

Application Note

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Macro Thermogravimetric Analysis **of Epoxy Resins and Filler Content**

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Abstract

The ability to precisely measure minute amounts of moisture and/or volatiles, by thermogravimetric analysis, can be vital to the production of a quality product. To accurately accomplish this, a large sample should be used. This allows for more surface area to be presented. This also allows for a more representative sample to be used. The Shimadzu TGA-51 allows the user to analyze a sample with a weight of up to ten (10) grams. The larger sample size facilitates accuracy and precision in weight losses of less than one percent ($>1.0\%$). The samples analyzed included glasses, films, fibers, and resins. In each sample, repeatable minute weight changes are detected that were not readily observed on a standard TGA.

Introduction

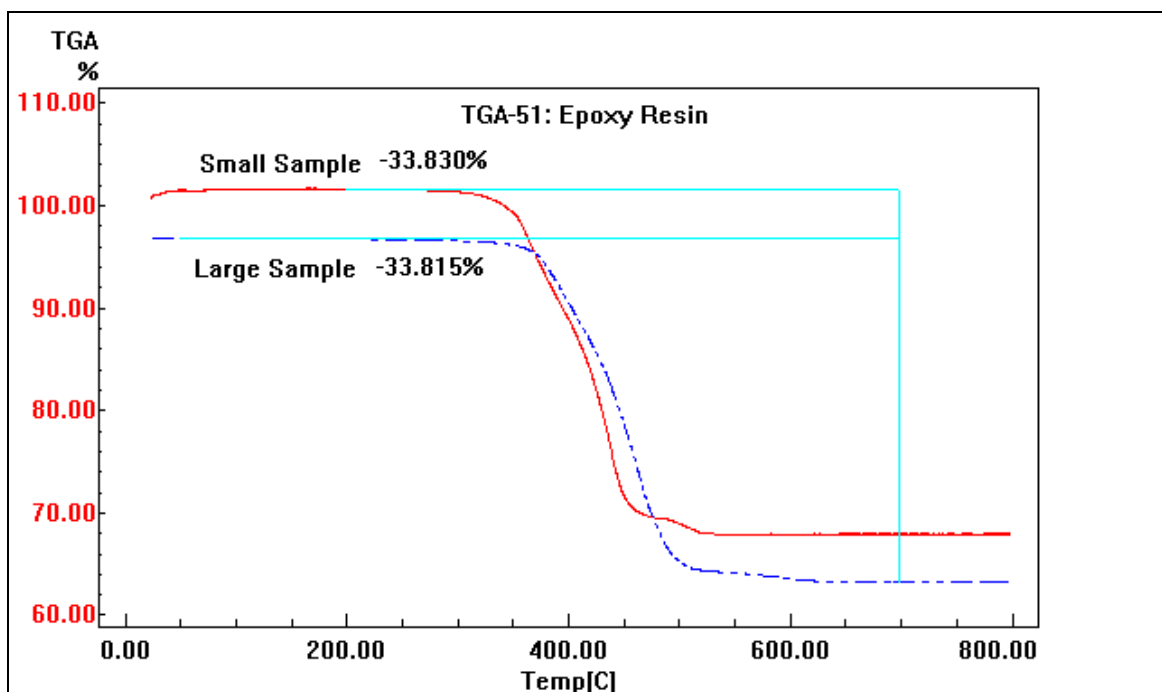
Theoretically, Thermogravimetric Analysis (TGA) measures changes in weight that occur to a sample as a function of temperature ($^{\circ}\text{C}$) over time. These changes usually occur as a loss in weight, but a gain in weight can be seen if the sample goes through an adsorption (i.e. oxidation). The ability to measure these changes in a larger sample size (i.e. > 1 gram) is very vital in samples in which the surface moisture and/or volatiles compose less than one percent ($>1.0\%$) of the total sample weight. In many instances this result can be in the parts per million range. The larger sample cell that is utilized in the TGA-51 also allows the user a more representative sample and a better opportunity to accurately determine the percentage of minute materials in the sample.



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Experimental



Determining the amount of filler in epoxy resin is a common application. In this application, the percentage of quartz, which is used to strengthen the resin, is determined. Epoxy resin will begin to degrade at 300°C in the presence of air, and will be completely degraded by 600°C. In this application two (2) samples, cut from the sample block, were analyzed from ambient to 800°C at 20 °C/min with an air atmosphere flowing at 20 ml/min. The first sample is a relatively small sample of 21.97 mg, and shows a weight loss of 33.83% at 700°C. From this it can be determined that the percentage of quartz is 66.17%. The second sample was much larger, having a sample weight of 694.4 mg. The second sample had a weight loss of 33.815%, resulting in a quartz content of 66.185%. Resin can be difficult to cut, so getting a small sample can be difficult, and the quartz filler isn't always uniformly mixed in the resin. This was done to illustrate that any sample size will give accurate and precise results. Based on this premise, it can be concluded that a large sample can and should be used when analyzing resins.

Conclusions

As is evident in the applications discussed above, the TGA-51 can be a very useful tool in the measurement of minute moisture and/or volatiles. It is also well suited to the analysis of materials that are irregular in shape and do not allow for representative sampling. Its ability to accurately measure large samples, both in weight and volume, give the operator another tool to help determine a sample's chemical makeup. The ease of use and compact design of the TGA-51 will make it a valuable addition to any thermal laboratory.



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