

TESTING THE CURE OF UV/EB CURABLE COATINGS

Before we discuss testing cure, let's briefly answer the question - what is UV/EB curing?

UV curing is a process involving 100% solids chemistry that is converted from liquid to solid in a fraction of a second when exposed to UV light. UV light is generated from high intensity ultraviolet lamps. Photoinitiator components of the chemistry absorb the UV light energy, forming free radicals, which cause polymerization and cure.

EB or electron beam curing is different in that the process utilizes a stream (or beam) of electrons produced by a high voltage electron generator. The high energy electrons are capable of initiating cure without the need for photoinitiators as a component of the similar 100% solids liquid chemistry. Cure is rapid, taking place in milliseconds.

The adequate curing of a UV curable coating is dependent on a number of variables including coating thickness, opacity, formulated cure speed, and substrate. Number and condition of UV lamps, type, age, focus, intensity of lamps, and reflector condition also affect cure.

Similarly, the adequate curing of an EB curable coating is dependent on application and EB process variables being optimized.

Cure may be said to be inversely related to the extractable reactive monomer and oligomer level remaining after cure has taken place. Conversion of multi-functional chemicals in UV formulations is said to be no greater than 95%, while EB closely approaches 100%.

Proper cure or functional cure can be best defined as the cure obtained when the target performance parameters of the coating film have been optimized for the end user.

UV coatings are challenging to test for cure. They typically cure from the top down and rarely cure uniformly throughout. The top most film layer has its cure inhibited by oxygen in the air. Internal cure takes place as the UV light penetrates to cause polymer formation. Adhesion to a substrate takes place when UV light reaches the interface. Good adhesion requires good surface wetting and low film shrinkage, which occurs due to polymer formation. Cure is approximately 80% complete when UV light exposure stops. Post cure takes place over the next 24 hours or longer producing a plastic solid that continues to crosslink and become more rigid and less flexible. Adhesion typically improves as a result of post cure.

When we consider proper functional UV cure, it is necessary to consider a balance between top surface cure and through cure.

Because post cure can be an important cure factor, timely production line testing should always be run press-side and also 24 hours later for meaningful end user correlation.

Determining proper functional cure is not easy, however, certain simple tests have been developed which have proven useful in place of expensive quantitative extractable testing.

So when we consider whether cure is adequate - what should we test for and how?

TESTING THE CURE

MEK RUB TEST - This test checks the relative solvent resistance of a cured coating sample. The procedure calls for multiple layers of cheesecloth either wrapped around a finger or more ideally around the ball end of a 2 pound ballpeen hammer. Felt may be substituted. The fabric is saturated with MEK and the sample is rubbed in a back and forth 2 - 4 inches (double rub) constant motion. The hammer handle should be held level with the sample with no downward pressure applied. The number of double rubs to when the coating wears through is noted. MEK rubs may be used as a relative indicator of cure and a given coating's expected performance. See RadTech Test Methods available from Radtech International, Northbrook, IL., 847-480-9576

PERMANGANATE STAINING TEST - This test measures residual unsaturation or the unreacted double bonds in a molecule and it directly indicates the amount of cure. The intensity of stain color is proportional to the amount of unsaturation. The test is conducted using a 1% solution of potassium permanganate (KMNO₄) in distilled or dionized water. The solution is applied to a coated sample so that a 1/2 inch diameter is covered. The solution is allowed to remain on the surface for 5 minutes after which it is rinsed away with water. A brown stain will be seen. The more unreacted material in the coating, the darker the stain. The stain color intensity may be compared to a standard, the color of which is indicative of a well cured coating. Care should be taken in that a stain standard can fade over time. Test is qualitative only, see RadTech Test Methods.

SCRATCH TESTING - These tests can assist in determining that a coating is undercured. Remembering that adhesion to a substrate develops when enough UV light reaches the interface, good adhesion should be shown immediately after a coated piece exits the cure unit. A quick adhesion test may be done by rubbing the edge of a nickel coin across the coated sample or by fingernail scratching. A cured coating should not peel or flake off. Differences may be observed by scratching inked and uninked areas. Adhesion failures can be caused by undercure, overcure, film shrinkage, film brittleness, poor substrate or ink wetting.

SCOTCH TAPE TESTING - Tape adhesion tests are not able to tell much about cure. Adhesion is without a doubt a function of cure, but too many other factors affect the test procedure to make it a reliable indicator of cure. Slip additives that migrate to the surface can prevent the tape from sticking effectively. The test, however, can have value as an indication of expected coating performance. Scotch brand #610 tape is used. ASTM D 3359 describes the procedure of attaching a 3" strip of tape to a coated sample. Remove within 90 seconds by pulling the free end of the tape back rapidly upon itself, close to a 180 degree angle, without jerking. Look for any coating and/or underlying ink removed.

FLAIR PEN TEST - This quick test can indicate a coating that is undercured. It is based on the pen's ink immediately beading up and reticulating on a coating's surface, indicating wax and silicone bloom, which is an indicator of adequate cure. The ink will leave a solid line (no reticulation) when cure is inadequate or when no silicone and little wax is present.

COMPUTERIZED CURE ANALYZER - This test features a PC based unit that measures the degree of polymerization of UV/EB cured materials. Proper cure, undercure and overcure can be assessed. The test is based on a test solvent solution being able to penetrate a coating. Penetration will be less into a cured tightly crosslinked coating, whereas it will be greater into an undercured loosely crosslinked coating. A resulting index number becomes the basis for comparative analysis. The system is available from UV Process Supply, Inc., Chicago, IL. 1-800-621-1296

ABRASION TESTING - Taber, GA-CAT, and Sutherland testing is conducted to ascertain a coating's resistance to abrasion. The tests are also an indicator of surface cure and slip. In general, undercured coatings offer poorer abrasion

resistance. However, coating thickness affects results. Also a good surface cure and a high slip surface can yield good wear results and yet hide a lack of through cure. These tests are best used as an indication of a coating's acceptable target performance being achieved for an end user.

Remember, while valuable in assessing degree of cure, these tests do not actually test absolute cure, but only test relative cure response and the coating properties that develop as a result of cure.

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