Albizia saman: A Green Route for the Reduction of Bulk TiO$_2$

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Abstract: Recently, Metal oxide nanoparticles have been paid much attention as they are possessing fascinating properties such as semiconducting, photovoltaic, photocatalytic, magnetic, electronic and antimicrobial activity. This is due to their high fraction of atoms and their high surface area. Each and every nanoparticle differs in their size, shape, composition and crystalline nature. Among the metaloxides, TiO$_2$ has its applications in degrading dyes and in purifying water. In this present investigation, an eco-friendly method was adopted to reduce the bulk TiO$_2$ to nanoscale by using the aqueous leaf extract of Albizia saman. The reduced particle was characterized by performing XRD. The average grain size formed after the bioreduction was determined by using the Scherrer’s formula, \(d = \frac{0.89 \lambda}{\beta \cos \theta}\). The estimated size for the bulk TiO$_2$ was 79nm whereas the bioreduced TiO$_2$ nanoparticle possessed the grain size of 41nm. The bioreduced TiO$_2$ should be explored for its potential applications in the future.

Keywords: Metaloxide nanoparticles, TiO$_2$, Albizia saman, Bioreduction, XRD

Introduction

Nanotechnology is one of the growing interdisciplinary areas covering all fields of science and technology. Now-a-days, nanoparticles made its foot prints in most of the applications as they possess considerable surface area to volume ratio. This determines their characteristics properties such as catalytic, mechanical, thermal, antimicrobial, degrading dyes, etc.

Metaloxides plays a critical role in major areas of physics, chemistry and material science$^1$. Particles in bulk materials are highly unstable as they possess high surface free energy. Those particles once synthesized as individual nanostructure may have low surface free energy, which makes them to acquire structural stability. Such stability has been found in TiO$_2$ and few more metal oxides.

TiO$_2$ are versatile metal oxide as they have wide range of applications from sunscreen lotions to wall paints. Apart from the commercial applications, they too possess biological applications, which includes degradation of organic dyes such as ethidium bromide in aqueous solution$^2$, anthraquinone dye$^3$, organic pesiticial pollutants like aldicarb$^4$, Alachlor and Fenitrothion$^5$, penicillin in water$^6$, etc.,

Generally, Metal and metaloxide nanoparticles can be synthesized by physical and chemical methods. But, both the methods employs the usage of expensive substrate, higher temperature, higher pressure and also results in the generation of harmful end products. To overcome such complications, there should be an alternative method, which should be cost effective, easy to handle, simple to perform and should result in the environmentally benign by products. Such conditions can be fulfilled by the diversified presence of microbial and plant sources on our planet.
Synthesizing nanoparticles using the plant system is currently under exploitation as the method does not cause lethal effects to the environment. Few researchers have reported the biosynthesis of TiO$_2$ nanoparticles using the leaf extract of Nyctanthes$^7$, Eclipta prostrata$^8$, Cantharanthus roseus$^9$. Albizia saman is a rain tree, which is not so far exploited for the green synthesis of TiO$_2$ nanoparticles. This tree exhibits antioxidant, antiplasmodial and cytotoxic properties$^{10,11,12}$. It was reported that its aqueous leaf extract contains tannins, flavonoids, saponins, steroids, cardiac glycosides and terpenoids$^{13}$. Such tree can be used for the green synthesis of metaloxide nanoparticles. Hence, the present work was focused to utilize the leaves of A. saman as a considerable source to bioreduce bulk TiO$_2$.

Materials and methods

Collection and processing the leaves of A. saman

![Fig. 1(a): Fresh leaves of Albizia saman](image)
![Fig. 1(b): 5mM TiO$_2$ solution](image)
![Fig. 1(c): Aqueous leaf filtrates of Albizia saman](image)
![Fig. 1(d): Dirty, white deposits of TiO$_2$ nanoparticles](image)

The fresh leaves of A. saman were collected in a polythene bag from the university campus of Sathyabama and were processed in the university laboratory. As an initial step, the leaves were washed thoroughly in running tap water and were rinsed with sterile distilled water twice. The spills of water were blotted and dried for 2-3 minutes. Then, the leaves (Fig 1(a)) were used for preparing the aqueous extract.

Preparation of 5mM TiO$_2$ solution

0.039 g of TiO$_2$ was weighed and it was mixed in 100 ml of distilled water in 250 ml Erlenmeyer flask. The preparation was mixed and swirled properly. The flask was then plugged with non-absorbent cotton, covered with aluminium foil and stored at 4$^\circ$C for future use (Fig 1(b)).

Preparation of aqueous leaf filtrate of A. saman

About 2gms of leaves were taken and were homogenized in mortar and pestle by adding 5 ml of distilled water. The resultant solution was made up to 20 ml and was kept in the water bath at 65$^\circ$C for 30 minutes. The solution was filtered using whatmann no.1 filter paper and the resulting filtrate (Fig 1(c)) was used for the bioreduction of bulk TiO$_2$.

Bioreduction of TiO$_2$ nanoparticles

The leaf filtrate was taken in an Erlenmeyer flask and TiO$_2$ solution was added in drops under constant stirring at room temperature. Then, the mixture was left in the magnetic stirrer at 50$^\circ$C for 24 hours.

Characterization of TiO$_2$ nanoparticles

The TiO$_2$ solution treated with the aqueous leaf extract of A. saman was observed after 24 hrs. There was a change of colour from milky white to dirty, white deposits. In order to confirm the as so formed nanoparticle, the sample was subjected to XRD measurements.
Results and Discussion

The aqueous leaf extract of A. saman was mixed with 5mM TiO\textsubscript{2} solution. To which, a magnetic pellet was added and kept on the magnetic stirrer for the reduction of TiO\textsubscript{2}. During the reaction, the milky coloured TiO\textsubscript{2} solution was gradually changing its colour to dirty-white deposits (Fig 1(d)).

![Fig. 2(a) – XRD analysis of bulk TiO\textsubscript{2}](image)

![Fig. 2(b) – XRD analysis of bioreduced TiO\textsubscript{2} nanoparticles](image)

The bulk TiO\textsubscript{2} as well the bioreduced TiO\textsubscript{2} nanostructure synthesized by employing the aqueous leaf filtrate of A.saman were characterized with the aid of X-ray diffraction measurements. The bulk TiO\textsubscript{2} showed crystalline nature with 2\(\theta\) peaks lying at 2\(\theta\) = 25.347° (Fig. 2(a)) and at 25.257° (Fig. 2(b)) for the bioreduced TiO\textsubscript{2} nanoparticles. The preferred orientation corresponding to the plane (101) is observed in the TiO\textsubscript{2} NPs. The result is in same proximity with the XRD peak at 2\(\theta\)=25.25° (101), which confirm the characteristic facets for anatase form of TiO\textsubscript{2} [14].

All the peaks in the XRD pattern can be indexed as anatase phase of TiO\textsubscript{2} and the diffraction data was in good agreement with the COD (Crystallography Open Database) file no. 9008213. The crystalline size was obtained by Debye-Scherrer’s formula, which is given by the equation, 

\[ d = \frac{k\lambda}{\beta\cos\theta}, \]

Where, \(d\) = crystal size,
\[ \lambda = \text{wavelength of the x-ray radiation (} \lambda = 0.15406\text{nm)} \text{ for CuK}_\alpha \text{. k is normally considered as 0.89 and } \beta \text{ is the line width at half-maximum height.} \]

The crystallite size obtained by using this formula ranged between 34nm and 58nm for the bioreduced TiO\textsubscript{2} nanoparticles whereas in the case of the bulk TiO\textsubscript{2} nanoparticles, the size was ranged between 68nm and 83nm. The average grain was estimated as 41nm for the bioreduced TiO\textsubscript{2} nanoparticles and 79nm for the bulk TiO\textsubscript{2} nanoparticles. The aqueous leaf extract of \textit{Eclipta prostrata} mediated synthesis of titanium dioxide nanoparticles ranged from 36 to 68nm with an average grain size of 49.5nm [9].

The sharp peaks confirmed the crystallinity of the bulk TiO\textsubscript{2} as well the bioreduced TiO\textsubscript{2} nanoparticles. The unidentified peaks assured the absence of other biomolecules in the aqueous plant extract, which in turn indicates the purity of the bioreduced nanoparticles.

**Conclusion**

The current study suggests that the aqueous leaf filtrate of \textit{A.saman} can be used to bioreduce the bulk TiO\textsubscript{2} nanoparticles even less to nanoscale level as the method is simple, easy and eco-friendly. Further, these TiO\textsubscript{2} nanoparticles should be subjected to SEM and FTIR analysis in order to find out the surface topography and the biomolecules of leaf filtrate involved in the reduction reaction respectively. The potential application of nano sized TiO\textsubscript{2} nanoparticles should be explored in the near future.

**Acknowledgement**

The authors sincerely acknowledge the Chancellor, Directors, Vice chancellor, Dean, Faculty Head of Bio and Chemical Engineering of Sathyabama University for their constant source of support, encouragement and inspiration to carry out the research work in the Department of Biotechnology, Sathyabama University, Chennai - 119.

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