# New Developments in Radiation-Curable Powder Coatings

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*Radiation-curable powder coatings are now in the early stages of commercialization. For years, the coatings industry has sought ways to lower the curing temperature of powder coatings. UV-curable powder coatings offer the possibility of curing powder coatings at temperatures as low as 120°C. Radiation-curable powder coatings also offer high cure speed and relatively low energy consumption. As each application requires its own coating performance, two different maleate-vinyl ether-based binders for UV-curable powder coatings have been developed. With these binders the balance between flexibility and hardness of the coating can be adjusted with proper formulation. In this paper, the curing characteristics and coating performance of several UV-powder coating formulations are described. In addition, a comparison between conventional powder coatings and UV-powder coatings is provided.*

For sometime, it has been thought that the UV curing of powder coatings has potential.[1] In the meantime several publications about UV-powder coatings have appeared.[2] Recently, UV-powder coatings are reported to be in the early stages of commercialization.[3] Why did it take so long for UV-powder coatings to become commercially available, since the use of UV-powder coatings may have the following advantages: virtually no emission of volatile organic compounds, high cure speed, maximum material utilization by re-using the overspray, minimal health risk by application of high molecular weight materials, and application of thick layers compared to standard liquid coatings?

Part of the answer might be due to the fact that UV-powder coating is not just another powder coating, and the perception that the technology is just too new. Admittedly, it is a completely new technology. UV-powder technology not only requires different resins than standard powder coatings, sometimes slightly different processing of the resins is required to obtain a good powder coating. Likewise, special components need to be selected or developed, such as efficient solid photoinitiators, pigments, and UV-stabilizing packages. In addition, the curing technology differs from standard thermal curing. Therefore, paint manufacturers, resin suppliers, and those who apply the new UV-powder coatings should become familiar with UV curing, including the effect of different UV lamps, the influence of level and type of photoinitiator, and the possibilities for pigmentation. In this technology the availability of equipment plays an important role. Equipment for the application of powder coatings, including IR lamps and UV lamps, have been in existence for a long time. It is only recently, however, that the parts have been combined, and the complete system developed and optimized to obtain good coating properties. Furthermore, equipment to cure UV-powder coatings on three-dimensional objects is now being set up for research purposes.[4] In conclusion, the development of this new technology will require close cooperation between all parties involved.

Originally, UV-powder technology was thought to provide an excellent opportunity for coating wooden substrates.[5] However, the first reported commercial application is on metal substrates.[3] Potential advantages for the use of UV-powder coatings might be in applications where it is impossible to heat the entire object to 180-200°C, as, for example, large objects, composites, or components that contain temperature sensitive materials like grease or oil or electronic components. In addition, possibilities for application on coil have been mentioned.[6] The start of this new technology on metal might be due to the fact that most of the powder coating manufacturers and companies who apply powder coatings have more experience with metal substrates than with wood. In addition, wooden substrates require very low temperatures (below 100°C) and natural wood, like beech, oak, and maple, is more difficult because it is a "living" material (in contrast to composite materials such as fiber boards). Since each application requires its own coating performance, different binder systems for UV-powder coatings have been developed. Binder systems for application medium density fiber board (MDF) and on metal substrates are described in this paper.

## PHOTOPOLYMERIZATION OF UV-POWDER COATINGS

In general, two types of photopolymerization reactions may be used for UV-powder coatings: cationic photopolymerization and free radical photopolymerization. Solid bisphenol A type epoxy resins[7] or vinyl ethers may be used for the cationic photopolymerization. In most cases the polymerization of UV-powder coatings is based on free radical polymerization of unsaturated compounds, for example methacrylated polyesters,[8] or the combination of an unsaturated polyester with a solid urethane acrylate.[9] Binder systems for UV-powder coatings based on unsaturated polyesters and vinyl ether crosslinkers have also been developed and are discussed in this paper.[10]

In theory, the polymerization mechanism of the unsaturated polyester containing maleate or fumarate groups with the vinyl ether crosslinker may proceed via two mechanisms (*Figure* 1). The first mechanism is the free radical initiated

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1. Formation of Donor Acceptor complex:

   MA + VE → [MA – VE]

   Homopolymerization of Donor Acceptor complex:

   PI → R • + [MA - VE] → R-MA-VE• + [MA - VE] → R-MA-VE-MA-VE•

2. Copolymerization via cross-propagation:

   PI → R• + MA → R-MA• + VE → R-MA-VE• + MA → R-MA-VE-MA•

*Figure 1—Mechanism of maleate-vinyl ether alternating copolymerization.*

1:1 copolymerization,[11] which involves a copolymerization via homopolymerization of the maleate-vinyl ether donor-acceptor complex. The other mechanism proceeds via the cross-propagation of the electron-rich vinyl ether with the electron-poor maleate groups.

Evidence for the 1:1 copolymerization can be drawn from Real Time Fourier Transform Infrared (RT-FTIR) experiments. RT-FTIR is a versatile technique that allows monitoring of the photocuring reaction in an experimental set-up. This technique provides information about the reaction rate of the photopolymerization and the induction time related to oxygen or moisture inhibition. In a specially designed RT-FTIR apparatus (*Figure* 2),[12] the UV curing of a molten powder coating can be studied. In this apparatus the powder can be heated to different temperatures using a heating rod. The infrared (IR) light is directed through the molten powder via mirrors and detected. This technique provides information about the curing reaction, but does not allow direct translation to the results of the curing of a UV-powder coating on a real IR-UV line; these lines are usually equipped with different UV lamps (intensity, emission spectrum) and IR heating instead of heating via conduction. It does, however, allow the monitoring of different types of monomers that are polymerizing.

*Figure* 3 shows the rates of consumption of double bonds of the vinyl ether groups and fumarate groups of an experimental UV-powder clear formulation. The rates of double bond consumption are of the same magnitude. In this set-up, the rate of double bond conversion is approximately 70-80%[13] after six seconds at 110°C. *Figure* 3 also shows that there is almost no induction period in this photopolymerization reaction. It is known that maleate-vinyl ether systems are less prone to oxygen inhibition than acrylate systems.[13] As a result of the 1:1 copolymerization, a minimum of unreacted double bonds will remain after curing, when a stoichiometric balance of maleate and vinyl ether groups is used initially.

## MANUFACTURING OF THE UV-POWDER COATING

UV-powder coating formulations usually consist of the maleate resin, the vinyl ether crosslinker, a flow additive, and a suitable photoinitiator. These ingredients are mixed via melt-extrusion under normal conditions. As the UV-powder coating is designed to flow at a relatively low temperature, the melt viscosity of the UV-powder coating is much lower compared to a standard powder coating system.[15] Therefore, the extrusion should be performed at a temperature of 70°C. At this temperature the binder system allows for standard processing in a conventional powder coating manufacturing process. If the resulting extrudate is very sticky, the throughput of the extruder and the chilling capacity require adjustment. A high throughput may lead to an increase in temperature in the extruder due to increased internal friction, which in turn leads to a lower viscosity of the extrudate. During the manufacturing of the UV-powder coating, we have used standard conditions with respect to ambient light. No precaution to shield away the UV light is necessary during processing of the powder coating. Even UV powder that has been standing at ambient temperature in transparent boxes in the lab proved to be stable and no preliminary polymerization could be detected. To store the powder coating for an extended time, it is advisable to store the powder under dry and dark conditions.

The UV-powder coating is chemically stable during storage because polymerization will only be initiated by UV radiation. It has been shown by photo differential scanning calorimetry experiments that the crosslinking only proceeds after UV radiation at elevated temperature. Irradiation of the powder with UV light at room temperature possibly only leads to partial consumption of the photoinitiator. At room temperature, the molecular mobility is too low to allow

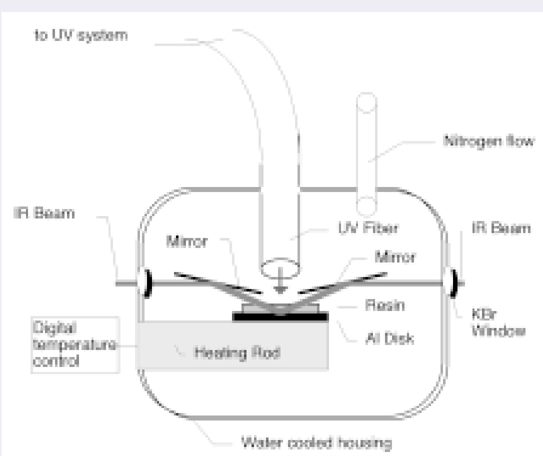


*Figure 2—Set-up for RT-FTIRmeasurements.*

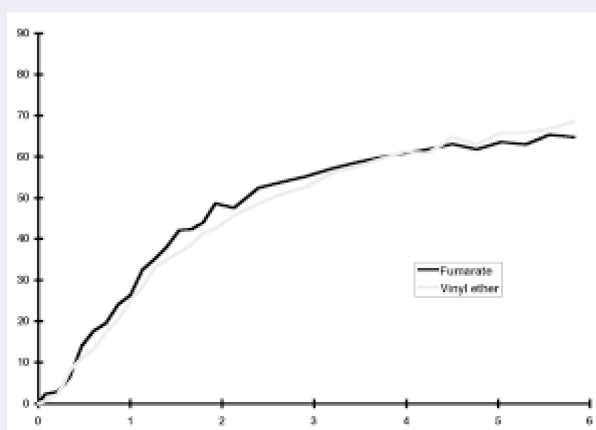


*Figure 3—RT-FTIR conversion of double bonds of an experimental fumarate-vinyl ether binder system*

for network formation. Above 80°C, however, the molecular mobility is high enough to ensure a good curing.[15] In our investigations, we have seen that the UV-powder only cures when exposed to UV light in the molten stage in a thin film. In clear coatings, we have used the hydroxy-acetophenones photoinitiators like Irgacure™ 184 or 2959. For the curing of pigmented formulations, the combination of the photoinitator, the pigment, and the UV lamp should be optimized.[16] Since the pigment absorbs UV light, a photoinitiator should be chosen which absorbs at different wavelengths than the pigment. To obtain good curing, a UV lamp should then be chosen that emits UV light at wavelengths where the photoinitiator absorbs. In general, good results with white pigmented coatings have been obtained with the bisacylphosphinoxide (BAPO) photoinitiators[17] such as Irgacure™ 819 or 1800, which absorb at longer wavelengths. In general, longer wavelength UV light seems to penetrate deeper into the coating, thereby facilitating the through cure. For price reasons, usually mixtures of the BAPO photoinitiators and hydroxyketone photoinitiators are used, although it is possible to cure a coating with Irgacure™ 819 alone.[15] In our study we used Kronos™ 2160 $TiO_2$, a standard outdoor durable pigment for powder coatings. The radical-induced polymerization of maleate-vinyl ether systems will not be hampered by high humidity or basic components, in contrast to cationic curing which may be inhibited by atmospheric moisture as well as by certain water containing pigments additives or substrates.

## APPLICATION, MELTING, AND CURING

The UV-powder coating may be applied via conventional spray application. On metal substrates, corona charging gives good results. On wooden substrates or when thick layers are applied, tribocharging is found to be more effective. The UV-curable powder coatings are cured by a combination of IR and UV radiation (*Figure* 4). The powder particles are melted with IR radiation (or a combination of IR and convection heating) and a smooth film is obtained after coalescence of the particles. While the film is still warm, the panel moves into the UV unit. UV radiation activates the photoinitiator and, with the generation of free radicals, the photopolymerization starts. After a few seconds, the film is cured. The coating reaches its full properties after the coating has cooled down to a temperature of <50°C; the time depends on the type and size of object used. The exact curing conditions can have a pronounced influence on the coating appearance and properties as described earlier.[15]

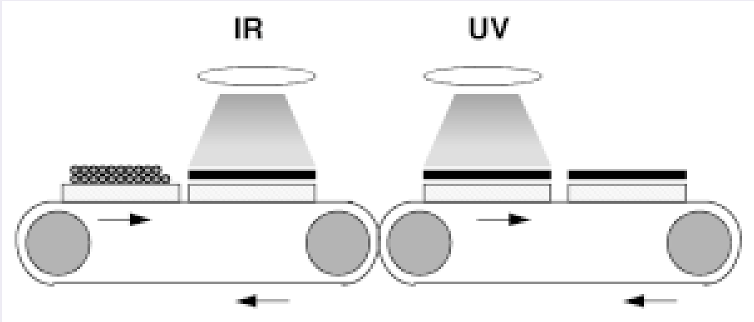


*Figure 4—Combined IR-UV curing line.*

## COATING PROPERTIES

As each application requires its own coating performance, different vinyl ether crosslinkers have been developed. The use of different crosslinkers allows for formulation freedom and the adjustment of the balance between hardness and flexibility. Coatings on wooden substrates generally require scratch resistance, hardness, and a high chemical resistance. On metal substrates, however, more flexibility is required to allow for impact resistance and bending of the coating.

## COATINGS ON MEDIUM DENSITY FIBERBOARD

In *Table* 1, the coating properties of a clear and a pigmented (15% $TiO_2$) coating on medium density fiberboard (MDF) are summarized. In this case, a stoichiometric ratio of the maleate and vinyl ether groups was used. The coatings were applied at a thickness of approximately 100 µm. After melting the powders at 100°C, good flow was obtained (determined visually). The solvent and chemical resistance of both systems is good, as the coatings withstand more than 100 acetone double rubs. The main difference is the hardness as de-

**Table 1—Properties of the Formulations for MDF Coatings**

| Formulation with Crosslinker 1 | Clear | 15% $TiO_2$ |
|---|---|---|
| Flow | good | good |
| Appearance | good | good |
| MEK resistance[a] | ++ | ++ |
| Acetone resistance[a] | ++ | ++ |
| Pendulum hardness (s)[b] | 188[c] | 145[d] |
| Adhesion[e] | 0 | 0 |

The clear formulation contains 81.6 wt% Uracross P3125, 16.7 wt% Uracross P3307, 1 wt% Irgacure 184, and 0.66% BYK 361. The pigmented formulation contains 15 wt% $TiO_2$ Kronos 2160, 68.3 wt% Uracross P3125,14.0 wt% Uracross P3307, 2 wt% Irgacure 1800 and 0.66% BYK 361. Extrusion: Prism 16 mm extruder, 200 rpm, 70°C. Application on MDF with a tribo gun. 90 s melt at 100°C under IR lamp, UV-cure: 1000mJ/cm², IL 390B light bug).
(a) ++ : no damage after 100 double rubs. ± : surface slightly damaged.
(b) Pendulum hardness König, DIN 53157, measured on MDF.
(c) The pendulum hardness on aluminium is 215 sec.
(d) After one day 81 sec, after 3 weeks 92 sec, after 3 months 145 sec.[18]
(e) Gitterschnitt scale 0-5; 0 is excellent, 5 is poor, DIN 53153/ISO 2409.

**Table 2—Clear Formulation for Metal Coating**

| Formulation with Crosslinker 2 | Clear |
|---|---|
| Flow | + |
| Appearance | good |
| Acetone resistance[a] | ++ |
| Pendulum hardness (s)[b] | 90 |
| Adhesion (GT-A)[c] | 0 |
| ESP (mm, alu)[d] | >6.0 |
| Impact (ip, alu)[e] | 40 |

The clear formulation contains 53.1 wt% Uracross P3125, 45.2 wt % Uracross P3898, 1 wt% Irgacure 184, and 0.66% BYK 361. Extrusion: Prism 16 mm extruder, 200 rpm, 70°C. Application on aluminum with a corona gun. 90 sec melt at 120°C under IR lamp, UV cure: 1000mJ/cm², IL 390B light bug).
(a) ++ : no damage after 100 double rubs. ± : surface slightly damaged.
(b) Pendulum hardness König, DIN 53157, measured on aluminum.
(c) Gitterschnitt scale 0-5; 0 is excellent, 5 is poor, DIN 53153/ISO 2409.
(d) Cupping test, Ericksen slow penetration, ISO 1520, scale 0-6.0 mm on aluminum, 6.0 is maximum.
(e) Reverse impact, ISO 6272, scale on aluminum 0-60 in.-lb, 60 maximum.

**Table 3—Comparison Between Standard Powder Coatings and the UV-Powder Coating**

| Test | UV-Powder (Crosslinker 2) | Hybrid PE-Epoxy[f] | Standard PE, TGIC[g] | Super Durable PE, TGIC[h] |
|---|---|---|---|---|
| Flow | 120°C, + | 180°C, + | 200°C, ± | 200°C, ± |
| Appearance | ok | ok | ok | ok |
| Acetone resistance[a] | ++ | ± | ± | ± |
| Pendulum hardness (s)[b] | 90 | 210 | 210 | 210 |
| Adhesion (GT-A)[c] | 0 | 0 | 0 | 0 |
| Impact (ip, alu)[d] | 40 | 60 | 60 | 20 |
| ESP (mm, steel)[e] | >8.0 | >8.0 | >8.0 | >8.0 |

(a) ++ : no damage after 100 double rubs. ± : surface slightly damaged.
(b) Pendulum hardness König, DIN 53157, measured on aluminum.
(c) Gitterschnitt scale 0-5; 0 is excellent, 5 is poor, DIN 53153/ISO 2409.
(d) Reverse impact, ISO 6272, scale on aluminum 0-60 in.-lb, 60 maximum.
(e) Cupping test, Ericksen slow penetration, ISO 1520, scale 0-8.0 mm on steel, 8.0 is maximum
(f) The formulation contains ratio resin/crosslinker 50:50 Uralac P5125 and epoxy resin (Araldite GT 7004 or DER 663 or Epikote 3003), flow additive BYK 365 and benzoin. Curing 12 min 80°C.
(g) The formulation contains ratio resin/crosslinker 93:7 Uralac P2400 and TGIC (Araldite PT 810), flow additive BYK 365 and benzoin. Curing 10 min 200°C.
(h) The formulation contains ratio resin/crosslinker 93:7 Uralac P6600 and TGIC (Araldite PT 810), flow additive BYK 365 and benzoin. Curing 15 min 200°C.

scribed elsewhere.[18] The $T_g$ of the cured clear coating is 55-60°C, measured with a DSC 2920 from TA Instruments with a heating rate of 5°C/min.

## COATINGS ON METAL

Especially for coatings on metal substrates, another crosslinker has been developed. The coating characteristics of a prototype UV-curable powder coating with this crosslinker are described in *Table* 2. In this case a thickness of 80 µm was applied on aluminum. The coating was molten at 120°C to obtain a good flow. This coating also has a high solvent resistance. The pendulum hardness is lower compared to the coating on MDF. The adhesion on aluminum measured with a Gitterschnitt test is good. On aluminum, the maximum value is obtained in the cupping test (Erichsen slow penetration, (ESP)), and an impact of 40 in.-lbs.

*Table* 3 provides a comparison between the newly developed UV-powder coating, a hybrid polyester-epoxy coating, a standard polyester-TGIC coating and a super durable polyester-TGIC coating (super durable means clearly better in outdoor durability, usually defined with the U.S. quality standard AAMA 603.8-92 or 605.2-92). The flow of the UV-powder coating at 120°C is comparable with a hybrid at 180°C and better than a standard polyester at 200°C. The solvent and chemical resistance of the UV-powder coating is clearly better compared to the standard thermal powder coatings. The pendulum hardness of the UV-powder coating is lower compared to the standard powder coatings. The adhesion and cupping test results on steel are good in all cases. The impact of the prototype UV-powder coating lies generally between the values of a standard polyester -TGIC and a super durable polyester-TGIC coating. In comparison with standard powder coatings for metal applications, UV powders may offer better flow, higher solvent resistance, quicker curing at lower temperature, and comparable flexibility.

To adjust the flexibility and the hardness of the UV-powder coating, mixtures of the two crosslinkers may be applied. In *Table* 4, different ratios of the vinyl ether crosslinker 1 and 2 are described. In all cases, the amount of resin has been adjusted to the total amount of vinyl ether groups. The formulations vary between a hard coating and a flexible coating with a good adhesion on metal. Depending on the application and the required coating characteristics, the optimum between hardness and flexibility might be chosen.

**Table 4—Different Ratios of Crosslinkers**

| Crossliner 1/ Crosslinker 2 (wt%) | Acetone Double Rubs[a] | Pendulum Hardness (s)[b] | Adhesion (GT-A)[c] | Impact (ip, alu)[d] | ESP (mm, alu)[e] |
|---|---|---|---|---|---|
| 100/0 | >100 | 195 | 5 | <4 | 4.0 |
| 40/60 | >100 | 182 | 0 | <4 | >6.0 |
| 20/80 | >100 | 144 | 0 | 10 | >6.0 |
| 7/93 | >100 | 112 | 0 | 20 | >6.0 |
| 0/100 | >100 | 90 | 0 | 40 | >6.0 |

The clear formulation contains ratio of resin/crosslinker as in *Table 4*, 1 wt% Irgacure 184, 0.66% BYK 361. Extrusion: Prism 16 mm extruder, 200 rpm, 70°C. Application on aluminum with a corona gun. 90 sec melt at 120°C under IR lamp, UV-cure: 1000mJ/cm$^2$, IL 390B light bug).
(a) ++ : no damage after 100 double rubs. ± : surface slightly damaged.
(b) Pendulum hardness König, DIN 53157, measured on aluminum.
(c) Gitterschnitt scale 0-5; 0 is excellent, 5 is poor, DIN 53153/ISO 2409, tested on aluminum.
(d) Cupping test, Ericksen slow penetration, ISO 1520, scale 0-6.0 mm on aluminum, 6.0 is maximum.
(e) Reverse impact, ISO 6272, scale on aluminum 0-60 in.-lb, 60 maximum.

## CONCLUSIONS

Several solid maleate-vinyl ether binder systems for UV-powder coatings have been described. With these binder systems, the balance between hardness and flexibility of the coating can be optimized depending on the requirements. In the UV-powder coating technology, the coatings are cured by a combination of IR and UV radiation at temperatures as low as 120°C. In this way application areas that were not viable for powder coatings might now be open to UV-powder coatings. UV-curable powder coatings for MDF, paper,[19] and metal substrates have been prepared.

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