

Complete curing ensures optimum release coating performance

Facts on file

Curing is a crosslinking process. Complete curing of SYL-OFF™ silicone release coatings ensures that uncrosslinked silicones do not migrate from the release liner to the adhesive where they could affect adhesive performance. Completeness of cure is also an important factor in determining the suitability of a release liner for food contact applications.

Evaluating the degree of release coating cure

A number of variables can affect release coating cure time, temperature, humidity, the type and age of the substrate, coating weight, degree of surface penetration, degree of functionality, etc. A precise and objective tool for evaluating the degree of release coating cure can help you manage variables and improve the quality of your end-use application. The subsequent adhesion tests give a general indication of cure. Measuring the percent of extractables in the cured silicone coating gives you a more precise and objective picture of your state of cure.

Percent of extractables as a measure of cure

The percent of extractables is the amount of uncrosslinked silicone that disengages from a cured release-coated sample in the presence of a solvent. The lower the percent of extractables, the more complete the cure. The most desirable percent of extractables for most applications is 5 percent or less.

How to measure extractables in silicone release coatings using atomic absorption

1. Measure the coating weight on the liner to be tested in pounds per ream.
2. Cut a 5" × 6" liner test sample into 1/2" × 1/2" pieces.
3. Put the sample pieces in a 100-mL bottle with 40 mL methylisobutyl ketone; seal the bottle and put it on a mixing wheel for 30 minutes.
4. Decant the solvent and analyze by atomic absorption for silicon; report the concentration in micrograms per milliliter.
5. Calculate the percent of extractables as shown in Figure 1.

As a rule, cure becomes more complete over time. To develop a profile of the lowering levels of extractables in your silicone release coating application over time, conduct the test immediately and at 1, 7, 14 and 30 days post-cure.

How to measure extractables in silicone release coatings using a Benchtop XRF instrument

The extractable component of a cured silicone release coating may also be determined using the following procedure designed for an Oxford Lab-X 3000 XRF analyzer. (Note: This procedure may be modified for other manufacturers' benchtop XRF instruments, but Dow experience is limited to the instrument described here.)

1. Cut 3 sample discs from the substrate using a sample punch and place the discs on a clean sheet of paper; use tweezers to handle the discs at all times.
2. Determine the silicone coat weight on each sample using the Oxford Instruments Lab-X 3000 Benchtop XRF analyzer; a sample spinner is required on the Lab-X 3000 to give the most accurate results.
3. Keep elapsed time between preparation of the coated substrate and contact with methylisobutyl ketone extraction solvent at a consistent and minimum time to prevent post cure; the shortest practical elapsed time is 5 minutes.
4. Place the 3 discs in a 100-mL bottle containing 40 mL of methylisobutyl ketone solvent; seal the bottle and put it on a mixing wheel for 30 minutes; after 30 minutes, remove the discs from the bottle using tweezers and place them on clean tissue paper, silicone coated surface up.
5. Allow the solvent to evaporate from the sample discs; DO NOT wipe or blot the sample discs.
6. Allow the sample discs to air dry for 10 minutes or longer to ensure complete evaporation of the methylisobutyl ketone.
7. Measure the final coating weight of each sample disc.

$$\frac{X \times 10^{-6} \text{ g Si}}{\text{mL}} \times \frac{40 \text{ mL}}{30 \text{ sq in}} \times \frac{74 \text{ g PDMS}}{28 \text{ g Si s}} \times \frac{144 \text{ sq in}}{\text{q ft}} \times \frac{3000 \text{ sq ft}}{\text{ream}} \times \frac{\text{lb}}{454 \text{ g}} \div \frac{\text{Y lb}}{\text{ream}} \times 100$$

The calculation reduces to: % extractables = (X/Y) 0.335

Where: X = Si concentration in methylisobutyl ketone, micrograms/milliliter
Y = Coating weight, pounds/ream

The extractable silicone present in the coating after preparation under specified conditions is determined. The quantity of extractables is expressed as a percentage of the initial coat weight.

Calculate the percent of extractables as shown below:

$$\% \text{ extractables} = \frac{(a - b)}{a} \times 100$$

Where: a = Initial coat weight (before contact with methylisobutyl ketone)

b = Final coat weight (after contact with methylisobutyl ketone)

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Factors affecting release performance

State of cure: If a coating is "fully cured," it should reach a state of constant physical parameters in terms of hardness, elasticity, strength, etc., and inertness. Release performance should also be consistent and repeatable, and release values should be stable. This state is easier to achieve with some polymer types than others. Also, some adhesives can tolerate significant undercure, while others cannot.

Because a state of total cure is achieved at the cost of long exposure to curing stimulation, the substrate itself may suffer significantly in terms of impoverished physical properties. Consequently, total cure is not always desirable. In reality, very few release coatings are cured to such a state, and all are undercured to some degree or another.

The impact of undercure: The impact of undercure is totally adhesive dependent. Most rubber-based adhesives, solvent or hot melt, are relatively inert to unreacted reactive groups in the silicone. Crosslinkable solvent acrylic adhesives, on the other hand, can and do react strongly with SiH functionality in the silicone. This phenomenon is known as acrylic lockup. Most water-based adhesives, either acrylic or rubber-based, exhibit a similar tendency but to a lesser degree.

With undercured silicone, release forces may be a little higher with even inert adhesives, simply because the elasticity of the silicone is higher due to a lower crosslink density or a softer silicone.

Minimum cure: For any given construction, there is a minimum degree of cure that will produce an acceptable product. This degree of cure is usually the point most users achieve, and any greater curing effort is perceived as a threat to the substrate. Release stability will always be improved with improved curing, and release values will usually be lower. Other properties such as adhesive tack, subsequent adhesion strength and extractables content of the silicone film will all improve with better curing.

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