Exponential Business and Technologies Company

Bridge You and Nano

Thermogravimetric Analysis of Calcium Oxalate

Thermogravimetric analysis (TGA) is a type of analysis that determines the mass change of a sample over time as it is heated. This analysis requires that the test instrument be able to accurately measure mass, temperature, and temperature change. Typically, samples are analyzed in an inert atmosphere although an oxidizing or reducing atmosphere can be used when necessary. TGA is widely employed in research and development, testing and characterization of all kinds of materials from metals and alloys to polymeric and ceramic composites. Typical applications of TGA may include determination of polymer degradation and decomposition temperatures, moisture content of materials, oxidation resistance and dynamics, volatile and non-volatile components, thermal stability, etc.

Thermal decomposition is the process in which a substance decomposes due to the application of heat. The phenomenon is common to most organic substances and occurs in many inorganic substances as well. Certain substances can undergo multiple decomposition reactions, each at a different temperature. Using thermogravimetry, the decomposition temperature and mass loss of each reaction can be determined.

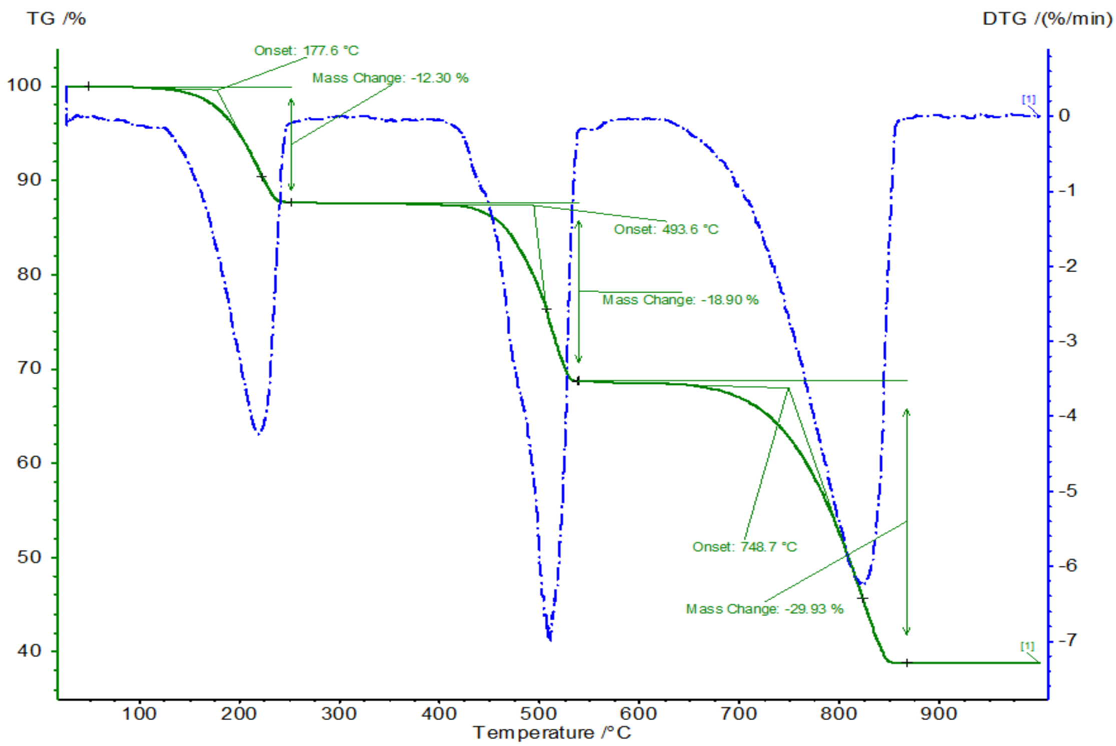

Figure 1. Thermal Decomposition of Calcium Oxalate Monohydrate.

Calcium Oxalate Monohydrate, CaC_2O_4•H_2O, is a useful industrial compound used to make oxalic acid, organic oxalates, and glazes. Thermal decomposition data for CaC_2O_4•H_2O was obtained using a Simultaneous TG-DTA/DSC Apparatus STA 449 F3 Jupiter (Netszch, Germany).

Thermal decomposition of CaC_2O_4•H_2O occurs in three distinct steps, as can be seen in Figure 1. The theoretical mass loss during each step can be calculated using the molar masses of the individual components. Table 1 shows the decomposition reactions that occur at each step as well as the theoretical and measured mass loss for each step.

Table 1 Thermal Decomposition Reactions of Calcium Oxalate Monohydrate

Step	Reaction	Theoretical Mass Loss	Measured Mass Loss	% Error
1	CaC_2O_4•H2O → CaC_2O_4 + H_2O	12.33%	12.30%	-0.24%
2	CaC_2O_4 → $CaCO_3$ + CO	19.17%	18.90%	-1.41%
3	$CaCO_3$ → CaO + CO_2	30.12%	29.93%	-0.63%

The measured mass losses during steps 1 to 3 closely match the predicted mass losses, which verify the theoretical predictions of the thermal decomposition for the calcium oxalate monohydrate. It is interesting to note the slight difference in the mass loss between theoretical calculation and actual measurement for decomposition in step 2. According to research, this is most likely due to disproportionation (a type of redox reaction during which a reactant is simultaneously oxidized and reduced, thus forming two different products) of CO into CO_2 and carbon. This disproportionation is highly dependent on the impurities within the sample as well as the cleanliness and material of the sample holder.