



Bismuth titanate as an infrared reflective pigment for cool roof coating



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ABSTRACT

A pale-yellow color nano pigment $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ (BTO) with impressive infrared (IR) reflectance ($R = 95\%$) was synthesized using a simple hydrothermal method and characterized its crystalline nature, structural property, morphology and optical properties using X-ray diffraction technique (XRD), Raman spectroscopy, Fourier Transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), UV–Vis–NIR spectrophotometer. The Near infrared (NIR) reflectance was measured using UV–Vis–NIR diffuse reflectance spectrophotometer at a wavelength range of 700–2500 nm. The band gap of the synthesized BTO nano pigment increases from 2.74 to 3.05 eV by increasing the calcination temperature from 600 °C to 800 °C. For comparison study, the IR reflectance property and the thermal performance of synthesized BTO nano pigment was compared with the conventional infrared reflective white pigment TiO_2 . The BTO nano pigment shows higher NIR reflectance property and better thermal performance than the TiO_2 pigment. The potential application of the BTO nano pigment as the “cool pigment” was also demonstrated by coating on to the steel substrate for the energy saving application.

1. Introduction

Solar spectrum consists mainly, 44.7% of visible radiation, 6.6% of ultraviolet radiation and 48.7% of infrared radiation. One part of solar spectrum is the infrared (IR) radiation, especially near-infrared radiation (NIR). The earth's atmosphere has a highly transparent window in infrared (IR) wavelength region between 8 and 13 μm , this region is called as “atmospheric window”. The material with high IR emission in 8–13 μm region is considerable for radiative cooling application [1]. The effect of NIR radiation on the coating will reflect on the performance of usability and durability of the coating. The interaction of pigments with the wavelength other than visible light has interesting properties on the coating.

Buildings are responsible for at least 40% of energy usage in most of the countries. An outdoor coating with high NIR property brings significant energy savings for building applications [2,3]. When an object absorbs light waves, the energy carried by the light waves are converted into the heat energy whereas approximately one-third of interior heat gain comes from the roof of the building. A study revealed that by increasing the heat reflectivity of the roof, homeowners can save an average of 23% of their cooling costs. According to the U.S. Department of Energy the color of the building can significantly affect the interior temperature of the building and also shows an effect on the cost of electricity due to the usage of high power consuming electric equipments like air conditioner to reduce the interior temperature.

Usually dark colors absorb more heat energy from the direct sunlight while lighter colors reflect the solar heat back into the atmosphere. A building with dark colors will absorb 70–90% of radiant energy from the direct sunlight that strikes the wall of the building [4]. Some of this collected heat will be conducted into the building. In contrast, light colored buildings reflect most of the heat away from the exterior surfaces resulting in less heat build-up. Due to these reasons of especially less heat build-up, light colored exterior coating is more preferable than the building with dark colored roof.

In recent days many NIR reflective inorganic pigments have been reported. But the current interest in the pigment industry is to develop a non-toxic and cheaper cool pigments that help in energy savings [5]. Recently an ultramarine pigment with good NIR reflectance value of 65% has been reported [6]. NIR reflective pigments with different shades of yellow are recently reported with shade of pale yellow [7] and bright yellow [8–11] with the reflectance value of 90%–93.7%. Light color shade NIR reflective pigments like Fe-doped MgTiO_3 [12], chromium doped alumina [13] and Mn-doped ZnAl_2O_4 [14] with NIR reflectance of 74%, 70.5% and 50.55% are also reported recently with low NIR reflectance values. In current scenario a cool pigment with high NIR reflectance, cheaper in cost and less toxic is more needed.

Recently, Bismuth Titanate, $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ (BTO) has attracted wide attention for its photo catalysis [15], ferroelectric material [16], piezoelectric transducers [17] and electrocatalysts [18]. Even though BTO has numerous applications, its near infrared reflectance property has

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not been studied yet to the best of our knowledge.

In this paper, we have reported a BTO pigment as an IR reflective pigment for the first time. The BTO pigment was synthesized using simple hydrothermal method and analysed their crystalline structure, morphology, composition and optical properties and also compared its reflectance properties and thermal behaviour with the conventional white IR reflective pigment, TiO₂. The thermal study was carried out by exposing the IR reflective BTO pigment coated stainless steel on Infrared Lamp to confirm the usage of BTO as IR reflective cool pigment.

2. Experimental section

2.1. Materials required

The chemical reagents bismuth nitrate (Merck) Butyl titanate (Sigma Aldrich), Nitric acid (Rankem), Ammonium hydroxide and Sodium hydroxide (Rankem) are analytical grade and used without further purification.

2.2. Synthesis of bismuth titanate pigment

The acidic solution of bismuth nitrate (4 mmol) were mixed with the titanium source of butyl titanate (3 mmol) under constant stirring and heated on a hot plate for 1 h. Then the sodium hydroxide solution was added drop wise to the above solution till the precipitation get complete. Then the precipitate was transferred to the Teflon lined stainless steel autoclave and heated to the temperature of 180 °C. The resulting powders were repeatedly washed using centrifugation and decantation with deionised water and then the powder were dried in an oven for 24 h at 80 °C. Dried powders were ground using a mortar and pestle before further calcination and characterization.

2.3. Characterization techniques

The purity and the phase analysis of the samples were performed by powder X-ray diffraction using a Ni-filtered Cu K_α radiation with a powder X-ray diffraction (XRD) (Bruker). Data were collected by a step-scanning from 2θ angle of 10°–90°. The surface morphology was analysed using the scanning electron microscope (SEM) (HITACHI, S-3000H) and the SAED pattern was also analysed using Transmission Electron Microscope (TEM) (FEI, The Netherlands) (Tecnai 20 G2 (FEI make)). The TEM samples were well dispersed in ethanol solution using ultrasonicator; drop cast on the copper grid and dried. The raman studies were measured using Laser Raman Microscope (Renishaw Invia Raman Microscope). FT-IR measurements were measured using IR-Spectrometer (Bruker, TENSOR 27). The NIR reflectance of the pigments and pigment coated steel substrate were measured using UV-Vis-NIR Spectrophotometer (VARIAN CARY 500 Scan) with an integrating sphere attachment using poly-tetrafluoroethylene (PTFE) as a reference at a wavelength range of 200–2500 nm. The NIR solar reflectance (R*) in the wave-length range from 700 to 2500 nm was calculated in accordance with the ASTM standard number G173-03 as described elsewhere [19]. Then, the NIR solar reflectance or the fraction of solar radiation incident at wavelengths between 700 and 2500 nm that is reflected by a surface is the irradiance-weighted average of its spectral reflectance, r(λ) can be determined that is

$$R^* = \frac{\int_{700}^{2500} r(\lambda) i(\lambda) d(\lambda)}{\int_{700}^{2500} i(\lambda) d(\lambda)}$$

where r(λ) is the spectral reflectance (W m⁻²) obtained from the experiment and i(λ) is the solar spectral irradiance (W m⁻² nm⁻¹) obtained from ASTM standard G173-03. The color of the pigment was measured based on The Commission Internationale del' Eclairage (CIE) through L*a*b* 1976 color scales (CIE-LAB 1976 color scales). In this

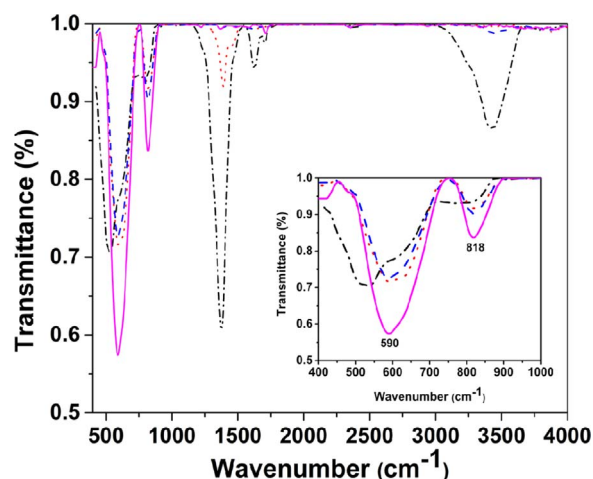


Fig. 1. FTIR spectrum of Bismuth titanate calculated at different temperature – as synthesized (· · ·); 600 °C (· · · ·); 700 °C (— — —); 800 °C (— — — —).

method the L* denotes the lightness axis (L* = 0 for black and L* = 100 for white), a* denotes green (-)/ red (+) axis, b* denotes blue (-)/ (+) yellow axis. The parameter C* (Chroma) denotes the saturation of the color and the hue angle (h°) is expressed in degrees and ranges from 0° to 360° and can be calculated using the formula $h^{\circ} = \tan^{-1}(b^*/a^*)$.

3. Results and discussion

3.1. FTIR

Fig. 1 shows the FTIR spectrum of as synthesized and calcinated BTO at different temperatures. The FTIR spectra was recorded at room temperature in the range of 400–4000 cm⁻¹ for the as synthesized and calcinated BTO at 600 °C, 700 °C and 800 °C were compared. The peak at FTIR spectrum in the wavenumber of below 830 cm⁻¹ is due to the cationic position of bismuth titanate. The sharp peak at 1376 cm⁻¹ is due to the N-O stretching vibration of nitrate which may present in the precursor solution and the broad peak at 3431 cm⁻¹ is assigned to the O-H stretching vibration. After calcination process the peaks in the range of 2800–3700 cm⁻¹ and 1771–1250 cm⁻¹ get disappears due to the evaporation of water, organic residues and nitrates. The calcinated BTO nanopowders show only two predominant peaks at ~ 588 cm⁻¹ and ~ 817 cm⁻¹. The sharp peaks at ~ 817 cm⁻¹ and ~ 588 cm⁻¹ are attributed to the Ti-O stretching vibration present in the BTO, which are in accordance with the results of BTO [19]. The presence of Ti-O stretching vibration with the characteristic band below 830 cm⁻¹ gives an idea of formation of a titanate structure in BTO.

3.2. Raman study

The formation of titanate structure of BTO is also confirmed by Raman Spectroscopy. Raman spectroscopy is a useful tool to investigate the structural changes in the formation of bismuth nitrate. Raman selection rule allows 24 raman active modes for orthorhombic bismuth titanate with space group of *Fmmn* [20]. The raman spectrum was recorded for both as synthesized and calcinated bismuth titanate nanopowders. Fig. 2 shows raman spectra of the BTO samples recorded in the range of 200–1000 cm⁻¹. The raman data of our bismuth titanate powder is in good agreement with the earlier results [21]. The formation of titanate structure was further confirmed by the raman studies from the peaks appeared at 540 cm⁻¹, 616 cm⁻¹ and 850 cm⁻¹. The raman peaks at 540 cm⁻¹, 616 cm⁻¹ and 850 cm⁻¹ are due to high intra-group binding energy into the TiO₆ octahedral. The raman peak at 271 cm⁻¹ is due to Ti-O torsional bending vibration in TiO₆ octahedral of BTO. The peaks at 566 cm⁻¹, 850 cm⁻¹ correspond to O-Ti-O

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