XG sciences -Dispersion Notes

General

xGnP® Graphene Nanoplatelets are very thin, (5 – 10 nanometers in thickness) flat particles with quite large diameters. Like other nanoparticles, the small size gives rise to certain handling issues. Because of the flat shape of these particles, they are especially sensitive to van der Waals attractive forces and have a tendency to re-aggregate in the dry state. For this reason, we ship our platelets in a granular form, which limits relative motion of the individual platelets during shipping. These granules are friable collections of individual platelets that prevent agglomerations and are easily broken with mechanical agitation.

Even though your material may appear to be agglomerated, and may contain large chunks of material, please consider that we have done a great deal of testing to determine that this is the most effective form of dry shipment for effective dispersion. We ship our material this way on purpose because we have found that this approach results in material that is most easily dispersed.

XG Sciences supplies xGnP® Graphene Nanoplatelets in various sizes and grades. We have found that the untreated material disperses well in many polymer systems, but results will, of course, vary depending on the host system. In general, smaller particles disperse better than larger particles; dispersion is better in low viscosity materials, and surface-treatment can improve dispersion and/or adhesion with various resins. Also, once the platelets are dispersed in a non-solid system, they will settle and possibly re-aggregate. The rate of settling will depend on whether a surface treatment is used and on the system's viscosity.

Below are hints to aid dispersion of xGnP® Graphene Nanoplatelets into various systems.

Dispersion into Non-Aqueous Solvents

Organic solvents are very effective in obtaining a good dispersion but, in most cases, are not practical for our customer to use. Much of the early development work with xGnP® in thermoplastic resins was done by dispersing the platelets in an aromatic solvent, dissolving the polymer in the same solvent , mixing the two solutions and then evaporating the solvent to obtain the thermoplastic with an excellent dispersion of platelets.

Isopropanol (IPA) is a fairly good solvent which is inexpensive and easy to use, but may not be as effective as some stronger solvents. Some very effect solvents include N-methylpyrrolidone (NMP), dimethylformamide (DMF), tetrahydrofuran (THF), and chloroform. However, these are very strong and toxic materials and should be handled with great care by experienced personnel. In any case, if solvents are used to aid dispersion into a polymer system, care should be taken to make sure that the solvent is removed prior to further processing.

Dispersion into Aqueous Systems

xGnP® Graphene Nanoplatelets are hydrophobic and will not disperse in water without a dispersion aid. Three dispersion aids that we have proven to be useful are:

- Sodium dodecylbenzene sulfonate - (SDBS) – (solid)
- Linear or branched poly(ethyleneimine) - ((PEI) –(50% H_2O solution)
- Poly(sodium styrene sulfonate) – (PSS) – (~70k Mw, 30% H_2O solution)

These materials are available at the chemical supply house Sigma Aldrich - www.sigmaaldrich.com

Two methods have been used:

1. Add dispersant to water at 1-2% level and then add xGnP® with the use of an ultrasonic mixer. The rate of xGnP® addition to the liquid is critical. It should be added with continuous mixing, at a rate so that the xGnP® does not coalesce on or in the liquid. A continuous feeding system (screw or vibratory) will be helpful. The addition rate may need to be adjusted during addition since the receiving liquid may change viscosity during this operation due to concentration and temperature changes. Careful xGnP® addition should result in a stable suspension. Experimentation with the amount of dispersant will be necessary to determine the optimum level for your system.
2. Continue to sonicate the suspension from step for a few minutes or longer. Filter and wash the coated platelets. Now re-disperse the coated xGnP® in the desired amount of water to obtain a stable suspension.

In addition to the above solvents, a mixture of 15 wt% IPA to 85 wt% water should result in a carrier solution into which our nanoplatelets can be dispersed with the aid of sonication.

Normally, particles that settle out of a solution can be redispersed with the aid of an ultrasonic probe.

Dispersion into Thermoplastic Matrix

While solvent dispersion will give the best results, it is not practical for volume production. Extrusion should yield good results, especially for lower viscosity thermoplastic resins, but it may be necessary to experiment to determine the best method for your resin system.

- Some manufacturers have introduced specially designed screws for nanocomposites.
- If available, counter-rotating screws have shown good results in many materials.
- Lower melt viscosity will improve dispersion. This can sometimes be achieved by increasing processing temperatures or by switching to a polymer with a higher melt index.
- In general, mixing graphite nanoplatelets with powdered polymers, rather than pellets, result in better dispersion.
- Especially with resins in a powder format, it may be advisable to mix xGnP® Graphene Nanoplatelets with powder before feeding into the extruder.

- Although feeding of pastes or liquids is not common, there may be special cases where a pre-dispersion of the nanoplatelets into a suitable carrier material could improve the results of the final composition. If the end use is in a polyolefin resin, the use of a pure hydrocarbon solvent would insure compatibility and a minimum of interference of any retained solvent in the final polyolefin properties

Because of the importance of good dispersion and the difficulty involved, we recommend that you purchase a pre blended master batch. This will allow you add xGnP® to your system with a pelletized concentrate. Ovation Polymers offers their ExTima™ brand of Graphene/Polymer Master Batches in a variety of base resins. (www.opteminc.com) and other compounders have also developed techniques for blending xGnP® nanoplatelets in thermoplastic resin systems.

Dispersion into Thermoset Resin Matrix (Epoxies, Urethanes, etc)

For most resin systems, traditional mixing techniques should prove adequate. Sonication with an ultrasonic probe works well. Also, good results have been obtained with a high-shear 3 roll mill. A balance must be stuck when using high shear mixing since prolonged use cause the platelets to deform.

The rate of xGnP® addition to the resin is critical. It should be added with continuous mixing, at a rate so that the xGnP® does not coalesce on or in the liquid. A continuous feeding system (screw or vibratory) will be helpful. The addition rate may need to be adjusted during addition since the receiving resin may change viscosity during this operation due to concentration and temperature changes. Careful xGnP® addition should result in a stable suspension.

Do not store the dispersed blend for long periods since the platelets will settle over time. The rate of settling is dependent on the viscosity of the subject resin. To prevent settling and re-agglomeration react the resin suspension as soon as possible. Normally, particles that settle out of a solution can be redispersed with the aid of an ultrasonic probe or other high-energy mixing system.