

## **SYNTHESIS AND CHARACTERIZATION OF CALCIUM TITANATE (CaTiO<sub>3</sub>) POWDER FOR PHOTOVOLTAIC APPLICATION**

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### **Abstract**

Calcium titanate (CaTiO<sub>3</sub>) powders with perovskite structure were synthesized by a co-precipitation method by using CaCO<sub>3</sub>, TiO<sub>2</sub> and NaOH as the starting materials. The effect of calcinations temperature in phase formation, morphology and particle size distribution of proposed powders were examined. The dried gels were calcined at 500°C, 600°C and 700 °C respectively. The Structural analysis was calculated by using X-ray Diffraction Method (XRD). The functional groups were examined by using Fourier Transformed Infrared Spectrophotometer (FTIR). The surface morphology was examined by Scanning Electron Microscopy (SEM). The crystallite phase and the morphology of CaTiO<sub>3</sub> were highly influence by the calcinations temperature.

**Keywords:** nanopowder, CaTiO<sub>3</sub>, co-precipitation

### **Introduction**

Among the metal oxide, titanium dioxide (TiO<sub>2</sub>) is well known n-type semiconductor and non-toxic, cheap, highly efficient photovoltaic material. It has three crystalline forms anatase, rutile and brookite. Different phases have different energy band gap. So it is extensively use in various applications like photoconductors, dielectric materials and solar cell etc . It is also use as an ingredient in sunscreen lotions and food products as a pigment. Calcium titanium oxide or Calcium titanate (CaTiO<sub>3</sub>) is the member of perovskite structure (ABO<sub>3</sub>) family and it was discovered by Gustav Rose in 1839 and named in honour of the Russian mineralogist Count Lev Alekseevich Perovski (1792-1856) . CaTiO<sub>3</sub> is ferroelectric with an orthorhombic phase at room temperature and at higher temperature 870K it exhibits tetragonal structure and at around 1273K it is transformed to cubic structure . Nowadays due to world high population global demand for energy is increasing rapidly. So the researchers are interested the photovoltaic perovskite solar cell to solve energy problem. CaTiO<sub>3</sub> is widely used in various electronics devices as a result of dielectrics, photo luminescent, photovoltaic and electronic conduction properties. Moreover there is no need for extremely high temperature and long reaction time producing CaTiO<sub>3</sub> powders possible for large scale synthesis. Due to these distinctive properties and numerous uses, CaTiO<sub>3</sub> has lots of attraction among the researchers. CaTiO<sub>3</sub> powders can be synthesized by several methods such as solid state reaction method, chemical co-precipitation method, wet chemical method (so-gel), hydrothermal technique, milling, physical and chemical vapour processing, precipitation from a precursor solution in a strong alkaline water, chemical bath deposition and many others. Although all these methods have their own advantage and most of them need high temperature and pressure. Among of them the co- precipitation method is very practical, low cost, simple and precise control of the composition so we have been used this method for the synthesis of calcium titanate (CaTiO<sub>3</sub>) powders.

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In this work the synthesis of  $\text{CaTiO}_3$  powder is carried out through the mixtures containing titanium dioxide, calcium carbonate, sodium hydroxide as precursor and the effect of calcinations temperatures on the structural properties are operating.

### **Experimental Procedure**

$\text{CaTiO}_3$  powders were synthesized by co-precipitation method. Calcium carbonate ( $\text{CaCO}_3$ ), titanium dioxide ( $\text{TiO}_2$ ) and sodium hydroxide ( $\text{NaOH}$ ) were used as starting materials. 0.2 mol of  $\text{NaOH}$  was dissolved in 100ml distilled water and 0.1 mol of  $\text{CaCO}_3$  was added to the  $\text{NaOH}$  solution. Then the mixture was magnetically stirred for one and half hours and added to 100ml distilled water and 0.1 mol of titanium dioxide solution. The mixture was magnetically stirred for 3 hours. The mixture has been filtered with filter paper for removing water. The resulting precipitations were kept at room temperature for 24 hours. Final products were dried in hot air oven at 80 °C for 15 hours. The dried precipitations were collected and grounded in an agate motor for 2 hours. The resulting synthesized powder were calcined at 500 °C, 600 °C and 700 °C for 2 hours in a muffle furnace and as synthesized  $\text{CaTiO}_3$  powders were obtained.

### **Result and Discussion**

#### **XRD Analysis**

The structural properties of  $\text{CaTiO}_3$  powder were studied by X-ray diffraction from which significant differences in the diffraction patterns of each calcined temperature at 500°C, 600°C and 700°C, as shown in Figure 1 (a-c). At calcined temperature 500°C of  $\text{CaTiO}_3$  powder was still dominated by the phase of the starting materials especially  $\text{TiO}_2$ . Phase of  $\text{CaCO}_3$  is also appeared with weak intensity. At calcined temperature 600°C the sample was also needed to cause a complete reaction to produce crystalline phase of  $\text{CaTiO}_3$ . But furthering calcined temperature at 700°C the perovskite  $\text{CaTiO}_3$  phase were formed. At that temperature the dominated phase of  $\text{CaTiO}_3$  pattern indicated by the characteristic with very high intensity and pure perovskite  $\text{CaTiO}_3$  were formed, as shown in Figure 1(c). All of  $\text{CaTiO}_3$  peaks appeared in diffraction pattern as can be identified at an angle ( $2\theta$ ) of 33.02°, 47.46° and 59.16° corresponding to the phase of diffraction (h,k,l) respectively to (020), (024), (132) and (204). All the peaks in XRD pattern match well with reported characteristic reflection peaks of  $\text{CaTiO}_3$ JCPDS file No, (01-089-8033). The crystallite size have been estimated from the X-ray peak of (h,k,l) diffraction using Scherrer formula. The average crystallite size of  $\text{CaTiO}_3$  calcined powder at 500°C, 600°C and 700°C were 39 nm, 36 nm and 26 nm respectively. As the calcinations temperature increased the crystallite size also decreased.

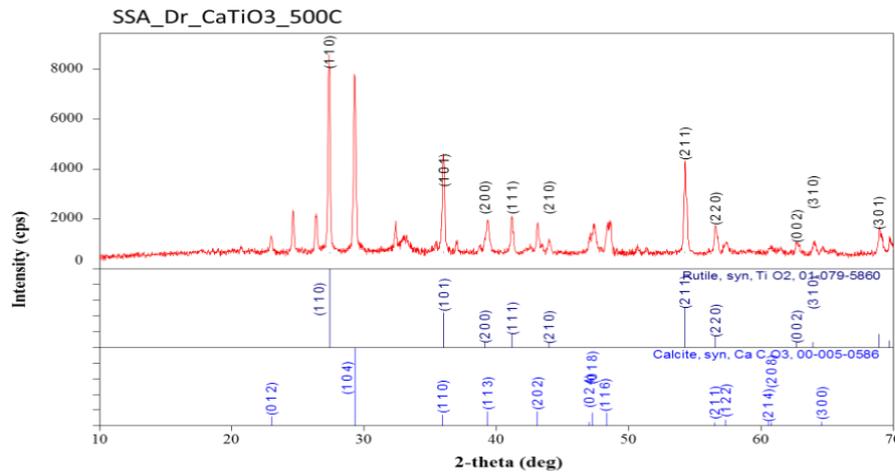


Figure 1 (a) XRD Pattern of CaTiO<sub>3</sub>powders prepared at 500°C

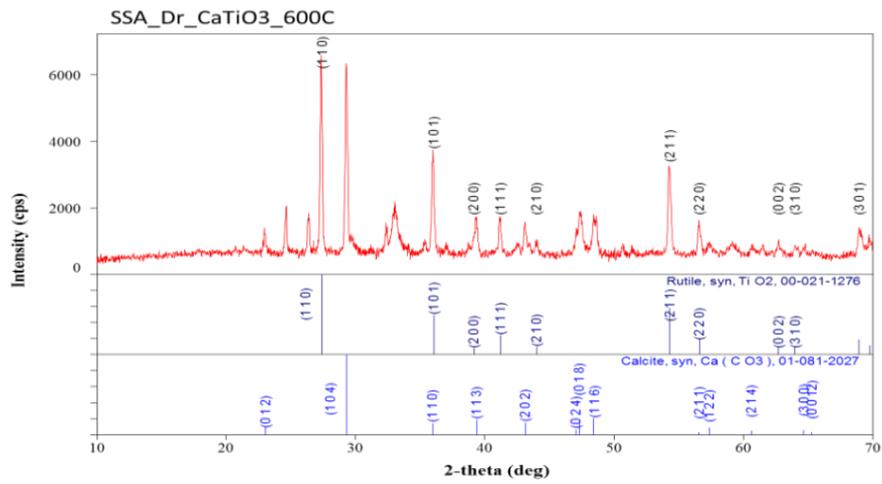


Figure 1 (b) XRD Pattern of CaTiO<sub>3</sub>powders prepared at 600°C

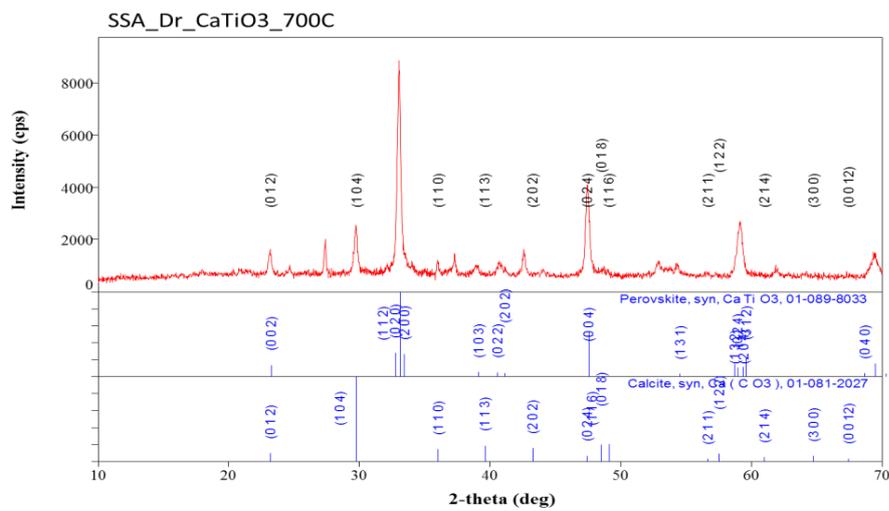


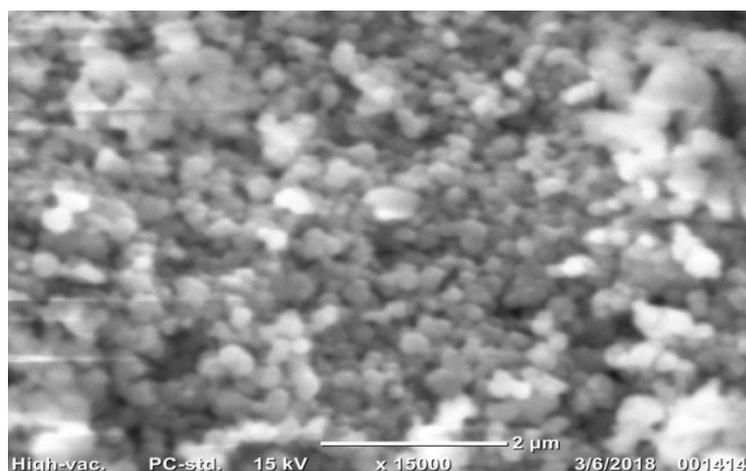
Figure 1 (c) XRD Pattern of CaTiO<sub>3</sub>powders prepared at 700°C

## SEM Analysis

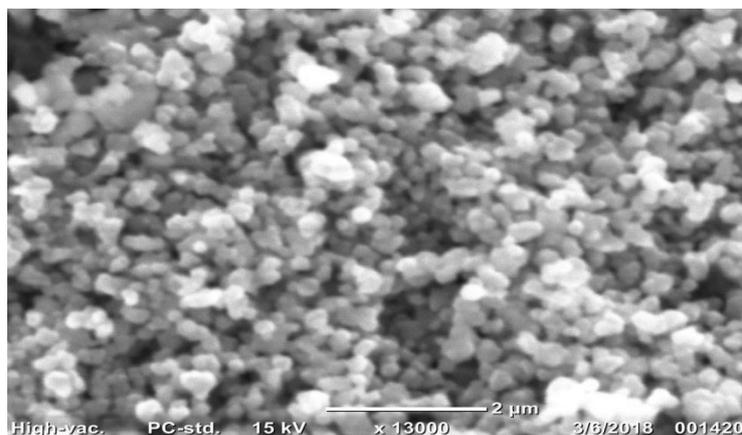
Calcium titanate nanoparticles were obtained by calcinations of the gels of the composite carbonate. Figure 2(a-c) shows the SEM analysis of  $\text{CaTiO}_3$  particles sintered at  $500\text{ }^\circ\text{C}$  –  $700\text{ }^\circ\text{C}$  exhibit grained microstructure and nanostructure with small crystallite size. The grain sizes were calculated by using well known bar code system. The average grain size of  $\text{CaTiO}_3$  particle was found to be about  $0.21\text{ }\mu\text{m}$ - $0.28\text{ }\mu\text{m}$ . This figure indicated that most of the grain size was regular structure and a few number of large grain size were found. It looks fairly dense and rough.

This fact indicated that structural properties were influenced by different temperatures. From the images, it was clearly found that the little amount of pores and grain growth were examined with the increase in process temperatures. The orientation of grain was towards left for all images. The grain size of  $\text{CaTiO}_3$  particles seen to be uniform but some of the grain size were slightly large. From detail analysis of figure 2(a) showed the dense particle with fine grains and without any cracks calcined at  $500\text{ }^\circ\text{C}$ .

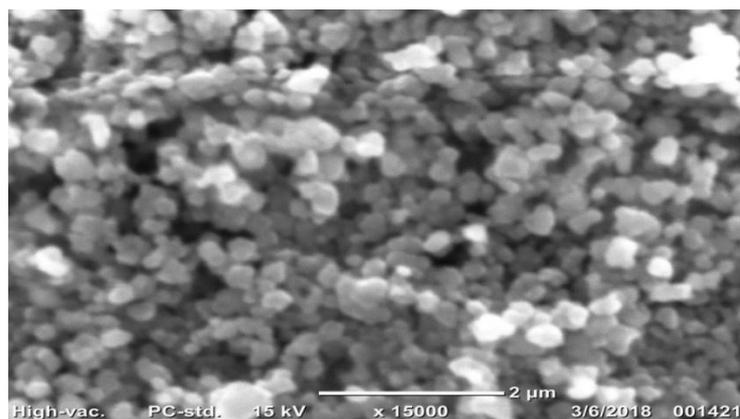
In figure 2 (b), the SEM photograph was examined to be non-uniform and crack free, this photo also indicated that the particles were dense, smooth and fine grain at  $600\text{ }^\circ\text{C}$ . SEM studied on the particle at  $700\text{ }^\circ\text{C}$  was represented at figure 2(c), this figure indicated the dense particle with fine grains, uniform and smooth but some pores were observed. All figures exhibited the smooth in morphology.



**Figure 2 (a)** SEM photograph of  $\text{CaTiO}_3$  powders prepared at  $500\text{ }^\circ\text{C}$



**Figure 2 (b)** SEM photograph of  $\text{CaTiO}_3$  powders prepared at  $600^\circ\text{C}$



**Figure 2 (c)** SEM photograph of  $\text{CaTiO}_3$  powders prepared at  $700^\circ\text{C}$

### FTIR Analysis

FTIR analysis were performed for the powder sample calcinations temperature at  $500^\circ\text{C}$ ,  $600^\circ\text{C}$  and  $700^\circ\text{C}$  in Fig (3) (a-c). The band around  $655.82\text{ cm}^{-1}$ ,  $653.89\text{ cm}^{-1}$ ,  $601.81\text{ cm}^{-1}$ ,  $590.24\text{ cm}^{-1}$ ,  $580.59\text{ cm}^{-1}$ ,  $569.02\text{ cm}^{-1}$ ,  $418.57\text{ cm}^{-1}$ ,  $428.21\text{ cm}^{-1}$  and  $443.64\text{ cm}^{-1}$  in Figure 3(a-c) were possibly caused by the stretching vibration due to the interactions produced between the oxygen and metal bonds. The broad band's at  $873.78\text{ cm}^{-1}$  in Figure 3(a) and (b) were both assigned to the symmetric stretching of C-H bands in aromatic functional group. The bands at  $1161.19\text{ cm}^{-1}$ ,  $1168.90\text{ cm}^{-1}$ ,  $1118.75\text{ cm}^{-1}$  and  $1068.60\text{ cm}^{-1}$  in Figure 3(a-c) were C-O stretching vibration of carboxylic acid group. The C-H stretching band was occurred at  $1442.80\text{ cm}^{-1}$ ,  $1452.45\text{ cm}^{-1}$ ,  $1427.37\text{ cm}^{-1}$  and  $1413.87\text{ cm}^{-1}$  and  $1440.87\text{ cm}^{-1}$  in alkanes functional groups. Another C-H stretching band was also found at  $2982.05\text{ cm}^{-1}$  and  $2980.12\text{ cm}^{-1}$  of Figure 3(a) and (b) in alkanes group. The O-H stretching band was occurred in alcohol group at  $3661.01\text{ cm}^{-1}$ ,  $3659.09\text{ cm}^{-1}$  and  $3641.73\text{ cm}^{-1}$  in Figure 3 (a-c).

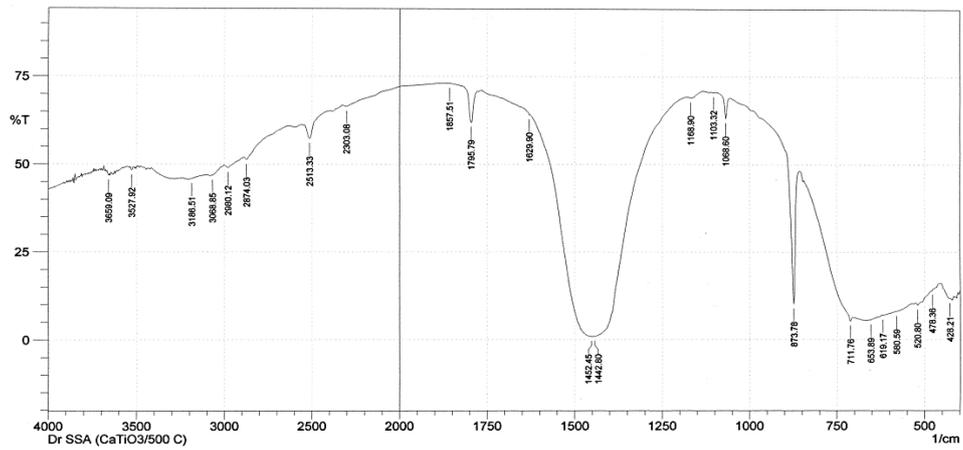


Figure 3 (a) FTIR spectra of CaTiO<sub>3</sub> powders prepared at 500°C

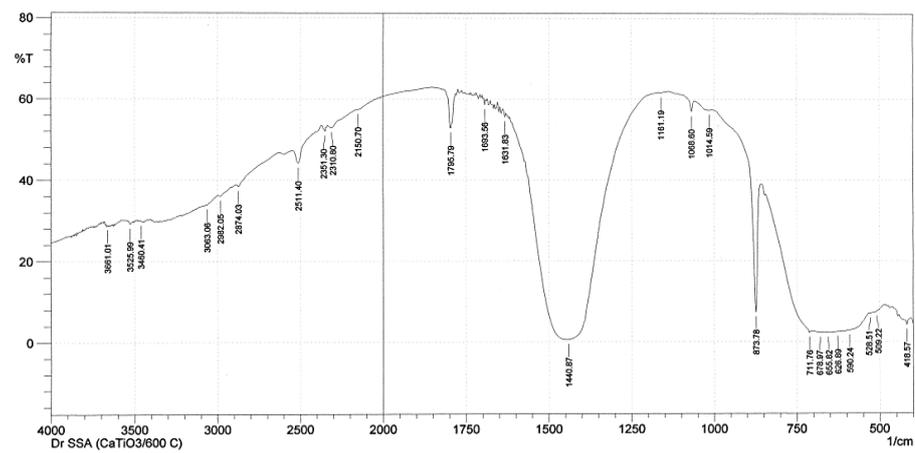


Figure 3(b) FTIR spectra of CaTiO<sub>3</sub> powders prepared at 600°C

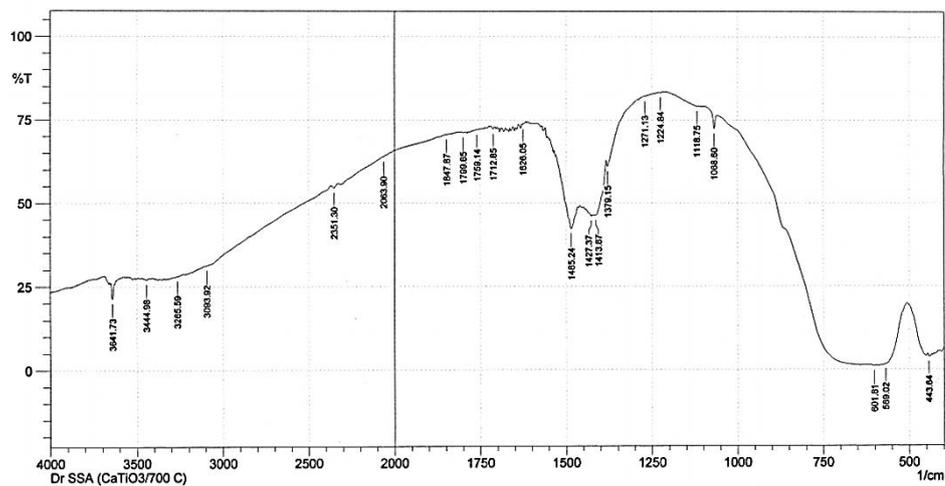


Figure 3 (c) FTIR spectra of CaTiO<sub>3</sub> powders prepared at 700°C

## Conclusion

Calcium titanate ( $\text{CaTiO}_3$ ) powders have been successfully synthesized by co-precipitation method, its surface morphology and structural properties have been studied. From this studied, the desire phase and crystalline size was obtained at 700 °C calcinations temperature. The XRD analysis showed that, as the temperature increase, the intensity of the peaks were clear and sharp. As a result, XRD analysis confirmed that the formation of pure  $\text{CaTiO}_3$  powders with perovskite structure. The SEM result showed that the higher the calcinations temperature, the greater the grain size due to solid-grain diffusion. According to FTIR analysis, all temperatures of  $\text{CaTiO}_3$  is suitable for their absorption band due to C-H bands in aromatic functional group, O-H stretching band in alcohol functional group and C-O stretching vibration of carboxylic acid group. According to the experimental results,  $\text{CaTiO}_3$  powder can be used in photovoltaic and thin films applications.

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