Cure dynamics of powder coatings

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Commercial application line operators, laboratory and customer service personnel working for powder coatings manufacturers, and even field engineers and application line designers have often relied on on-line trial-and-error methods and procedures to establish viable time and temperature data to assure that powder coatings are fully cured. This article offers a relatively simple formula to calculate the time-temperature dynamics for powder coating cure.

There is still a lot of confusion in the industry regarding oven dwell time (the total time a part remains in the cure oven) and actual cure time (the time it takes for powder finishes to cure up completely at a set and stable substrate or part temperature). Many people, especially those in a laboratory environment, still believe that oven dwell time and cure time are essentially the same. The formula featured here is for calculating cure time only, but when oven bring-up times (the time it takes for parts to reach cure temperature) are known or can be estimated with some accuracy, the cure-time formula can be modified to calculate oven dwell time.

Technical data sheets issued by powder coatings manufacturers generally show only one single bake schedule, for example 10 minutes at 400°F. However, engineers designing application lines and powder coating applicators need to consider cure-time schedules under different time-temperature conditions to optimize space, equipment, and process, to minimize costs, and to assure that the finished products meet the specifications set down by internal quality assurance and by the end user of the coated products.

Understanding reaction mechanisms

Following are scientific explanations of the complexity of chemical reactions and kinetics of organic binder systems used in powder coatings. The information is from the Web site [http://ibchem.com/IB/ibnotes/brief/kin-hl.html]. It has been modified to explain some details about powder coating chemistries and cure mechanisms. For a summary, see Table 1.

- Rate determining step: The slowest step in a reaction. It determines the rate of the overall reaction. This may apply for the cure mechanism in emissive polyurethane powder coatings—the blocking agent (usually E-caprolactam) has to dissociate (split off) from the curative before the resulting isocyanate groups can react with the hydroxyl groups of the polyester.

- Rate of reaction: Rate of reaction is concerned with how quickly a reaction reaches a certain point. Applied to powder coatings, this would be equivalent to cure time.

- Collision theory: Reactions take place as a result of particles (atoms or molecules) colliding and then undergoing a reaction. Not all collisions cause reactions, even in a system where the reaction is spontaneous. The particles must have sufficient kinetic energy and the correct orientation with respect to each other for them to react.

- Activation energy: This is the minimum energy that colliding particles must have to produce successful reaction. The energy of particles is expressed by their speed. The kinetic energy of molecules is related to heat energy; higher heat means higher collision energy. Higher heat also means lower viscosities for liquefied powder coatings, resulting in higher mobility of the resin and curative molecules to create favorable conditions for collisions. (See Collision theory.)

- Changing the conditions: Increasing the temperature of a substance increases the average speed (energy) of the particles and consequently the number of particles colliding with sufficient energy (see Activation energy) to react; it also reduces the viscosity of liquefied powder.

— More successful collisions, and therefore a faster reaction, occur at higher temperatures, which means shorter cure times for powder coatings.
Catalysts lower the activation energy by providing an alternative mechanism for the reaction and greater probability of proper orientation. This results in a faster reaction.

**Establishing a cure dynamics curve for powder coatings**

A rule of thumb in chemistry declares that the reactivity doubles (or is cut in half) whenever the temperature is increased (decreased) by 18°F (10°C). Given the facts that we have the reactions taking place in a relatively thin film on (mostly metal) substrates with different specific heat characteristics, and taking changing bake schedules into consideration, establishing a formula to eliminate guesswork and calculate cure time or total oven dwell time at various temperatures is at least a very difficult undertaking.

Oven dwell time, the total time (in minutes) parts spend inside a cure oven, consists of bring-up time (the time in minutes it takes for the parts to reach cure temperature) and cure time (the time in minutes it takes for a powder finish to reach full cure):

**Oven dwell time = Bring-up Time + Cure Time**

Depending on the chemical nature of the powder coating, a slow cure reaction may start during bring-up time after the powder liquefies and reaches temperatures of about 230°F-302°F (110°C-150°C). However, oven air and substrate temperatures must reach a certain and predetermined point for the cure reaction to proceed at a speed suitable to meet the equipment design specifications. In my experience, this formula is quite widely applicable for commercial application lines containing convection ovens, but results should be verified. Whenever time-temperature changes based on calculations from this formula are made on an application line, the resulting finishes need to be accurately calculated. This explains the relatively leisurely cure reaction speed of the higher molecular binder systems in paints and powder coatings.

All this makes it virtually impossible to establish a purely scientific formula that can be used to calculate cure time-temperature curves from one single time-temperature cure schedule set.

**Using an empirical formula**

Approximately 18-20 years ago, I established an empirical formula to calculate multiple time-temperature cure schedules for commercial application line convection ovens. At that time, I was involved in a series of new product line trials for appliance, under-the-hood automotive, functional epoxy, and general metals applications. This formula is based on years of laboratory and field observations, applicator feedback, and on-line trials and errors:

\[
t_1 = \frac{t_0}{1.024 \left(\Delta T \text{ (°F)}\right)}
\]

- \(t_1\) = New cure time after temperature change
- \(t_0\) = Cure time before temperature change
- \(\Delta T\) = New temperature minus initial (base) temperature

Temperature cure schedule set.

**Example.** Calculate the cure times in 10°F intervals between 350°F (177°C) and 400°F (204°C) for a polyurethane and a TGIC (triglycidyl isocyanurate) powder coating based on the following technical data sheet information and using the (non-metric) formula for degrees Fahrenheit:

- Polyurethane: 15 min at 375°F (191°C)
- TGIC: 10 min at 400°F (204°C)

See the cure times in Table 2.

**Discussing the accuracy of an empirical formula**

In my experience, this formula is quite widely applicable for commercial application lines containing convection ovens, but results should be verified. Whenever time-temperature changes based on calculations from this formula are made on an application line, the resulting finishes need to be accurately calculated.
should be tested for full cure, for example by using the MEK cure test. (See Table 3.)

Calculating cure temperatures below the activation energy, the energy needed to start the chemical reaction, is obviously nonsensical. Unless powder coatings were clearly labeled and designated as low temperature cure, commercial application line oven temperatures below 340°F-350°F (171°C-177°C) shouldn’t be considered for standard epoxy-polyester hybrid, (emissive) polyester-polyurethane, polyester-TGIC, polyester-HAA (beta-hydroxyl alkyl amide), and polyester-TMMGU (tetra methoxy methyl glycoluril) chemistries. Powder coatings manufacturers should be consulted for low- (and possibly high-) temperature limits when applicators are using specially formulated powder coatings designed for commercial cure below 300°F (149°C).

On the other end, cure-oven conditions resulting in substrate temperatures above 410°F-420°F (210°C-216°C) can be detrimental to the formation and performance of quality standard powder finishes (except for silicone resin based products or special higher functionality epoxies) because of heat yellowing of films, volatile inclusions (micro-bubbles) and micro-pinholing in the film as a result of rapid cure, or heat degradation of phosphate pretreatments.

Based on my experience over the years, this formula produces usable results within a ±50°F (±28°C) range from cure schedules featured by powder coatings manufacturers in technical data sheets and calculated within the upper and lower temperature limits discussed in the previous paragraph. Any results from calculations beyond this range, especially on the very low or very high bake temperature side, for example for powder coatings commercially curable between 280°F-390°F (138°C-199°C), must be regarded as raw approximations only and subject to verification through line trials.

### Table 2

<table>
<thead>
<tr>
<th>Temperature (°F)</th>
<th>Polyurethane cure time (minutes)</th>
<th>TGIC cure time (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>350</td>
<td>27</td>
<td>32-33</td>
</tr>
<tr>
<td>360</td>
<td>21-22</td>
<td>26</td>
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<tr>
<td>370</td>
<td>17</td>
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<td>380</td>
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<tr>
<td>390</td>
<td>10-11</td>
<td>12-13</td>
</tr>
<tr>
<td>400</td>
<td>8-9</td>
<td>10</td>
</tr>
</tbody>
</table>

Note: Data-sheet time-temperature: Polyurethane: 15 min at 375°F (191°C); TGIC: 10 min at 400°F (204°C)

### Table 3

**Recommended procedure for solvent cure test**

**Step 1:**
Use 100 percent MEK for epoxy powder coatings or powder coatings that exhibit a high degree of solvent resistance and are relatively unaffected by strong solvents, such as MEK: mix 10 percent MEK and 90 percent xylene by volume for other powder coatings, including hybrids, polyester urethanes, and TGIC-cured polyesters; mix other ratios of MEK and xylene to more clearly discern cure in certain instances.

(MEK and xylene are flammable solvents. Consult Material Safety Data Sheets and supplier’s Technical Data Sheets. Be sure to provide adequate ventilation, consistent with accepted lab practice, to prevent solvent vapors from accumulating to dangerous levels.)

**Step 2:**
Fill squeeze bottles with MEK or MEK-and-xylene blends. Be sure to properly label each container.

**Step 3:**
Fold a soft cloth, such as cheesecloth or cotton, into a 3-inch by 3-inch square approximately ½-inch thick.

**Step 4:**
Attach the cloth to the ball end of a 2-lb ball-peen hammer with No. 18 copper wire, for example. (The hammer provides a constant force on the test panel and helps eliminate operator dependence, which can affect test results.)

**Step 5:**
Saturate pad with solvent blend.

**Step 6:**
Use either 100 percent MEK or blend of MEK and xylene on test panels.

**Step 7:**
Stroke, or slide, cloth-covered ball-peen hammer back and forth on test panel, looking for obvious signs of powder coating failure.

Results from this test should always be compared with known cured panels representing the same system under evaluation.

Note: This test procedure is adapted with the kind permission of the Powder Coating Institute from Technical Bulletin #8: Recommended Procedure for Solvent Cure Test (Alexandria, Va.: Powder Coating Institute, 1987).
This cure time-temperature formula is a useful tool for engineers and technicians running oven temperature profilers to estimate the percentage of cure. However, line operators are primarily interested in determining oven dwell times over a given temperature range. Therefore, once oven bring-up times are known over a preferred temperature range (for example, by using oven temperature profilers), or can be estimated with a degree of certainty, the cure time-temperature formula can be modified to determine oven dwell time:

\[
t_{DT} = (t_{BUT} + t_1) = t_{BUT} + \frac{t_0}{1.024 \left( \frac{\Delta T}{900} \right)}
\]

\(t_{DT}\) = Oven Dwell Time
\(t_{BUT}\) = Oven Bring-Up Time (known)

For Celsius, use the same formula; however, use 1.0436, instead of 1.024.

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References


**Editor’s note**

For further reading on the topics discussed in this article, see *Powder Coating* magazine’s Web site at [www.pcoating.com]. Click on Article Index and search by subject category. Have a question? Click on Problem solving to submit one.

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