Comparison of Tio2 Nano Particles Produced By Sol Gel Synthesis and Biological Synthesis

Arun Raju [1], P.Prathap [2], Eldhose Mathew [3]
[1, 2, 3]Department of Mechanical Engineering
[1]Sri Guru Institute Of Technology, Saravanampatti, Coimbatore-641110, Tamil Nadu, India
[2, 3] Kathir College Of Engineering, Neelambur, Coimbatore-641062, Tamil Nadu, India

Abstract- *Titanium dioxide naturally occurs in the form of an oxide of titanium whose chemical formula is given as TiO2. TiO2 pigments are used in paints and coatings, plastics, paper, building materials, cosmetics, pharmaceuticals, foods and many other commercial products.TiO2 when ground to fine particles can be used as pigments for paints, it also acts as a strong antimicrobial agent so it has a wide application in the antimicrobial coatings. TiO2 nano coating was carried out by using sol gel method .In sol gel synthesis the precursor used was Titanuim isobutoxide, Cetyl Trimethyl Ammonium Bromide (CTAB) was used as the surfactant for improving formation synthesis of TiO2 nano particles. Deionised distilled water was used as the reducing agent to reduce Titanium iso butoxide into TiO2 nano particles.TiO2 nano particles were biologically synthesized by drop by drop addition of leaf extract into Titanium tetrachloride solution. Characterization of nano particles were done by UV visible spectroscopy, Particle sizer, and Scanning electron microscopy were carried out and inference was derived from the results. The TiO2 particles showed good resistance towards staphylococcus bacteria when tested in laminar flow chamber.*

Keywords- Biological synthesis,TiO2, UV visible spectroscopy, antimicrobial, sol-gel synthesis.

I. INTRODUCTION

Titania is a very widely researched material due its stabile chemical structure, biocompatibility, physical, optical, and electrical properties. Photocatalytic properties of titania have been widely utilized in various environmental applications to extricate contaminants from both water as well as air. The titania nanoparticles have the characteristic properties such as increased surface area, size and morphology of the same when compared with the bulk titania particles are different and improved when compared to the bulk counterparts. Metal nano particles are extensively used because of their distinct physical properties, chemical reactivity. The potential fields of applications are research areas involving antibacterial, antiviral, diagnostics, anticancer and targeted drug delivery.

Metal nanoparticles are usually synthesized using various chemical method such as chemical reduction, solvo-thermal reduction, electrochemical techniques (Krishna, and Goia Dan, 2009 ; Saxena et al. 2010) and photochemical reaction in reverse micelles (Taleb et al. 1997). Among them, chemical reduction is the most frequently applied method. Previous studies showed that the use of a chemical reducing agent resulted in generation of larger particles and consume more energy. It was also reported that more side products were formed by chemical approaches which are not eco-friendly. Moreover, the chemically synthesized Nanoparticles were reported to show less stability and more agglomeration (Mukherjee et al. 2001). Hence there is a need to develop an eco-friendly protocol that could produce stable and dispersible nanoparticles of controllable size by consuming less energy.

Alternate methods are also adopted for the synthesis of metal and metal oxide nano particles which utilize bacteria, fungi and plant extracts as reducing agents. These biological methods, so called green synthesis methods, are not only benign and environment friendly but also cost effective, rapid, less laborious, easily scalable to large scale and more efficient than conventional methods. It also benefits us by being compatible for various biomedical and pharmaceutical applications as they do not use toxic chemicals for the synthesis.

Titanium dioxide (TiO2) nanoparticles have wide environmental applications such as air purification and waste water treatment. TiO2 nanoparticles possess potential oxidation strength, high photo stability and non-toxicity, are used in dye sensitized solar cells (Li et al. 2004; Salim et al. 2000; Ito et al. 1999). TiO2 nanoparticles are also used in industrial applications such as pigment, fillers, catalyst supports and photo catalyst due to optical, dielectric, antimicrobial, chemical stability and catalytic properties (Barbe et al. 1997; Carp et al., 2004; Ruiz et al. 2004).

Hibiscus rosa sinesis (commonly called shoe flower) is rich in polyphenolic photochemical like tannins and phenolic proteins, triterpenoids, 2,3-hexanediol, n-Hexadecanoic acid, 1,2-Benzenedicarboxylic acid and squalene (Mandade et al. 2011; Anusha et al. 2011). These compounds have good antimicrobial, anti oxidative and anti proliferative activity and therefore be used to treat cancer, especially lung cancer, cardiovascular disease, asthma and pulmonary function (Tsao et al. 2003; Feskanich et al. 2000; Kim et al. 2002). The Hibiscus flower extract may result in photochemical stabilized nanoparticles with better biomedical activity. Hence Hibiscus rosa sinesis extract was used and standardized a protocol to synthesize TiO2 nanoparticles

II. MATERIALS AND METHODS

A. Materials

All the chemicals and reagents used for the preparation of TiO2 nanoparticles were purchased from The Precision Scientific co. (CBE).

The flowers were collected from a village in Coimbatore district, Tamil nadu. The petals were seperated , washed with deionized & distiled water and then dried under shadow. The dried petals were identified as Hibiscus-rosa-sinensis L. of Malvaceae family.

III. METHODOLOGY

A. Preparation of Extract

For the biological synthesis of TiO2 nano particles 14g of air dried petals of Hibiscus rosa sinensis were taken in the beaker and extracted with 200ml water at 80□C for 4hrs. The extract was filtered using whatman filter paper and the filterate was used for the synthesis of nanoparticles.

B. Preparation of sol

For the sol gel dip coating procedure for TiO2 following procedure was adopted. 1M Titanium iso butoxide was dissolved in 90ml ethanol by stirring at 300 rpm. Cetyl Trimethyl Ammonium Bromide (CTAB) 0.9 g was added to the precursor solution as the surfactant. After 2 hrs of stirring distilled deionized water 620µL was added as the reducing agent. The solution was then heated at a temperature of 600C for a period of 1hr. The solution was then kept for ageing for 1 day. Thus the sol was obtained.

C. Preparation of TiO2 nanoparticles

For biological synthesis TiO2 nanoparticles were prepared as follows: To 0.1 M solution of Titanium tetrachloride in 50 ML of water, 50ml of flower extract was added to the solution in drop wise under continuous stirring. The pH of the solution was adjusted to 7 by continuous washing with water. The mixture was stirred for 3 hours and at a temperature of 800oC. After stirring, the nanoparticles obtained were separated by centrifuging at 7000 rpm for 15mins. The precipitate was washed repeatedly with water to remove the byproducts.

For sol gel synthesis TiO2 nanoparticles were prepared as follows:

1M Titanium iso butoxide was dissolved in 90ml ethanol by stirring at 300 rpm. Cetyl Trimethyl Ammonium Bromide (CTAB) 0.9 g was added to the precursor solution as the surfactant. After 2 hrs of stirring distilled deionised water 620µL was added as the reducing agent. The solution was then heated at a temperature of 600oC for a period of 1hr. The solution was then kept for ageing for 1 day. Thus the sol was obtained. A216 WCB steel was used as the substrate, which was dipped in the sol for a period of 15 mins. The retraction rate of the substrate was maintained to be 5mm/s. This process was repeated of 5 times on the same substrate. The coated substrate was then subjected to sintering at a temperature of 8000oC in the presence of argon gas for a time period of 5hours. The sintered specimen was then cleaned and subjected to testing.

D. Evaluation of Antibacterial Activity

The antibacterial activities of the both bio and chemically synthesized TiO2 nanoparticles were evaluated using the Staphylococcus aureus bacterial stain obtained from the Department of Microbiology, KMCH, Coimbatore. All cultures were biochemically tested for purity and were used to determine the inhibitory action of Metal oxide nano particles solutions.. Bacteria were grown overnight on Mueller Hinton agar plates (pH 7.3±0.2) and the activity was assessed by disc diffusion method. The plates were incubated at 37°C for 24 hours and the inhibition zone was measured and calculated. Beef infusion was used as a positive control for the bacterial growth. The experiments were carried out in triplicate. The results were recorded by measuring the zones of growth inhibition surrounding the disc. The incubated plates were examined for the interruption of growth over the inoculums. The size of the clear zone was measured to evaluate the inhibitory action of the Metal oxide nano particles solutions. The zone of inhibition was calculated by measuring the diameter of the inhibited growth around the wells.

IV. RESULTS AND DISCUSSION

A.UV spectrum analysis: Particles synthesized by biological method and particle of Sol Gel

The synthesized particles of TiO2 is been analyzed by using UV spectrum analysis and the result is shown as per the Fig. 1 and the TiO2 particle in the sol which is prepared for the dip coating process and the result is shown in the Fig.2

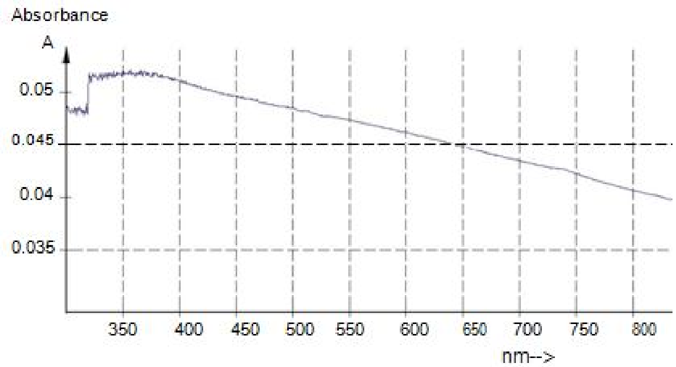

Fig.1. UV spectrum analysis for particle synthesized by biological synthesis method.

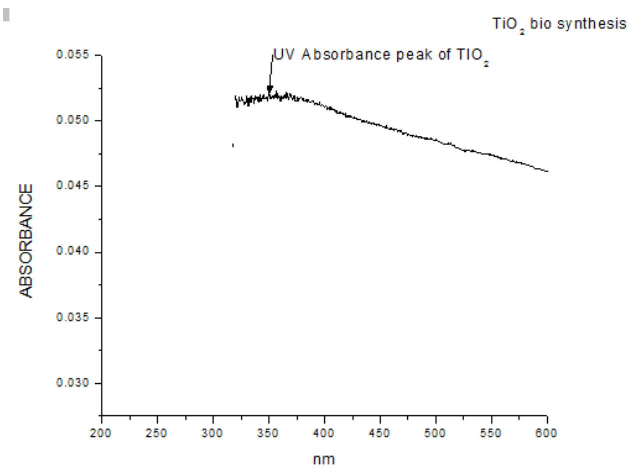

Fig.2.UV spectrum analysis for particle synthesized by sol gel synthesis method.

The UV spectrum analysis of the both biologically synthesized particles and the particles of sol prepared for the sol gel dip coating process was conducted and found that the absorbance peak is obtained in the rage of 300 nm to 400 nm from that the particles which is synthesized can be confirmed as the TiO2 nano particles.

B. Nanoparticles Characterization

The TiO2 nano film formed on the sample was studied by Scanning Electron Microscopy and the results are shown in Fig.3.The Scanning Electron Microscopy (SEM) analysis was conducted on the zirconium coated specimen with a magnification of 10K X and the microstructure of the coating is analyzed and found that the particles were found to be spherical with clearly defined edges and without accumulation.

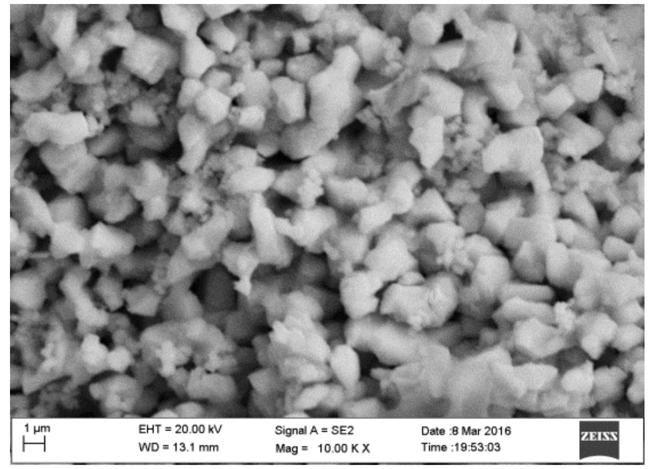

Fig.3. SEM micrograph of the TiO_2 coated on the A216 WCB steel by sol gel dips coating process.

C. Dynamic Light Scattering Method: Particles synthesized by biological method

MEASUREMENT RESULTS				
Measurement type		Particle		
Sample Nme		TiO2,biological synthesis		
Scattering Angle		173		
Temperature of the holder		25.0 o C		
T% before meas		3081		
Viscosity of the dipersion medium		0.895 mPa*s		
Form Of Didtribution		standard		
Representation of Result		Scattering Light Intensity		
Count rate		1775 kCPS		
Calculation Results				
Peak No	S.P Area Ratio	Mean	S.D	Mode
1	1	151.5 nm	50 nm	120.5 nm
2	...	...nm	...nm	...nm
3	...	...nm	...nm	...nm
Total	1	151.5nm	50 nm	120.5 nm

Fig.4 Dynamic Light Scattering analysis for particle synthesized by biological synthesis method and sol gel synthesis method

The nano particle of TiO2 which is been synthesized by biological method is been analyzed by dynamic light scattering method by using zeta size and the result of the analysis is in the Fig.3.

D. Dynamic Light Scattering Method: Particles synthesized by Sol gel method

The particle size of the biologically synthesized particle is 120.5 nm and we can confirm that the synthesized particle has size within the acceptable nano size

MEASUREMENT RESULTS				
Measurement type		Particle		
Sample Nme		TiO2, Sol		
Scattering Angle		90		
Temperature of the holder		24.9 o C		
T% before meas		11156		
Viscosity of the dipersion medium		0.895 mPa*s		
Form Of Didtribution		Standard		
Representation of Result		Scattering Light Intensity		
Count rate		1628 kCPS		
Calculation Results				
Peak No	S.P Area Ratio	Mean	S.D	Mode
1	1	102.9 nm	5.9 nm	101.6 nm
2	...	...nm	...nm	...nm
3	...	...nm	...nm	...nm
Total	1	102.9nm	5.9 nm	101.6 nm

Fig.5.Dynamic Light Scattering analysis for particle synthesized sol gel synthesis method

D. Particles of Sol Gel

The nano particles of TiO2 of the sol prepared for the sol gel dip coating process is been analyzed by Dynamic Light Scattering Method by using zeta size and the result of the analysis is in the Fig. 4.The particle size of the particles in the sol prepared for the dip coating process is 101.6 nm and we can confirm that the synthesized particle has size within the acceptable nano size.

V. CONCLUSION

In this paper we have prepared TiO2 nano particles by:

Using Hibiscus flower extract as capping agent and the nano particles which synthesized is been analyzed by UV spectrum analysis and the range of the absorbance peak is between 300 nm to 400 nm which confirms the synthesized particles is TiO2 nano particles. The biologically synthesized particle is then analyzed in by Dynamic Light Scattering Method for the particle size evaluation using Zeta size and the results showed that the size of the particle is 120.5 nm. Chemical method and the nano particles which synthesized is been analyzed by UV spectrum analysis and the range of the absorbance peak is between 300 nm to 400 nm which confirms the synthesized particles is TiO2 nano particles. The chemically synthesized particle is then analyzed in by Dynamic Light Scattering Method for the particle size evaluation using Zeta size and the results showed that the size of the particle is 101.6 nm. The A216 WCB steel which is dip coated in the chemically synthesized TiO2 sol is been characterized with the SEM analysis. The SEM image shows that the particles were found to be spherical with clearly defined edges and without accumulation.

REFERENCES

[1] Al-Salim, N. I., Bagshaw, S. A., Bittar, A., Kemmtt, T. and Mcquillan, A. J., Characterisation and activity of sol–gel-prepared TiO2 Photocatalysts modified with Ca, Sr or Ba ion additives, J Mater Chem.

[2] Anusha Bhaskar, Nithya V, and Vidhya V. G., Phytochemical screening and in-vitro antioxidant activities of the ethanolic extract of Hibiscus rosa sinensis L, Annals of Biological Research.

[3] Archana Maurya, Pratima Chauhan, Amita Mishra, and Abhay K. Pandey., Surface Functionalization of TiO2 with Plant Extracts and their Combined Antimicrobial Activities Against E. faecalis and E. Coli, Journal of Research Updates in Polymer Science, 1.

[4] Balantrapu Krishna, and Goia Dan., Silver nanoparticles for printable electronics and biological applications, Journal of materials research.

[5] Barbe, C. J., Arendse, F., Comte, P., Jirousek, M., and Gratzel, M. Nanocrystalline titanium xidelectrodes for photovoltaic applications. J Am Ceram Soc.,

[6] Bhainsa, K. C., and D'Souza S. F., Extracellular biosynthesis of silver nanoparticles using the fungus Aspergillus fumigatus, Colloids and Surfaces B: Biointerfaces.

[7] Bhumkar, D. R., Joshi, H. M., Sastry, M., and Pokharkar V.B., Chitosan reduced gold nanoparticles as novel carriers for transmucosal delivery of insulin, Pharm Res., 24, 1415-26 (2007)

[8] Carp, O., Huisman, C. L. and Reller, A., Photoinduced reactivity of titanium dioxide, Prog Solid State Chem., 32, 133 (2004).

[9] Daizy Philip, Biosynthesis of Au, Ag and Au–Ag nanoparticles using edible mushroom extract, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 374–381 (2009).

[10] Devasena, T. and Ravimycin, T., Ketoconazole coated Silver nanoparticles- A potent antidandruff agent, Int J plant sci., 4, 517-520 (2009b).

[11] Feskanich, D., Ziegler, R., Michaud, D., Giovannucci, E., and Speizer, F., et al., Prospective study of fruit and

vegetable consumption and risk of lung cancer among men and women. Cancer Inst.,

[12] Gou N, Onnis Hayden A, and Gu A. Z., Mechanistic toxicity assessment of nanomaterials by whole-cell-array stress genes expression analysis, Environ Sci Technol.

[13] Ikigai, H., Toda, M., Okubo, S., Hara, Y. and T Shimamura. Relationship between the anti-hemolysin activity and the structure of catechins and theaflavins, Jpn J Bacteriol., 45, 913—919 (1990).

[14] Kim, D. O., Lee, K. W., Lee, H. J., and Lee, C. Y Vitamin C equivalent antioxidant capacity (VCEAC) of phenolic phytochemicals, J Agric Food Chem.

[15] Li L., Sun X., Yang Y., Guan N. and Zhang F., Synthesis of anatase TiO2 nanoparticles with beta-cyclodextrin as a supramolecular shell, Chem Asian J.

[16] Li,Y.,White, T. J. and Lim, S. H., Low-temperature synthesis and microstructural control of titania nano-particles, Journal of solid state chemistry.

[17] Ito, S., Inoue, S., Kawada, H., Hara, M. and Iwaski, m., Low-temperature synthesis of nanometer-sized crystalline TiO2 particles and their photo induced decomposition of formic acid, Colloid Interface Sci., 59, 216 (1999).

[18] Jain, D., Daima, H. K., Kachhwaha, S., and Kothari, S. L., Synthesis of plant-mediated silver nanoparticles using papaya fruit extract and evaluation of their anti microbial activities. Digest J Nanomat Biostru., 4, 557-63 (2009).

[19] Kannan, P. and John, S. A., Synthesis of mercaptothiadiazole-functionalized gold nanoparticles and their self-assembly on Au substrates, Nanotechnology, 18, 0856