Testing Raw Materials I: Resins and Pigments

Paint manufacturers do less testing of raw materials than they did years ago, because most suppliers now guarantee that their products meet all specifications. They provide customers with batch certificates, data tables, and other information and may provide compensation if a product fails to perform and causes paint manufacturing problems or field failures. However, raw material testing still is needed to qualify suppliers and when problems arise. Internally produced materials such as resins also require testing, but only the larger paint companies make their own stuff.

Common resin tests include measurement of viscosity, usually by bubble tube (ASTM D1545, recently revised), percent nonvolatile (D2369), density as weight per gallon (D1475), color, and IR analysis to see if it is the correct resin. Molecular weight (MW) is a particularly important property, since paint viscosity depends on resin MW and its distribution. High solids paints require low MW resins, and even a small amount of high MW material can cause application problems. The standard technique for measuring MW and its distribution is gel permeation chromatography (GPC), also known as size exclusion chromatography (see D3016). High-temperature cone/plate melt viscosity measurements (D4287) of coatings or resins after solvents have been removed can be used to establish the presence of high MW material. Results of limiting viscosity number [η] (formerly called intrinsic viscosity) measurements (D2857) can be used to calculate MW.

One thing to watch out for with any of these techniques is the possibility of erroneous results due to removal of gel or very high MW material if the specimen solution is filtered. Particle or micelle size, size distribution, and dispersion quality of waterborne polymers can be measured by optical microscopy or light scattering. Beware of settling out of large particles or their removal by filtration before the measurements are carried out. Storage stability can be tested by viscosity measurements and microscopy before and after heat aging.

The glass transition temperature, T_g, of a solventborne resin affects the viscosity of the resultant paint and that of a waterborne resin affects the quality and temperature-dependence of film formation. The minimum film forming temperature (MFFT) is the lowest temperature at which a latex, emulsion, or dispersion polymer solution will uniformly coalesce when applied to a substrate as a thin film. It is measured by drawing down a specimen on a special temperature gradient bar, allowing the film to dry, observing the dividing line between clear, continuous film and opaque, fragmented pieces and reading the temperature at that point. Standard tests include ISO 2115 and ASTM D2354. The latter was withdrawn in 2007, but is still available from ASTM. The glass transition can give an estimate of the MFFT. The MFFT always is higher than the T_g, and there is a rule of thumb that MFFT ≤ (T_g + 10° C), which works fairly well for coatings with glass transitions greater than 0°C.

Tests on as-received dry pigments rarely are needed, but when there is a problem, there are tests for oil absorption (ASTM D281 or D1483, which has better precision) and water content. I have worked on problems where high pigment water content has contributed to defects such as popping or pinholing and one case where moisture from a pigment caused a urethane to gel. I have seen where a sack of pigment was mislabeled (IR analysis showed that it was the wrong material) and encountered batches of supposed easy-disperse pigment that were very difficult to disperse and appeared to not have had any surfactant adsorbed onto the particles. I also worked on a case where my employer was being sued by a contractor for adhesion failure of a maintenance coating, but x-ray diffraction of paint chips showed a pigment that we did not use in our formulations. We suggested that the contactor should sue someone else.

Rather than testing dry pigment, it is more likely that a pigment sample would be made into a paste and tested for fineness of grind (D1210) and the ability to tint a white or neutral base. A portion of paste would be let down to be examined for particle size and size distribution with an optical microscope (dilution may be necessary) or, after considerable dilution, by light scattering. Dilution must be done carefully, preferably with mill base; otherwise, flocculation may occur. There usually is an optimum viscosity range for dispersing a pigment, especially in a sand mill. If the viscosity is too high, the mill overheats and the paste kicks out. If the viscosity is too low, there is not enough of a shearing action on the pigment particles, and the result is a poor dispersion with large particles or clumps of particles. The viscosity depends on the mill, the pigment, the mill base, and other factors and usually is established by trial and error. With pigments, when in doubt, make a paste, then a paint.

Next month, I will discuss the testing of solvents.