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Original Research Paper

# Optical, electrical properties, characterization and synthesis of $Ca_2Co_2O_5$ by sucrose assisted sol gel combustion method

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ABSTRACT

Sol–gel combustion method has been used as an efficient and simple method to synthesize pure $Ca_2Co_2O_5$ (CCO-225) ceramic powder using sucrose which play a dual role as the gelling agent and combustion fuel. The advantage of this method is simple low cost and environmental friendly. The synthesized sample is sintered at various temperatures the products were characterized by powder X-ray diffraction (XRD), Thermogravimetric and differential thermal analysis (TGA-DTA), Fourier transformer infrared spectroscopy (FTIR), Scanning electron microscope (SEM) and UV–Visible diffuse reflectance spectroscopy (DRS). X-ray diffraction pattern of sintered sample at 800 °C confirmed the formation of single phase $Ca_2Co_2O_5$ and also it is proved in thermal analysis. SEM image indicates the obtained samples are diffused platelet like morphology and its grain size will be in the range of 150–300 nm. CCO-225 ceramic material has a wide range of optical and electronic applications due to its wide band gap energy of 3.50 eV. The dielectric constant, dielectric loss and AC conductivity were analyzed at different temperatures and frequencies of the applied field. The AC conductivity studies carried out in the frequency range of 50 Hz to 5 MHz at various temperatures from 30 °C to 400 °C. The result reveals that the space charge polarization leads for conduction mechanism.



## 1. Introduction

The layered calcium cobalt oxides ($Ca_2Co_2O_5$) have been used as electrical, optical, magnetic, superconducting and thermoelectric material [1–8]. Funahashi et al. reported that the single crystal of $Ca_2Co_2O_5$ in whisker structure have application in thermoelectric power generation [5]. Typically, $Ca_2Co_2O_5$ powders are synthesized by a solid state reaction between the individual component of calcium carbonate and cobalt oxide powders. This method generally requires repeated mechanical mixing and extensive heat treatment at high temperatures to achieve the desired phase purity. It has been accepted that the wet-chemical process offers advantages of good mixing of the starting materials and excellent chemical homogeneity of the final product [8–10].

By contrast with traditional solid state reaction, the sol–gel method is an efficient way to decrease the atom diffusion distance and to obtain the pure phase in a lower temperature range [11–13]. In sol–gel method, the reactant cations are intimately mixed on the atomic scale, so the rate of the reaction will be increased; leading to lower synthesis temperatures and smaller particles [12]. The soluble sucrose is a low-cost, abundant, renewable source and thus fulfills environmentally aware chemistry demands [13]. Sucrose has a dual role during the synthesis as fuel and gelling agent [14,15]. For practical applications, sol gel process that can introduce high strains into materials is highly desirable for reducing the production costs and times [16–18].

$Ca_2Co_2O_5$ is a well-known material of its electronic and electro-optic properties. Its optical properties have attracted considerable attention and many experimental and theoretical reports have been published on this CCO-225 bulk material. In the present investigation, for the first time we are reporting the synthesized $Ca_2Co_2O_5$ by sucrose assisted sol–gel combustion method and the study of electrical, optical and dielectric properties. The main advantage of sol–gel combustion method is using sucrose as a less toxic fuel and water as a solvent and single phase $Ca_2Co_2O_5$ product obtained at low temperature. The AC conductivity studies carried out in the frequency range of 50 Hz–5 MHz at various temperatures from 30 °C to 400 °C. The result reveals that the space charge polarization leads for conduction. The AC conductivity data have been used to estimate the apparent activation energy $E_a$ = 0.63 eV at a frequency of 5 kHz [20].

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## 2. Materials and methods

### 2.1. Experimental procedures

$Ca_2Co_2O_5$ prepared by sucrose assisted sol gel combustion method can be represent in a simple scheme as in Fig. 1. Polycrystalline samples of $Ca_2Co_2O_5$ were synthesized by the sol–gel cum combustion method using sucrose as a gelling agent and combustion fuel. The starting materials were analytical-grade ($C_{12}H_{22}O_{11}$) sucrose, $Ca(NO_3)_2{\cdot}4H_2O$ and $Co(NO_3)_2{\cdot}6H_2O$. Aqueous solutions of the reactants were prepared according to the molar ratio of metal nitrates mixed and then sucrose added to the 1:1 fuel-oxidant ratio corresponding to the stoichiometry solutions were mixed. The resulting pink transparent solution was heated to a temperature in the range 80–120 °C with constant stirring to obtain the pink gel. Subsequently, it was decomposed at 350 °C for 0.5 h in a preheated furnace, resulting in a large voluminous fluffy mass of black precursor. This precursor was then heated at 800 °C for 2 h to obtain the single phase product by removing carbonaceous material.

### 2.2. Characterization technique

Thermogravimetry and differential thermal analysis of precursor was carried out with a SDT Q600 V20.9 model. The powders were characterized by powder X-ray diffraction technique (XRD, Bruker D8 Advance, by Cu Kα radiation, $k\alpha$ = 1.5406 Å). FTIR spectrums were examined using JASCO 400 Infrared spectrometer. The surface morphology and the microstructure were studied by a scanning electron microscope (HRSEM FEI Inspect F50) instrument. Dielectric measurements were studied by a LCR meter (HIOKI 3532-50LCR meter HITESTER) in the frequency range from 50 Hz to 5 MHz for variation of temperatures. Optical band gap obtained by using Jasco V-670-UV–Visible diffused reflectance spectrometer.

## 3. Results and discussion

### 3.1. Thermal analysis

Thermogravimetric analysis (TGA) is an important tool to determine the stable product formation and decomposition step of carbonaceous organic compound present in the precursor. Fig. 2. Shows the TGA-DTA results of precursor. TGA graph shows three decomposition step, first step the weight loss of about 9% due to evaporation of adsorbed water at 100–190 °C. The endothermic peak reveals an endothermic process with small intensity between 180 and 200 °C can be observed. This appears to be due to the initiation of sucrose melting (the melting point of sucrose is 186 °C) [12,21]. When the second step weight loss 22% in the temperature range of 260–550 °C due to decomposition of organic material it

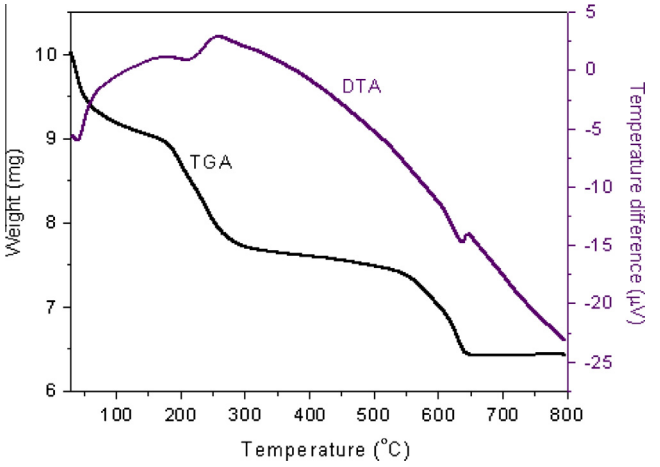


**Fig. 2.** TGA-DTA pattern of $Ca_2Co_2O_5$.

leads to form $CaCO_3$ and $Co_3O_4$ and the second endothermic peak at 630 °C due to decomposition of organic compounds it losses carbon dioxide. Finally, the third step weight loss of 10% due to decomposition of calcium carbonate in the temperature range of 800 °C it forms the stable $Ca_2Co_2O_5$ product it could be further proved by XRD. The TGA reaction governing the formation of $Ca_2Co_2O_5$ this can be represented in Fig. 3.

### 3.2. XRD analysis

Fig. 4 Shows the XRD pattern of precursor calcined at temperatures between 350–800 °C for 2 h. XRD pattern of 350 °C precursor shows the major 2$\theta$ peaks at 29.60°, it reveals the presence of calcium carbonate as impurity. After calcined at 600 °C for 2 h, $CaCO_3$ phase maximum intensity peak decreased. Increasing the temperature to 700 °C for 2 h it forms a CCO-225 product phase with amorphous less intense peak. Further increase the temperature to 800 °C for 2 h the XRD pattern proves the $Ca_2Co_2O_5$ single phase intense *C*-axis oriented planes (002) and (004) as orthorhombic crystal system and its lattice parameters $a$ = 11.1305 Å, $b$ = 10.7522 Å, $c$ = 7.4876 Å are consistent with those reported in JCPDS (card No. 37-0668) [3,7,19]. Using the XRD diffraction data, the crystallite sizes of samples were able to be estimated using the Scherer equation (1).

$$D = K\lambda/\beta\cos\theta \tag{1}$$

In this equation, $D$ is the crystallite size (nm); $K$ is the so-called shape factor, which usually takes a value of about 0.9; $\lambda$ is the X-ray wavelength; $\beta$ is the full width at half maximum of the diffraction peak at 2$\theta$ is the diffraction angle. Since the peak from the

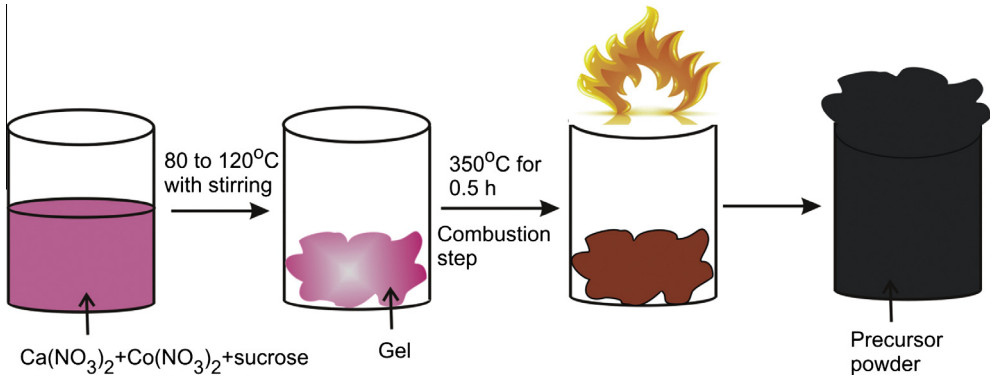


**Fig. 1.** Schematic representation of sol gel combustion synthesis of $Ca_2Co_2O_5$.