Dispersion and Analysis of Magnetic Particles With The Elzone

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Determining particle size information of materials that have a magnetic attraction is a challenge to producers of magnetic products. The magnetic attraction causes particles to agglomerate and form new particles that do not represent the true particle size of the sample. Several methods have been proposed to disperse magnetic materials so they are less likely to agglomerate during analysis, but these methods are often accompanied by undesirable side-effects. One such method is to coat the particles with long chain organics such as sodium oleate. This works for small colloidal material, but presents a problem with materials used in permanent magnets. Another approach includes heating the sample and then cooling it again to remove the magnetic properties, but this can cause the sample to change particle size due to sintering.

Discussed here is another method that can be applied using the electrozone sensing technique of particle size analysis. This method, performed on the Micromeritics Elzone, was found to be useful in dispersing magnetic particles, yet does not produce the side-effects that accompany the other methods.

This technique is applicable for analyzing magnetic powder using the electrozone sensing principle, which requires a low concentration of particles dispersed in an electrolyte solution. During an Elzone analysis, the sample solution is placed in a sample vessel containing an electrode. An orifice tube (a tube that has a tiny, pinhole-shaped orifice through which particles can pass) is placed inside the sample vessel. Inside of the orifice tube is a second electrode. A steady electric current flows from the electrode in the sample vessel to the second electrode inside the orifice tube. The sample solution is pulled through the orifice by a partial vacuum applied to the exit end of the tube. A particle blocks part of the orifice while passing through it, displacing the conductive electrolyte. This creates a change or pulse in the otherwise steady voltage. The number of pulses reflects the number of particles, and the size of the pulse determines the volume, and therefore the particle size. The Elzone's analytical method simultaneously yields both size distribution and particle concentration per unit volume of carrier electrolyte.

Dispersion and Analysis of Magnetic Particles

The dispersion and analysis technique begins when a small portion of the sample is placed into about one cubic centimeter of pure commercial honey. The powder and the honey should be mixed well to obtain a homogenous suspension. A drop of the mix is then placed on a microscope slide. A second slide is placed half way over the first one. Next, the two slides are pressed together which creates a thin film that will spread out uniformly under the slides. Since the particles in the material between the slides is probably too concentrated, the two slides are then separatedby pulling them apart in a longitudinal plane, shearing the agglomerates apart. Another slide is placed over one of the coated slides and, again, the slides are pressed together and separated in the manner described above.. The compression and resulting shear disperses the magnetically attracted particles. The combined slides are then placed on a microscope and examined for the quality of the dispersion and concentration. If the concentration is too high, the above process must be repeated to reduce it. If the concentration is moderate and the dispersion is good, the exterior surfaces of the slides are rinsed with filtered water. The instrument is prepared for analysis and the two washed slides are separated. One is placed into the stirring electrolyte in the instrument platform and the analysis is begun.

This technique created the necessary conditions for analyzing magnetic particles with the Elzone. These conditions are:

- Loose agglomerates are broken.
- Individual particles in a moderately high concentration must be held at a finite distance from their neighbor.
- The quality of the initial viscous phase dispersion must be capable of being viewed under a 100X dark field microscope.
- The initial viscous phase dispersion must be stable for 15 minutes or more.

The final analytical aqueous particle suspension should not exceed a 1% coincidence rate at the instrument's detector orifice, as this condition is necessary to achieve a high resolution analysis. Also, large inter-particle distances within the circulating electrolyte are maintained.

Results and Conclusions

The success of this technique is based upon the fact that sample dispersion is achieved by shear forces and the material transmitting the shear holds the dispersed particles apart until they are analyzed. The particles on the slide which were originally in surface contact with the circulating electrolyte were slowly liberated from the dissolving honey into the electrolyte. The change in volumetric dilution vastly increased the inter-particle distances thereby promoting suspension stability for a long enough period to complete the analysis.

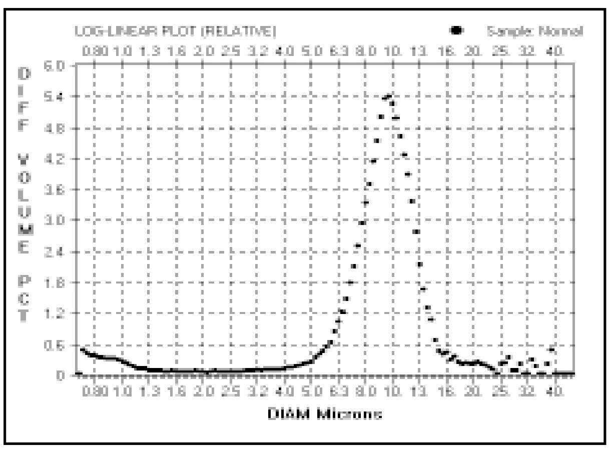

Figure 1. Particle size distribution of a ferrite material dispersed with normal technique (ultrasonic bath).

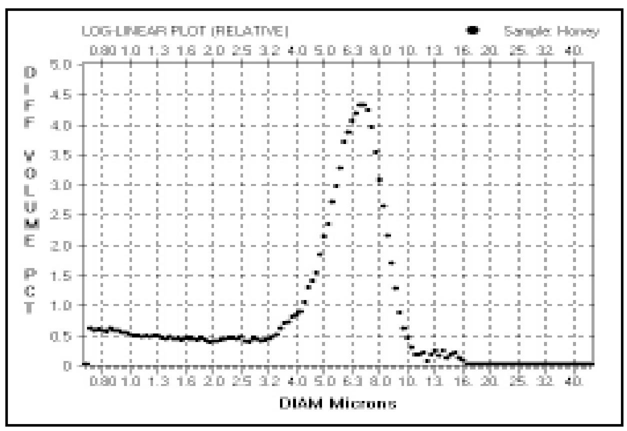

Figure 2. Particle size distribution of ferrite material dispersed with honey-dispersion technique.