Synthesis and Characterisation of In$_2$O$_3$ Nanoparticles from *Astragalus gummifer*

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Abstract

Exploitation of green chemistry approach for the synthesis of Indium Oxide nanoparticles using green synthesis has received a great attention in the field of nanotechnology. To demonstrate a biogenic method that involves the Katira gum (*Astragalus gummifer*) leading to the formation of different morphological In$_2$O$_3$ using the precursor Indium (III) Acetylacetonate and TG-DTA is characterised for calcination temperature and it is found to be above 500$^\circ$C. Different techniques such as XRD, UV-VIS, SEM and EDAX have been used for the characterisation of In$_2$O$_3$ nanoparticles. The average crystallite size of Indium oxide nanoparticles is determined as 19 nm by using Scherrer’s Equation and PSA and studying optical properties.

Keywords

In$_2$O$_3$ Nanoparticles, Bio Synthesis, XRD, UV-Vis, SEM and EDAX, PSA, RAMAN FTIR and PL

1. Introduction

These days nanotechnology is one of the important research fields. For several years, scientists have constantly explored different bio synthetic methods to synthesize nanoparticles. The gum mediated synthesis of nanoparticles is eco friendly, efficient and easier when compared to chemical mediated or microbe mediated synthesis. For the production of nanoparticles plant extract is an alternative method to chemical and physical methods and it eliminates the elaborate process of maintaining cell cultures.

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Among the many transparent conducting oxide materials like SnO$_2$, Indium oxide is physically stable chemically inert and hence it is superior for applications in several aspects. Usually, Indium oxide crystallizes into a cubic bixbyite structure with a melting point temperature of 1910°C. It is highly conductive and exhibits a direct band gap between 3.55 and 3.75 eV, which is an unusual property for wide band gap material. Indium Oxide also has very interesting optical properties. It absorbs IR light waves beyond 900 nm and transmits visible light of wave length 400 to 700 nm. It has interesting properties such as high transparency to high electrical conductance, visible light and strong interaction between certain poisonous gas molecules and its surfaces [1]-[3]. Indium Oxide with these properties makes an interesting material for various applications, including solar cells [1][2], Panel Displays [4], Organic Light Emitting Diodes [5], Photo Catalysts [6], Architectural Glasses [7], Field Emission [8]. Moreover, Indium Oxide is an important material for Semiconductor Gas Sensors [9]-[14]. Recently, investigations on preparation of Indium Oxide nano structures with various forms such as Nantubes [15], Nanobelts [16]-[18], Nanofibers [19]-[20], Nanowires [5][8][21]-[26] and Nanoparticles [27]-[29] have been widely emphasised to extend their technological usages. Nanoparticulate form of Indium Oxide nanostructures has been used as a promising material for gas sensor applications. Tragacanth is a natural gum (Botanical name Astragalus gummifer) obtained from the dried sop of several species of Middle Eastern legumes of the genus Astragalus. It is tasteless, odourless and viscous and is obtained from the root of the plant and dried. The gum comes from a thorny shrub. It has been used as treatment for burns, cough, texturant additive and also as a binder to hold all the powdered herbs together.

2. Experimental

For the preparation of Indium Oxide nanoparticles, the chemical materials used were Indium (III) Acetylacetonate (99.99+% purity, Sigma Aldrich) and Astragalus gummifer (Katira Gum) bought from local Unani (davaasaas) Shop. The Astragalus gummifer (0.05 gm) and Indium (III) Acetylacetonate (0.4 gm) were mixed and crushed into fine powder using mortar and pistle. The precursor was characterised by TG-DTA to determine the calcination temperature and is found to be above 450°C (Figure 2). Then the precursor was calcinied in box furnace at 500°C for 2 hours in air. The yellowish nano-powder of In$_2$O$_3$ as described in (Figure 1) was obtained.

Physical Characterization

The precursor was characterised by TG-DTA (HITACHI Spectrophotometer) to determine the thermal decomposition and crystallite temperature was found to be at 500°C (Figure 2). The dried precursor was ground and

Figure 1. Flow chart of preparation of In$_2$O$_3$ nanoparticles.
Figure 2. TG-DTA curves of thermal decomposition of In$_2$O$_3$ precursor at a heating rate of 10°C/min in static air.

subsequently calcined in box furnace at 500°C for 2 hours in air. The dried precursor and calcined samples of In$_2$O$_3$ were characterised for crystal phase identification by powder XRD using Cu$_{K\alpha}$ radiation (λ = 0.15406 nm). The optical absorption spectra were measured in the range of 200 - 800 nm using UV-Vis scanning spectrometer. Photo Luminiscence (PL) measurement was carried on a luminescence spectrometer with xenon lamp as the excitation source at 25°C temperature. The samples were dispersed in using ethanol and the excitation wave length used in PL measurement was 353 nm.

3. Results & Discussion

3.1. TG-DTA Analysis

The TG-DTA analysis curves of as prepared In$_2$O$_3$ precursor are shown in Figure 2. The TG curve in Figure 2 shows a major weight loss step from 200°C up to about 350°C. The major weight loss is due to the combustion of organic matter (CO and OH groups) present in the precursor and slight weight loss (combustion of remaining carbonyl group) was observed at 470°C. On the DTA curve (Figure 2) a main exothermic effect was observed between 300°C and 400°C with maximum at about 320°C indicating that the thermal events can be due to the burning of organic species in the precursor powders from the amorphous component.

3.2. XRD Analysis

The formation of nano crystalline In$_2$O$_3$ as decomposition product was confirmed by XRD pattern in Figure 3. All of the detectable peaks (Figure 3) can be indexed as the In$_2$O$_3$ cubic structure in the Standard Data (JCPDS: 06-0416). The cubic lattice parameter “a” can be calculated from the XRD spectra is 10.222 Å which is close to those of lattice constants α = 0.32488 nm and C = 0.52066 nm in the Standard Data (JCPDS: 06-0416). The crystallite sizes of the powders were estimated from X-ray line broadening using Scherrer’s Equation [30] (i.e., $D = \frac{0.89\lambda}{\beta\cos\theta}$), where λ is the wavelength of the X-ray radiation, k is a constant taken as 0.89, 2θ is the diffraction angle (30.286°), β is the Full Width at Half Maximum (FWHM = 0.4723) and is obtained to be 19.25 nm for In$_2$O$_3$ sample calcined at 500°C. The particle size and lattice parameter of In$_2$O$_3$ sample is summarised in Table 1.
Figure 3. XRD pattern of nano crystalline In$_2$O$_3$ sample calcined in air for 2 hours at 500°C.

<table>
<thead>
<tr>
<th></th>
<th>Average Particle size (nm) from XRD</th>
<th>Cubic lattice parameter (nm)</th>
<th>Morphology from SEM</th>
<th>Estimated Band gap UV PL</th>
<th>PSA</th>
<th>EDAX</th>
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<th></th>
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<tbody>
<tr>
<td></td>
<td>19.25 nm</td>
<td>1.0222 nm</td>
<td>Spherical</td>
<td>3.86 eV</td>
<td>24 nm</td>
<td>O/In = 2.2</td>
<td></td>
<td>93</td>
</tr>
</tbody>
</table>

3.3. EDAX & SEM Analysis

The SEM images in Figure 4 showed that the In$_2$O$_3$ nanoparticles are formed as cubic crystals. However, the particle size as well as agglomeration increased and the smaller grains coalesced to form larger size particles [31]. The EDAX data at 500°C sintering temperature suggested the stoichiometry of In$_2$O$_3$ nanoparticles with the elemental composition of oxygen and Indium and their atomic and weight percentages are given Table 1. The atomic ratio of (O/In) calculated as 2.2 indicated the stoichiometric composition of In$_2$O$_3$, which is in good agreement with the theoretical value of 1.5.

3.4. PSA Analysis

The average particle size of the In$_2$O$_3$ nanoparticle from the particle size analyzer, as in Figure 5, was found to be 24 nm as shown in Table 1.

3.5. RAMAN Studies

The In$_2$O$_3$ Raman spectrum (Figure 6) shows the expected vibrational modes at 109, 475 cm$^{-1}$, which in turn is an unambiguous signature of the cubic In$_2$O$_3$ structure [32]. The higher frequency (1500 cm$^{-1}$) is due to super position of the contribution of the In-O vibration modes with frequency 630 cm$^{-1}$. The intensity peaks which are known to be related to the pure In$_2$O$_3$ vibrational modes which confirm the In$_2$O$_3$ cubic like feature.

3.6. FTIR Analysis

The IR spectra of In$_2$O$_3$ indicating that the bands around 3506 cm$^{-1}$ and 1623 cm$^{-1}$ are attributed to the absorptions of hydroxyls from absorbed water or alcohols and those at 1416 cm$^{-1}$ can be ascribed to the C-H vibrations of the organics in Figure 7. The band at 1050 cm$^{-1}$ is due to C-O vibrations. While the absorptions around 1500 cm$^{-1}$ are due to the In-O vibrations [33]. The weak absorption at 1568 cm$^{-1}$ is due to C-O vibrations from the...
Figure 4. EDAX & SEM images of the nano crystalline In$_2$O$_3$ sample.

Figure 5. Particle size analysis of the nano crystalline In$_2$O$_3$ sample.

<table>
<thead>
<tr>
<th>S.P. Area Ratio</th>
<th>Mean</th>
<th>S.D.</th>
<th>Mode</th>
</tr>
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<tbody>
<tr>
<td>1.00</td>
<td>24.3 nm</td>
<td>11.3 nm</td>
<td>17.9 nm</td>
</tr>
</tbody>
</table>

Figure 6. Raman Spectra of nanocrystalline In$_2$O$_3$ sample calcined in air for 2 hours at 500°C.
Acetylacetone species [34]. The results indicate the presence of few acetyl acetone species on the surface species of the In$_2$O$_3$ nanocrystals.

### 3.7. UV-Vis Absorbance

Now let us consider the optical properties of the In$_2$O$_3$ samples. The UV-visible absorption spectra of all the In$_2$O$_3$ samples (Figure 8) exhibit a strong absorption below 450 nm (2.7 eV) with a well defined absorbance peak at around 320 nm (3.88 eV). This value is greater than that of 3.6 eV for the In$_2$O$_3$ reported in the literature [2].

### 3.8. PL Spectrum

Figure 9 shows the room temperature PL spectra of the nanocrystalline In$_2$O$_3$ samples measured using a

![Figure 7. FTIR nano-crystalline In$_2$O$_3$ sample calcined in air at 500°C.](image)

![Figure 8. Room temperature optical absorbance spectra of nano crystalline In$_2$O$_3$ sample calcined in air for 2 hours at 500°C.](image)
Figure 9. Room temperature photo luminescence spectra of the synthesized nano crystalline In$_2$O$_3$ sample calcined in air for 2 hours at 500˚C.

Xenon laser of 270 nm as an excitation source. The spectra of all the samples mainly consist of a strong UV emission broad band having emission maximum at ~353 nm (3.51 eV). It is well known that the bulk In$_2$O$_3$ cannot emit light at room temperature [38]. However PL emissions of our nano crystalline In$_2$O$_3$ samples are possibly due to the effect of the oxygen vacancies as reported in literatures [14] [17] [19] [20] [27] [28] [36]-[38]. In the present work, oxygen vacancies would generally act as deep defect donors and cause the formation of new energy levels in the band gap of In$_2$O$_3$ samples. Thus the PL emission from In$_2$O$_3$ nanoparticles results from the radiative recombination of electron occupying oxygen vacancies with a photo excited hole which is analogous to the photo luminescence mechanism of ZnO and SnO$_2$ semi conductors [19] [38].

4. Conclusion

We have synthesized nanoparticles of In$_2$O$_3$ by a green method using Astragalus gummifer. Structural, morphological chemical composition and optical properties of the green synthesized nanoparticles were characterised. XRD, RAMAN, SEM analysis showed that the In$_2$O$_3$ samples were cubic with particle size of 19 nm. The morphology and size of In$_2$O$_3$ materials were affected by the calcination temperature. The prepared In$_2$O$_3$ nanoparticles showed a strong PL emission in the UV region. The strong emissions of In$_2$O$_3$ are attributed to the radiative recombination of electron occupying oxygen vacancies with a photo excited hole.

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Nanocrystalline In(OH)₃ to In₂O₃. Inorganic Materials, 41, 609-612. [http://dx.doi.org/10.1007/s10789-005-0178-x]


