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P Mariselvi
Department of Chemistry, Rani Anna Govt. College for Women, Tirunelveli – 08, TN, India.

G Alagumuthu
Post Graduate and Research Department of Chemistry, Sri Paramakalyani College, Alwarkurichi, Tirunelveli – 627 412, TN, India

Correspondence
G Alagumuthu
Post Graduate and Research Department of Chemistry, Sri Paramakalyani College, Alwarkurichi, Tirunelveli – 627 412, TN, India

Synthesis, characterization and antibacterial activity of illite/TiO_2 Nanoncomposites

P Mariselvi, G Alagumuthu

Abstract
Titanium dioxide (TiO_2) occurs in three basic modifications anatase, rutile and brookite. Anatase is widely studied due to its very good photocatalytic properties, which may be used in additives to paints and construction materials, for degradation of organic pollutants in air and water etc., In the present work, the synthesis, characterization and antibacterial activity of illite/ TiO_2 nanocomposites were carried out by using solvothermal method. Synthesized nanocomposite was characterized by using XRD, SEM, TEM and UV-vis absorption spectroscopy. The XRD pattern reveals the course of composites formation and illite/ TiO_2 Nanocomposites containing anatase phase of TiO_2 were found. The spherical morphology of illite/TiO_2 nanocomposites was observed in SEM analysis. The TEM results indicate that the nanocomposite is spherical in shape. The nanocomposites size can be calculated from Debye-scherrer formula. The antibacterial assay were also done on *staphylococcus aureus, E.col*i and *Bacillus*.

Keywords: Illite, TiO_2, Nanocomposites, XRD, SEM, TEM.

Introduction

Nanoscale materials are ranging from 1 to 100 nm, as defined in the chemistry context, which have contributed to the development of Nanoscience and Nanotechnology at the exponential rate in recent years. Nanomaterials often have a significant degree of difference in physico-chemical and biological properties to their macroscale counter-part in spite of the similar chemical composition they possess [1-2]. Nanocomposites are composites in which at least one of the phases shows dimensions in the nanometer (1nm=10^9m) [3]. Nanocomposites materials have emerged as suitable alternatives to overcome limitations of microcomposites and monolithic, while posing preparation challenges related to the control of elemental composition and stoichiometry in the nanoclusterphase, they are reported to be the materials of 21st century in the view of possessing design uniqueness and property combinations that are not found in conventional composites. The general understanding of these properties is yet to be reached [4], even though the first inference on them was reported as early as 1992 [5]. Titanium dioxide is known for its chemical stability, photocatalytic characteristics, durability and antimicrobial activity, which could be attributed to its crystal structure [6]. Anatase has stronger antimicrobial and photocatalytic activity than rutile [7-9]. Clays, such as Montmorillonite, Rectorite, and Kaolinite have attracted much attention in recent years for their applicability, in waste water treatment. These natural materials possess layer structures, large surface areas, high cation exchange capacities and can adsorb organic substances either on their external surfaces or within their interlaminar spaces by interaction or substitution. It has been reported that dispersion TiO_2 particles into layered clays can improve catalyst performance because such composites structures can stabilize TiO_2 crystals for access by various molecules [10, 11]. Enhancement in material research significantly supports application of titanium dioxide in the field of photocatalytic materials for potential applications in civil engineering. Titanium dioxide is widely used to modification building materials because it has been used as a white pigment for many years. The pure applications of titanium dioxide based on photocatalysis in the field of building materials are self-cleaning and self-disinfecting. The advantage of using solar light and rainwater as driving force has opened as new domain for environmentally friendly building materials [12]. However, in recent years TiO_2 has been used widely for the preparation of different types of nanomaterials, including nanoparticles, nanorods, nanowires nanotubes and mesoporous and nanoporous TiO_2containing materials [13]. Regardless of scale, TiO_2 maintains its photocatalytic abilities, and in addition,, nanoscale TiO_2 has a surface reactivity that fosters its interactions with biological molecules, such as phosphorylated proteins and peptides [14], as well as some nonspecific binding with DNA [15]. In this work, we have taken the illite clay, which is a non-expanding clay crystalline mineral. Illite is a secondary mineral precipitate phyllosilicate or layered alumino-silicate. Its structure is 2:1 clay of silica tetrahedron – alumina octahedron – silica tetrahedron layers. In the present investigation, we report the synthesis and characterisation of illite/TiO_2 nanocomposites by the solvothermal method. The TiO_2 nanocomposites are characterised by X-Ray diffraction, scanning electron microscopy (SEM) transmission electron microscopy (TEM) and UV-vis absorption spectroscopy. The antibacterial assays of nanocomposites were also studied.

2. Material and Methods

Clay-water dispersion (1% w/w) was stirred for 2 hours. An aliquot of TiO_2 sol was added to the dispersion, to obtain a final TiO_2 content of 70% w/w. The slurry was stirred for 24 hours. The resulting dispersion was centrifuged at 3,800 rpm for 10 minutes. The solid phase was washed with nanopure water followed by triplicate centrifugation. The resulting clay-TiO_2 composite was dispersed in 1:1 water: ethanol solution, prior to hydrothermal treatment in an autoclave at 180 °C for 5 hours. The product was centrifuged once again at 3,800 for 15 minutes, and oven-dried at 60 °C for 3 hours [16].

3. Instrumental Characterizations

For determination of crystallite size, Scherrer analysis of XRD is commonly used. XRD measurements are performed using a Philips diffractometer of 'X'pert company with mono chromatized Cu Kα (λ=1.54060Å) radiation. A double beam UV-Vis (Jascow-500) spectrophotometer with 1 mm optical path length quart cells was used for all absorbance measurement in the range of 200 nm – 800 nm. SEM image was obtained with HITACHI-S-3400H model. The transmission electron microscopy (TEM) analysis performed using a Philips CM-200 electron microscope with operating voltages 200 KV, resolution 2.4 Å.

4. Agar well diffusion assay

The antimicrobial activities of the clay/TiO_2 nanocomposite species were checked by using agar well diffusion method [17]. The antimicrobial activity against *Escherichia coli, Staphylococcus aureus* and *Bacillus*. The nanocomposites were dissolved in the solvents like ethanol, methanol respectively. Mueller Hinton agar was prepared, sterilized and poured into petri plates. The test organisms were spread on these plates on which wells were made using a sterile cork borer. To each well, 30 µl of each nanocomposites are added and plates were incubated at 37 ^{0}C for 24 hrs. After incubation, the results were recorded by measuring the diameter of zone of inhibition surrounding the well.

5. Results and Discussion

5.1. XRD Analysis

The XRD Pattern of the nanocomposites obtained by solvothermal method is shown in Fig (1). The spectra showed crystalline nature with 2θ peaks lying at 2θ=25.25° (101), 2θ=37.8° (004), 2θ=47.9° (200), 2θ =53.59° (105) and 2θ =62.36° (204). The preferred orientation corresponding to the plane (101) is observed. All the peaks in the XRD pattern have been indexed as anatase phases of TiO_2 and the diffraction data were in good agreement with JCPDS files 21-1272 [18]. Crystallite size was obtained by Debye – Scherrer formula. (Equation 1).

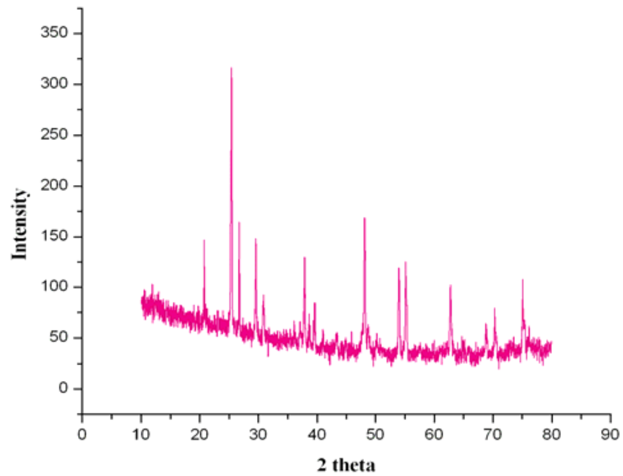

Fig 1: XRD pattern of Illite/TiO_2 nanocomposites

$D=K\lambda/(\beta \cos \theta)$ ---------- (1)

Where D is the crystal size ; λ is the wavelength of the X-ray radiation (λ =0.15406nm) for CuKλ.K is usually taken as 0.89; and β is the line width at half-maximum height [19]. The crystallite size was calculated and value 81 nm was observed.

5.2. SEM Analysis

Scanning electron microscopy (SEM) was used to investigate the surface morphology of illite/TiO_2 nanocomposites. The SEM image of the nanocomposites is shown in figure. 2. has spherical shape and well dispersed. The SEM image of the studied sample proved matrix consisting of micro sized illite particle having layered structure with sub – micron particle of TiO_2 attached on the surface of the clay matrix.

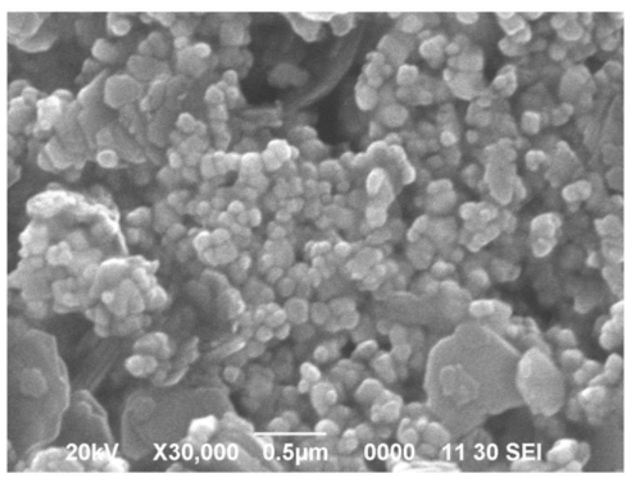

Fig 2: SEM image of Illite/TiO_2 nanocomposites

5.3. TEM Analysis

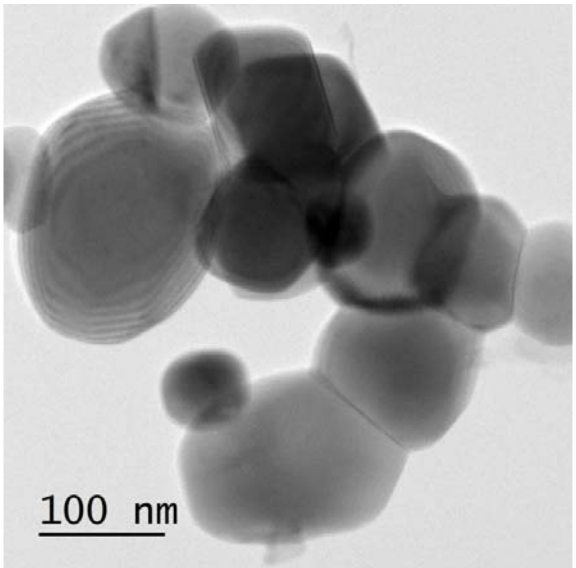

Fig 3: TEM image of Illite/TiO_2 nanocomposites

The morphology and structure of the as prepared product were further investigated with TEM analysis. TEM image of illite/TiO_2 nanocomposites is shown in figure.3. Clear spherical and non – homogenous structures [20] can be seen in figure.3. The average size of the nanocomposites was estimated from the TEM image is about 82 nm, which is in good agreement with the calculated value.

5.4. UV-vis absorption spectroscopy

The absorption edge shifted towards lower wavelength for illite/TiO_2 nanocomposites. This clearly indicated a increase in the band gap energy of illite/TiO_2 nanocomposites. The band gap energy can be estimated from the following equation [21].

$$\alpha = \frac{k(h\nu - E_g)^{n/2}}{h\nu} \text{ -------- (2)}$$

Where, $h\nu$ is the photon energy. A and n are constants. Where n is 2 for direct energy gap and ½ for an indirect energy gap.

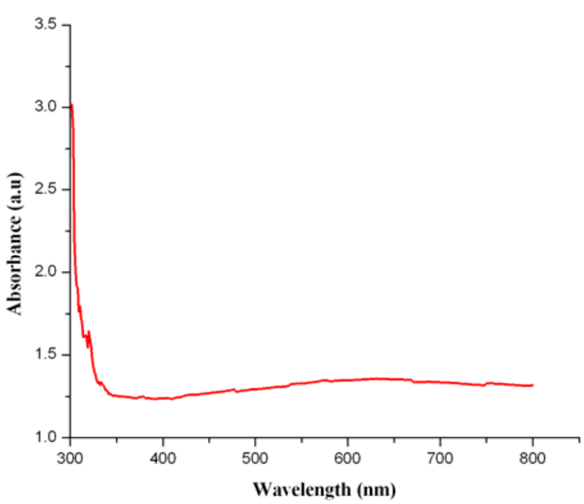

Fig 4: UV-vis absorption spectrum of Illite/TiO_2 nanocomposites

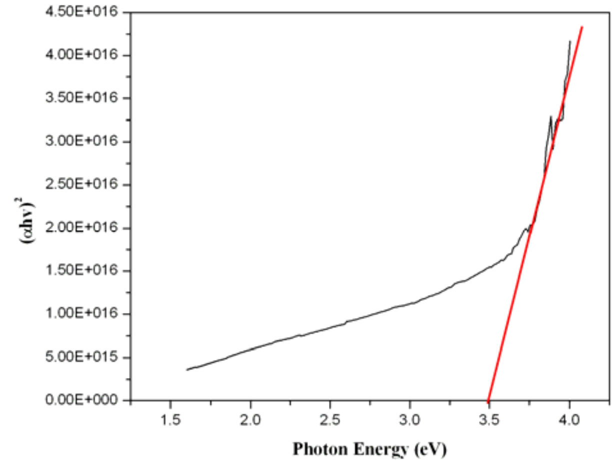

Fig 5: Band gap energy of Illite/TiO_2 nanocomposites

Plots of $(\alpha h\nu)^2$ versus photon energy (hv) for illite/TiO_2 nanocomposites is shown in figure.5. The band gap energy is estimated from the intercept of the tangents of the plot is 3.5 eV. This showed that the blue shifted when compared with the bulk TiO_2 (3.2 eV). The blue shift might be caused by nanosize effect and structural defect of nanomaterials.

5.5. Antibacterial Activity

Fig 6: Antibacterial activity of Illite/TiO_2 nanocomposites

The minimum inhibitory concentration of nanocomposite was tested and reported by agar well diffusion method. The fresh bacterial strains *Escherichia coli, Staphylococcus aureus* and *Bacillus* were cultured in Mueller Hinton Broth. After incubation the results were analysed the nanocomposites inhibit the growth of *E.coli* sp (15mm) *S.aureus* sp (18mm) and *Bacillus sp* (13mm) with respect to the control ampicillin. Hence, the obtained nanocomposites have very good antibacterial activity on the given pathogens, viz *E.coli, S.aureus and Bacillus.*

6. Conclusion

In conclusion, the illite/TiO_2 nanocompoites have been successfully synthesized by using solvothermal method. The synthesized nanocomposites were characterised by using various analytical tools like XRD, SEM, TEM and UV –Vis absorption spectrum. The XRD result reveals that the presence of anatase phase of TiO_2.The synthesized nanocomposites size and morphology of the sample were characterised by using TEM and SEM. SEM proved the spherical shape and well dispersed on the clay surface. TEM showed that the particles are clear spherical and non-homogenous structures. The average size of the nanocomposites was estimated from TEM image is about 82 nm. The band gap energy of this nanocomposite is 3.5 eV, which is larger than the value of 3.2 eV for bulk TiO_2. Based on the antibacterial studies, the illite/TiO_2 nanocomposites show good accountability on the degradation of growth of pathogens.

7. References

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