Experiment Number: HS-2009----------- Beamline: ID01--

Starting date and time:07/07/03; 7:00 - Finishing date and time: 12/07/03; 7:00--------------

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Report

Introduction

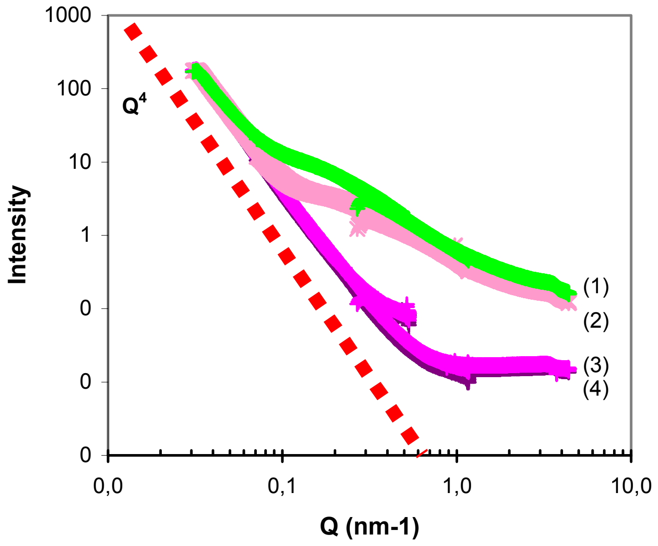
Magnetic nanocomposites are useful for a broad variety of applications including information storage, magnetic shields, printing, refrigerants, soft magnets, and, specially, for biomedical applications.[1,2] The benefits of magnetic nanocomposites in these areas is largely dependant of a control of crystalline phase, particle size, size dispersion, aggregation, and organization. Iron oxide nanoparticles are abundant in rock, soils and marine deposits, in the Earth and other planets. Therefore, the interpretation of their magnetic properties is of great interest for geological studies. Moreover, the influence of finite size, surface and shape on the magnetic moment and magnetic anisotropy of nanoparticles is a key issue in nanomagnetism.[3] We are involved in the development of: 1) methods of production of magnetic nanocomposites; and 2) models relating magnetic properties and structure. We have produced nanocomposites of: a) several iron oxide phases, b) several polymer matrixes, c) different particle sizes, and d) different particle shape. And, we have measured the magnetic properties of most of these samples. The characterization of all these samples by TEM would be extremely lengthy and costly. We have examined some of them by XRD, HRTEM, EELS, Mössbauer, IR, DTA, and SQUID magnetometer. The purpose of this study is to interpret the SAXS measurements of samples with a known structure, and to use this information to characterize a large number of samples with unknown structure in a short time.

Samples

Three kinds of materials have been examined: iron oxide powders, pure polymers, iron oxide and polymer powders mixed in a mortar, and iron oxide nanocomposites prepared by “in situ” precipitation. Iron oxide phases were goethite, akaganeite, maghemite and magnetite. Polymer matrixes were: PVP, PVA and dextran). The shape of particles in reference samples was: spherical, rodlike, or needle like. Particle size ranged for 2 to 20 nm in spherical particles, and from 20 to more than 100 nm in elongated particles. Most of the samples for SAXS observation were prepared by powdering the materials in a mortar, and then pressing them into pellets. The pellets were about 0.1 mm thick and 1 mm wide. Other samples were thin films of as prepared materials. Three camera distances were used in the experiments: 0.53 m, 2.20 m, and 4.00 m.

Results

Figure 1 shows that both pure polymer and composite films follow a Porod regime at small angles. At large angles, the polymer shows a constant scattering, while the nanocomposite shows a bump characteristic of polydispersed particles. Figure 2 shows plots from nanocomposites containing particles with different shape. That containing spherical particles show a Guinier region at high angles. That containing rod particles shows and initial Porod region, then a straight line with a smaller slope and a bending region. The beginning of the intermediate region corresponds well with the thickness of the particles and the end corresponds with the length. The intermediate region for needle like particles continues to the end of the plot. Figure 3 shows a plot of nanocomposite samples containing spherical particles with different sizes. Three regions are clearly evidenced from the plots. The first and third regions follow a Porod regime, while the intermediate region follows a Guinier regime. The intermediate region is displaced to smaller angles as the particle size increases. These results are similar to those found in porous media[4], and they can interpreted in the same way.



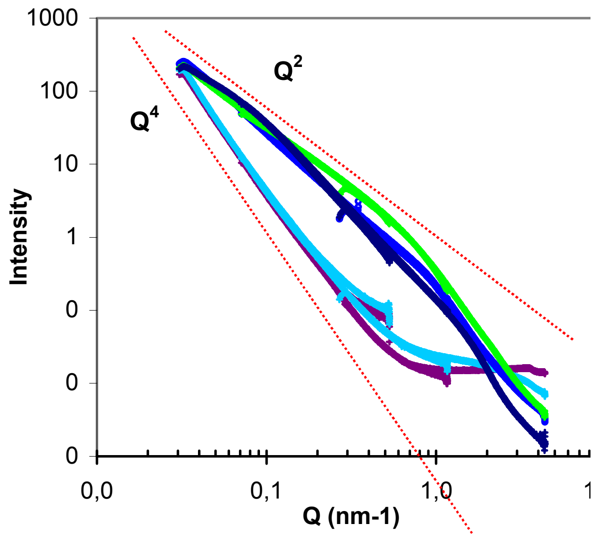


Fig. 1. SAXS plots of (1) as prepared nanocomposite film, (2) PVP polymer pellet, and (3) Fe-PVP coordination compound.

Fig. 2. SAXS plots of nanocomposites containing (1) needle like particles, (2) rod like particles, and (3) spherical particles.

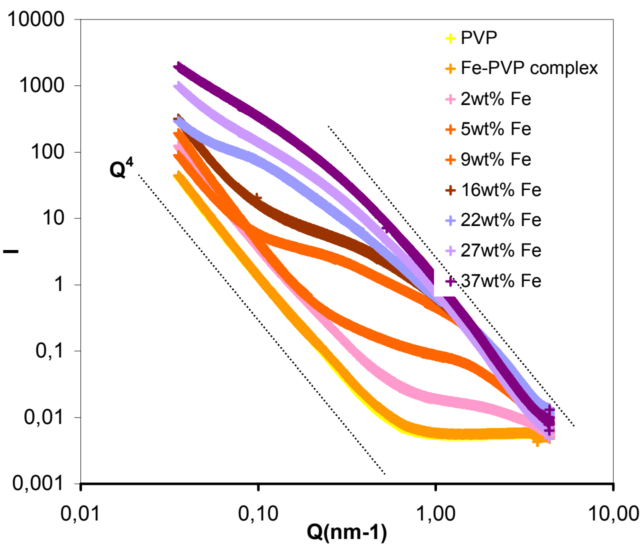


Figure 3. Scattering plots of nanocomposite samples prepared from different iron/polymer ratios.

Conclusions

It has been found that SAXS of oxide-polymer nanocomposites yields a clear information about particle size and shape. According to these results the method of production used here is useful for the obtention of nanocomposites with a variable size. We are now using these data to elaborate structure-magnetic properties models for nanocomposites.

References

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