A GREEN METHOD FOR THE SYNTHESIS OF TITANIUM DIOXIDE NANOPARTICLES USING CASSIA AURICULATA LEAVES EXTRACT

Dr. G. Valli*1 and S. Geetha2

*1Associate Professor & Head, 2M.Phil Scholar, Department of Chemistry, S.F.R. College, Sivakasi -626123, Virudhunagar District, Tamilnadu.

ABSTRACT

Knowing the importance of titanium dioxide nanoparticles and Cassia auriculata as revealed by various literature resources, Green synthesis of titanium dioxide nanoparticles using Cassia auriculata leaves were carried out. The formation of titanium dioxide nanoparticles were identified first by observing the color changes of the extract. Secondly titanium dioxide nanoparticles were confirmed by the spectral studies like UV-Visible, FT-IR, X-Ray Diffraction and Field Emission Scanning Electron Microscopy. UV absorbance at 447.3 nm and M-O stretching at 516.92 cm⁻¹ by FT-IR for the TiO₂ nanoparticles were observed. XRD and FE-SEM analysis of TiO₂ nanoparticles showed that they were found to exhibit spherical shape with an average size of 38.0 nm.

KEYWORDS: Cassia auriculata, UV-Vis, FT-IR, XRD and FE-SEM.

INTRODUCTION

Cassia auriculata Linn (Family: Caesalpiniaceae) commonly known as Tanners senna, is distributed throughout hot deciduous forests of India and holds a very prestigious position in Ayurveda and Siddha systems of medicine. It was profoundly used in ayurvedic medicine as a tonic, astringent and as a remedy for diabetes, conjunctivitis and ophthalmia. Cassia auriculata have been shown to possess antibacterial, antifungal, antiprotozoal, antipyretic, hepatoprotective, antidiabetic, antiperoxidative and antihyperglyceamic activities. The flowers are used to treat urinary
discharges, nocturnal emissions, diabetes and throat irritation.\cite{7} They are one of the constituent of polyherbal formulation ‘Diasulin’ in the concentration range of 40 mg/dl which is proven to have antidiabetic activity.\cite{8} Cassia auriculata medicinal properties are due mainly to the content of hydroxyanthraquinone derivative. The uses of Cassia auriculata plant bio-active compounds for pharmaceutical purpose have been gradually increased.\cite{9} Titanium dioxide (TiO2) is thermally stable, non-flammable, poorly soluble and it occurs in nature as well-known minerals rutile, anatase and brookite.\cite{10} The most important application areas are paints and varnishes as well as paper and plastics, which account for about 80% of the world's titanium dioxide consumption. Titanium dioxide is also used as a material in the memristor, which can be employed for solar energy conversion based on Titanium dioxide solar cells using conjugated polymers as solid electrolytes.\cite{11} Synthetic single crystals and films of Titanium dioxide are used as a semiconductor.\cite{12} A significant amount of research on TiO2 have been performed over the last five decades and a number of reviews on various aspects of TiO2 have been published\cite{13-18} to understand and summarize the progress in this field. The properties of TiO2 that make it suitable to act as a photo catalyst and various methods including dye sensitization, doping, coupling and capping are used to improve its efficiency.

Knowing the importance of Cassia auriculata leaves extract and titanium dioxide nanoparticle’s biological importance, a green method for the synthesis of titanium dioxide nanoparticles using cassia auriculata leaves extract was performed.

**MATERIALS AND METHODS**

a) Materials

Cassia auriculata leaves (collected from the Jameen Nathampatti village, Rajapalayam, virudhunagar district), titanium tetra isopropoxide and ethanol were used.

b) Methods

i) Preparation of Cassia Auriculata Leaves in Ethanol Extract

Dried leaves of Cassia auriculata were grinded to fine powder. The ethanol extract was prepared by refluxing 15gm of leave powder and 150ml of ethanol solution by using soxhlet apparatus to maintain carefully the temperature level at 50 – 60°C for 3 hours. Then, the extracts were collected in an airtight bottle and were kept in deep freezer for further use.
ii) Synthesis of TiO\textsubscript{2} Nanoparticles from Leaves Extract

For the synthesis of titanium dioxide nanoparticles, the Erylenmeyar flask containing 50 ml of Cassia auriculata ethanol leaves extract, 50 ml of double distilled water were added to 0.4 ml of titanium tetra isopropoxide and subjected to under stirring at 50\textdegree{}C. After four hours of continuous stirring, the formed TiO\textsubscript{2} nanoparticle was acquired by centrifugation at 10000 rpm for 15 minutes. Then the centrifuged particles were washed with ethanol and again subjected to centrifugation at 5000 rpm for 10 minutes. Separated titanium dioxide nanoparticles were grinded and calcinate at 500\textdegree{}C in muffle furnace for about 2 hours. The calcined titanium dioxide nanopowder was used for further analytical characterization.

iii) Characterization of Titanium dioxide nanoparticles

a) UV- Visible Spectrophotometer Analysis:

The UV-DRS spectrum was recorded in Shimadzu UV- 2400PC series. Absorption spectra of titanium dioxide nanoparticles (Figure-1) have absorbance peak at 447.3 nm.

![Figure-1: UV-Visible Spectrum of TiO\textsubscript{2} Nanoparticles](image)

b) Fourier Transform - Infrared Spectroscopy

The FT-IR spectrum was recorded in Shimadzu FT-IR spectrometers 7600 and the measurement of the samples were performed in transmission mode. In order to identity the binding groups of the plant extract with titanium dioxide, FT-IR spectra of Cassia auriculata leaves extract (Figure-2) and titanium dioxide nanoparticles (Figure-3) were recorded.
c) X-Ray Diffraction Analysis

For the XRD (BRUKER ECO D8 ADVANCE) spectral analysis, prepared particles in solution was purified by centrifugation at 5000 rpm for 20 minutes. An XRD spectrum was recorded at Kalasalingam University and the spectra was given as Figure-4. The dried mixture of titanium dioxide nanoparticles collected for the formation of titanium dioxide nanoparticles by X-ray diffractometer operated at a voltage of 40 kV and a current of 20 mA with Cu Kα radiation in 0-20 configuration. Morphology of the interplanar distance spacing was calculated using Bragg’s equation. ($n\lambda = 2d \sin \theta$).
d) Field Emission Scanning Electron Microscopy

FE-SEM images of the green synthesized titanium dioxide nanoparticles obtained using Cassia auriculata leaves extract recorded at Sastra University was shown in Figure-5.

The titanium dioxide nanoparticles exhibit good uniformity with the average diameter of about 38 nm. The titanium dioxide nanoparticles sizes were found in the range 38.0 – 44.2 nm.

RESULT AND DISCUSSION

Green synthesis of titanium dioxide nanoparticles using Cassia Auriculata leaves extract was first identified by color changes of the extract. Secondly titanium dioxide nanoparticles were confirmed by the spectral studies of UV-Visible Spectroscopy, FT-IR Spectroscopy, X-Ray Diffraction and Field Emission Scanning Electron Microscopy.
a) Ultraviolet Visible Spectrophotometer Analysis (UV-DRS)
UV-Visible Spectroscopy could be used to examine formation of titanium dioxide nanoparticles. Absorption spectra of titanium dioxide nanoparticles formed in the reaction media have shown the absorbance peak at 447.3 nm.

b) Fourier Transform - Infrared Spectroscopy Analysis (FT-IR)
On comparing the FT-IR spectra of Cassia auriculata leaf extract (Figure-2) and the titanium dioxide nanoparticles (Figure-3) revealed the following observation. The FT-IR spectra of Cassia auriculata leaf extract showed a broad band at 3317 cm\(^{-1}\) indicating the presence of bonded – OH group. Stretching at 1643 cm\(^{-1}\) indicated the presence of >C=O group. The FT-IR spectra of titanium dioxide nanoparticles showed the following stretching frequencies.

(i) at 1606 cm\(^{-1}\) (diminished carbonyl stretching)
(ii) 516.92 cm\(^{-1}\) (due to formation of Ti-O bond)

On comparing the IR spectra of both the extract and its assisted titanium dioxide nanoparticles formation revealed the following observation. During the formation of titanium dioxide nanoparticles assisted by Cassia Auriculata, the O-H bonds present in the extract (observed by 3317 cm\(^{-1}\)) diminishes that showed that O-H group, Oxygen may binds to titanium dioxide in titanium dioxide nanoparticles. >C=O stretching frequency at 1643 cm\(^{-1}\) of the extract shifted to 1606 cm\(^{-1}\) in titanium dioxide nanoparticles that showed that carbonyl oxygen enter into the binding with titanium dioxide nanoparticles. M-O stretching at 516.92 cm\(^{-1}\) also confirms the existence of Ti-O bond. Hence these observations indicated the formation of titanium dioxide nanoparticles in which Ti binds strongly to the oxygen atom present in the phytoconstituents of Cassia Auriculata extract.

c) X-Ray Diffraction (XRD)
Titanium dioxide nanostructures were confirmed by the characteristic peaks observed in the XRD pattern. The analysis was carried out 20 value ranging from 10\(^{0}\) to 90\(^{0}\), with step size 0.020. All diffraction peaks correspond to the characteristic face centered cubic observed at 20 angle at 21.3\(^{0}\), 31.5\(^{0}\), 39.9\(^{0}\), 43\(^{0}\), 46.3\(^{0}\), 64\(^{0}\) and 75.9\(^{0}\) respectively. Morphology of the interplannar distance spacing was calculated using Bragg’s equation \((n\lambda = 2d\sin\theta)\).
d) Field Emission Scanning Electron Microscope (FE-SEM)
FE-SEM observation of titanium dioxide nanoparticles proved that the average size was from 38.0 - 44.2 nm. (as given in Figure-5). However, further observation with high magnification revealed that these TiO$_2$ nanoclusters were assembled by smaller nanoparticles, which exhibit good uniformity and the average diameter was about 38.0 nm.

CONCLUSION
Green synthesis of titanium dioxide nanoparticles using Cassia auriculata leaves extract revealed the following observation. Formation of titanium dioxide nanoparticles was confirmed by color changes of Cassia auriculata leaves extract and then characterized by UV-Vis, FT-IR spectral studies and their structure and size by XRD and FE-SEM analysis. UV absorption studies of titanium dioxide nanoparticles showed the absorbance at 447.3 nm and FT-IR studies showed the stretching frequency of M-O bond at 516.92 cm$^{-1}$. Titanium dioxide nanostructure was confirmed by the characteristic peaks observed in the XRD pattern. All diffraction peaks correspond to the characteristic face centered cubic observed at 20 angle at 21.3$^0$, 31.5$^0$, 39.9$^0$, 43$^0$, 46.3$^0$, 64$^0$ and 75.9$^0$ respectively. FE-SEM observation with high magnification reveals that these TiO$_2$ nanoparticles were found to have an average diameter about 38.0 nm.

REFERENCE


