the cigarette smoke was chosen as model of air pollution. A glass rod was dipped into a formulation of 100 mL of water based coating and 1 g of prepared sample powder for one minute before drying at room temperature. The cigarette smoke was collected in Bucherner funnel using a vacuum pump (Fig. 1(a)). It was then photo irradiation at room temperature using visible light (Fig. 1(b)). The smoke degradation over times was measured.

Results and discussions

The synthesis TiO_2 nanoparticles and TiO_2/G nanocomposite was successfully fabricated by modified Alkaline Fusion method from synthetic rutile in anatase phase/structure. The reaction between synthetic rutile and NaOH during the fusion process at high temperature (550 °C) lead to the breaking of Ti–O–Ti bond and then the broken bond was replaced with Na and OH to form a new bond; Ti–O–Na and Ti–OH bond. This results was further confirmed by FTIR spectrum as shown in Fig. 2. The washing and filtration process after this process was removed the unwanted Na and other impurities in order to get the purity of fusion product.

Next, for leaching process are consist of three stage; 1) recrystallization, 2) removal of impurities, and 3) reduce the crystallite size. The molarity of H_2SO_4 (2 M) and constant temperature (80 °C) is important factor to breaking and reforming the new particle in nano size. The FTIR spectrum of both samples showed the intense broad band in the range 400–1000 cm^{-1} common in the spectra of all samples is assigned to the stretching vibration of Ti–O bond. The band at wavenumber 3381 cm^{-1} are showed the terminal of OH bond which are characteristic of metal oxides. The presence of carbon group C–H was confirmed in spectra at the peaks of 2921 cm^{-1} and 2849 cm^{-1} [17,19,4].

So, to further confirmed, XRD analysis was carried out for both sample TiO_2 nanoparticles and TiO_2/G nanocomposite. Fig. 3 shows the comparison of XRD analysis between synthesized (TiO_2 nanoparticles and TiO_2/G nanocomposite) and commercial (TiO_2 Rutile and TiO_2 P25), where the unknown element were identified by comparing diffraction data opposed to a database maintained by International Centre for Diffraction Data (ICDD).

XRD data in Fig. 3 was confirmed synthesized TiO_2 nanoparticles and TiO_2/G nanocomposite showed formation of anatase phase (ICDD PDF 01-071-1166) due to obvious peak at 2θ = 25.2° (1 0 1), 37.6° (0 0 4), 47.9° (2 0 0), 54.2° (1 0 5) and 62.15° (2 0 4). The intensity at peak 2θ = 26.5° G (0 0 2) attributed to graphite (ICDD PDF 41-1487) in Fig. 3(b) was formed during addition of graphite powder in leaching process are clearly seen in TiO_2/G nanocomposite sample [6,9]. Although, there some impurities in the TiO_2 nanoparticles samples which