Crosslinkers

Power to the Powder

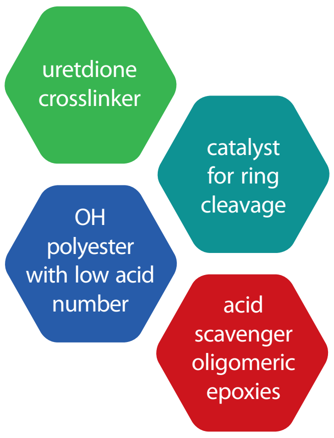
The second generation of LTC (Low Temperature Cure) PUR powder coatings is exhibiting significant improvements in storage stability. A proper design of the polyol is a new key element for overall performance of LTC systems. This powder coating technology is targeted for heat sensitive substrates like MDF, pre-assembled parts, electronic applications, aluminum substrates, etc.. Also, heavy metal parts can be powder coated with more energy efficiency.

Powder coatings and their success story have been the subject of much discussion since the 1960s. The more than 1 million metric tons produced worldwide are far from reaching the saturation limit. Growth is still predicted to be good, particularly in the Far East. Producers intend to increase their market share, even in well established powder coating markets, by opening up new market segments. To do so, significant innovations are being sought in these regions.

A general limitation of powder coating technology has been the coating of heat-sensitive parts, particularly with weather-stable systems. A new approach for doing this is provided by a recently discovered catalyst system with an additional stabilizer and activator for blocking agent-free polyurethane systems, which has already been discussed here.

With the so-called second generation of this development, we have now eliminated some crucial drawbacks.

To recap once more, four “elements” are needed as keys to this system.



uretdione crosslinker

catalyst for ring cleavage

OH polyester with low acid number

acid scavenger oligomeric epoxies

The catalyst tetraethylammonium carboxylate (VESTAGON® EP-RC 8020) induces ring cleavage of uretdione crosslinkers, for example, VESTAGON® EP-BF 9030, at considerably lower temperatures (120–150 °C) than purely thermal cleavage at ca. 170–180 °C (time/temperature function).

However, this catalyzed ring cleavage occurs at 120–150 °C only when no COOH groups are in the reaction mixture, namely, the powder coating. The hydroxyl polyesters generally used for manufacturing PUR powder coatings contain small amounts of carboxyl groups, which thus initially reduce reactivity and elevate storage stability.

To achieve high reactivity, they must then be removed from the reaction mixture for the above-stated reason. An acid scavenger (i.e. triglycidylisocyanurate or homologs) are used for this purpose.

The low-temperature system presented here offers some obvious improvements over the preceding system.

- Better surfaces with adequate storage stability and good mechanical properties
- Reduction of the crosslinking ratio NCO:OH from 2.0–1.7:1 to 1.4–1.2:1

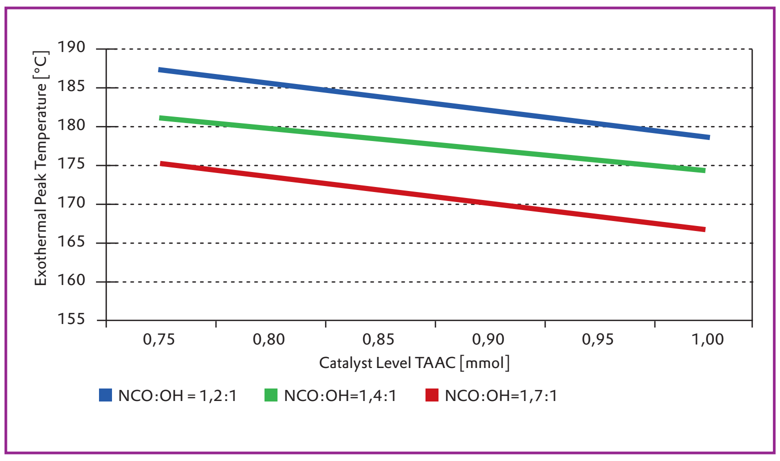
The following Table 1 shows some starting formulation examples with different NCO:OH ratios and a catalyst concentration of 0.75 mmol TAAC:

	NCO:OH 1.2:1	NCO:OH 1.4:1	NCO:OH 1.7:1
VESTAGON® EP-BF 9030	18.99	21.14	24.01
Fine-Clad® M 8078	46.20	43.82	40.64
VESTAGON® EP-RC 8020	2.06	2.29	2.60
Araldit® PT 912	1.25	1.25	1.25
Kronos® 2160	30.00	30.00	30.00
Resiflow® PV 88	1.00	1.00	1.00
Benzoin	0.50	0.50	0.50
Total	100.00	100.00	100.00

Table 1

These mixtures of catalyst with crosslinker, crosslinker with polyester and polyester acid with acid scavenger TGIC or homologs are extremely sensitive and influence the following parameters:

1. Reactivity
2. Storage stability
3. Smoothness
4. Mechanical properties



Graph 1: Influence of Catalyst Level on Exothermal Peak Temp.
Comp. of EP-BF 9030 plus Fine-Plus M 8078 at Different Stoichiometric Ratios

Graph 1 illustrates the catalyst-crosslinker and crosslinker-polyester coherences.

As we see from this figure, an increase in catalyst from 0.75 to 1.0 mmol reduces the peak temperature by approximately 7 °C. Likewise, an increase in the crosslinking ratio from 1.2:1 to 1.4:1 or from 1.4 to 1.7:1 causes a peak temperature decrease of approximately 6 °C in each case. As mentioned further above, a clear cut advantage over the first generation of the low temperature system here is the potential to appreciably reduce the uretdione crosslinker in the NCO:OH ratio from 1.7–2.1:1 to 1.4:1 and even 1.2:1. This reactivity is reflected in the achievable curing temperatures at which desired mechanical properties are obtained. One must take into account that a further increase in reactivity is detrimental to storage stability and surface appearance at elevated storage temperatures.

In Table 2, a comparison of generation 1 and 2 of the low temperature system illustrates the distinct advantages of the new generation in terms of storage stability at high temperatures.

In the following graphs 2 and 3, we find coating data for the system VESTAGON® EP-BF 9030 with Fine-Plus M 8078 and 0.75 mmol catalyst and different crosslinking ratios.

Storage Stability of New LTC Formulations
0.75 mmol catalyst; NCO:OH = 1.4:1

	Cure Conditions 30 min@150 °C:			
Storage@40 °C	Initial	1 Week	2 Weeks	4 Weeks
PCI Smoothness (1–10)	4–5	4	3	2
60° gloss	93	92	92	92
Impact (D/R) (in. -lb.)	160/140	120/120	160/140	70/40
Cupping test (mm)	>10	>10	>10	>10
Storage@23 °C	Initial	1 Week	2 Weeks	4 Weeks
PCI Smoothness (1–10)	4–5	4	4	4
60° gloss	93	92	92	92
Impact (D/R) (in. -lb.)	160/140	110/80	160/160	160/160
Cupping test (mm)	>10	>10	>10	>10

Graph 3: Properties of recommended formula after 7, 14 and 28 days of storage at 23 and 40 °C

In other words, we always obtain sprayable powder coatings, though the most sensitive parameter, smoothness, changes depending on formulation, storage time, and storage conditions. The storage at room temperature is extremely stable, showing very little change in all values throughout the entire four week period. The elevated storage temperature of 40 °C again illustrates the improvements in the system with Fine-Plus M 8078. In this case, storage of up to two weeks at 40 °C with only minor changes in these values is within the realm of possibility, particularly when our recommendations are followed and a crosslinking ratio NCO:OH of 1.4:1 is used. The formulator can control final properties within certain limits by means of varying the above discussed coating parameters. The achieved properties make it possible to open up uses for powder coating in applications that were previously ruled out. Nonetheless, our goal is to further improve low temperature curable, storage-stable powder coatings to find increase the novel uses for polyurethane powder coatings.

Table 2: Evaluation of storage stability at 40 °C based on DIN ISO 8130-8

	First Generation	Second Generation
Storage Time, Days (d)	VESTAGON® EP-BF 9030 Crylcoat 2839-0 1.7:1	VESTAGON® EP-BF 9030 Fine-Plus M 8078 1.4:1
1	5	1
7	5	2
14	5	2
28	5–6	2

Key
1 Very good fluidity
2 Good fluidity
3 Low agglomeration
4 Severe agglomeration, no longer completely pourable even after tapping
5 Agglomerated into a single piece, dispersion possible only by machine
6 Product sintered, volume reduction

Properties and Stability of New LTC Formulations
0.75 mmol catalyst

	NCO:OH 1.2:1		NCO:OH 1.4:1		NCO:OH 1.7:1	
Initial Coating Properties:						
Cure Temperature, 30 min	130 °C	150 °C	130 °C	150 °C	130 °C	150 °C
PCI Smoothness (1–10)	5	5	4–5	4–5	4	4
60° gloss	86	94	83	93	82	92
Impact (D/R) (in. -lb.)	40/<10	80/20	60/<10	160/140	140/60	160/160
Cupping test (mm)	10	10	>10	>10	>10	>10
After 1 Week, 40 °C Exposure:						
Cure Temperature, 30 min	130 °C	150 °C	130 °C	150 °C	130 °C	150 °C
PCI Smoothness (1–10)	4–5	4	3	4	3–4	3–4
60° gloss	77	94	76	92	74	93
Impact (D/R) (in. -lb.)	40/<10	140/120	100/<60	120/120	100/40	160/100
Cupping test (mm)	>10	10	10	>10	>10	>10

Graph 2: Values at different crosslinking ratios initial and after 7 days of storage at 40 °C

Technical Contact:

Europe + Middle East
werner.grenda@evonik.com

Americas + Asia
corey.king@evonik.com

www.vestagon.com