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A study of thermal, dielectric and magnetic properties of strontium malonate crystals

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ABSTRACT

Crystals of strontium malonate ($SrC_3H_2O_4$) were grown in silica gel by the single diffusion technique. The thermo gravimetric (TG), differential thermal analysis (DTA) and differential scanning calorimetric (DSC) studies were carried out to investigate the thermal stability of the crystal. The dielectric behavior of the title compound crystal was investigated by measuring the dielectric parameters – dielectric constant, dielectric loss and AC conductivity as a function of four frequencies – 1 kHz, 10 kHz, 100 kHz and 1 MHz at temperatures ranging from 50 to 170 °C. Results indicate that the title compound is thermally stable up to about 409 °C and is a promising low ε_r-value dielectric material. The magnetic behavior of the crystal was also explored using a vibrating sample magnetometer.

1. Introduction

Strontium malonate and other strontium salts are widely used as ingredients for pharmaceuticals, vitamins and nutritional supplements. Malonate complexes of strontium exhibit remarkable magnetic properties [1,2]. The three dimensional structural network aspects of strontium malonate anhydrate are reported by Kenny Stahl and co-workers [3]. Strontium malonate and other metal malonates are generally prepared by the precipitation technique. The authors have reported the growth and spectroscopic characterization of strontium malonate crystals grown by the gel technique [4]. Growth of crystals in gel is the most versatile and inexpensive technique. Further, it is a self-purifying process, free from thermal strains, which is common in crystals grown from the melt [5].

Malonate, the dianion of 1,3-propanedioic acid can function as a versatile bridging ligand. The intriguing structural complexity of malonate is associated with the simultaneous adoption of chelating bidentate or unidentate and the different bridging coordination modes like syn–syn, syn–anti and anti–anti. The ability of the bridging ligand to mediate magnetic coupling between the paramagnetic centers that it links plays a fundamental role in the magnetic behavior of metal malonates. Thus malonate ligand is a promising candidate in designing extended magnetic networks [6–9].

Thermal studies of transition metal malonates and malonic acid have been reported [10,11]. But the reports related to dielectric studies are scanty in literature [12,13]. In this report, we present the thermal, dielectric and magnetic studies of strontium malonate crystals grown by the gel method.

2. Materials and methods

Strontium malonate crystals were grown by the gel technique using the single diffusion method. Silica gel was prepared by mixing an aqueous solution of sodium meta silicate of density 1.033 g/cc with 1 M malonic acid solution. After the gel was set, strontium chloride solution (0.5 M) was slowly poured over it. Transparent crystals of strontium malonate were grown after a period of six weeks. The growth details are discussed elsewhere [4]. Powdered samples were used for X-ray diffraction studies using a Philips Pan analytical X'pert-Pro diffractometer with Cu-Kα radiation in 2θ ranging from 0 to 70°. The measured X-ray diffraction (XRD) profile of the title compound is shown in Fig. 1.

Thermo gravimetric (TG) and differential thermal analysis (DTA) runs of the crystal were taken in nitrogen atmosphere at a heating rate of 10 °C per minute on Perkin Elmer Diamond thermal analyzer in the temperature range of 40 to 800 °C. Differential scanning calorimetric (DSC) curve of the title crystal was recorded using a Mettler Toledo DSC 822e instrument in the temperature range of 40–550 °C. The dielectric constant (ε_r) and dielectric loss ($\tan\delta$) were estimated using an Agilent 4284-A LCR

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meter [14,15]. The crystal was powdered and pelletized to a diameter of 13 mm. The opposite faces of pellet were coated with graphite in order to obtain good ohmic contact. Using the LCR meter, the capacitance of the crystal was measured for frequencies 1 kHz, 10 kHz, 100 kHz and 1 MHz at various temperatures ranging from 50–170 °C. The dielectric constant of the crystal was calculated using the expression $\varepsilon_r = C_{crys}/C_{air}$, where C_{crys} is the capacitance of the crystal and C_{air} is the capacitance of same dimension of air.

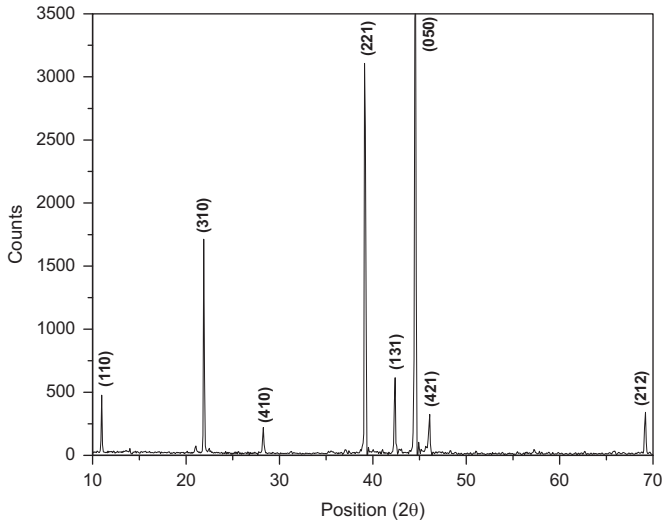

Fig. 1. XRD pattern of strontium malonate crystals.

The magnetic behavior of the material was studied using a vibrating sample magnetometer, model Lakeshore 7300 VSM [16] at room temperature with a maximum applied magnetic field of 10 kOe.

3. Results and discussion

3.1. Thermal studies

Fig. 2 (a–c) shows TG, DTG and DTA curves for strontium malonate crystal at a heating rate of 10 °C/min. The thermal studies indicate that the title compound is thermally stable up to about 409 °C. There is only one stage of decomposition as the compound is anhydrous and crystalline [17]. DTA shows an endotherm at 412.36 °C corresponding to DTG peak at 409.39 °C with a shoulder in DTG at about 420 °C. Since any intermediate formed could not be detected, so the shoulder is not attributed to any stable intermediate species but to a change in pyrolysis of gaseous decomposition products only or to a phase transformation. TG shows a change in slope at a mass loss of 15%. It may be due to the removal of one CO molecule. The end product formed is strontium carbonate with a mass loss of 22.15%.

Fig. 2d shows the DSC curve for strontium malonate in nitrogen atmosphere at a heating rate of 10 °C/minute. The endotherm at 411.35 °C corresponds to the DTA curve at 412.36 °C.

3.2. Dielectric studies

Fig. 3 shows the variation of dielectric properties of strontium malonate crystal with temperature at four different frequencies, viz. 1 kHz, 10 kHz, 100 kHz and 1 MHz.

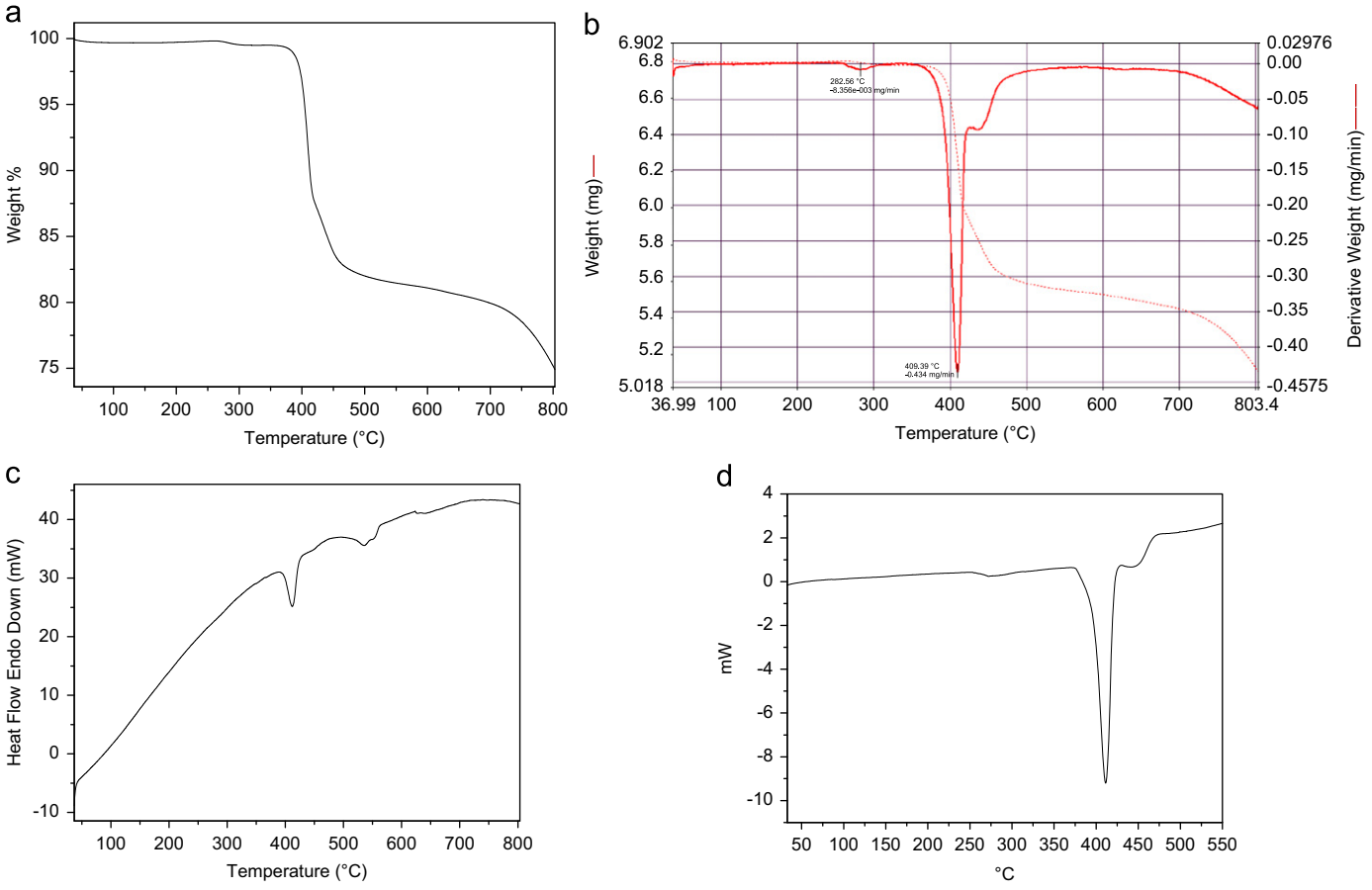

Fig. 2. TG, DTG, DTA and DSC of strontium malonate crystal. (a) TG profile. (b) DTG profile. (c) DTA profile. (d) DSC profile.