Successful Use of Fumed Silica in Liquid Systems

Hydrophilic and hydrophobic AEROSIL fumed silica grades are used in many liquid systems for viscosity control, anti-sag and anti-settling behavior, and for general rheological enhancement. To optimize efficiency and consistency of the behavior for all fumed silica grades, the following conditions are important for success.

1. Finding desired addition level
2. Applying proper dispersion
    - Equipment and design features
    - Tip speed considerations
    - Length of shearing time
    - Temperature build-up
    - Sequence of addition
    - Masterbatch vs. direct addition
    - Grind values vs. dispersion
    - Preventing under/over shearing
3. Completing the formulation

Finding the Desired Addition Level

In non-polar to semi-polar systems, hydrophilic AEROSIL 130, 200, 300, 380 grades are used routinely for rheological improvement. In semi-polar to polar systems, hydrophobic AEROSIL R972, R974, R812, R812S, R202 and R805 grades are used. In many systems, hydrophobic grades are slightly less efficient but impart other properties to the film such as water resistance, improved leveling, and suspension of pigments with less viscosity increase and improved corrosion resistance.

Whatever the use, loading levels in most coating and ink applications are relatively low. In the final formulation, < 1.0% by weight (based on total system) is usually suggested as a starting point. Loading levels for anti-settling properties may approach 1.5% if the formulation contains high loadings of other pigments and heavy fillers. For adhesive and sealant applications, loading levels are typically higher, in most cases 4-8% by weight (based on total system), because the desired thickening and thixotropy is higher. The actual percentage is dependent on the initial viscosity condition of the system, the desired condition, the desired length of storage stability and the polarity of the final formulation. The optimum loading is determined by trial and error and is very system specific.

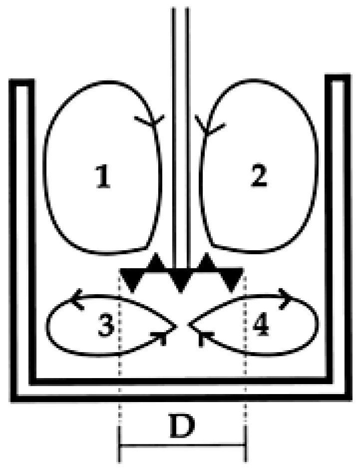

Figure 1

Applying the Proper Dispersion

To maximize fumed silica efficiency and to ensure performance consistency, proper dispersion is required. Low shear dispersion with a propeller or stirring blade is insufficient shear for all AEROSIL grades and in general for all fumed silica. Peripheral velocity (tip speed) for this type of mixing is typically 5-20 ft/sec. At these rates, minimal energy is used to wet the fumed silica. Using this low shear rate, resulting thickening performance can be very inconsistent, batch-to-batch. Other results of low shear rate are loss of efficiency, whereby more AEROSIL fumed silica needs to be added, poor grind behavior, settling, and poor storage stability.

High shear dispersion (HSD) using an aggressive saw-tooth type blade is the minimum shear that is required for AEROSIL grades with surface areas of 90-200 (both hydrophilic and hydrophobic). Higher surface area fumed silica grades usually require more energy intensive equipment for optimizing dispersion. Peripheral velocity for HSD equipment should be more than 30 ft/sec. For most industrial applications, Degussa suggests tip speeds above 30 ft/sec (9-10 m/sec), for sufficient shear.

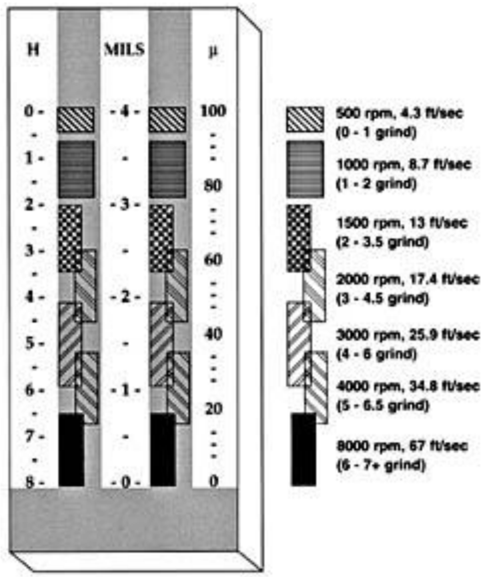

Figure 2

A crucial aspect for adequate dispersion includes the blade:vessel ratio (see Figure 1). The blade:vessel diameter should be 1:2-1:3. With this ratio, a strong vortex should be observed straight down to the dispersing blade. When the ratio approaches 1:4, material often clings to the sides of the tank and a small vortex is noticed. When a vortex is observed, where only the shaft is visible, wet-in of powders takes longer. The blade should be positioned 0.5-1 D from the bottom of the mixing tank. The blade should be positioned where four mixing zones are created; the top two mixing zones are pulled down into the dissolver blade and the bottom two mixing zones are pulled up into the dissolver blade, according to INSCO Blade Shop literature. Blade sharpening and tightening of belts should be part of routine maintenance. Both contribute to efficiency and consistency of dispersion. It is also best to have an rpm/torque read-out on the equipment, instead of an variable resistor with arbitrary settings from 1-10. This is to ensure the shaft is revolving at the same shear rate batch to batch.

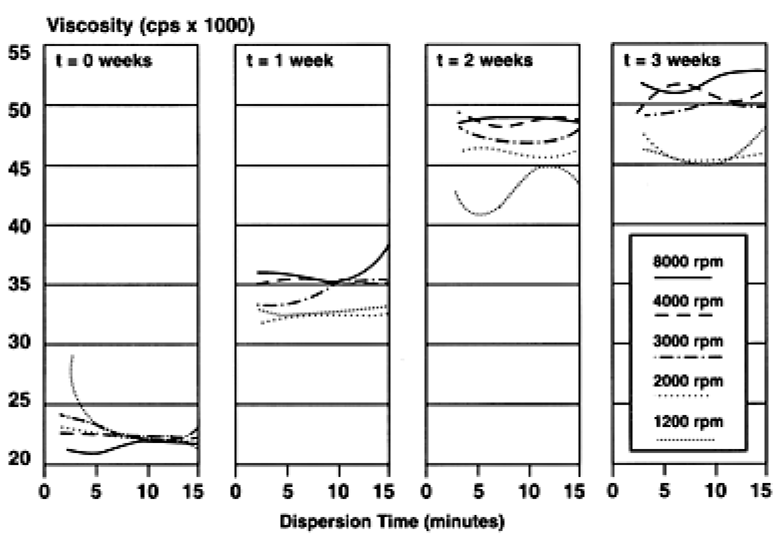

Figure 3

High intensity mills, sand mills, media mills and roller mills are sufficient and suggested for high surface area AEROSIL (>300 m2/g grades) and for all applications requiring the highest thickening efficiency and anti-sag properties, best grind values and highest clarity. Performance of all fumed silica grades normally improves with increasing shear forces.

Length of dispersing time should be kept to a minimum to prevent excessive temperature buildup. For most systems, longer dispersing times result in lower mixing viscosity due to the higher mixing temperatures. Extended dispersion at higher temperatures (normally defined as 20degF higher than what is normal for the batch) may do more harm than good to most systems. This is due to higher temperatures resulting in faster wetting of pigments. This is an environment where overdispersion of fumed silica may occur.

Overdispersion results when fumed silica aggregates are broken down past an optimum aggregate size. Aggregates then become so homogeneously dispersed, that the distance between adjacent aggregates becomes too great, and aggregate-to-aggregate interaction is reduced. This results in a total loss of thickening and thixotropy. A normal observation by a plant worker may be that the batch went "water thin." This behavior may occur in manufacturing sites where time and temperature of batches are not frequently monitored and operators take extended breaks, leaving batches unattended.

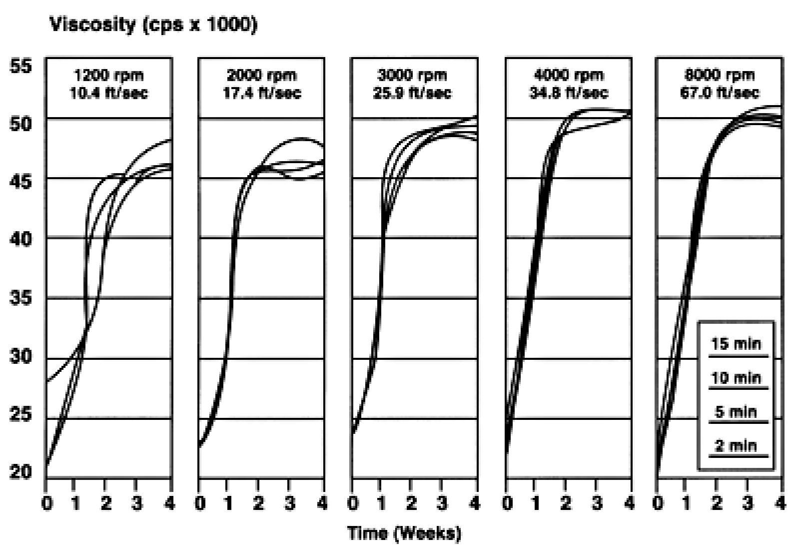

Figure 4

Optimum mixing time and temperature are system specific and must be established for each formulation. Results from testing show that once sufficient energy is put into the system (rpm/tip speed), processing time becomes less critical (see Figures 2-7). Also, slight changes in processing procedures or parameters have less of an impact on the consistency of the final product, with sufficient shear rate. When insufficient energy is put into systems, all changes in processing can drastically affect the efficiency of fumed silica and the consistency of the final product.

Specific time should be allotted for wet-in of fumed silica packed in paper bags. Wet-in time is considered the time required for the fumed silica to be wetted by the vehicle. At this stage, low shear mixing is often used. Once all the fumed silica is wetted (brought under the surface of the liquid), the mixing speed is increased and dispersion begins. Fumed silica should be wetted in as quickly as possible. It should be added into a vortex, with the mixer running at a slow speed. The time it takes bags to be broken and material wet-in can be operator dependent. Different operators can be faster or slower at this point. If time is not allowed for each of these periods, some batches may receive more or less dispersion time depending on the operator.

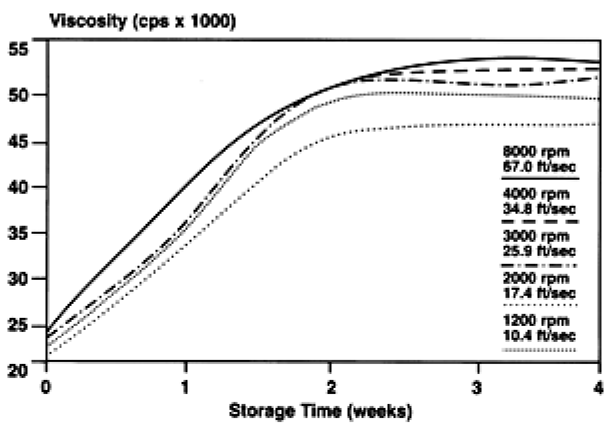

Figure 5

The sequence of AEROSIL addition is crucial in many processes. Tests show that fumed silica should be one of the first components added, after the resin or vehicle is introduced into the batch vessel. Some resins show preferential wetting of fumed silica. In these cases, it is best to disperse fumed silica into the better wetting resin, then bring the rest of the formulation into this mixture.

In water-reducible systems, sequence of addition is especially crucial for hydrophobic AEROSIL grades. It is suggested that the fumed silica be added into the vehicle first, without the water adjustment. Once the fumed silica is wetted in, the other pigments, fillers and additives can be added. If water encapsulates unwetted hydrophobic fumed silica, further dispersion becomes very difficult.

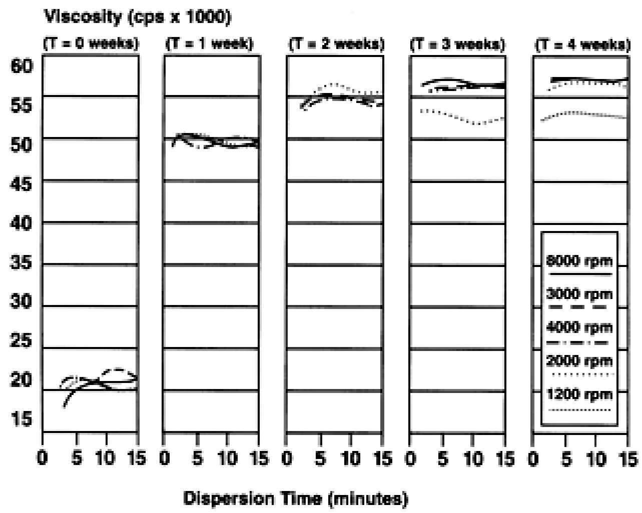

Figure 6

In emulsion systems, both AEROSIL 200 and R972 are successfully used for improving pigment suspension and anti-sag properties. Incorporation is suggested at the highest resin solids state, preferably higher than 38-40%. Incorporation of hydrophobic grades becomes very difficult once the resin solids drop below 30%. After wet-in and proper dispersion, final letdowns with water can be conducted.

Dispersion of fumed silica in the pigment grind portion is always recommended. Making concentrated masterbatches of AEROSIL fumed silica, pigments and fillers has proven to give the best long-term storage stability and higher overall thickening and thixtropy vs. direct addition into the letdown. We do not recommend that fumed silica be added into the letdown or as a post-add in powder form due to lower shear forces used for mixing. If post-adjustments are needed, it is suggested that users take a 5-gal pail of the final system, disperse fumed silica under HSD conditions in this vessel, then return this concentrate to the batch.

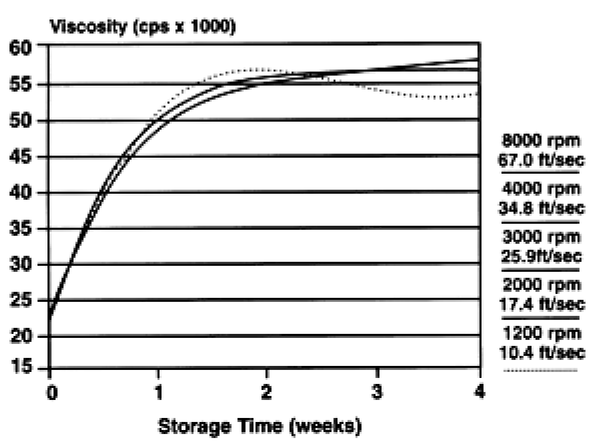

Figure 7

Under-shear is more common than over-shear, due to inadequate energy input during the dispersing stage. However, as mentioned previously, over-shear may occur in cases where very long dispersing times are used or where very high temperatures in the process are generated, which gives added wetting characteristics to the resins. In these instances, more fumed silica is needed to increase rheology. Once over-dispersion has occurred, it is very difficult to "bring it back" without adding more fumed silica.

Completing the Formulation

After dispersion is complete, remaining components can be added under letdown or lower shear mixing conditions. At this point, necessary applications tests need to be conducted to ensure that the loading level and dispersion process was adequate to meet performance requirements. Long term stability testing is also needed to ensure a proper dispersion procedure is established. Poor long-term stability can be an indication that dispersion, loading level and/or sequence of addition have not been optimized.

For more information on fumed silica, contact Degussa Corp., Akron Technical Center, 3500 Embassy Parkway, Suite 100, Akron, OH 44333; phone 888/SILICAS; visit www.aerosil.com; or Circle Number 137.

http://www.pcimag.com/articles/86041-successful-use-of-fumed-silica-in-liquid-systems