Using NMR Relaxation to Optimize Use Concentration of a Dispersant: An Example Using Graphene

Nanocarbon materials such as graphene are increasingly attracting a lot of attention because of their stable physico-chemical properties, low cost, excellent chemical stability, wide operating temperature range, and long cycle life. Nanocarbon materials are being investigated for use in all types of applications to deliver enhanced performance capabilities to products such as reinforced composites, the development of new generation super-capacitors, sensors, catalyst supports, and electrode materials. It has been suggested that graphene could entirely replace carbon fiber – for example, in aerospace and defense applications – thanks to its higher strength and lightness.

Single and multiple graphene layers can be produced as dispersions in a variety of liquids but when starting as a dry powder they must be correctly wetted, dispersed, and then stabilized (see Mageleka Application Note #3). Some graphene powders can be dispersed more easily than others but, because of their general tendency to agglomerate, most benefit from the use of a dispersant. Note that true dispersants do not reduce the surface tension of liquids (i.e., they are not "surface active"), but instead work to chemically aid separation of agglomerated particles by increasing the electrostatic repulsive forces between the particles. By improving liquid penetration into the inter-particle spaces, dispersants enhance the separation process, thereby creating a better dispersion.

Choosing the most appropriate dispersant can have important effects on the quality of formulations and the economics of production. The application of nuclear magnetic resonance (NMR) relaxation measurements, such as those made by the Mageleka *M*agno*M*eter XRS™, has proved invaluable in this regard (see Mageleka Application Notes #4 and #5). However, beyond the choice of dispersant, the appropriate concentration must also be considered.

In this Application Note, we show how the *M*agno*M*eter can (additionally) be used to optimize the "use" concentration of any dispersant, which can result in significant savings in production costs. The case study we present here involves a series of 2 wt% aqueous dispersions of graphene, for which the wetted surface area was calculated from relaxation time data (see Mageleka White Paper #1). Four suspensions of dry nanographite powder were prepared by small bench-scale milling (at constant mechanical energy) in the laboratory with increasing addition of a polymeric surfactant/dispersant – a naphthalene polycondensate.

Figure 1 shows the wetted surface area for the graphene suspensions, and the sample

> *"NMR relaxation measurements made it easy to determine the optimum concentration of dispersant."*

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names reflect the percentage of dispersant used: 0%, 1%, 5%, and 10%; the "SP" sample is a dispersion prepared at 20 wt% using production dimension equipment, but with no surfactant (i.e., the same as with sample S0). Since adsorption of a polymeric material can influence the value for the specific surface relaxivity parameter, k_A, the values calculated here for the wetted surface area are relative, not absolute (see Mageleka White Paper #1).

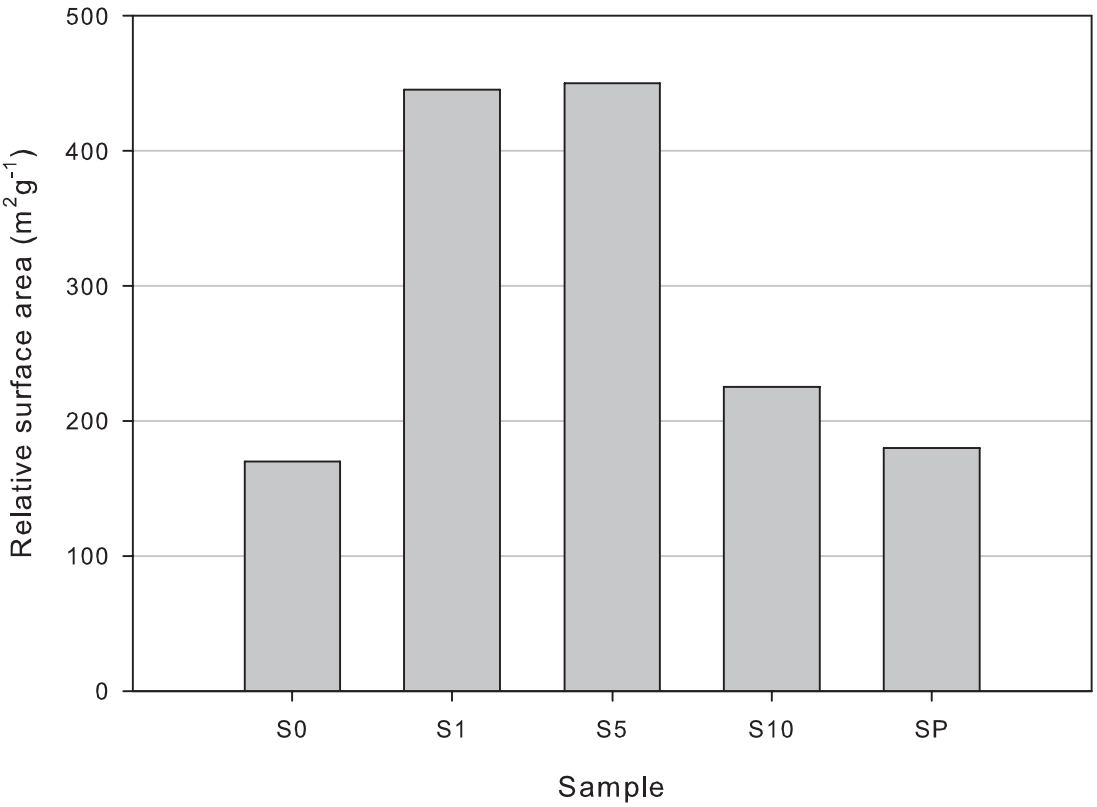

Figure 1. Wetted surface area, calculated from nuclear magnetic resonance relaxation time values (average repeatability <1.0% for all samples), of a series of samples of graphene powder dispersed at 2 wt% using a polymeric surfactant/dispersant at concentrations of 0%, 1%, 5%, and 10%. The "SP" sample was prepared at 20 wt% using production dimension equipment, but with no surfactant and gave similar results to S0.

The data in Figure 1 show that as the concentration of dispersant was increased in these samples the wetted surface area also increased. But it is clear that the S10 sample has been overdosed because of its *smaller* wetted surface area. This is likely to be a result of particle aggregation through a repulsive interaction between an adsorbed polymeric surfactant layer and the high solution concentration of polymeric material in a good solvent (in this case, water).

Overdosing with surfactant impacts both the performance of the final product and also the economics of the process. Thus, the *M*agno*M*eter is a fast and simple analytical tool to determine that this is the case, and so its use is highly

advantageous both in method development and in quality control.

Another valuable observation is that comparing the data for the SP sample with that for the S0 sample suggests that the laboratory preparation procedure using a small bench-scale mill is a good model of the commercial-scale production process, even though the milling takes place at rather different particle concentrations (2 wt% *vs.* 20 wt%).

In this example, NMR relaxation measurements, such as those produced by the Mageleka *M*agno*M*eter XRS, made it easy to determine that the optimum concentration of dispersant for this graphene material was no more than 5%. Optimizing dispersant concentration not only saves money by reducing the amount used but also results in a better quality dispersion, which can have important implications for product performance. Moreover, the data show that scaling-up to production levels can be monitored or verified using NMR relaxation techniques.

This case study highlights how a next-generation benchtop NMR instrument such as the *M*agno*M*eter provides a simple and flexible tool for *M*agno*M*eter key characteristics of dispersions (see Mageleka Technical Note 3). Although the data presented here are for a carbon nanoparticulate material, the *M*agno*M*eter is suitable for use with any solid-liquid or liquid-liquid dispersion at all industrially-relevant concentrations (see Mageleka Technical Note 1).

For more information, to send samples, to arrange a demonstration of the MagnoMeter at your facility, or to talk to one of Mageleka's technical applications specialists, please email roger@mageleka.com

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