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BIO FABRICATION AND CHARACTERISATION OF METAL OXIDE NANOCOMPOSITE USING *PIPER BETEL* LEAVES

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ABSTRACT

In recent years nanotechnology is used as a wide platform for beginners on research works and it has various ranges of advantages over any other technology. Nano based materials are used in a variety of manufacturing processes, healthcare, paints, filters and lubricant additives. Green synthesis is one of the best and eco-friendly method for synthesizing nanomaterials. In this work, Copper/Titanium dioxide nanocomposite was successfully synthesised by using *Piper Betel* leaf extract. The *piper betel* leaves naturally contains the antioxidant and antimicrobial activities. The biosynthesized copper titanium dioxide nanocomposite was characterized using UV-Vis analysis, FTIR analysis, X-ray diffraction analysis (XRD), Scanning Electron Microscopy (SEM) analysis and EDAX analysis. The optical property of the nanocomposite was studied by using UV-Vis Spectroscopy. FTIR analysis shows the presence of functional groups in *piper betel* plant which is responsible for the reduction of metal/metal oxide nanocomposites. Using XRD analysis the crystallinity of the synthesized nanocomposite was determined. SEM image shows that the synthesized Cu/TiO_2 NCs were flake like shape. EDAX analysis shows presence of elemental composition of Cu, Ti and O.

Keywords: Nanocomposites, SEM, XRD, EDAX

1. INTRODUCTION

In recent years nanotechnology gained utmost importance in industrial, pharmaceutical, environmental and health care applications due to their unique physical and chemical characteristics [1]. Titanium dioxide (TiO_2) is one of the most important metal oxide semiconductors, which find applications in various fields of solar energy conversion, water purification, PEC splitting of water into Hydrogen and Oxygen, photocatalysis, ceramic material, filler, coating, pigment [2], cosmetics [3] etc. Reports of TiO^2 with different shapes such as nano-particles, thin films [4], nanorods, nanowires and nanotubes have spurred a great interest in studies on TiO_2 nanostructure synthesis and their applications. The doping of various metal ions like Ag, Ni, Co, Au, Cu, V, Ru, Fe, La, Pt, Cr, Ce, etc. in titanium dioxide have been found to be influencing the band gap, surface area, particle size, thermal property, etc.[5]. In this present work, the transition metal copper was doped with titanium dioxide to produce cu doped titanium dioxide nanocomposite.

Green synthesis has become one of the most preferred applications in various fields, including chemistry, because of its eco-friendly approach. With the application of green synthesis to nanochemistry, another area of study had emerged that has gained increasing value: Green nano synthesis. Green nano synthesis allows for a nano material to be synthesized in a way that is friendly to both humans and to the environment [6].

The betel leaf when taken alone has several medicinal benefits. Apart from being a mild stimulant, betel leaf is used for various medicinal purposes. Betel leaves possess good diuretic properties. The betel leaves are also beneficial in treating nervous pains and debility. Betel leaf has been in use since ancient times for healing wounds. Also, recent studies have shown that the leaf contains components that have chemo-preventive and anti-cancer properties [7].

Green Synthesis of Copper-Titanium dioxide nano-composite has considerable attention due to their electrical, catalytic properties. The Green syntheses of Titanium oxide nanoparticle using plants have already been reported. In this work, we report synthesis of Copper-Titanium dioxide nanocomposite using the extract of *Piper* betel leaves.

The Scope of the present work is to synthesize the Cu/TiO_2 nanocomposite using the extract of *Piper* betel leaves. The optical property, nature of metal oxide bond and biomolecules, size and crystallinity, elemental composition present in the nanocomposite and morphology of the nanocomposite was characterized by using UV- Vis spectroscopy, FT-IR spectroscopy, XRD, EDAX and SEM respectively.

2. MATERIAL AND METHODS

2.1. Material

Copper Sulphate and Titanium Dioxide with AR grade were used in the study, Fresh *Piper Betel* leaves were collected from in and around Thoothukudi District.

2.2. Preparation of the *Piper betel* Leaves extract

The collected *Piper betel* leaves were incised into small pieces, washed well with double distilled water to dirt and other foreign materials. About 10 grams of thus dried *Piper betel* leaves were weighed and transferred into 250mL beaker containing 100mL of water and boiled well for 30 minutes. The extract obtained was filtered through Whatman No-40 filter paper and the filtrate was collected in a 250mL beaker and stored in refrigerator for further use. All the further experiments were carried out using this extract.

2.3. Green synthesis of Copper/Titanium Dioxide Nanocomposites

In this method, Titanium Dioxide was used as a precursor and *Piper betel* leaves extract as a reducing and stabilizing agent for the synthesis of Cu/TiO_2 nanocomposite. For the green synthesis of Cu/TiO_2 nanocomposite, 50mL of previously prepared *Piper betel* leaves extract was taken in a 100mL beaker. To this, 0.2g of Titanium Dioxide and 0.4g of Copper Sulphate was added and the solution in the beaker was stirred in a heating magnetic stirrer at 70ºC until the ash colour paste was obtained. Then the paste was collected in a ceramic crucible and calcinated in Muffel Furnace at 350ºC. A black coloured powder of Cu/TiO_2 nanocomposite was obtained and this was carefully collected and preserved in the air-tight sample tubes for further studies.

2.4. Characterization Techniques

2.4.1. UV- Visible spectroscopy

UV- visible spectral analysis was carried out on a JASCO V-530, UV visible spectrometer at Ayya Nadar Janaki Ammal College, Sivakasi. UV- visible spectrophotometer with a resolution of 2nm between 200- 800nm possessing a scanning speed of 200nm/min was used.

2.4.2. Fourier Transform Infrared spectroscopy (FT-IR)

FT-IR measurements were carried out in order to obtain information about Ti-O stretching and biomolecules that present around the Copper/Titanium Dioxide nano-composite for their reduction and stabilization. The material was grinded with KBr and the Copper/ Titanium Dioxide nanocomposite pellets were then analyzed. FT-IR spectra were recorded at Ayya Nadar Janaki Ammal College, Sivakasi.

2.4.3. X-Ray Diffraction analysis

XRD technique was employed to visualize crystal structure including lattice constants, geometry, identification of unknown phases etc., The scanning was done in the region of 2θ from 0^o to 100^0 and by using monochromatic Cu K α radiation with a wavelength of 1.54Å. XRD was recorded at Manonmanium Sundaranar University, Tirunelveli.

The size of Cu doped Titanium Dioxide nanocomposites were calculated by using Debye- Scherer's formula (i.e)

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad \text{-------- (eq.1)}$$

Where D is the mean crystalline size

K is the constant (Shape factor). K= 0.94

λ is the Wavelength of X- ray. λ = 0.154 nm

β is the FWHM of the diffraction peak.

And θ is the Bragg diffraction angle

2.4.4. Energy Dispersive X- Ray Spectroscopy (EDAX)

EDX was used for identifying the elemental composition of specimen. The Energy Dispersive Spectra was operated at operating voltages 0 to 8KeV. EDAX was recorded at Karunya University, Coimbatore.

3. RESULTS AND DISCUSSION

3.1. UV Visible Spectroscopy

The Cu/TiO_2 nanocomposite was synthesized from *Piper betel* leaf extract by sunlight induced method. The formation of Cu/TiO_2 Nanocomposite from copper sulphate and Titanium Dioxide was monitered by UV-Visible spectroscopy. UV-Vis spectra of synthesized Cu/ TiO_2NCs from *Piper betel* leaf extract are shown in fig.1.

The peak observed at 281 nm indicated that the absorbance of TiO_2. The peak absorbed at 386nm indicates the absorbance of CuO [8].

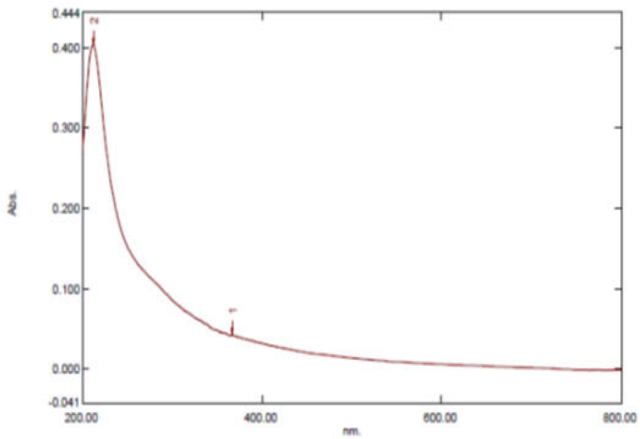

Fig. 1: UV- Vis spectra of Cu/TiO_2NCs synthesized from the extract of *Piper betel* leaves

3.2. FTIR analysis

Fourier transform infrared spectroscopy gives data of functional group in leaves extract that interact with metal oxide and also gives the information about Ti-O-Ti bond. The identification of functional groups leads to determine the reducing agent and the capping agent responsible for synthesis and stability of metal oxide nanocomposite.

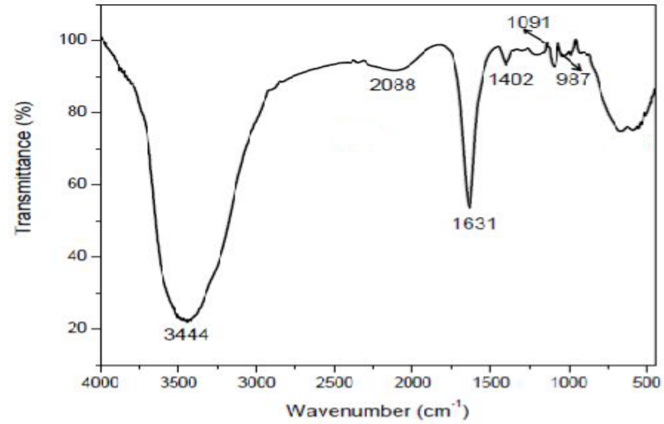

Fig. 2(a): FTIR spectrum of *Piper betel* leaves extract

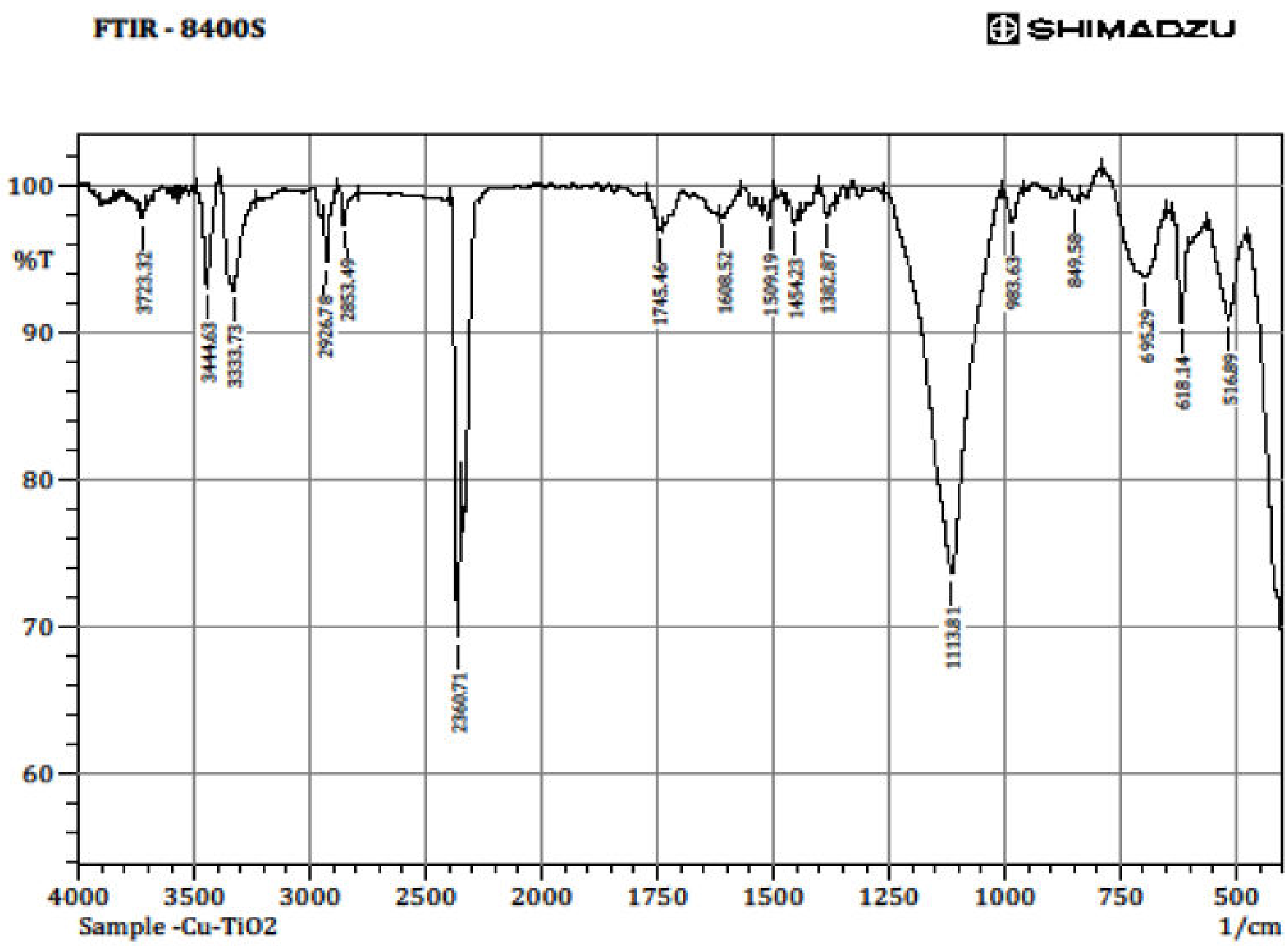

Fig. 2(b): FTIR spectrum of Cu/TiO_2NCs synthesized using extractof*Piper betel* leaf

Table 1: Frequency Interpretation Data of FTIR spectra

Wavenumber(cm^{-1})		Functional group(s)
***Piper betel* leaves extract**	**Cu/TiO_2**	
3444	3445.63	O-H stretching
	3445.73	
2088	2360.71	C-H Stretching of Alkene
	2926.78, 2853.49	C-H Stretching of Alkane
1091	1382.87	C-O Stretching
	1745	C=O Stretching
987	1608.52	Aromatic Ring
-	983.63	Ti-O-Ti
	695.29	
	618.14	
-	849.58	TiO_2 group

The FTIR spectrum of *Piper betel* leaves extract shown in Fig. 2 (a). The peak at 3444cm^{-1} indicates the presence of O-H stretching of phenolic compound [9]. The peak at 2088 cm^{-1} is due to the presence of C-H stretch [10]. The peak at 1091 cm^{-1} is due to the presence of C-O Stretch of phenol [11]. The peak at 987cm^{-1} is due aromatic C-H out of plane bending [12]. The FTIR spectrum of Cu/TiO_2 nanocomposites using *Piper betel* Leaf extract is shown in Fig. 2(b). The peak at 3444.63 and 3333.73 cm^{-1} indicates the presence of O-H stetching of phenolic compound. The peak at 1745cm^{-1} is due to presence of C=O stretching. The peak at 2926.78 and 2853.49 cm^{-1} shows C-H stretching of alkane. The peak at 2360.71cm^{-1} is due to C-H stretching of alkene. The peak at 1382.87cm^{-1} is due to C-O stretch. The peak at 1608.52cm^{-1} is due to aromatic ring. The peak at 983.63, 695.29 and 618.14 cm^{-1} indicates the presence of Ti-O-Ti stretching group. The peak at 849.58 cm^{-1} indicates the presence of TiO_2 group.

3.3. X- Ray Diffraction Analysis (XRD)

X-ray diffraction (XRD) measurement was carried out to confirm the crystalline structure, phase composition and preferential orientation of formed copper/titanium dioxide nanocomposites. The XRD pattern for is shown in Fig. 3.

From the Fig. 3 it is seen that the XRD peaks appear at 27.5487, 35.5802, 38.7809 and 69.9121 correspond to the (111) and (220) agree well with JCPDS card file no: 80-1916 [13]. This indicates FCC crystal nature of Cu nanoparticle. Other XRD peaks are appeared at 48.9845, 54.4014 and 62.8619 correspond to the (200), (211) and (002) agree well with JCPDS card file no: 21-1272 [14]. The peak at 48.9845 indicates that TiO_2 is in anatase phase. The XRD pattern of Cu/TiO_2NCs clearly shows the crystalline nature.

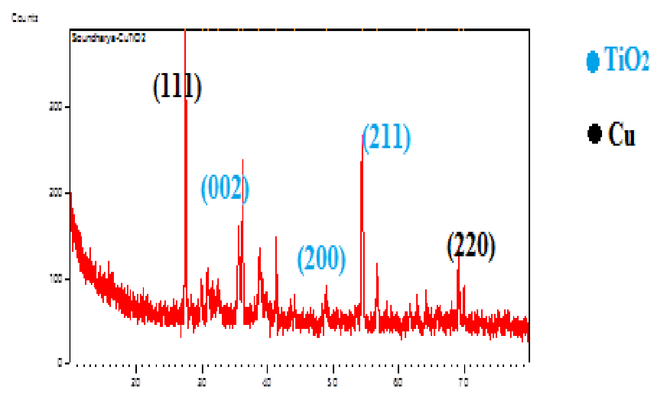

Fig. 3: XRD pattern of synthesized Copper/ Titanium dioxide nanocomposites using *Piper betel leaf* extract.

Table 2: Calculation of average particle size of Cu/TiO_2 nanocomposites using *Piper betel* leaf extract

2 θ	Θ	Cos θ	FWHM Β	FWHM (rad)	D (nm)
27.5487	13.7743	0.9712	0.1171	0.002042	69.89
35.5802	17.7901	0.9521	0.2007	0.003500	41.59
38.7809	19.3904	0.9432	0.2676	0.004666	31.50
41.3540	20.6770	0.9355	0.1338	0.002333	63.51
56.7164	28.3582	0.8799	0.1338	0.002333	72.14
69.0964	34.5482	0.8236	0.1338	0.002333	72.14
69.9121	34.9560	0.8195	0.1338	0.002333	72.52

The size of Cu/TiO_2 nanocomposite was calculated using the Debye-Scherrer's formula (as said in eq.1). It was found that the average size of Cu/TiO_2NCs synthesized by *Piper betel*was 41 nm.

3.4. Scanning Electron Microscopy

The surface morphology of the Cu/TiO_2 nano-composites was studied by Scanning Electron Micro-scopy (SEM) analysis.

The Fig. 4 shows the SEM image Cu/TiO_2NCs synthesized by *Piper betel* leaf extract. It shows a granular shape. This shape of the Cu/TiO_2 nanocomposite may be due to the phenolic compounds present in extract which may act as structure directing agent.

3.5. Energy Dispersive X-Ray analysis (EDAX)

Energy dispersive X-Ray analysis was carried out to find out the elemental composition of the synthesized Cu/TiO_2 nanocomposites. The EDAX graph of Cu/TiO_2 NCs was shown in Fig. 5 and the EDAX data of Cu/TiO_2NCs was shown in Table 3. Indicate the Copper/Titanium dioxide nanocomposite synthesis using *Piper betel* Leaf extract.

EDAX analysis proves the presence of elemental Titanium oxygen and Copper and atomic percentage of Titanium and oxygen is 12.74%, 68.18% and 10.57% respectively. S, Cl and K also present in the nanocomposite due to the impurities present in the chemicals used. The environment in which the *Piper betel* leaves grew up is also the reason for the presence of other elements such as S, Cl, and K.

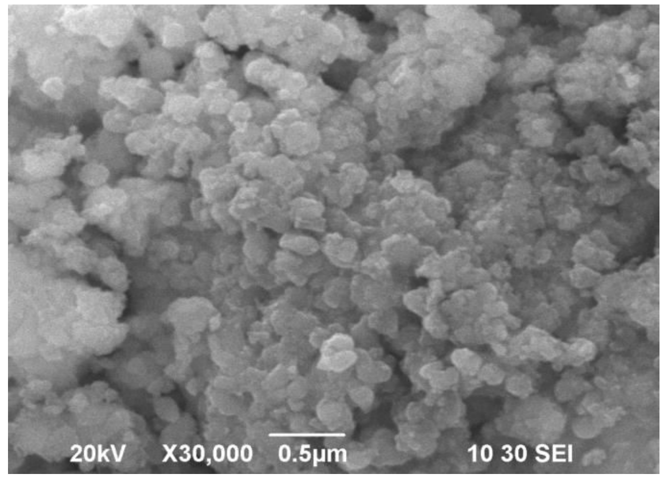

Fig. 4:SEM image of Cu/TiO_2 NCs

Table 3: EDX data of Cu/TiO_2NCs synthesized from *Piper betel* leaf extract

Element	Series	App Conc.	Intensity corrn	Weight%	Weight % Sigma	Atomic%
O	K	17.57	0.5150	40.70	0.90	68.18
S	K	2.08	0.9079	2.74	0.18	2.29
Cl	K	2.41	0.8109	3.54	0.17	2.68
K	K	4.77	1.1029	5.16	0.18	3.54
Ti	K	16.85	0.8832	22.78	0.45	12.74
Cu	K	17.91	0.8529	25.07	0.60	10.57

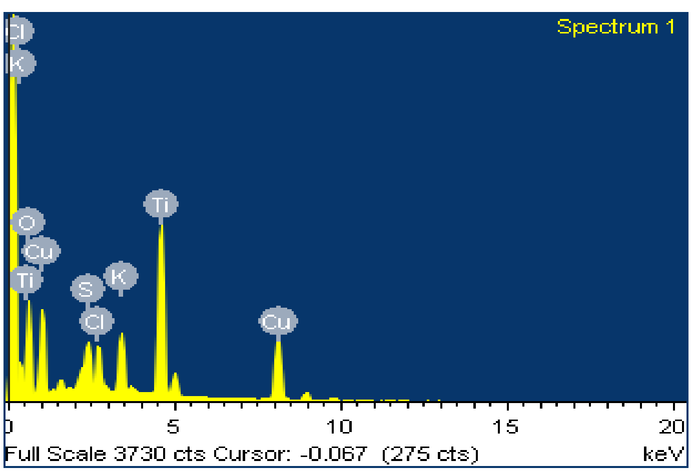

Fig. 5: EDX graph of Cu/TiO_2NCs synthesized from *Piper betel leaf extract*

4. CONCLUSION

The copper titanium dioxide nanocomposite was successfully synthesized by using the extract of *Piper betel* which provides cost effective, easy and proficient method for synthesis of Cu/TiO_2 NCs. The synthesized copper titanium dioxide nanocomposite was characterized using UV-spectrophotometer, FITR, SEM with EDAX and XRD. The blue shift in wavelength indicates the smaller size of nanocomposite was confirmed by UV-VIS spectrophotometer. The metal-oxide bond was confirmed by FTIR analysis. XRD data clearly shows the crystalline nature of copper titanium dioxide nanocomposites. SEM analysis shows the morphology of copper titanium dioxide nanocomposites.

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