Silicone Hybrid Resins for High Solids Topcoats

May 3rd 2018 | DSCT FOCUS Conference
Jim Reader, Brian Casey and Heiko Alzer
Presentation Overview

- Hybrid polyol technology
  - Si – polyester chemistry
  - Coating benefits
  - Application Areas
    - Performance properties as Main Binder
    - Performance properties as Co-binder
- Wrap – Up
Silicone Polyester Polyol

- Aliphatic polyester polyol
- Silicone resin
- Silicone aliphatic polyester resin

Hybrid topcoat

Substrate

Polyisocyanate curing agent
Silicone Aliphatic Polyester Resins

**advantages**
- high flexibility
- good mechanical properties
- high pigment loading capacity
- good adhesion
- chemical resistance

**advantages of hybrid resins versus cold blends**
- homogenous resin → no phase separation
- excellent weather and chemical resistance due to chemical bonding

**advantages**
- high solids content
- good chalk resistance
- increased weather stability
- increased stain resistance

aliphatic polyester part

silicone part
# Comparison of Silicone Aliphatic Polyester Polyols

<table>
<thead>
<tr>
<th></th>
<th>LA – B 1519</th>
<th>LA – B 1520</th>
</tr>
</thead>
<tbody>
<tr>
<td>solid content (butyl acetate)</td>
<td>90%</td>
<td></td>
</tr>
<tr>
<td>OH content on solid binder</td>
<td>~ 6%</td>
<td></td>
</tr>
<tr>
<td>OH number on solid binder [mgKOH / g]</td>
<td>~ 200</td>
<td></td>
</tr>
<tr>
<td>acid value on solid binder [mgKOH / g]</td>
<td>&lt; 9</td>
<td></td>
</tr>
<tr>
<td>viscosity 25°C [mPa*s]</td>
<td>~ 9000</td>
<td>~ 7000</td>
</tr>
<tr>
<td>stone chip resistance</td>
<td>⭐⭐⭐</td>
<td>⋅</td>
</tr>
<tr>
<td>flexibility (indirect impact test)</td>
<td>⭐⭐⭐</td>
<td>⋅</td>
</tr>
<tr>
<td>flexibility (ERICHSEN cupping test)</td>
<td>⭐⭐⭐</td>
<td>⭐⭐⭐</td>
</tr>
<tr>
<td>dry time (drying recorder)</td>
<td>⭐⭐⭐</td>
<td>⭐⭐⭐</td>
</tr>
<tr>
<td>degree of dryness</td>
<td>⋅</td>
<td>⭐⭐⭐</td>
</tr>
<tr>
<td>chemical resistance</td>
<td>⋅</td>
<td>⭐⭐⭐</td>
</tr>
<tr>
<td>hardness (pendulum)</td>
<td>⋅</td>
<td>⭐⭐⭐</td>
</tr>
<tr>
<td>weather resistance</td>
<td>⭐⭐⭐</td>
<td>⭐⭐⭐</td>
</tr>
<tr>
<td>VOC (ready to use)</td>
<td>⭐⭐⭐</td>
<td>⭐⭐⭐</td>
</tr>
</tbody>
</table>

- ⭐⭐⭐ especially recommended
- ⋅ recommended
Coating Benefits

Both systems provide:

► Ability to formulate high solids (low VOC) coatings
  ▶ Si – polyester hybrids have a high solids content of 90% (butyl acetate)
  ▶ Si – polyol resins have low viscosity at high solids content & enable high film thickness application (up to 200m)
► Very good appearance, film build & image development
► Single coat layer with less reaction blisters
► Excellent chemical & weathering resistance
► High compatibility with other resins
Silicone Hybrid Resins as Main Binder
High Solids and Low Viscosity

- Viscoity of pure binder adjusted with butyl acetate
- Si – polyol resins show better dilution properties & provide a lower viscosity profile at high solids contents
- Able to formulate low VOC coatings with because of good processability

→ Low VOC at low viscosity
Top Coat Guiding Formulation

Mill Base Component

<table>
<thead>
<tr>
<th>Raw Material</th>
<th>Parts by Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>LA – B 1520</td>
<td>27.2</td>
</tr>
<tr>
<td>Deaerator</td>
<td>0.3</td>
</tr>
<tr>
<td>Dispersant</td>
<td>0.9</td>
</tr>
<tr>
<td>Bentonite Clay (modified)</td>
<td>0.5</td>
</tr>
<tr>
<td>Aluminosilicate</td>
<td>0.2</td>
</tr>
<tr>
<td>Titanium Dioxide</td>
<td>31.8</td>
</tr>
<tr>
<td>Barium Sulfate</td>
<td>8.2</td>
</tr>
<tr>
<td>Butyl Glycol Acetate</td>
<td>1.5</td>
</tr>
<tr>
<td>Butyl Acetate</td>
<td>2.9</td>
</tr>
<tr>
<td><strong>Total Mill Base</strong></td>
<td><strong>73.5</strong></td>
</tr>
</tbody>
</table>

Let Down Component

<table>
<thead>
<tr>
<th>Raw Material</th>
<th>Parts by Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>LA – B 1520</td>
<td>19.9</td>
</tr>
<tr>
<td>Catalyst (1% in Butyl Acetate)</td>
<td>3.8</td>
</tr>
<tr>
<td>UV Absorber / Light Stabilizer</td>
<td>1.5</td>
</tr>
<tr>
<td>Butyl Acetate</td>
<td>1.3</td>
</tr>
<tr>
<td><strong>TOTAL</strong></td>
<td><strong>100.0</strong></td>
</tr>
</tbody>
</table>

Mixing Ratio

Coating System : Hardener 100 : 27.7

Hardener → Aliphatic Polyisocyanate

Solid content approximately 85% by weight
Coating density of 1.52 g/mL
Cup efflux time of 40 – 50 s
Calculated VOC approximately 215 g/L
- Based on Part A without hardener
Calculated VOC approximately 160 g/L
- Based on ready – to – use formulation
Drying Properties

**Drying Time – As Supplied**

<table>
<thead>
<tr>
<th>Product</th>
<th>Touch Dry</th>
<th>Through Dry</th>
</tr>
</thead>
<tbody>
<tr>
<td>LA-B 1519</td>
<td></td>
<td></td>
</tr>
<tr>
<td>LA-B 1520</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Commercial A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Commercial B</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Commercial C</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Conditions**

- Measured with dry time recorder at room temperature
- Degree of crosslinking at 105% for LA – B 1519 & LA – B 1520
- Commercial products cured per supplier TDS
- Applied at 3 mil WFT over glass
### Drying Properties as Applied

#### Conditions
- **DILUTED TO SPRAYABLE VISCOSITY**
- Measured with dry time recorder at room temperature
- Degree of crosslinking at 105% for LA – B 1519 & LA – B 1520
- Commercial products cured per supplier TDS
- Applied at 3 mil WFT over glass

#### Drying Time – As Applied

<table>
<thead>
<tr>
<th>Product</th>
<th>Touch Dry</th>
<th>Through Dry</th>
</tr>
</thead>
<tbody>
<tr>
<td>LA-B 1519</td>
<td></td>
<td></td>
</tr>
<tr>
<td>LA-B 1520 (5% solvent)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Commercial A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Commercial B (15% solvent)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Commercial C (15% solvent)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- Touch Dry
- Through Dry

-Drying Time / Hours

---

11 Public | DSCT Focus 2018 | Silicone Hybrid Resins for High Solids Topcoats
Hardness Build

Pencil Hardness

- LA-B 1519
- LA-B 1520
- Commercial A
- Commercial B
- Commercial C

Conditions

- Performed in accordance with ASTM D 3363
- Applied at 3 – 4 mil DFT over epoxy primer
- Conventional spray applied of the solvent reduced formulations

Hardness Increase

1 Month □ 1 Week □ 24 Hrs

Performed in accordance with ASTM D 3363
Applied at 3 – 4 mil DFT over epoxy primer
Conventional spray applied of the solvent reduced formulations
Pendulum Hardness Development

hardness development after two weeks

<p>| | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
<td>2</td>
<td>7</td>
</tr>
<tr>
<td>Pendulum Hardness</td>
<td>0</td>
<td>40</td>
<td>140</td>
</tr>
<tr>
<td>Time [days]</td>
<td>1</td>
<td>2</td>
<td>7</td>
</tr>
</tbody>
</table>

Degree of crosslinking: 100% and 120%

Clearcoats cured with VESTANAT® HT 2500 LV\*1 and TIB KAT® 218 at room temperature

DFT: ~90 µm

\*1 According to DESMODUR® N 3600
Crosshatch Adhesion

<table>
<thead>
<tr>
<th>LA – B 1519</th>
<th>LA – B 1520</th>
<th>Commercial A</th>
<th>Commercial B</th>
<th>Commercial C</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1" alt="Image" /></td>
<td><img src="image2" alt="Image" /></td>
<td><img src="image3" alt="Image" /></td>
<td><img src="image4" alt="Image" /></td>
<td><img src="image5" alt="Image" /></td>
</tr>
<tr>
<td>24 Hrs</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><img src="image6" alt="Image" /></td>
<td><img src="image7" alt="Image" /></td>
<td><img src="image8" alt="Image" /></td>
<td><img src="image9" alt="Image" /></td>
<td><img src="image10" alt="Image" /></td>
</tr>
<tr>
<td>1 Month</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Adhesion to Plastic Materials

Adhesion on plastic surface directly

<table>
<thead>
<tr>
<th>Substrates</th>
<th>SILIKOTOP® E 900</th>
<th>SILIKOTOP® E 901</th>
<th>Competitor</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABS, PC</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>GRP, PVC</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Blend (PPE, PS, PA)</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>PA</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>PMMA</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

Comments

- Top coat formulation based on SILIKOTOP® applied on plastic materials directly
- Plastic surface pretreatment: Roughened with soft touch pad
- Competitor: Elastified 2-pack PU topcoat standard material
Abrasión Testing

### Taber Abrasion

<table>
<thead>
<tr>
<th>Product</th>
<th>Weight Loss (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LA-B 1519</td>
<td></td>
</tr>
<tr>
<td>LA-B 1520</td>
<td></td>
</tr>
<tr>
<td>Commercial A</td>
<td></td>
</tr>
<tr>
<td>Commercial B</td>
<td></td>
</tr>
<tr>
<td>Commercial C</td>
<td></td>
</tr>
</tbody>
</table>

### Conditions

- Performed in accordance with ASTM D 4060
  - CS – 10 rolls
  - 500 grams per roll
- Applied at 3 – 4 mil DFT direct – to – metal
- Conventional spray applied of the solvent reduced formulations
Elasticity - Stone Chip Resistance

High stone chip resistance

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>LA-B1519</td>
<td></td>
<td>Rating 0.5</td>
</tr>
<tr>
<td>competitor</td>
<td></td>
<td>Rating 2.0</td>
</tr>
</tbody>
</table>

Comments

Ranking
1. LA-B1519
2. LA-B1520
3. Commercial high solids polyester

Stone chip resistance according to DIN EN ISO 20567-1

Abrasive material: chill cast iron, angular 4-5 mm according to ISO 11124-2
2x 500g material for 10 seconds each, with 2 bar
White Top Coat with 120% degree of crosslinking and DFT ~ 80 µm
Rating according to DIN EN ISO 20567-1
Corrosion Protection - Early Water Resistance

Early water resistance

<table>
<thead>
<tr>
<th>Drying Condition</th>
<th>LA-B1519</th>
<th>LA-B1520</th>
<th>Benchmark</th>
</tr>
</thead>
<tbody>
<tr>
<td>6 h RT</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Forced curing + 1 h RT</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Comments

- application on steel (Gardobond 26S 6800 OC)
- panels were half dropped in water for 24 hours after different drying conditions
- forced curing: 20° 70°C
Salt Spray Corrosion Test Results
Chemical Resistance

<table>
<thead>
<tr>
<th></th>
<th>LA-B 1519</th>
<th>LA-B 1520</th>
<th>common HS polyester</th>
</tr>
</thead>
<tbody>
<tr>
<td>sodium hydroxide solution (10 %)</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
</tr>
<tr>
<td>acetic acid (10 %)</td>
<td>⬤</td>
<td>⬤⬤⬤</td>
<td>⬤</td>
</tr>
<tr>
<td>hydrochloric acid (10 %)</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
</tr>
<tr>
<td>sulfuric acid (10 %)</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
</tr>
<tr>
<td>hydraulic oil</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
</tr>
<tr>
<td>anti-freeze</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
</tr>
<tr>
<td>pancreatin (1:1 in water)</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
<td>⬤⬤⬤</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>3</th>
<th>5</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>coffee (4 %)</td>
<td>3</td>
<td>5</td>
<td>3</td>
</tr>
<tr>
<td>mustard (medium-hot)</td>
<td>4</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>ketchup</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>red wine</td>
<td>4</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>black tea (1 %)</td>
<td>2</td>
<td>5</td>
<td>3</td>
</tr>
</tbody>
</table>

comments

- ⬤⬤⬤ very good
- ⬤ good
- ⬤ poor

Tested for 8h at room temperature, after 24h regeneration

White topcoat formulation with 120% degree of crosslinking

Food contact according to DIN 68861-1

- 5 very good
- 3 poor
- 1 worse
**Chemical Resistance**

**Spot Test**

<table>
<thead>
<tr>
<th>Material</th>
<th>Resistance Increase</th>
<th>Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>LA-B 1519</td>
<td></td>
<td>Performed in accordance with ASTM D 1308</td>
</tr>
<tr>
<td>LA-B 1520</td>
<td></td>
<td>Applied at 3 – 4 mil DFT direct – to – metal</td>
</tr>
<tr>
<td>Commercial A</td>
<td></td>
<td>Conventional spray applied of the solvent reduced formulations</td>
</tr>
<tr>
<td>Commercial B</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Commercial C</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- Bases
- Acids
- Solvents

Resistance Increase

**Variables:**
- Commercial A
- Commercial B
- Commercial C
- LA-B 1519
- LA-B 1520
QUV Weathering

<table>
<thead>
<tr>
<th>Product</th>
<th>ΔE Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>LA – B 1519</td>
<td>ΔE = 5.67</td>
</tr>
<tr>
<td>LA – B 1520</td>
<td>ΔE = 3.64</td>
</tr>
<tr>
<td>Commercial A</td>
<td>ΔE = 3.53</td>
</tr>
<tr>
<td>Commercial B</td>
<td>ΔE = 3.60</td>
</tr>
<tr>
<td>Commercial C</td>
<td>ΔE = 4.97</td>
</tr>
</tbody>
</table>
Weather Resistance - Xenon Weathering

Color Stability

Residual Gloss 20°

comments

<table>
<thead>
<tr>
<th>light stabilizer:</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5% TINUVIN® 292 and</td>
</tr>
<tr>
<td>1.0% TINUVIN® 400</td>
</tr>
</tbody>
</table>

→ very high color and gloss retention
Reflow effect

SILIKOTOP® E900 and E901 show reflow effect.

Crock meter scratches after 2h @ 80°C / 175°F heat treatment (Between red stickers)

Crock meter scratches without heat treatment

Clear coat based on SILIKOTOP® cured with Vestanat® HT 2500 LV 20 min @ 140°C / 285°F
Contact angle

Lower contact angle for recoatability

<table>
<thead>
<tr>
<th>Exposure time</th>
<th>SILIKOTOP® E 900</th>
<th>SILIKOTOP® E 901</th>
<th>High solids polyester</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 s</td>
<td>78°</td>
<td>82°</td>
<td>82°</td>
</tr>
<tr>
<td>180 s</td>
<td>75°</td>
<td>79°</td>
<td>74°</td>
</tr>
</tbody>
</table>

comments

top coat white, guiding formulation No. GF TOP E 900 9 001
DFT: 80 µm
**degree of crosslinking:** 120%
Contact angle measured immediately and after 3 minutes
Silicone Hybrid Resins as Co-Binders for Clear Coating

High Solids Clear Coating
Silicone Hybrid Resins as Co-binders for 2K Clear Coating: Non volatile content

Polyol-NCO Clear Coating

Polyol:
Nuplex Setalux 1767-VV65
LA-B 1520

Isocyanate:
Vestanat HT 2500 LV

Already hardened clear coating set on same viscosity of 18" DIN4 cup at 23°C/73°F

Nvc Test
1h @ 125°C/260°F; 1g material
Silicone Hybrid Resins as Co-Binder for 2K Clear Coating: Reflow effect

Polyol-NCO Clear Coating

Polyol:
Nuplex Setalux 1767-VV65
LA-B 1520

Isocyanate:
Vestanat HT 2500 LV

DFT
40 µm

Curing
10 min @ 23°C/73°F
30 min @ 80°C/175°F
Silicone Hybrid Resins as Co-Binder for 2K Clear Coating: Keonig Hardness Development

Polyol-NCO Clear Coating

Polyol:
Nuplex Setalux 1767-VV65
SILIKOTOP® E 901

Isocyanate:
Vestanat HT 2500 LV

DFT
40 µm

Curing
10 min @ 23°C/73°F
30 min @ 80°C/175°F
Silicone Hybrid Resins as Co-Binders for White Pigmented Top Coat

High Solids System for Low Bake
Silicone Hybrid Resins as Co-Binders for Pigmented Top Coat:
Non volatile content

Polyol-MF Top Coat (70:30)
White pigmentation

Polyol:
Nuplex Setalux 1756-65
LA-B 1520

Melamine:
Cymel 303LF

Catalyst:
PTSA

Set on same viscosity of 30" DIN4 cup at 23°C/73°F
Silicone Hybrid Resins as Co-Binders for Pigmented Top Coat: Viscosity

Polyol-Mel top coat (70:30)
White pigmentation

Polyol:
Nuplex Setalux 1756-65
LA-B 1520

Melamine:
Cymel 303LF

Catalyst:
PTSA

Set on same nvc of 68%.
Silicone Hybrid Resins as Co-Binders for Pigmented Top Coat: Reflow Effect

Polyol-Mel top coat (70:30)
White pigmentation

Polyol:
Nuplex Setalux 1756-65
LA-B 1520

Melamine:
Cymel 303LF

Catalyst:
PTSA

Curing conditions:
10 min @ RT
20 min @ 125°C/260°F (oven)
13 min > 120°C/250°F (object)
Silicone Hybrid Resins as Co-Binders for Pigmented Top Coat: Koenig Hardness Development

Pendulum hardness of a white heat curing coating in relation to the concentration of SILKOTOP® E 901

Polyol-Mel top coat (70:30)
White pigmentation

**Polyol:**
Nuplex Setalux 1756-65
LA-B1520

**Melamine:**
Cymel 303LF

**Catalyst:**
PTSA

**Curing conditions:**
10 min @ RT
20 min @ 125°C/260°F (oven)
13 min > 120°C/250°F (object)
Summary

Silicone Hybrid Resins can be used to:

- Increase the formulation solids content or
- Reduce the formulations VOC content
- Improve formulation scratch resistance by the reflow effect
- Positively influence the corrosion resistance
- Positively influence the humidity tests
- Positively influence the stone chip resistance

- Silicone Aliphatic Polyester Polyols can be cured with Isocyanate or with Melamine based crosslinker
- Silicone Aliphatic Polyester Polyols can be used as Main Binder or as Co-Binder
## Application Areas

- **heavy – duty protective coatings**
- **marine coatings (above the water line)**
- **ACE (agriculture, construction & earthmoving)**
- **trucks & public transport**
- **wood coatings**