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Synthesis and Characterization of Indium Tin Oxide with Neem Extract for Antioxidant Applications

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Indium tin oxide was prepared by using simple co precipitation method with Neem extract as reducing agent and it was characterized by using X-ray diffractometer, with the applications of antioxidant effect, the prepared sample was directly calcined at 400ºC and then characterized. Morphological studies were analyzed by using Transmission Electron Microscopy and Selective area diffraction pattern. The crystal sizes were calculated and it value is nearly 12 nm. Here, Williamson-Hall (W–H) have been used to investigate the particle size and the intrinsic strain from the XRD peak broadening analysis.

Keywords: Indium tin oxide; Azadirachta indica; cell line and antioxidant.

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1. INTRODUCTION

Transparent conducting oxide (TCO) nanoparticles of tin, indium and zinc oxides (doped and undoped) have been extensively studied due to their high optical transmittance and electrical conductivity. These particles are useful in photovoltaic and photo thermal applications [1–3]. Unlike the more commonly used indium tin oxide, zinc oxide is a non-toxic, inexpensive and abundant material. It is chemically and thermally stable in hydrogen plasma processes which are commonly used for the production of solar cells [4]. As the development of nano materials proceeds, commercial applications may start with car mirrors, thereafter sun roofs in cars, and finally, windows in buildings. In other words, there would be a continual increase in the available active nano particle area. In addition to the energy regulation, visible colour changes may be exploited architecturally, i.e. so called `fancy windows' [5]. ITO has been extensively studied due to its wide applications in industry as coating material for energy saving of Lamp [6], transparent electrode for watch, LCD and indication element for office equipment and automobile measuring instrument, and coating material for screen panel, and is expected to use as selectively permeable membrane and transparent conductive for solar cell [7]. Neem (Azadirachta Indica) a member of Meliaceae family is a fast growing tropical evergreen tree and its tremendous therapeutic, domestic and agricultural and ethno medicinal significance and its proximity with human environment. Thus, resistance to antibacterial agents poses threat in many areas of the world especially in the developing countries. The antioxidants in Neem extract, fruits extracts and cocoa and dark chocolate have been linked to impressive health benefits such as less inflammation and reduced risk factors for heart disease.

2. EXPERIMENTAL PROCEDURE

2.1 Preparation of Precursor Materials

Calculated amount of $In(NO_3)_3$ (5.0 M), dissolved in minimum volume of water was added to the aqueous solution of $Sn(NO_3)_2$ obtained by stirring the In-metal ingots with concentrated HNO_3 for 40 h. The volume of $In(NO_3)_3$ solution and the weight of $Sn(NO_3)_2$ were taken by maintaining the In/Sn atomic ratio 90:10. The mixture was diluted with water and the resultant solution was stirred magnetically for 2 h. Neem extract of 10 mL and Glycene was added to the above mixture till a gel of hydrated indium tin oxide appeared. The pH of the above solution was maintained in the range 8.0–8.5 at this stage. The solution was stirred again for ½ h to ensure the complete precipitation. The precipitation was placed in a hot plate at 100°C until it turned as gel. The resulting gel was then fired at a temperature higher than 300°C until complete decomposition of the residues was achieved. The above procedure was repeated under different experimental conditions. The powder was heated at 300°C for 1 h to examine the crystallization behavior [8].

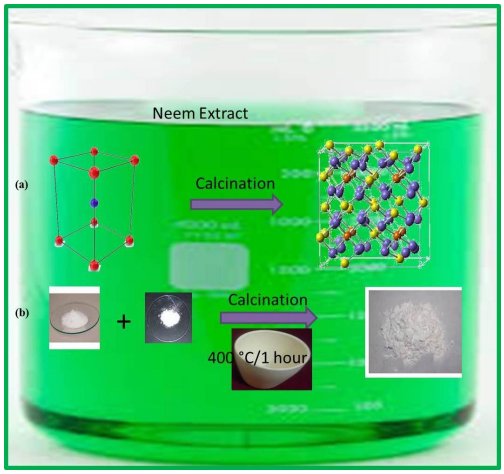


Fig. 1. Mechanism of phase transformation from BCC to BCT structure in synthesis of Indium tin oxide with Neem extract: (a) Phase transformation (b) prepared sample

3. RESULTS AND DISCUSSION

Whenever the both P block neighborhood friend elements so called post-transition metals Indium (In) (P – Block of 49z and 115_A) and Tin (Sn) (P – Block of 50z and 118_A) associate with diatomic nonmetallic oxygen element, gives ternary composition InSn:O with better crystalanity nature under nano synthesis. Even in the bitter herbal background, this nanocomposition shows the well crystalline peaks with strong intensity prominent peak $(2\ 2\ 2)_{hkl}$ in the Fig. 1. The JCPDS data (PDF No-89-4598) is well agree with the observed crystallographic data of entitled composition constructed by the body centered cubic lattices [9]. The body centered tetragonal structured bulk crystalline indium (In) and tin (Sn) salts are successfully converted in to cubic body centered crystal structure during the synthesis in the Neem extract medium. This phase transformation does not affected by the external extract background. Since the dislocation of atoms without change in the relative position and diffusion takes place during the synthesis, this structure phase transformation has done at constant calcination temperature 400°C. Hence the synthesized compounds changed itself also switch over from BCT (P – Block of 49z and 115_A) to BCC (P – Block of 50z and 118_A) structure at higher temperature. The mechanism of herbal assisted metals oxide formation is clearly depicted in the Fig. 2.

The JCPDS data clearly describing that the observed value is most relevant and matches with standard data and has cubic phase with body centered where all lattice are equal with constant value of 10.13 Å. From the Debye Scherrer equation, **D=** $\frac{0.94\lambda}{\beta Cos\theta}$ crystallite size of the ITO nanocrystals is observed that 11.573 nm which is an expected result reveals the nanocrystaline particles formation evenly in the nano range. When the particle size decreases, the number of energy as well as its sub levels increases so the carriers easily jump from valence band to conduction is one of the important behavior of semiconductor. Some basic crystallographic parameters and its corresponding values are shown in Table 1 and its structural parameters. Moreover the extract actively acts as an excellent reducing agent during the synthesis which means it oxidized by releasing more electron. The crystallite and dislocation density and lattice constant values are also tabulated in Table 1.

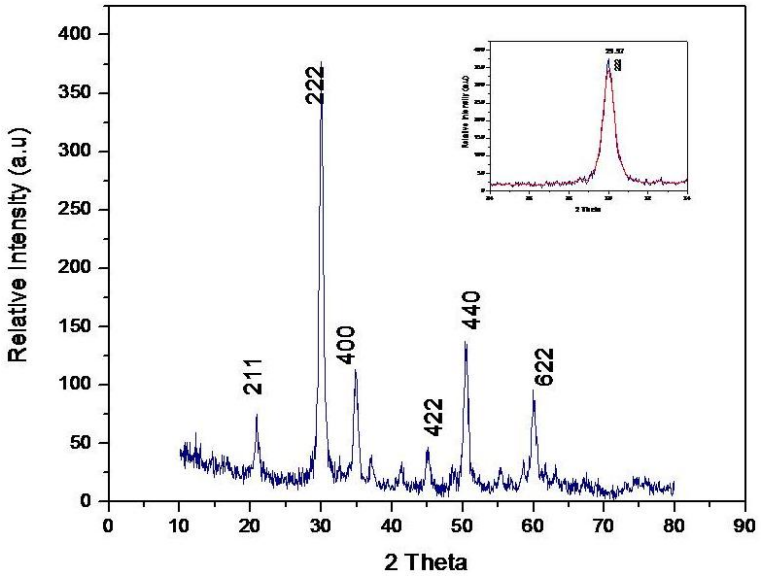


Fig. 2. XRD Pattern of ITO

Table 1. Structural parameters of the prepared ITO Green synthesized NPs

Asprepared concentrations from EDAX	Chemical compositions from Refined structure	Average crystallite size (nm)	
		XRD	Refined structure
$\mathbf{In_{1.98}Sn_{0.017}O_{3.01}}$ (90:10)	$In_{1.94}Sn_{0.063}O_2$	12.383	12.21

Scherrer formula considers only the effect of crystallite size on the XRD peak broadening, which gets developed in the nanocrystals due to the point defect, grain boundary, double layered junction and stacking faults.

The possible reaction of synthesis of ITO shown in equation (1)

$$In(NO_3)_3 + Sn(NO_3)_4 \xrightarrow{\triangle} In_2O_3;\ SnO_2 + 4(NO)_3 \quad (1)$$

Most of both prominent peaks of observed and standard intensity are coincident nearly cent percentage as shown in the Fig. 3. The distortion due to the presence of extract composition removed as well as refined and shown in the diagram Fig. 4 [10].

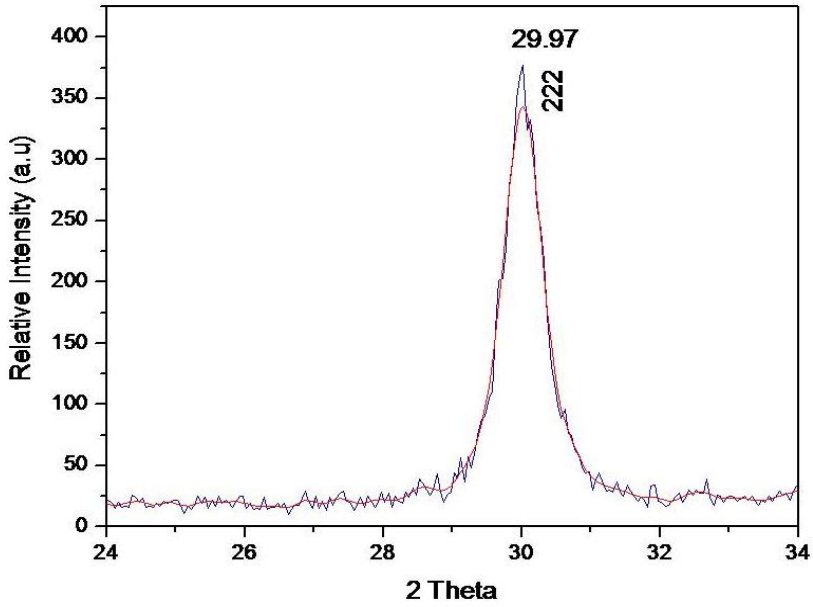


Fig. 3. 2θ position of most prominent crystalline peak of green synthesized ITO nanocomposition

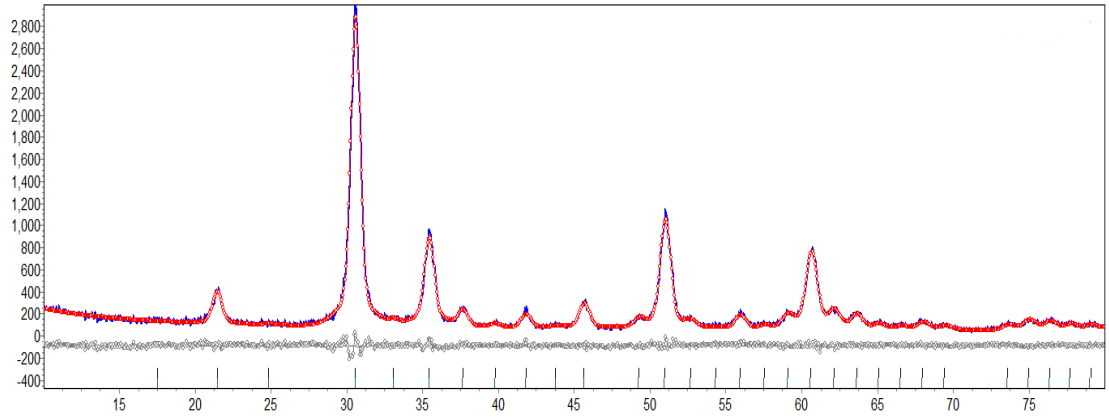


Fig. 4. Refined diffractogram of ITO

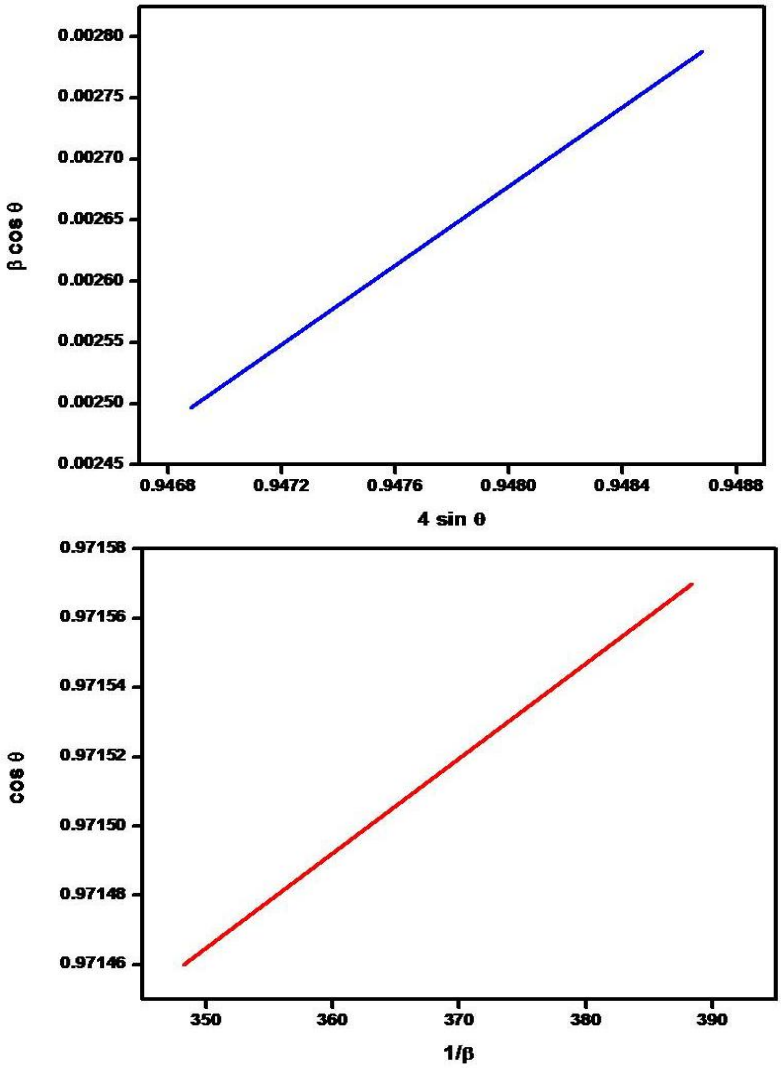


Fig. 5(a-b). Williamson hall analysis of linear graphs

The powder is then characterized to determine the phase structure and the crystallite size using XRD technique and Williamson Hall analysis helps to fix the linear proportion of the NPs shown in Fig. 5(a-b) [11]. Uniform distribution of the particles considers uniform strain throughout the crystallographic direction, which gets introduced in the nanocrystals due to crystal imperfections. In other words, strain, actually effects the physical broadening of the XRD profile and this the strain induced peak broadening can be expressed as,

$$\beta strain = 4\varepsilon.\tan\theta \quad (2)$$

So, the total broadening due to strain and size in a particular peak having the hkl value, can be expressed as,

$$\beta_{hkl} = \beta_{size} + \beta_{strain} \quad (3)$$

Where, β_{hkl} is the full width at half of the maximum intensity for different diffraction planes.

Antioxidants are substances may protect our cells against free radicals, play a role in heart disease, cancer and other diseases. Free radicals are molecules produced when our body breaks down food or exposed to radiation. Such vitamins C and E and carotenoids help to protect our cells from damage caused by free radicals. Antioxidants include flavonoids, tannins, phnols and lignans. Plant based extract such as (Azadirachta Indica) Neem extract are the best source, alos it includes fruits, vegetables, whole grains, nuts, herbs and seeds [12].

Gomes et al., compared the antioxidant activity of trihydroxyflavones containing hydroxyl groups in different positions. It was established that the *ortho*-dihydroxy group is required for free radical scavenging activity. Despite the numerous studies of separate flavonoids and plant extracts enriched by those compounds, the relationship between anticancer and antioxidant activity remains unclear and still has to be studied more thoroughly [13].

Hydroxyflavones are very attractive compounds for anticancer activity studies since they have low toxicity and may interact with DNA, this hydroxyflavones are existed in the (Azadirachta Indica) Neem extract [14].

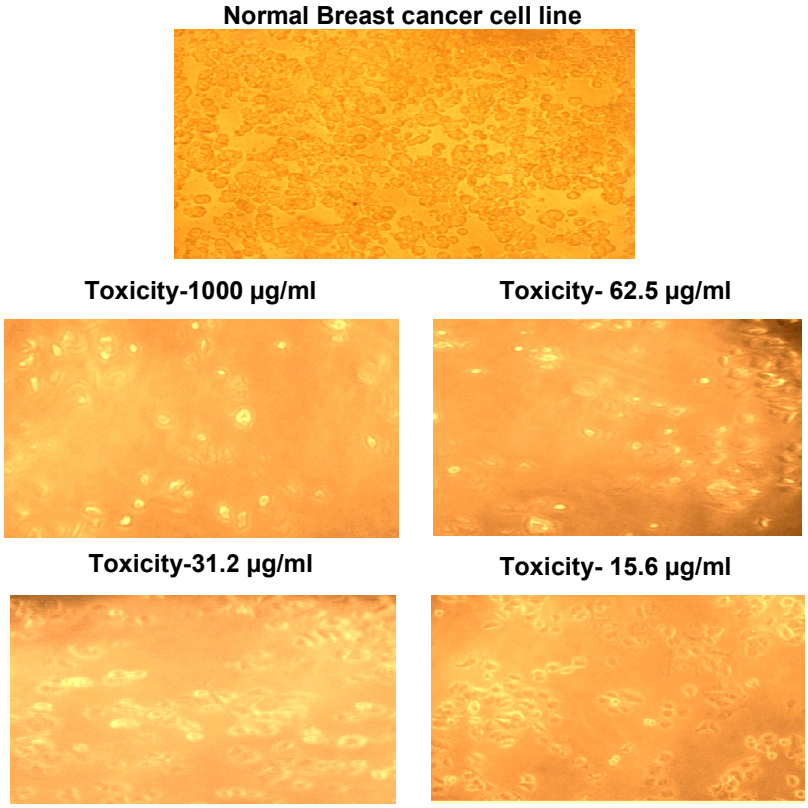
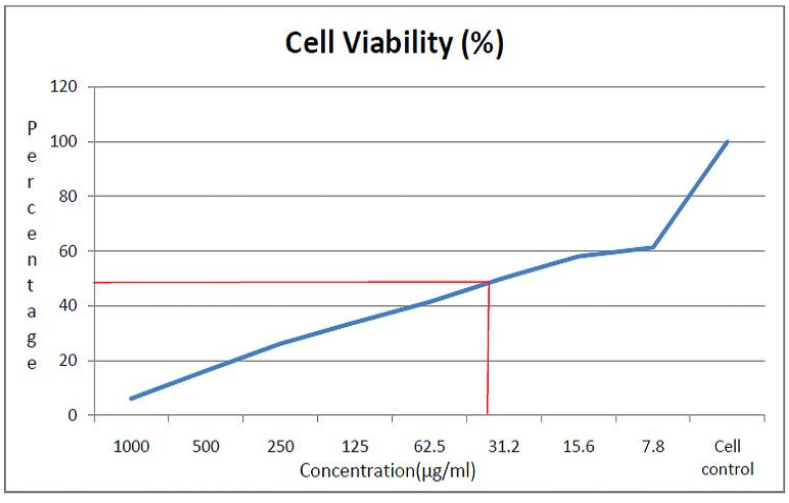


Fig. 6. Antioxidant activity of breast cancer cell line



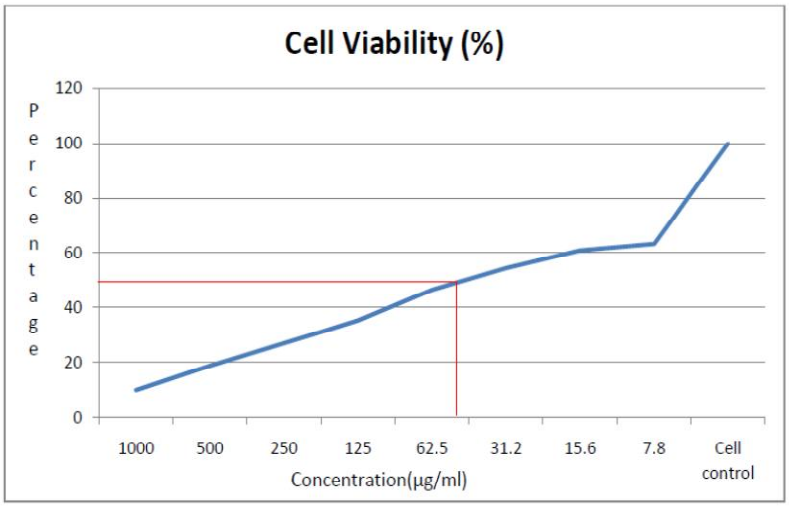


Fig. 7(a-b). Graphical representation of cell viability of ITO

Table 2a. Cell viability of A549 cell line against ITO with Neem extract nanoparticles

S.No	Concentration (µg/ml)	Dilutions	Absorbance (O.D)	Cell viability (%)
1	1000	Neat	0.052	9.92
2	500	1:1	0.098	18.70
3	250	1:2	0.142	27.09
4	125	1:4	0.185	35.30
5	62.5	1:8	0.243	47.36
6	31.2	1:16	0.285	54.38
7	15.6	1:32	0.319	60.87
8	7.8	1:64	0.332	63.35
9	Cell control	-	0.524	100

Table 2b. Cell viability of A549 cell line against ITO with Neem extract nanoparticles

S. No	Concentration (µg/ml)	Dilutions	Absorbance (O.D)	Cell viability (%)
1	1000	Neat	0.032	6.10
2	500	1:1	0.085	16.22
3	250	1:2	0.137	26.14
4	125	1:4	0.178	33.96
5	62.5	1:8	0.217	41.41
6	31.2	1:16	0.263	51.23
7	15.6	1:32	0.304	58.01
8	7.8	1:64	0.321	61.25
9	Cell control	-	0.524	100

The present study was designed to investigate the cellular and molecular mechanisms by which Azadirachta Indica with ITO NPs of cytotoxic effects in the human cervical cancer (A549) cell line. Both Azadirachta Indica with ITO significantly suppressed the viability of A549 cells in a dose-dependent manner by inducing cell cycle arrest accumulation and down-regulation of the cell cycle regulatory proteins. Characteristic changes in nuclear morphology, presence of peak and staining pointed to apoptosis as the mode of cell death. Increased generation of reactive oxygen species with decline in the mitochondrial transmembrane potential and

release of cytochrome *c* confirmed that the Neem extract with ITO transduced the apoptotic signal via the mitochondrial pathway. Altered expression of the normal family of proteins, inhibition of cell activation and over-expression of caspases and surviving provide compelling evidence that Azadirachta Indica with ITO induce a shift of balance toward a pro-apoptotic phenotype. Antioxidants such as Azadirachta Indica with ITO that can simultaneously arrest the cell cycle and target multiple molecules involved in mitochondrial apoptosis offer immense potential [12].

The result, thus support the use of the plants traditionally to treat cancer cells and suggest its usage in the formulation of new antioxidant and antibacterial drugs.

4. CONCLUSION

Azadirachta Indica with ITO NPs can be prepared by using Green Synthesis method. It was analysed by using X-Ray diffractometer to find the crystallite size of the NPs. The crystallite size of the NPs can be determined by Scherer equation and its value is equal to 12 to 13 nm. Cell control and its viability was observed as 47.36 and 51.23 showed the good results of the sample which control the cell line of A549. The size of ITO NPs exhibit a particle decrease in size will give the better antioxidant effects comparatively with reported researchers.

CONSENT AND ETHICAL APPROVAL

It is not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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