## SIMULTANEOUS THERMAL ANALYSIS (TGA-DTA, TGA-DSC)

Thermal Analysis is the measurement of changes in physical properties of a substance as a function of temperature while the substance is subjected to a controlled temperature program. Of the many techniques, Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC), and Thermogravimetric Analysis (TGA) are the most common.

Typically, a sample is heated at a linear rate and the temperature is continuously recorded so that the temperature range of any reaction can be accurately measured. In DTA or DSC, the temperature of a specimen is compared to a thermally inert material so that heat absorption, during endothermic reactions, or heat emission, during exothermic reactions, is recorded. In TGA, the weight of a specimen is continuously monitored to record any weight change of the material over temperature or time in a specified atmosphere.

Interpretation of the thermal pattern can provide a wealth of information about a material. Simultaneous TGA-DTA or TGA-DSC provides information about what type of reaction is taking place:

| Observation<br>Endothermic<br>weight loss | <ul> <li><u>Reaction Example</u><br/>Free H<sub>2</sub>O</li> <li>a) <u>Hygroscopic - water</u> retained by surface tension within a<br/>powdery mass. Loss occurs from Room Temperature (RT) -<br/>200°C.</li> </ul>  |
|---|--|
|   | <ul> <li>b) <u>Interstitial - water</u> retained in or occurring in interstices that<br/>are defined as small, fine cracks, pores, between parts or<br/>things of a powdery mass. Loss usually occurs from 75-<br/>600<sup>o</sup>C (in some cases up to 1000C)</li> </ul> |
|   | <u>DEHYDRATION</u> (Water of Crystallization, Chemically Bound<br>Water) - H <sub>2</sub> O molecules retained within the crystal lattice.   |
|   | DEHYDROXYLATION (Basic, OH-) - negatively charged OH-<br>present in the crystal lattice. A dehydroxylation can occur<br>from RT-1600°C.  |

DECARBOXYLATION - CO2 loss, for example from CaCO3

| Exothermic weight loss              | Oxidation with evolved gas, e.g., organic combustion            |
|-------------------------------------|---|
| Exothermic weight gain              | Oxidation consuming a gas, e.g., Ca to CaO                      |
| Endothermic,<br>no weight<br>change | Melt; Solid state reaction, Crystal transformation or formation |
| Exothermic, no                      | Solidification, solid state reaction, crystal transformation or |
| weight change                       | formation   |

A wide variety of conditions are available:

Atmosphere - air, nitrogen, argon, carbon dioxide, nitrogen/carbon dioxide mixture, vacuum

Heating Rate - from 0.2 to 25°C per minute

Temperature Range - 25 to 1650°C

The temperatures are accurate to  $\pm$  5° C and the weight measurement is accurate to  $\pm$  0.01%.

<u>Sample size</u> - Less than a gram of material is generally a sufficient. Larger sample size provides sufficient material for further study by x-ray diffraction or microscopy. This combination of instrumental techniques (simultaneous TGA-DTA and TGA-DSC) can accurately characterize complex systems.

A sample can be a powder, slurry, paste, liquid, paper, metal chip, metal foil, plastic piece, or a small aggregate.

In summary, Thermal Analysis is a versatile technique for studying materials as well as a research technique to investigate reactions, reaction rates, and optimum thermal conditions.

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