

# UV LED Curing The Booster Resin Approach

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# CYTEC

- Introduction
- The Booster Resin Approach
- Application Program
  - Clear Coatings

#### Reactivity

- LED Line settings (Distance LED to substrate, LED orientation)
- Coating Thickness
- Formulation Viscosity & Dilution
- Dosage of Booster Resin

Gloss, Yellowing

Stain resistance

- Pigmented Coatings
- Conclusions



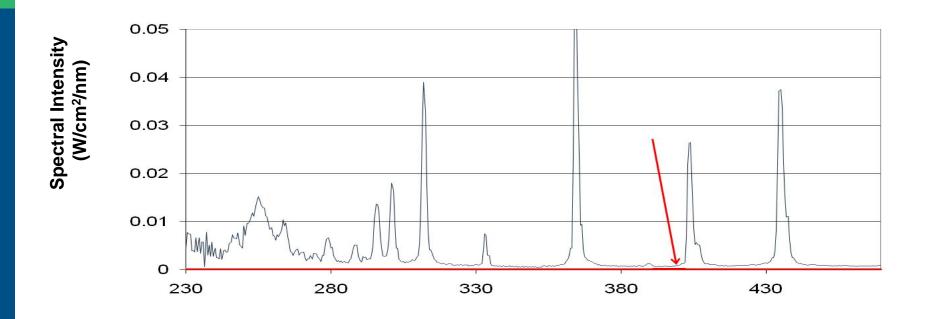
#### **Value Proposition LED**

- ✓ Allows coating of heat sensitive surfaces
- ✓ Instant on, instant off
- ✓ Ability to coat small surfaces
- ✓ No ozone generation
- ✓ Others...

Could enable new applications which up to now were not feasible with standard high powered lamps



# Comparison spectral intensity of a 80W/cm Mercury lamp with a 1W/cm<sup>2</sup> LED 395nm



Energy output of LED is much lower!



#### The first trials

Urethane Acrylate, 3f	85	85	85	75	85
DPGDA	10	10	10	10	10
Amine modified PEA, 4f			5	10	
Amine synergist, 2f					5
Phosphine oxide PI	5	5	5	5	5
Inert atmosphere	Yes	No	No	No	No
Reactivity Finger Nail Resistance (m/min)	> 80	4x5 NOK	4x5 NOK	4x5 NOK	4x 5 NOK

Application : 30μ by Barcoater on Leneta paper

Curing: LED 8W/cm<sup>2</sup> 395 nm, 1cm distance from substrate to LED

(perpendicular orientation)

Surface curing is an issue!



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- Transform a UV curable formulation into a UV LED curable by replacing part of the oligomer by a « booster » resin to assist in cure
- This led to the development of the new Booster Resin
- The approach is also valid for other low energy curing UV technologies (e.g. UV-A curing)



#### **Example**

Urethane acrylate, 3f	85	65
DPGDA	10	10
Booster Resin		20
Phosphine oxide PI	5	5
Reactivity Finger Nail Resistance (m/min)	4x 5 NOK	1x 5

Application : 30μ by Barcoater on Leneta paper

Curing: LED 8W/cm<sup>2</sup> 395 nm, 1cm distance from substrate to LED

(perpendicular orientation)

The Booster Resin is a co-resin, used to increase surface cure



#### **Booster Resin- Physical Data**

- Appearance
- Viscosity, 25°C, cP
- Colour
- Boiling point
- Vapour Pressure
- Flash Point
- Stability

Liquid

210

pale, pinkish

>200°C

< 1.33 hPa @ 20°C

> 100°C Cleveland Open Cup

>10 days @ 60°C



#### **Booster Resin - Compatibility**

Urethane acrylate, 6f	75	55			
Urethane acrylate, 4f			45		
Urethane acrylate, 3f				45	
Urethane acrylate, 2f					45
HDDA			10	10	10
Booster Resin	20	40	40	40	40
Phosphine oxide PI	5	5	5	5	5
Thioxanthone PI	0,1	0,1	0,1	0,1	0,1
Fingernail resistance	1 x 5	1 x 10	1 x 25	1 x 15	1 x 20

Application :10µ on Leneta

Cured with 8W /cm<sup>2</sup> 395nm LED at 2cm substrate lamp distance

**Good Compatibility with Selected UA** 



#### **Booster Resin - Compatibility**

Epoxy acrylate, 2f	65	45		
Polyester acrylate, 4f			75	55
HDDA	10	10	10	10
Booster Resin	20	40	20	40
Phosphine oxide PI	5	5	5	5
Thioxanthone PI	0,1	0,1	0,1	0,1
Fingernail Resistance	1 x 35	1 x 50	< 1 x 5	1 x 10

Application :10µ on Leneta

Cured with 8W /cm² 395nm LED at 2cm substrate lamp distance

**Good Compatibility with Selected EA and PEA** 



#### **Booster Resin - Compatibility**

Polyester acrylate, 4f	34
Polyester acrylate, 6f	10
Pigment Paste	36
Booster Resin	20
Acidic adhesion promoter	4
Phosphine oxide PI	4
Thioxanthone PI	0.5
Viscosity (mPa.s)	390

Application : 20 µm by barcoater on sanded untreated steel Cured with Panacol 254 UV-H (UVA-lamp) at 15 cm substrate lamp distance

**Good Compatibility with Acidic Adhesion Promotors** 



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## **LED Equipment**



8W/cm<sup>2</sup> LED 395 nm (2.5cm x 22.0cm)





#### **Clear Coating - Starting Point Formulation**

Urethane acrylate, 3f	65
DPGDA	10
Booster Resin	20
Phosphine oxide PI	5
Viscosity (mPa.s)	4000

Application: Barcoater on Leneta paper, thickness as mentioned Curing: LED 8W/cm<sup>2</sup> 395 nm, geometry LED and distance LED to substrate as mentioned

Urethane Acrylate is 3f with balanced properties



#### **Reactivity – Distance to Substrate & Lamp Orientation**

•	"perpendicular"	"in-line"
		G Promise Control of the Control of
Distance LED to substrate (cm)		
0.5	1X 5	1x 15
1	1x 5	1x 15
2	2X 5	1x 15

SPF, 30  $\mu$  on Leneta Reactivity (m/min) - finger nail resistance

Lower Influence of distance to substrate with "in-line" orientation





#### **Reactivity – Coating thickness**

	10μ	30μ
Distance LED to substrate (cm)		
0.5	3x 5	1x 15
1	3x 5	1x 15
2	4x 5	1x 15

SPF, on Leneta, thickness as mentioned

LED orientation: "in length"

Reactivity (m/min) - finger nail resistance

Significantly lower reactivity for thin coatings (<20μ)



#### **Reactivity - Viscosity & Dilution**

SPF

Urethane acrylate, 3f	49	57	65
DPGDA	26	18	10
Booster Resin	20	20	20
Phosphine oxide PI	5	5	5
Sum	100	100	100
Viscosity (mPa.s)	1000	2000	4000
Reactivity-Finger Nail Resistance (m/min)	1x5	1x10	1x15
Gloss 60°	90	90	90

Application : 30  $\mu$  on Leneta

Cured with 8W /cm² 395nm LED at 1cm substrate lamp distance, LED "in-line" with the substrate (22cm)

Cure speeds drops with monomer dilution



#### Reactivity – Dosage of Booster Resin at iso formulation viscosity

Urethane acrylate, 3f	65	65	65
DPGDA	10	10	10
Booster Resin		20	40
Polyester acrylate, 4f	40	20	0
Phosphine oxide PI	5	5	5
Viscosity, 25°C, mPa.s	2300	2400	2500
Reactivity – Finger Nail Resistance (m/min)	4 x 5 NOK	1 x 5	> 1 x 40

Application : 30  $\mu$  on Leneta

Cured with 8W /cm<sup>2</sup> 395nm LED at 1cm substrate lamp distance, LED "in-line" with the substrate (22cm)

#### > 40m/min with 40 parts Booster Resin



#### **Reactivity – Oligomer Functionality**

Urethane acrylate, 3f	57	
DPGDA	18	
Urethane acrylate, 6f		75
Booster Resin	20	20
Phosphine oxide PI	5	5
Viscosity (mPa.s)	2000	1400
Reactivity-Finger Nail Resistance (m/min)	1x 10	1x 40

Application : 30  $\mu$  on Leneta

Cured with 8W /cm<sup>2</sup> 395nm LED at 1cm substrate lamp distance, LED "in-line" with the substrate (22cm)

Higher surface reactivity is obtained with higher functional oligomers



#### **Reactivity - Summary**

Effect of Inc	_	Coating thickness	Monomer dilution	Booster Resin	Oligomer Function- ality
Formulation reactivity	Higher Lower				

e.g. a 30 $\mu$  hard coating will be easier to cure than a 5 $\mu$  flexible coating



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#### **Gloss**

Urethane acrylate, 3f	60
DPGDA	40
Booster Resin	20
Phosphine oxide PI	5
Silica matting agent, wax treated	8
Silica matting agent, untreated	2
Polypropylene wax	2
Viscosity, 25°C, mPa.s	2150
Reactivity – Finger Nail Resistance (m/min)	2 x 5
Gloss (60°)	65

Application : 30-35  $\mu$  on Leneta Cured with 8W /cm² 395nm LED at 1cm substrate lamp distance, LED "in-line" with the substrate (22cm)



#### Yellowing

	Hg (80W/cm)	LED	LED
Urethane acrylate, 3f	85	85	65
DPGDA	10	10	10
Booster Resin			20
Benzophenone/α-hydroxy ketone (50/50) PI	5		
Phosphine oxide PI		5	5
Yellowing, b-value 24 h (initial)	0 (1.4)	/	0.5 (2.8)
Reactivity Finger Nail resistance (m/min)	1x 25	4x5 NOK	1x 5

Application : 30μ by Barcoater on Leneta paper

Curing: LED 8W/cm<sup>2</sup> 395 nm, 1cm distance from substrate to LED

(perpendicular orientation)



	Hg (80W/cm)	LED	
Urethane acrylate, 3f	85 65		
DPGDA	10	10	
Booster Resin		20	
Benzophenone/α-hydroxy ketone (50/50) PI	5		
Phosphine oxide PI		5	
Viscosity (mPa.s)	10,100	4,020	
Acetone DR	>100	>100	
Eosine, red dye (16 hours)	4	1	
NH <sub>3</sub> , 10% (16 hours)	5	5	
Mustard (16 hours)	1	1	
Ethanol 50% (16 hours)	5	5	

Application : 30μ by Barcoater on Leneta paper

Curing: LED 8W/cm<sup>2</sup> 395 nm, 1cm distance from substrate to LED (**perpendicular** orientation)

Very similar results, except for the eosine resistance



#### **Pigmented systems**

Urethane acrylate, 3f	45	35	25
DPGDA	10	10	10
Booster Resin	20	20	20
TiO <sub>2</sub>	20	30	40
Phosphine oxide PI	5	5	5
Viscosity (mPa.s)	3620	3520	3540
30-35 μ, Finger Nail Resistance (m/min)	1 x 5	1 x 10	1 x 15
Yellowing, immediately after cure, b-value	2.7	2.5	2.2

Application : 30-35  $\mu$  on Leneta

Cured with 8W /cm<sup>2</sup> 395nm LED at 1cm substrate lamp distance, LED "in-line" with the substrate (22cm)

Good surface & deep curing possible in white pigmented systems

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#### **Conclusions**

- The Booster Resin Approach is about transforming UV formulations into LED curable formulations
- This approach is also valid for other low energy curing UV technologies (e.g. UV-A cure)
- The Booster Resin is a co-resin and replaces part of the UV oligomers in the formulation
- Application results have shown that the Booster Resin is very effective in increasing reactivity of UV LED curing systems.
- UV LED technology opens opportunities for new applications.
- Further development will be necessary to achieve specific application requirements





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#### Thank you for your attention!

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