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Synthesis and Characterization of Fabric coated with Nano Titanium oxide prepared using sol-gel, hydrothermal & Sonochemical technique

Jositta Sherine[1]*, Gnaneshwar.P.V[1], Pandiyarasan Veluswamy[2]

[1]Department of Physics and Nanotechnology, SRM University, Katankulathur, Chennai-603203, India
[2]Research Institute of Electronics, ShizuokaUniversity, Japan.

Abstract : Textiles is the most widely used and purchased materials all over the world for many purposes, every year there is a new improvement is made in the textile industries. Titanium dioxide (Tio_2), it is a semiconductor material having various number of applications and its been extensively studied in the past few decades .Nano titanium oxide is synthesised using many number of synthesis techniques like hydrothermal, sol-gel & sono chemical. Nano titanium dioxide ware prepared by sol-gel, sonochemical–hydrothermal process from a precursor of titanium isopropoxide in the presence of polyvinyl alcohol aqueous solution. Sonication is conducted using bath-sonicator until completely precipitated product was reached. As the intermediate precipitate is formed transfer it into Teflon-lined stainless steel autoclave for hydrothermal process with 10M NaOH aqueous solution and heated under different temperature 80-120ºC. The product is calcinated and coated on a cotton fabric and the synthesized products were characterized and other properties where studied. The structural morphology ,chemical properties, optical properties and thermal properties of a cotton fabric before and after the coating of nano titanium oxide has been studied using various techniques like scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier transform infrared spectro scopy (FTIR), X–ray photoelectron spectroscopy (XPS).

Introduction

In recent years, Nano structured materials have received much attention to their superior properties which differ from those of bulk material. Titanium dioxide(Tio_2) also known as Titania is a versatile oxide semiconductor material accompanying its exceptional optical band gap of 3.2ev[1]. Titanium dioxide (Tio_2) nanoparticles have recently attracted increasing attention because of its unique characteristics. Tio_2 nano particles are studied extensively in the past in the vast range of scientific and industrial fields due to its good photo-catalytic activity[2],high & long term chemical stability, low cost, non –toxicity, antifogging[3], self-cleaning devices[4], strong ultraviolet absorption and high energy conversion efficiency. Tio_2nano particles are widely studied in various research fields. For example, Tio_2 nano particles beads have been known as effective absorbents specific for phosphorylated peptides, and Nano crystalline Tio_2 electrodes have been applied in the research of solar cells. The ability of Tio_2 nano particles in photocatalytic reduction is applied in metal reduction

to remove heavy metal in waste water. Furthermore, titania material also have anti-microbial activities. There are many studies made that nano titanium dioxide materials can inhibit the cell growth of microorganisms via photochemical reaction. High photo-catalytic property of Tio_2 nano particles /coating has been widely used for water and air purification ,sterilization/disinfection ,photo-induced water splitting, organic compound degradation, dye-sensitized solar cell(DSSC),super hydrophilic effect and self-cleaning effects, Imparted by nano-Tio_2 to textile substrates. There are many different methods and techniques have been developed for preparation of Tio_2 nanomaterials such as co-precipitation process[5], sol-gel process[6], electrospinning[7], sonication, hydrothermal technique[8], and sonochemical process[9]. The hydrothermal technology has been used to fabricate functional composite materials, such as mesoporous materials, photocatalysts, nanoparticles, inorganic powders, and gas sensors. Also, there has been a great interest in controlling the structural properties of materials and finding enhanced properties of materials by employing a variety of preparative methods.

Current scientific research involving antimicrobial coatings for textiles is gaining attention in the healthcare industry due to the increased risk of healthcare associated infections (HAIs) (Gouveia, 2010). Reducing the number of pathogenic microorganisms in a patient's environment is now a high priority in all healthcare institutions. Antimicrobial coated surfaces can potentially reduce these troublesome infections. In recent years, several studies have been published on textile substrates treated with Tio_2 nanoparticles[10]. Studies on the UV-blocking property of Tio_2 treated cotton fabrics[11]. The treated cotton fabrics possessed a high UPF rating of 50+ and provided excellent protection according to the Australian/New Zealand Standard. Moreover, Tio_2 films on cotton fabrics showed excellent durability up to 55 home launderings. In a work reported as anatase Tio_2 nanocrystallites were successfully grown on cotton fabrics through a hydrothermal process for 3h after the fabric treatment using ethanol-based sol-gel titania[12,13]. Later, Reported the preparation of self-cleaning wool–polyamide, polyester and cotton textiles treated with Tio_2[14,15,16,17,18]. In their work, the Tio_2 colloidal solution was prepared using an isopropanol-based sol-gel process[19] or using an isopropanol-based sol-gel process at followed by hydrothermal treatment at 100˚C and 220˚C for 16h[20], and then applying the colloidal solution to textiles. However, it was amorphous[22] Tio_2 or rutile[21] Tio_2 that was attained on the textiles, which were less photoactive than anatase Tio_2[20]. In a typical sol-gel process[19], a colloidal suspension or a sol is formed due to the hydrolysis and polymerization reactions of the precursors, which on complete polymerization and loss of solvent leads to the transition from the liquid sol into a solid gel phase .This can be used to produce thin coatings/films on textile substrate using dip coating . The wet gel film can be converted into anatase nano crystals with further drying and hydrothermal treatment at elevated temperature. Anatase Tio_2 is reported to have the highest activity among the crystalline phases of Tio_2. Several techniques such as chemical precipitation, micro emulsion[21], hydrothermal crystallization[22] and chemical vapour deposition[23] have been reported for the activity of the anatase Tio_2, however these techniques are complex and required very high temperature for the formation of this structures. Sonochemical-hydrothermal synthesis is a prospective method to obtain nanostructures. Sonochemical hydrothermal technique is one of effective techniques for synthesizing a great variety of materials. where polymorphism, particle size, crystallinity, morphology, and processing time could be very well controlled as required, comparing to any other techniques. More recently, there have been related works employing the potential of sonochemical-hydrothermal process to synthesize nanostructure of Tio_2. The sonochemical synthesis of nanocrystalline Tio_2 by hydrolysis of titanium alkoxides is reported[24]. A simple hydrothermal process for preparing Tio_2 nanomaterials using concentrated hydrochloric acid[25]. Prepared Tio_2 nanoparticles from titanium (IV) isopropoxide diluted in isopropyl alcohol[26,27]. In this contribution we describes an effective method to produce Tio_2 coated cotton fabrics at ambient temperature from powered Tio_2 prepared from various methods like sol-gel, hydrothermal and sonication process. And the Tio_2 coated samples are further studied for anti-bacterial, anti-microbial activities, photo-catalytic and self-cleaning properties for future applications.

Experimental part

Materials

The following chemicals were of analytical grade and purchased from Merck specialities, Mumbai. Titanium tetraisopropoxide (97%) was used as a precursor for the synthesis of Tio_2 nano particles .deionised water was used for hydrolysis of Ti(O-iPr)4 and for the preparation of all sols and solutions. Sodium hydroxide anhydrous pellets (NaOH) and polyvinyl alcohol (PVA) which are typically used as general capping agent for promoting the anisotropic growth of nanoparticles[27].

Synthesis of Tio_2 nano particles

10 mL solution of titanium (IV) isopropoxide in 100 mL deionized water was used as precursor of Tio_2 nano particle. 1gm Sodium hydroxide anhydrous pellets (NaOH) and 0.5gm polyvinyl alcohol (PVA) is taken as precursor sample (Stock sample 2). This solution and sample (Stock 1&2) is prepared 4 times for different sets of preparation process.

Synthesis using Sonication

Stock solution 1 is taken in 100ml beaker, started stirring for 15 minutes at a rate of 2000 rpm. Stock 2 is taken and added while stirring the stock 1 solution. Stirring is continued for 10 minutes after the stock 2 is added. After the stirring is completed change the solution in a conical flask and place in a liquid bath for sonication at 50Hz for 30 minutes .After sonication is completed wash it using acetone solution and deionised water. Subject the sample for centrifugation at 3000 rpm for 5minutes and repeat the centrifugation process till the complete sample is done .Dry the sample at 80˚C for 12 hours .

Synthesis using Hydrothermal

Stock solution 1 is taken in 100 ml beaker and stirred for 20 minutes then the sample is transferred into a Teflon coated autoclave .The autoclave is subjected in furnace at 120˚c for 8 hours .The furnace rate should be set at 15˚C per minute .The sample is taken after the furnace is cooled down to the room temperature .Then the sample is transferred in to beaker and washed with acetone and deionised water. Subject the sample for centrifugation at 5000 rpm for 5 minutes and repeat the centrifugation process till the complete sample is done .Dry the sample at 80˚C for 12 hours .

Synthesis using sonochemical

Sonochemical process is a combination of hydrothermal and sonication process. Stock solution 1 and 2 are made stirring for 10 mintuts and 15 minutes separately. Then the samples are subjected to sonication for 20 minutes respectively .Then these two samples are transferred into autoclaves and place in furnace at 100˚C and 120˚C for 8 hours. Then samples are taken out after the temperature reached room temperature. Then the sample is transferred in to beaker and washed with acetone and deionised water. Subject the sample for centrifugation at 5000 rpm for 10 minutes and repeat the centrifugation process till the complete sample is done. Dry the sample at 80˚C for 12hours .

Result and Discussion

In order to determine the uv absorptivity of peaks uv-spectrometric analysis is done. To confirm the functional groups specific for Tio_2 FTIR is taken and To confirm the uniform coating on cotton fabric and tostudy the structure of nanoparticle formation SEM images are taken.

UV-visible Spectrum

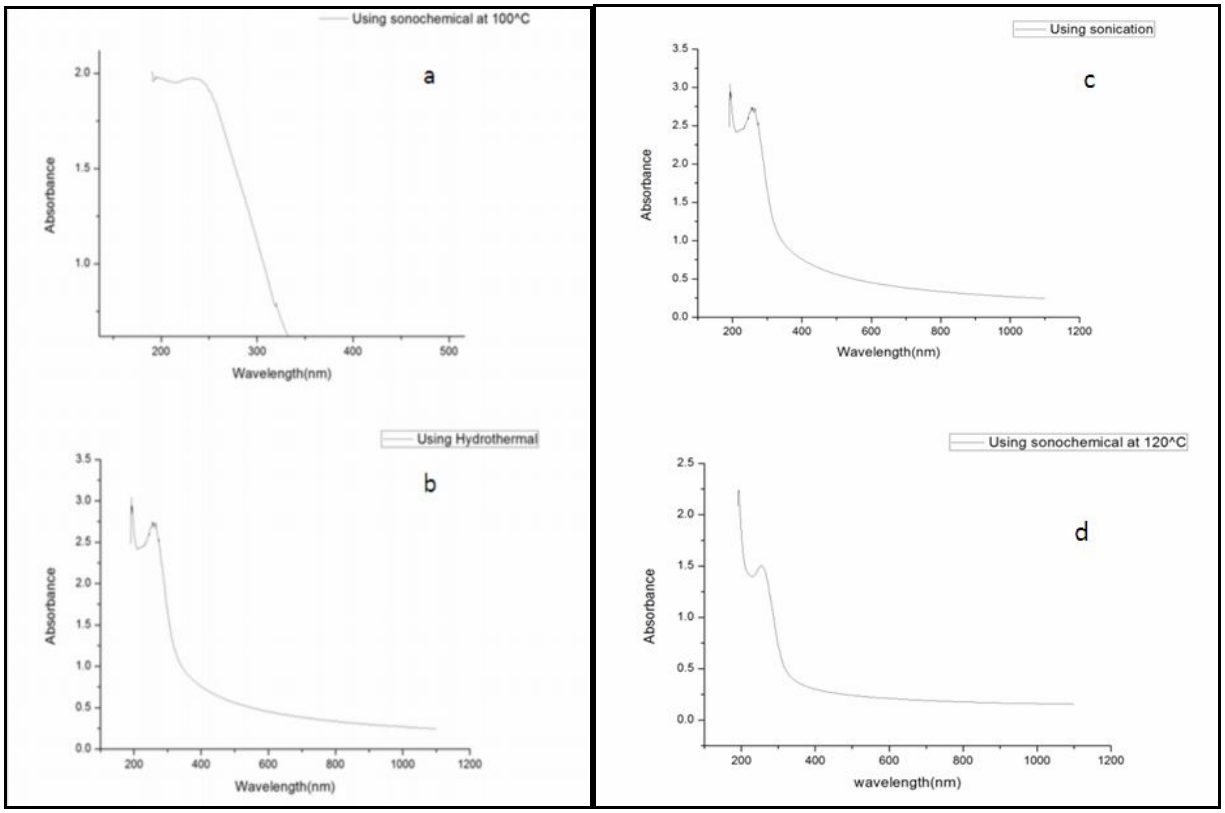

Figure 1: UV analysis for Tio_2 synthesized
(a),(b) are the samples prepared by sonication and hydrothermal respectively.
(c),(d) are the samples prepared by sonochemical at 100˚c and 120˚c respectively.

The Tio_2 samples by varying parameter samples were prepared as per procedure discussed in experimental part and UV-Spectrometer analysis is done to confirm the formation of Tio_2. The Fig:1 (a),(b),(c),(d) shows the significant peak near 230 nm for all four samples of various concentration.This confirms the presence of Tio_2.

FTIR

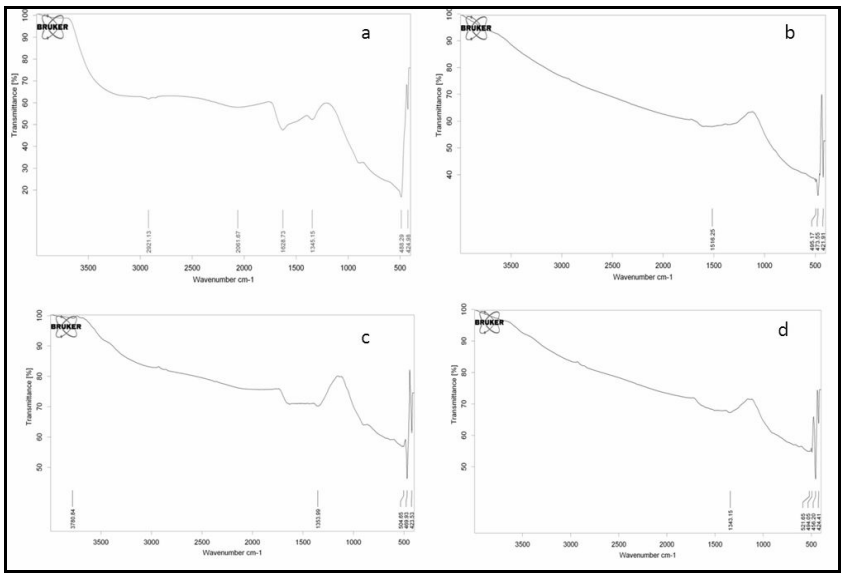

Figure 2: FTIR analysis for Tio_2 synthesized
(a),(b) are the samples prepared by sonication and hydrothermal respectively.
(c),(d) are the samples prepared by sonochemical at 100˚c and 120˚c respectively.

The presence of some functional group as revealed by IR spectral is shown in Fig .2 The FTIR Spectral analyses of Titanium dioxide nanoparticles show certain common absorption band at 1350 cm-1 indicate it is pure Tio_2 sample . The peak between 800 and 450 cm-1 was assigned to the Ti-O stretching bands. In sample one peak at 1628 cm-1 shows C=N compounds shows the presence of PVP.

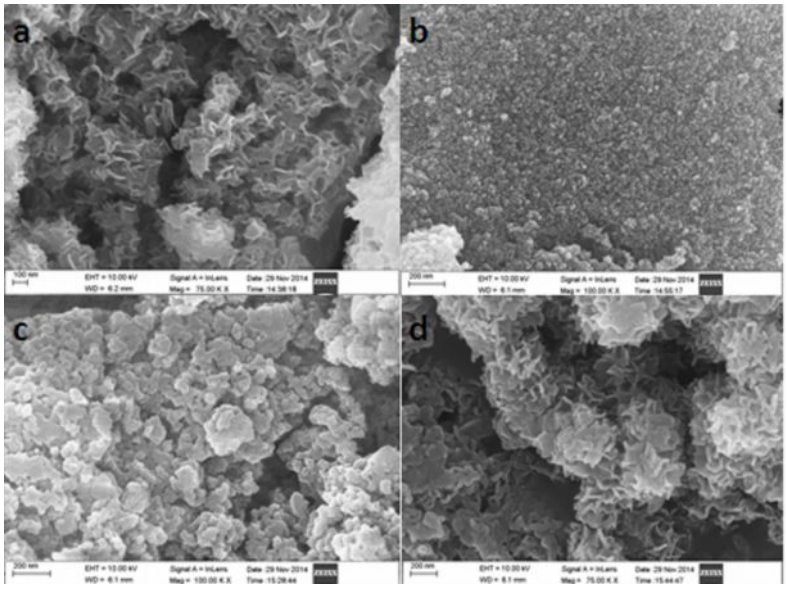

Figure 3: SEM images for Tio_2 :
(a)Synthesised using sonication method .
(b)Synthesised using hydrothermal method .
(c)Synthesised using sonochemical method at 100˚c .
(d)Synthesised using sonochemical method at 120^{o}C.

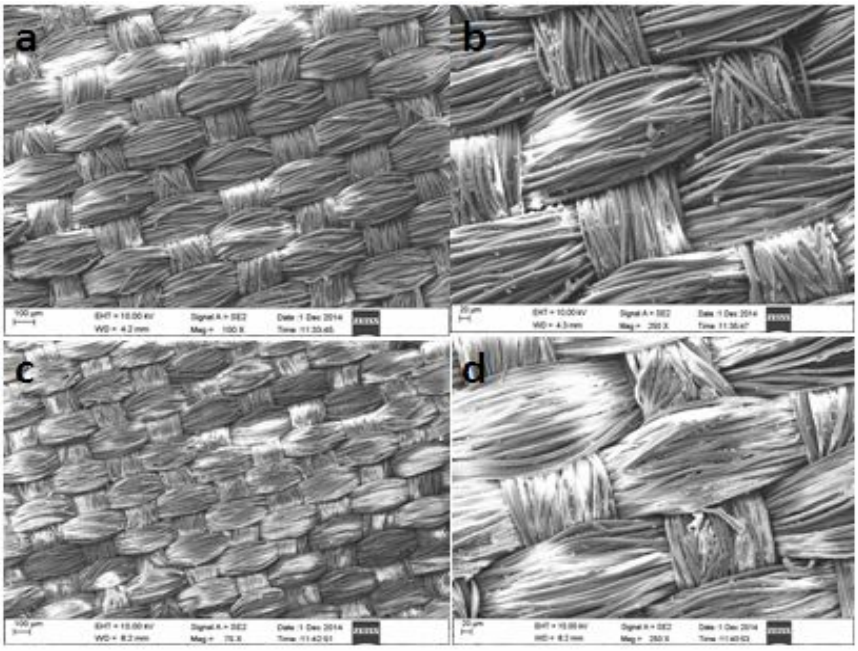

Figure 4: SEM Analysis for Tio_2 coated fabric
(a)Synthesised by sonication coated on cotton fabric at a resolution of 100µm.
(b)Synthesised by hydrothermal coated on cotton fabric at a resolution of 20µm
(c)Synthesised by sonochemical (100˚c)coated on cotton fabric at a resolution of 20µm
(d)Synthesised by sonochemical (120˚c)coated on cotton fabric at a resolution of 100µm .

SEM images of fig:3,(a) resembles swamped strings ,Fig : (3),b shows sphere shaped particles,Fig:3,(c) shows fresh cotton,Fig:3(d) resembles sea urchin,Fig: (4) a,b,c,d shows the Tio_2 coated cotton fabric.SEM images resembles that all the samples prepared by various methods gives different structures on individual ,

after coating to cotton fabric the SEM results were similar in all the samples despite of their individuals structures and coating was found to be even on the cotton fabric.

Conclusion

We described an effective method to produce Tio_2 and its coating on cotton fabric at ambient temperature. Powered Tio_2 prepared from synthesis methods like sol-gel, hydrothermal and sonication process. These samples are primarily analysed using FTIR, UV/VIS Spectroscopy and Scanning electron microscopy And the Tio_2 coated samples are further studied for anti-bacterial, anti-microbial activities, photo-catalytic and self-cleaning properties for future applications.

References

1. Isley, S.L., Jordan, D.S. and Penn, R.L., Titanium dioxide nanoparticles : Impact of increasing ionic strength during synthesis, reflux, and hydrothermal aging, Mater. Res. Bull., 2009, 44, 119-125.
2. Hussain, M., Ceccarelli, R., Marchisio,D.L., Fino, D., Russo, N. and Geobaldo, F. Synthesis, characterization and photocatalytic application of novel Tio_2 Nanoparticles, Chem. Eng. J., 2010,157: 45-51.
3. Law, W.S., Lam, S.W., Gan, W.Y., Scott, J. and Amal, R., Effect of film thickness and agglomerate size on the superwetting and fog-free characteristics of Tio_2films, Thin Solid Films, 2009, 517, 5425-5430.
4. Jiang, X., Tian, X., Gu, J., Huang, D. and Yang, Y., Cotton fabric coated with nano Tio_2-acrylate copolymer for photocatalytic self-cleaning by in-situ suspension polymerization, Appl.Surf. Sci., 2011, 257, 8451-8456.
5. Zanella, R., Giorgio, S., Shin, C.H., Henry, C.R. and Louis, C., Characterization and reactivity in CO oxidation of gold nanoparticles supported on Tio_2 prepared by deposition-precipitation with NaOH and urea, J. Catal., 2004, 222, 357-367.
6. Prasad, K., Pinjari, D.V., Pandit, A.B. and Mhaske, S.T., Phase transformation of nanostructured titanium dioxide from anatase-to-rutile via combined ultrasound assisted sol–gel technique, Ultrason. Sonochem., 2010,17, 409-415.
7. Wang, C., Tong, Y., Sun, Z., Xin, Y., Yan, E. and Huang, Z., Preparation of one- dimensional TiO2 nanoparticles within polymer fiber matrices by electrospinning, Mater.Lett., 2010,61, 5125-5128.
8. Byrappa, K. and Adschiri, T. Hydrothermal technology for nanotechnology, Prog.Cryst.Growth Charact, Mater., 2007, 53, 117-166.
9. Guo, S., Wu, Z., Wang, H. and Dong, F., Synthesis of mesoporous Tio_2 nanorods via a mild template-free sonochemical route and their photocatalytic performances.Catal.Commun, 2009,10, 1766-1770.
10. Bozzi, A., Yuranova, T., Guasaquillo, I., Laub, D., and Kiwi, J., Self-cleaning of Modified Cotton Textiles by Tio_2 at Low Temperatures Under Daylight Irradiation, J. Photochem. Photo- bio. A. Chem., 2005,174, 156–164.
11. Xin, J.H., Daoud, W.A. and Kong, Y.Y., A new approach to UV-blocking treatment for cotton fabrics, Textile Res J . 2004 74, 97–100.
12. Peblow, M., Clothes launder own fabric. Nature News, 2004, 429, 620.
13. Daoud, W.A. and Xin, J.H., Nucleation and growth of anatase crystallites on cotton fabrics at low temperatures, J. Am Ceram Soc. 2004, 87, 953–955.
14. Bozzi, A., Yuranova, T., Guasaquillo, I., Laub, D. and Kiwi, J., Self-cleaning of modified cotton textiles by Tio_2 at low temperatures under daylight irradiation, J PhotochemPhotobiol A: Chem, 2005, 174, 156–164.
15. Bozzi, A., Yuranova, T. and Kiwi, J., Self-cleaning of wool- polyamide and polyester textiles by Tio_2-rutile modification under daylight irradiation at ambient temperature. J Photochem Photobiol A: Chem, 2005,172, 27–34.
16. Yuranova, T., Mosteo, R., Bandata, J., Laub, D. and Kiwi, J., Self-cleaning cotton textiles surfaces modified by photo- active Sio_2/ Tio_2 coating, J Mol Catal A: Chem., 2006,244:, 160–167.
17. Meilert, K.T., Laub, D. and Kiwi, J., Photocatalytic self- cleaning of modified cotton textiles by Tio_2 clusters attached by chemical spacers, J MolCatal A: Chem., 2005,237, 101–108.
18. Fujishima, A., Hashimoto, K. and Watanabe, T., Tio_2 Photocatalysis: Fundamentals and Applications. Tokyo: BKC1999, 32,364.
19. Macwan, D.P., Dave, P.N., Chaturvedi, S., A review on nano- Tio_2 sol–gel type syntheses and its applications, Journal of Materials Science, 2011, 46, 3669-3686.

20. Prasad, K., Pinjari, D.V., Pandit, A.B. and Mhaske, S.T., Phase transformation of nanostructured titanium dioxide from anatase-to-rutile via combined ultrasound assisted sol–gel technique. Ultrason. Sonochem, 2010,17, 409-415.
21. DeBenedetti, B., Vallauri, D., Deorsola, F. A. and Martinez G. M., Synthesis of Tio_2 nanospheres through microemulsion reactive precipitation, Journal of Electroceramics , 2006 17, 37-40.
22. Yu, J. G., Yu, H. G., Cheng, B., Zhou, M. H. and Zhao, X. J., Enhanced photocatalytic activity of Tio_2 powder (P25) by hydrothermal treatment, Journal of Molecular Catalysis A: Chemical., 2006, 253, 112-118.
23. Byun, D., Jin, Y., Kim, B., Lee, J. K., Park, D., Photocatalytic Tio_2 deposition by chemical vapor deposition, Journal of Hazardous Materials, 2000,73, 199-206.
24. Guo, W., Lin, Z., Wang, X. and Song, G., Sonochemical synthesis of nanocrystalline Tio_2 by hydrolysis of titanium alkoxides microelectron, Eng., 2003, 66, 95-101.
25. Nguyen Phan, T.D., Pham, H.D., Cuong, T.V., Kim, E.J., Kim, S.W. and Shin, E.W. A simple hydrothermal preparation of Tio_2 nanomaterials using concentrated hydrochloric acid, J. Cryst. Growth., 2009, 312, 9-85.
26. Viana, M.M., Soares, V.F. and Mohallem, N.D.S., Synthesis and characterizationof TiO2 nanoparticles, Ceram. Int., 2010,36, 2047-2053.
27. Ranjbar-Karimi, R., Bazmandegan-Shamil, A., Aslani, A. and Kaviani, K., Sonochemical synthesis, characterization and thermal and optical analysis of CuO nanoparticales, Physica B., 2010, 405, 3096-3100.

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