

# Adhesives

The Adhesives Magazine of Evonik Industries

Issue 01 | 17



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## Dear readers,

Discover our “Power to create” for the adhesives and sealants industry. Permanently bonding a variety of materials and substrates to one another poses ever new challenges for users and producers of adhesives. Sustainability and efficient use of resources are also important considerations in our research.

Cutting-edge electronics demands nanotechnology, which allows handling of the finest components. Water-repellent sealants and transparent hybrid systems for the construction industry are becoming increasingly important, and adhesive joining techniques are being more widely used in the automotive industry.

To keep pace with these trends we rely on close collaboration with our customers. As a reliable partner, we endeavor to spot market developments early, understand them, and, jointly with our customers, develop innovative solutions for them. In this new edition of the Evonik Adhesives Magazine we want to present to you some of the results of this research and collaboration.

Learn more about our latest developments like transparent hybrid sealants, polyesters for two-component PU hot melt adhesives for car body construction, and new adhesives for microelectronics.

We’re sure you’ll be keenly interested in these and other innovations for your adhesives and sealants applications.

Enjoy reading!

Claus Rettig

Chairman of the Board of Management  
Evonik Resource Efficiency GmbH

# AEROSIL® & Dynasylan®: A perfect team in transparent hybrid sealants

As an important manufacturer of fumed silicas and organo-functional silanes (brand names AEROSIL® and Dynasylan®), Evonik constantly strives to offer innovative product solutions for the growing market demands and for increasing customer requests in the adhesives and sealants industry.



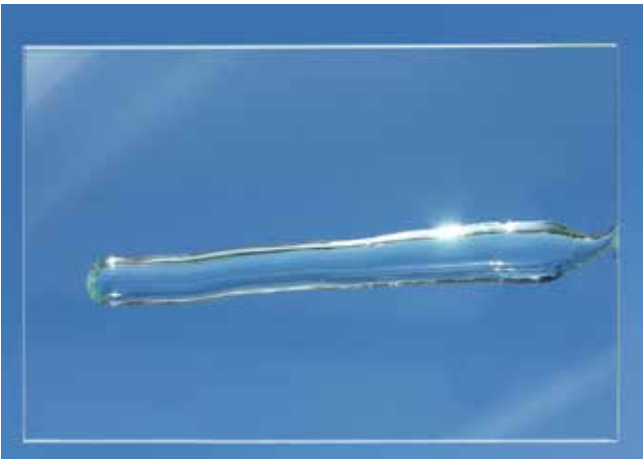
Wellness oasis. In modern bathrooms highly transparent sealants are very popular.

Adhesives and sealants based on silane modified polymers (also named SMP or hybrid) are a strongly increasing product class for a multitude of applications. By chemistry, they are based mainly on STPU and STPE polymers. Besides chalk filled systems, also transparent systems are becoming increasingly important. In these transparent adhesives and sealants, special hydrophobic AEROSIL® products are of essential importance with regard to transparency, reinforcement and

rheology, while organofunctional silanes as high performance additives are able to improve mechanical properties, adhesion, water resistance, and storage stability. The positive effects of especially suitable hydrophobic AEROSIL® products and special Dynasylan® products will be described in this article.

## When it comes to transparency and reinforcement, AEROSIL® fumed silica is essential.

Due to its fine particle size, AEROSIL® fumed silica leaves clear resins more transparent than many other additives or fillers do. Especially AEROSIL® products with high specific surface areas like AEROSIL® R 812 S, AEROSIL® R 805, AEROSIL® R 106, AEROSIL® R 974 or AEROSIL® R 8200 are recommended to formulate transparent products with excellent mechanical properties and good processabilities.



Highly transparent sealant based on MS Polymer™ with 10 wt.-% of AEROSIL® R 812 S

### STPU sealants



Transparent STPU sealants thickened with each 10 wt.-% of AEROSIL® R 974 or AEROSIL® R 106 resp. 20 wt.-% of AEROSIL® R 8200 in comparison to the reference without AEROSIL® (cf. Formulation 1).

Each drawdown, applied to glass, was 2 mm in thickness. The label placed below the glass plate provided a good method to compare each silica grade and loading level. The relatively high concentrations of fumed silicas (10 wt.-% of AEROSIL® R 974 / AEROSIL® R 106 resp. 20 wt.-% of AEROSIL® R 8200) did have only a minimal impact to transparency compared to the polymer without silica. Moreover, hydrophobic AEROSIL® products increased the tensile strengths of SMP sealants and improved also the elongation of the sealants significantly. The thickening effect of AEROSIL® R 106 was higher compared to AEROSIL® R 974 (cf. figure 2).

### Outstanding properties and effects of hydrophobic AEROSIL® products in transparent STPU and STPE sealants

| Product | Properties   | Effects   |
|---------|--|---|
| R 805   | high hydrophobicity  | excellent reinforcement and transparency                                  |
| R 812 S | high hydrophobicity and high specific surface area             | outstanding reinforcement and transparency                                |
| R 106   | medium hydrophobicity and high specific surface area           | excellent reinforcement, outstanding transparency (e.g. in STPU sealants) |
| R 974   | medium hydrophobicity  | excellent reinforcement and transparency                                  |
| R 8200  | very high hydrophobicity, structure modified -> low thickening | High loading possible -> for high strength/ high transparent sealants     |

Figure 1

### Fumed silicas in STPU formulation

|                  | Viscosity<br>Pas (0.1 s <sup>-1</sup> ) | Tensile<br>strength<br>N/mm <sup>2</sup> | Elongation<br>at break % |
|------------------|---|--|--------------------------|
| Without AEROSIL® | 9                                       | 0.2                                      | 60                       |
| AEROSIL® R 8200  | 7600                                    | 2.2                                      | 380                      |
| AEROSIL® R 974   | 2100                                    | 1.8                                      | 360                      |
| AEROSIL® R 106   | 3400                                    | 1.7                                      | 317                      |

Figure 2

**STPE sealants**

In a second formulation based on MS Polymer™ S 303 (STPE), following products have been tested as thixotropes and reinforcing agents: AEROSIL® R 106, AEROSIL® R 812 S, AEROSIL® R 805 (each 10 wt.-%) and AEROSIL® R 8200 (20 wt.-%). Excellent MS Polymer™ sealants with outstanding transparencies and mechanical properties could be formulated by using all four above mentioned AEROSIL® fumed silica grades.

AEROSIL® R 812 S and especially AEROSIL® R 8200 are specifically recommended for transparent MS Polymer™ sealants with high strengths, whereby higher loading levels up to 20 wt.-% can be achieved by using the structure modified grade AEROSIL® R 8200.

**Fumed silica in STPE formulation**

| AEROSIL® | Viscosity<br>Pa s<br>(0.1 s <sup>-1</sup> ) | Tensile<br>strength<br>N/mm <sup>2</sup> | Elongation<br>at break % | Modulus<br>100<br>N/mm <sup>2</sup> |
|----------|---|--|--------------------------|-------------------------------------|
| R 8200   | 11000                                       | 2.1                                      | 410                      | 0.61                                |
| R 812 S  | 9000  | 1.7                                      | 401                      | 0.56                                |
| R 106    | 9100  | 1.5                                      | 270                      | 0.69                                |
| R 805    | 7600  | 1.3                                      | 248                      | 0.67                                |

Figure 3



Hybrid sealants with AEROSIL® R 812 S showed the highest tensile strengths and elongations at break compared to AEROSIL® R 106 and AEROSIL® R 805 at the same concentration (cf. figure 3). The sealant with AEROSIL® R 8200 showed at a loading of 20 wt.-% the highest tensile strength and elongation, while the viscosity was only 20 % higher compared to those with AEROSIL® R 812 S; this sealant even showed a good processability. As an important consequence, also medium modulus hybrid sealants can be formulated with higher concentrations of silica.

**Excellent properties and effects of Dynasylan® products in transparent STPE/STPU sealants**

**Dynasylan® products in transparent STPE/STPU sealants**

| Product  | Properties  | Effects  |
|----------|---|--|
| VTMO     | Vinylfunctional   | Water scavenger<br>-> to improve the storage stability                     |
| AMMO     | Aminofunctional   | Adhesion promoter and co-crosslinker/-catalyst                             |
| 1146     | Oligomeric, diamino silane system, no labeling required | Adhesion promoter, especially for critical and also transparent substrates |
| SIVO 203 | New, multi-amino-functional silane system               | Adhesion promoter with significantly reduced yellowing                     |

Figure 4

**Positive effects with Dynasylan® 1146 and Dynasylan® SIVO 203 in transparent STPE sealants**

Organofunctional silanes play multiple, but always elementary roles in moisture-curing hybrid sealants based on SMP – a powerful product cannot be formulated without silanes! Vinylfunctional Dynasylan® VTMO traditionally acts as a water scavenger for water bearing raw materials, while Dynasylan® AMMO is used mainly as standard adhesion promoter and co-crosslinker.

However, if the technical requirements to substrates or secondary conditions are getting more and more challenging, the exchange of the adhesion promoter should be considered seriously. The oligomeric, aminofunctional Dynasylan® 1146 shows technical benefits as adhesion promoter, especially for critical substrates like non polar plastics, and achieves – because of its chemical structure – more flexible sealants at the same time. Further technical benefits of Dynasylan® 1146 for the adhesives and sealant manufacturers are:

- no labeling required even at higher concentrations
- the VOC content is significantly lower compared to silane monomers
- inherent hydrophobicity of the formulated hybrid products
- colour stability of liquid or cured formulations compared to Dynasylan® AMMO, even after storage/exposure to light

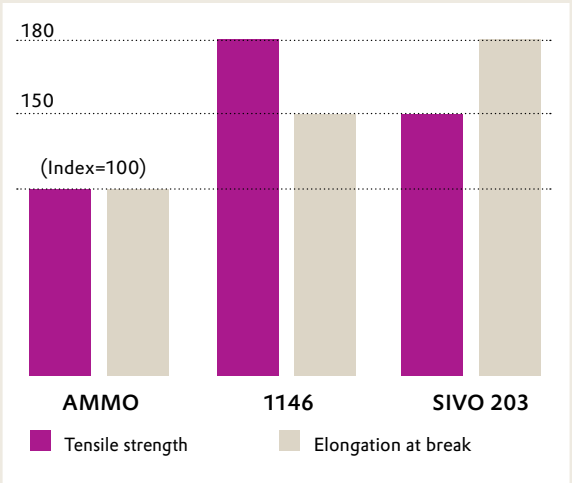
Remark: in few cases, formulations with Dynasylan® AMMO can show a slight yellowing when exposed to light or darkness.

Dynasylan® SIVO 203, a special designed, multifunctional silane system, did not cause any yellowing of the transparent STPE sealants in our lab trials – both after 10 months exposure to light and darkness - with and without stabilizer. Moreover, the STPE sealants formulated with Dynasylan® SIVO 203 did show also higher tensile strengths and elongations at break compared to those formulated with Dynasylan® AMMO.

**Dynasylan® SIVO 203 is specifically suitable both for flexible, transparent formulations, and also for transparent formulations which are not allowed to show any yellowing.**

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**Mechanical properties of cured transparent MS-polymer sealants**



**Test formulations**

|                              | Formulation 1                              | Formulation 2                                  |
|------------------------------|--|--|
| Polymer ST 48 (STPU)         | 56.7                                       |  |
| MS Polymer S 303 (STPE)      |  | 63.2   |
| Diisononylphthalate          | 29.0                                       | 22.5   |
| Hydrophobic AEROSIL®         | 10.0<br>R 974 resp. R 106<br>20.0 (R 8200) | 10.0<br>R 812 S, R 106, R 805<br>20.0 (R 8200) |
| Dynasylan® VTMO              | 3.0  | 1.5  |
| Dynasylan® adhesion promoter | 1.0<br>1146                                | 2.0<br>AMMO resp.<br>1146 resp.<br>SIVO 203    |
| Catalyst TD 18               | 0.3  | 0.8  |

Figure 5



# Waterproofed with POLYVEST® HT:

A liquid hydrophobic polyol for polyurethane-based waterproofing applications

With POLYVEST® HT Evonik provides a liquid hydroxyl-terminated polybutadiene (HTPB) that is used in a broad field of applications for sealants and adhesives.

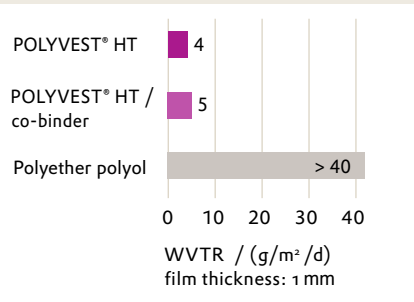
### Unique properties of POLYVEST® HT:

- Low glass transition temperature (-80°C)
- High hydrophobicity and low water uptake
- Good compatibility with bitumen and hydrocarbon oil
- Exhibits high loading of inorganic fillers

In particular, due to its nonpolar and hydrophobic polymer backbone and its low glass transition temperature (-80°C) POLYVEST® HT is highly suitable for being used as a resin in polyurethanes (PUR) for sealing and waterproofing applications in the construction area. It is applied in potting compounds, insulating glass sealants, crack-fillers, expansions joints, tank coatings and self-leveling waterproofing coatings - only to mention a few. POLYVEST® HT-based PUR sealants provide an excellent hydrolytic stability, superior low water uptake & low water vapor transmission rates (WVTR) combined with a high flexibility at low temperatures. In this respect, they surpass the performance of conventional PURs based on polyether polyols. Even in boiling water in a pressure cooker at 120°C the water uptake of a PUR sealants based on POLYVEST® HT is below 0.5 wt% after 24 h and the sealant maintains its high mechanical flexibility. Figure 1 exemplarily illustrates the WVTR of PUR films based on HTPB compared with conventional polyether polyols. By using POLYVEST® HT as the main binder also in combination with other co-binders (e.g. chain extenders and low molecular polyols) the vapor transmission rate is significantly reduced compared to conventional PURs.

### Water vapour transmission rates of unfilled two-component PURs

| Two-component PUR      | I            | II                       | III   |
|------------------------|--------------|--------------------------|---|
| Component A            | POLYVEST® HT | POLYVEST® HT / co-binder | Polyether polyol (polypropylene glycol based) |
| Component B            | Modified MDI |                          |   |
| OH/NCO ratio (mol/mol) | 1:1.15       |                          |   |



### Bitumen-modified polyurethanes

Because of its nonpolar polybutadiene backbone liquid polybutadiene resins also provide a good compatibility with hydrocarbon oil and bitumen and also accept high amounts of inorganic fillers. This facilitates the fabrication of two-component PURs extended with bitumen and asphalt for waterproofing sealants and coatings. In contrast to conventional one-component SBS-modified bitumen systems that need to be heated to high temperature before being applied, the bitumen-extended PURs can be applied at room temperature in a liquid form. The application viscosity and the mechanical properties of the cured compounds

can be varied in a broad range by variation of the composition (amount and type of bitumen, mineral oil, POLYVEST® HT, inorganic filler and isocyanate curing agent). Figure 2 shows an exemplary composition and the properties of a two-component bitumen-modified PUR with a high elongation at break of more than 1,000 %. The final cured sealants also exhibit the properties of an elastomer with high elastic recovery and good flexibility even at temperatures below -30°C.

### Prepolymers based on POLYVEST® HT

In addition to its application as a polyol component in two-component systems POLYVEST® HT can be converted with diisocyanates to form isocyanate-modified prepolymers that allow broadening the field of addressable applications. Such prepolymers provide a good flexibility at low temperatures and excellent hydrophobicity and can be applied as hardener components in two-component PURs, in combination with amine hardeners in polyurea compounds and also in moisture-curable one-component PURs.

Prepolymers with NCO contents in a range of 5-10 wt% are accessible via conversion of POLYVEST® HT with an excess of diisocyanates, e.g. MDI or IPDI. In order to yield MDI-prepolymers with a suitable low viscosity a NCO/OH ratio of >2.8 is recommended. Using non-reactive plasticizer and aliphatic diisocyanates with different reactivities of the two isocyanate moieties (e.g. IPDI) allow to further reduce the viscosity of the resulting prepolymers. Figure 3 shows the correlation of NCO-content and viscosity of MDI-modified prepolymers based on POLYVEST® HT.

### Bitumen-modified PUR for waterproofing: Composition and properties

| Composition              |                  |
|--------------------------|------------------|
| <b>Component A</b>       |                  |
| bitumen (160/220)        | 28               |
| mineral oil              | 10               |
| POLYVEST® HT             | 29               |
| Calcium carbonate        | 32               |
| <b>Component B</b>       |                  |
| Modified MDI             | 100              |
| OH/NCO ratio (mol/mol)   | 1:1.2            |
| <b>Properties</b>        |                  |
| Viscosity of Component A | 19.0 Pa s (20°C) |
| Shore A after 4d         | 33               |
| Tensile strength         | 1.4 MPa          |
| Elongation at break      | 1,010%           |

Figure 2

# POLYVEST® HT

These isocyanate-modified prepolymers provide an improved compatibility also with polar polyols (e.g. polyether and polyesters) and facilitates the combination of these polyol components in two-component PURs. The combination of POLYVEST® HT as a polyol component with isocyanate-modified prepolymers as the hardener component allows to fabricate two-part PUR sealants with high amount of polybutadiene and a low water uptake. Figure 4 shows that the water uptake of a basic two-component PUR based on polybutadiene components is significantly lower compared to a polyether polyol-based sealant (0.2 vs. 3.7 wt% after water immersion for 7d at 80°C).

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Prepolymers based on POLYVEST® HT can be used - also in combination with bitumen – in hydrophobic and flexible one-component PUR formulations.

Guided formulations for one- and two-component PUR are available on request.

## Two-component PUR sealants

| Composition                                    | I       | II      | III     |
|--|---------|---------|---------|
| <b>Component A</b>                             |         |         |         |
| POLYVEST® HT                                   | 99.99   |         |         |
| Polyether polyol                               |         | 99.99   | 99.95   |
| Catalyst (DBTL)                                | 0.01    | 0.01    | 0.05    |
| <b>Component B</b>                             |         |         |         |
| POLYVEST® HT prepolymer (NCO = 7,1 Gew.-%)     | 100     | 100     |         |
| Polyether polyol prepolymer (NCO = 6,5 Gew.-%) |         |         | 100     |
| Mixing ratio A:B (m/m)                         | 2:1     | 2.5:1   | 1.5:1   |
| <b>Properties A:B</b>                          |         |         |         |
| Shore A  | 42      | 12      | 22      |
| Tensile strength                               | 0.9 MPa | 0.4 MPa | 0.5 MPa |
| Elongation at break                            | 160%    | 340%    | 350%    |

I) POLYVEST® HT cured with POLYVEST® HT-based prepolymer  
II) Polyether polyol cured with POLYVEST® HT-based prepolymer  
III) Polyether polyol cured with Polyether polyol-based prepolymer

Water immersion at 80°C for 7d

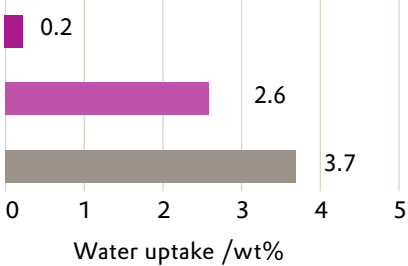


Figure 4

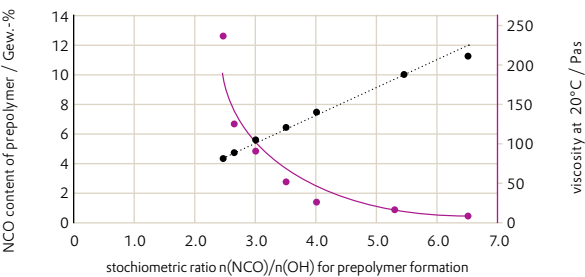


Figure 3: MDI-modified prepolymer based on POLYVEST® HT: Correlation of viscosity and NCO-content (POLYVEST® HT converted with 4,4'-MDI)

# NANOPOX®:

Smart electronic adhesives for microelectronics – enabled by low viscosity nanocomposite materials

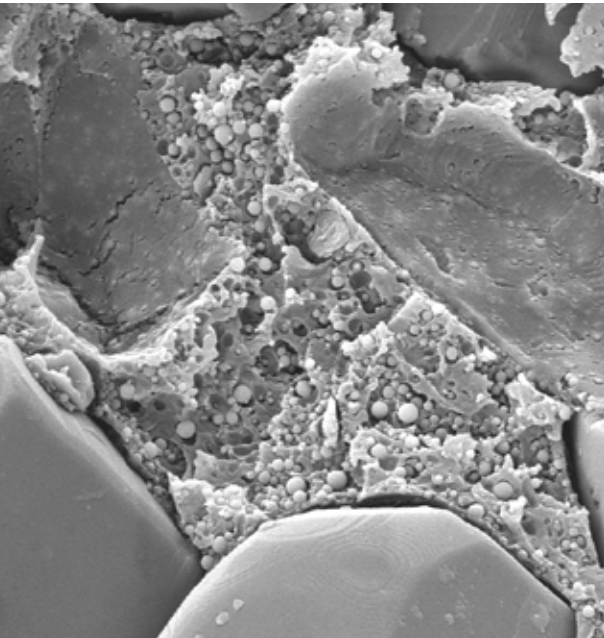


Figure 1: SEM micrograph of a cured specimen containing micron and nano scaled particles. The nano particles fill the interstitial spaces between the micron particles and thus enable extremely high loading levels.

The era of modern electronics began with the invention of the transistor. While in the 1970ies personal computers filled entire rooms, we have nowadays smart-watches with much higher computing power. One could say that the most general trend in electronics is miniaturization and increasing power density.

Today, the miniaturization of assemblies has advanced to such a degree that also potting materials, underfills and encapsulants have to fulfill high requirements e.g. increased level of heat conductivity and/or minimal thermal expansion (CTE), cure shrinkage and other mechanical properties.

As an example, the CTE of a silicon die, a common printed circuit board (PCB) and of a typical cycloaliphatic epoxy adhesives differ by factor 50. This huge difference can cause several problems, such as delamination or bond wire tear off. To avoid these problems usually high amounts of inorganic filler are used to shift the CTE of the adhesive as close as possible to the CTE of the die and substrate. This usually demands high filler loading levels. In order to gain maximum loading levels at minimal viscosity Evonik Nutrition & Care GmbH has developed nanosilica concentrates under the tradename NANOPOX® E, which are especially designed for the use in electronics.

Silica and alumina nano particle composites NANOPOX® E and NANOPOX® T provide new effects helping to overcome today's limitations and push back borders to meet tomorrow's needs.

Realizing highest filler content levels while keeping the viscosity under control as well as maximizing the thermal conductivity at the same time has a name: NANOPOX®.

Keeping viscosity at bay

NANOPOX® E series contains surface treated, spherical nanoparticles that allow high filler loading levels at much lower viscosity compared to conventional fillers. Moreover, nano particles can even penetrate smallest gaps between microchips and the board or finest filaments like in ignition coils or even glass fiber bundles inside PCBs. Thus, they provide improvements in CTE and mechanical properties.

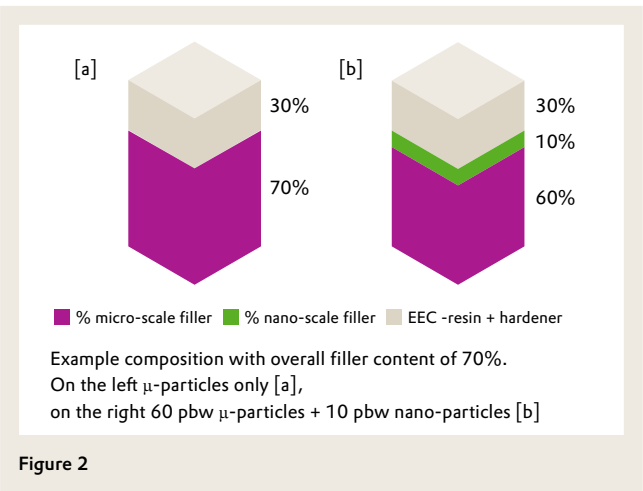


Figure 2 shows a composition of cycloaliphatic epoxy resin, anhydride as curing agent filled with ground quartz. In the right column a part of the filler is replaced with nano silica particles by using NANOPOX® E 601, a dispersion of 40 wt.% nano scaled silica particles in cycloaliphatic epoxy resin.

NANOPOX® nanoparticles can be used to significantly decrease the viscosity (up to 70 %!) at a given filler loading level. On the other hand NANOPOX® can also help to maximize the total filler content by filling the interstitial spaces between the micron particles (Page 11. Fig. 1).

| Properties of the example formulations            |                     |              |            |                  |
|---|---------------------|--------------|------------|------------------|
|   | Viscosity<br>[mPas] | CTE<br>[ppm] | Tg<br>[°C] | Modulus<br>[MPa] |
| [a]<br>70 % $\mu$ -particles                      | 20400               | 32           | 190        | 6670             |
| [b]<br>60% $\mu$ -particles<br>10% nano-particles | 5700                | 33           | 190        | 5704             |

Table 1: Properties

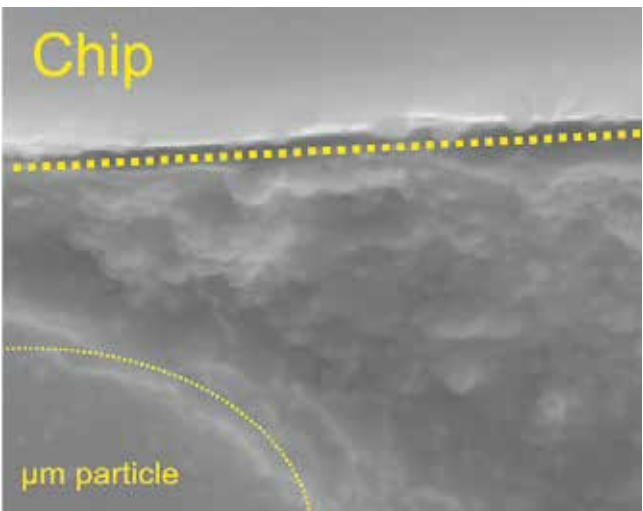


Figure 3: Nanoparticles have direct contact to the surface of the chip.

Preventing sedimentation

Due to stokes law micron scaled particles tend to settle down. This is especially true for highly filled, low viscosity base resins like cycloaliphatic epoxy. Sedimentation can occur over time during storage or after the jetting application at the beginning of the curing process. Specimen [a], which contains micron particles only, clearly shows sedimentation after curing. In contrast, nanoparticles do not agglomerate, thus show no sedimentation. Moreover, nanoparticles can even prevent micron particles from sedimentation (specimen b)! A vertical concentration gradient of filler particles between chip and substrate (Figure 4) caused by sedimentation can cause several negative effects, like:

- Thermal stress - causing inner tensions
- CTE in areas of low particle concentrations is higher
- Reduced thermal conductivity

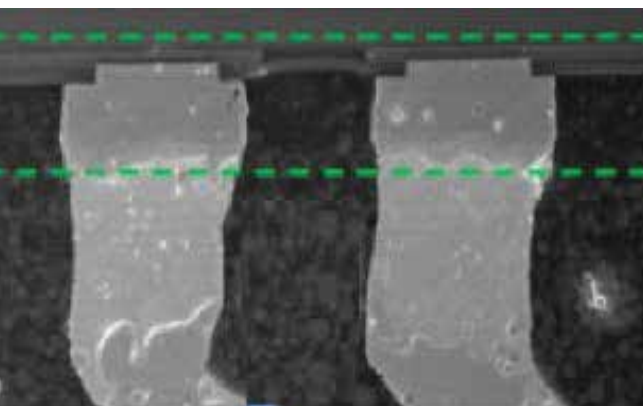


Figure 4: SEM micrograph of an assembly cross section (light-gray area = solder bonds, black area = resin, medium-gray dots = micron scaled filler particles). The underfill shown contains micron particles only. In the upper area, where heat conductivity is needed the most, there is a decreased particle concentration because of particle settlement.

Improved thermal conductivity

NANOPOX® E can even improve the overall heat transfer - not only by filling the interstitial spaces between the filler, but also by reducing the resistance to heat transfer at the interface chip/adhesive. Nanoparticles can get closer to the surface compared to micron particles (Figure 3) and therefore can foster the heat transfer from the die into the adhesive. This leads to increased overall heat removal in a given assembly. The thermal conductivity of pure EP resin is only ~0.35 W/mK. The bulk thermal conductivity of silica used in NANOPOX® E series is ~1 W/mK. For even further improved thermal properties Evonik Nutrition and Care GmbH has developed nano concentrates based on nano scaled alumina under the trade name NANOPOX® T. Al2O3 has a bulk heat conductivity of ~30 W/mK and thus NANOPOX® T improves the thermal conductivity of adhesives enabling the next generation of microelectronics.

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Figure 5: Specimen with  $\mu$ -particles only (a) and with micro and nano particles (b). Specimen a shows sedimentation, while NANOPOX® in specimen b prevent a settlement.





## SHELL GTL SARAWAX SX 80: That certain something for hot melts in food packaging

During the long lasting and trustful partnership with Shell MDS (Malaysia) and Evonik in the area of premium-grade synthetic GTL hard paraffin waxes, the product portfolio has expanded consistently. The latest addition to the Evonik wax portfolio is the Shell GTL Sarawax 80, which provides a unique property profile.

The synthetic GTL hard paraffin has a significantly low molecular weight and an extremely narrow molecular weight distribution. Along with a low drop point and additionally reduced melt viscosity, this combination results in a strongly pronounced crystallinity, thus allowing particularly precise control of industrial processes and further improvement in the accuracy with which specific product properties can be fine-tuned. A particularly interesting potential field of application for Shell GTL Sarawax SX 80 is in hot melt adhesives, where it allows significantly reduced setting times and excellent substrate wetting for adhesives with low softening points.

Especially hot melts for food packaging can be a target, as the short setting time allows for high speed packaging and low application temperatures can be achieved, which leads to a cost reduction due to lower energy consumption. Additionally, SX 80 is compliant to EU 10/2011 without specific migration limit as well as FDA regulations, thus saving the user from additional costs for food approval filing.

Solvent-based mold release agents (such as for PU foam) are another suitable application. Here, Shell GTL Sarawax SX 80 combines easy and stable dispersibility with excellent release effect and enables particularly precise and effective process control.

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### Technical properties at a glance

|                         | Method                               | Typical Values          | Specification               | Remarks                            |
|-------------------------|--------------------------------------|-------------------------|-----------------------------|------------------------------------|
| Congealing point        | ASTM D 938                           | 84°C                    | 81 - 86°C                   |                                    |
| Penetration number      | ASTM D 1321                          | 4 mm * 10 <sup>-1</sup> | 3 - 6 mm * 10 <sup>-1</sup> |                                    |
| Acid number             | ASTM D 1386                          |                         | < 1 mg KOH/g                |                                    |
| Purity                  | Sulfuric acid test                   | OK                      |                             |                                    |
| Viscosity               | Cone & Plate (30.5 s <sup>-1</sup> ) | 12 mPa s<br>6 mPa s     | < 10 mPa s                  | at 100°C<br>at 120°C               |
| Melting enthalpy T [3%] | DSC                                  | 237 J/g<br>49 °C        |                             | Temp. when 3% of the wax is molten |

## TEGOPAC®: Tracking the trend Methanol-free and low viscous hybrid adhesive & sealant formulations

TEGOPAC® products represent a new class of silane-modified polymers that are used for production of neutral curing adhesives and sealants. The polymers are based on a unique polymer technology with lateral crosslinking groups. Typical applications for TEGOPAC® polymers are parquet adhesives, assembly adhesives or waterproofing membranes.

Adhesive and sealant formulations with TEGOPAC® polymers show excellent through cure properties, improved water & thermal resistance and excellent elastic recovery properties. The different behaviour compared to existing silane-terminated binders in the market is linked to the reactive groups in lateral position which are responsible for a special polymer network construction during the curing process. Anyhow curing starts in presence of moisture and a catalyst.

In comparison to silane-terminated polymers which release methanol TEGOPAC® polymers release ethanol during the curing process and thus allow to formulate methanol-free adhesive formulations. Reduction of methanol evaporation is required for adhesives which are applied onto large surface areas under conditions where air exchange is limited in order to decrease health risks.

Methanol-free formulations based on TEGOPAC® polymers show good mechanical properties and good reactivity for example in an assembly adhesive formulation. A standard assembly adhesive formulation consists of a polymer binder (e.g. TEGOPAC® Bond 150 or Bond 251), fillers (e.g. calciumcarbonate), a plasticizer, silanes (water scavenger & aminosilanes) and a catalyst.

Figure 1 shows typical mechanical properties of different methanol-free assembly adhesive formulations based on TEGOPAC® Bond 150. Skin-over-time and Shore A hardness can be adjusted to different application needs, cure-tough properties are excellent (Figure 2). Reactivity and curing speed of methanol-free formulations based on TEGOPAC® polymers is comparable to systems based on silane-terminated polymers.

The latest products in the TEGOPAC® family are reactive diluents. TEGOPAC® RD 1 and TEGOPAC® RD 2 are polymers of low viscosity which allow to decrease formulation viscosity when replacing a part of the main polymer by the reactive diluent. Additionally TEGOPAC® reactive diluents can be used to substitute migrating

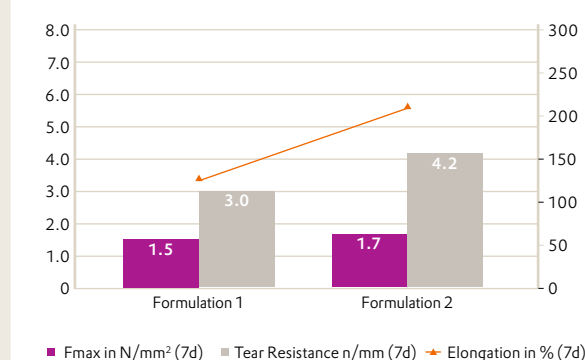


Figure 1: Methanol-free assembly adhesive formulations based on Tegopac® Bond 150; mechanical properties

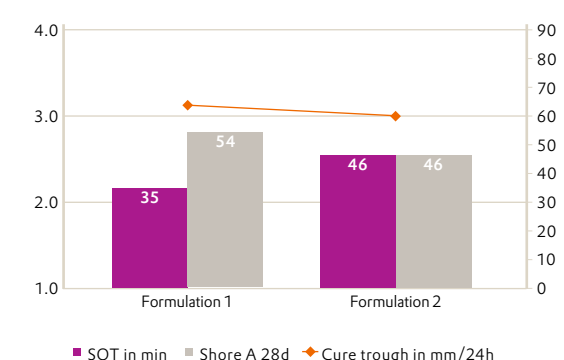


Figure 2: Methanol-free assembly adhesive formulations based on Tegopac® Bond 150; Skin-over-time & Shore A hardness



plasticizers in adhesive and sealant formulations. TEGOPAC® reactive diluents will be implemented into the polymer network during the curing process so that development of staining-free formulations is possible.

**TEGOPAC® RD 1** is used to reduce viscosity of formulations and to improve cure through properties (Figure 3) with only marginal effects on mechanical properties of the adhesive & sealant product.

**TEGOPAC® RD 2** is addressing polymer structures with a high crosslinking density. Development of formulations with improved curing properties (Figure 3) and increased hardness & tensile strength is possible (Figure 4).

With TEGOPAC® reactive diluents it is possible to formulate pasty formulations as well as self-levelling products. It is recommended to combine TEGOPAC® RD products with a co-binder (e.g. with different types of silane-modified polymers).



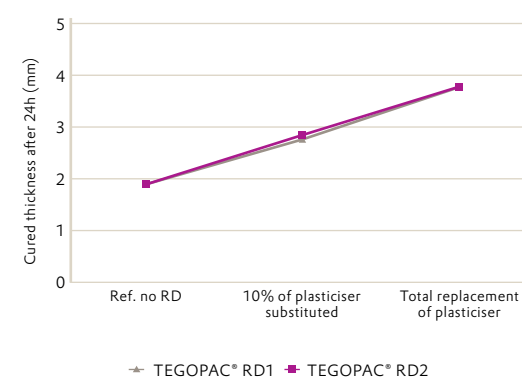
TEGOPAC® reactive diluents are interesting raw materials for the development of adhesives and sealants for flooring applications or for the development of liquid membranes that are used for different construction demands:

- roof sealing/ sealing of balconies
- moisture/air barriers
- sealing of construction joints & connections
- barrier layers to optimise thermal conditions

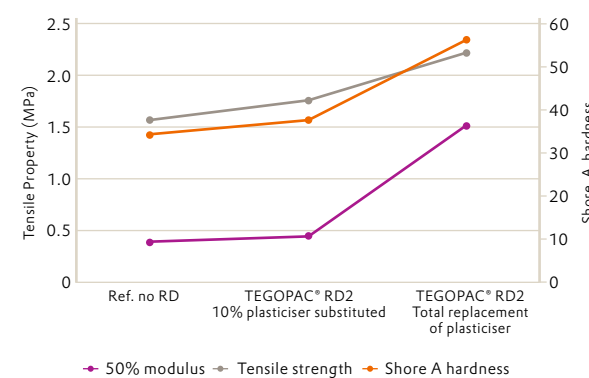
With TEGOPAC® reactive diluents it is possible to develop self-levelling formulations that can be applied easily via a brush or roller.

The application of liquid waterproofing membranes often requires good adhesion properties between different polymer/membrane layers. Either formulations are used for repair work on already existing cured membrane layers or the waterproofing membrane consists of different layers (first adhesive layer, a fabric, second adhesive layer). With a formulation based on TEGOPAC® reactive diluents and TEGOPAC® polymers (a variety of these with viscosities between 10-50 Pas exists in the portfolio) good inter-layer adhesion will be reached easily.

Cured formulation thickness 24 hours after application



**Figure 3:** The addition of TEGOPAC® RD to a formulation allows to speed-up the in-depth cure.

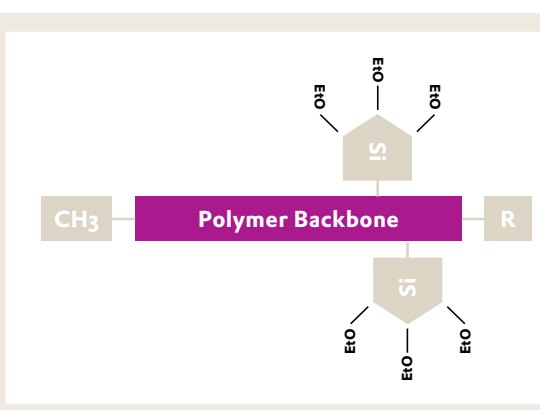


**Figure 4:** TEGOPAC® RD 2 increases the crosslinking density of a formulation. Increase of tensile strength and Shore A hardness takes place.

#### Technical parameters

|             | TEGOPAC® RD 1       | TEGOPAC® RD 2 |
|-------------|---------------------|---------------|
| Appearance  | transparent, liquid |               |
| Viscosity   | 1 Pa.s              | 1.5 Pa.s      |
| Density     | 1g/cm³              |               |
| Plastisizer | no                  |               |
| Solvent     | no                  |               |

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The TEGOPAC® portfolio with different polymers and reactive diluents offers new formulation possibilities for SMP-based adhesives and sealants.

With varying effects TEGOPAC® polymers are an interesting portfolio extension to silane-terminated polymers in the market.

# TEGOPREN®: A Package – Defoamer and Wetting Agent with Food Contact Compliance

A new baby in the family of food contact  
compliant antifoams

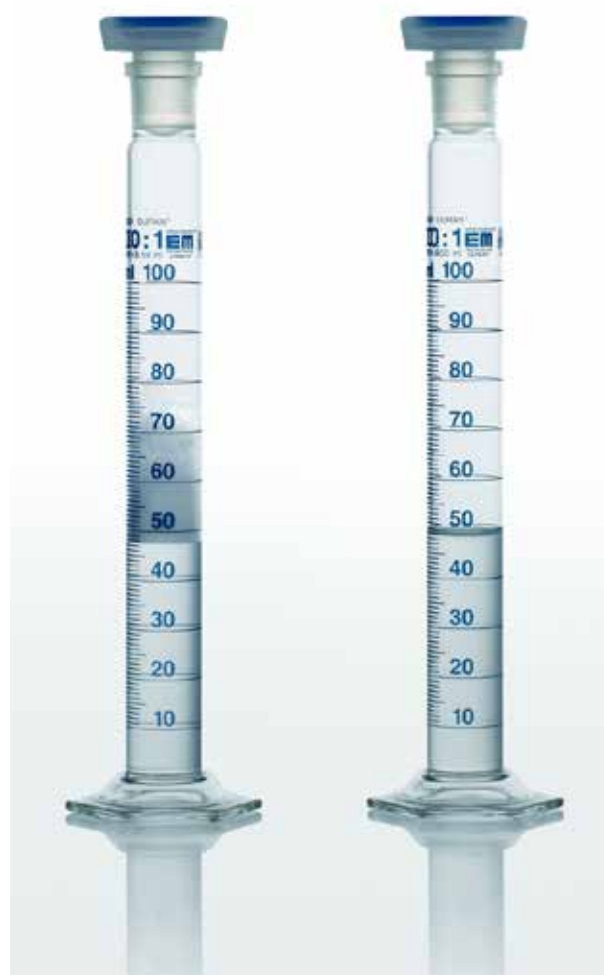


Figure 1: Shaking test

Evonik Nutrition & Care expanded the portfolio of de-foamers and wetting agents with food contact compliance to support manufacturers of polymer dispersions and adhesive formulations who have special requirements with such legislations.

## TEGO® Antifoam 4-88

Manufacturers of polymer dispersions based on acrylics, SBR, polyvinyl acetates or polyurethanes need to produce resins with multiple requirements, in respect to performance but also legislation. It might be that the addressed end application of the dispersion requires already in the manufacturing step of the resin the selection of food compliant raw materials, e.g. defoamers. However, manufacturers of polymer dispersions require even independently from the end application during the stripping process food contact compliant defoamers to avoid in their plants special cleaning operations or failures. Conclusively well performing and robust defoamers with broad food contact compliance are needed. Considering the requirements of a formulator of adhesives a package of defoamer and wetting agent is required with such a compliance.

Evonik Nutrition & Care has expanded the range of defoamers with broad food contact compliance which contains TEGO® Antifoam 2291 (100% defoamer based on paraffinic oil) and TEGO® Antifoam 4-94 (40% active defoamer based on polyether siloxane). TEGO® Antifoam 4-88 is a new grade and also based on an emulsion of a polyether siloxane with an

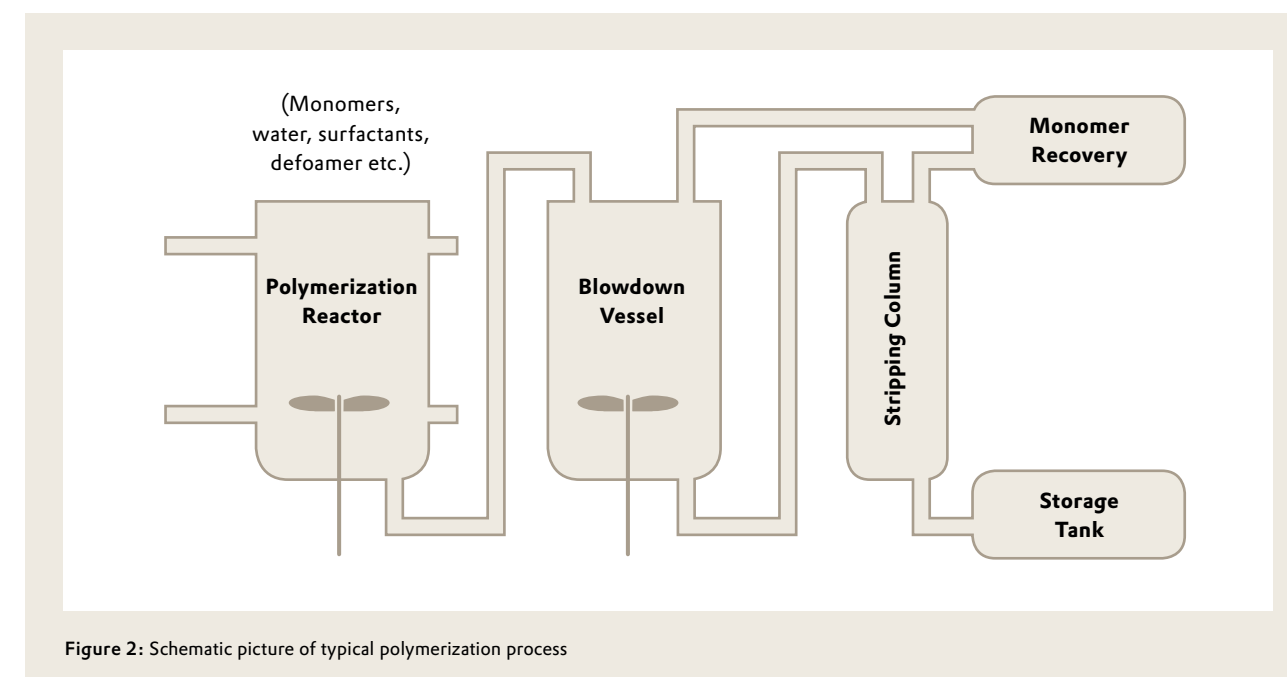


Figure 2: Schematic picture of typical polymerization process

active content of 40%. Due to that new product customers working in the field of manufacturing of dispersions for the paper industry can choose now between two products based on polyether siloxane technology - TEGO® Antifoam 4-88 and TEGO® Antifoam 4-94. This is of importance since the paper industry faces the situation that the widely used mineral oil based defoamers are blamed for their migration potential. In general, the novel antifoam is suitable for all manufacturers of acrylics, SBR and PU and not only for those supplying the paper industry, since the grade has a well-balanced compatibility and defoaming efficiency. Therefore, also formulators will appreciate the use of TEGO® Antifoam 4-88. Formulators have also the need that the applied defoamer needs to keep the defoaming properties after storage of the formulation, this can be guaranteed with polyether siloxanes. TEGO® Antifoam 4-88 has been specifically developed to fulfill global food contact compliance paragraphs. On the one hand the European legislation EU 10/ 2011 as well as a variety of FDA paragraphs. Regarding FDA it should be mentioned that the product is especially FDA 175.105 for adhesives, FDA 176.170 and FDA 176.180 which are relevant for paper applications, conform.

## TEGOPREN® 5890

Manufacturer of adhesive formulations usually prefer wetting agents with low foaming appearance, since the widely used sulfo succinates – also available from Evonik Nutrition & Care in form of Rewopol® SB DO 75- often strongly foam and need to be applied at high dosage levels.

TEGOPREN® 5890 is a low foaming wetting agent based on polyether siloxane technology which can be applied with 0.3-0.5% in order to receive good substrate wetting. It can be well combined with the above mentioned defoamers and is even compliant with EU 10/2011 leading to a high interest for adhesive formulations.

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# ALBIDUR® and ALBIPOX®: Essentials for structural adhesives!

The challenges of the 21st century regarding reduced energy consumption, especially for transportation, preserving resources and resource efficiency are enormous. Lightweight construction is a key technology to cope with these tasks. Adhesive bonding as a joining technology is a prerequisite for many lightweight construction applications.



Figure 1: Double hat profile test specimen used to test automotive adhesives regarding crash performance

For demanding and highly stressed adhesive joints so-called structural adhesives are used; most of them are based on epoxy resin formulations. A typical example for such an application is automotive engineering. For the body-in-white, the integral car body, so-called body-structure adhesives and hem flange adhesives are used. These heat-curing, one-component paste adhesives exhibit a demanding property profile: they need to be applicable by a spray robot but shall not creep away after being applied – hence they need a well-defined flow behavior. The adhesives need to be water-resistant in the uncured stage as the car body with the adhesive passes the cathodic immersion prime coating (EDC) and the adhesive is curing later when the coating is cured in the oven. Especially important is the capability to bond various substrates like high strength or ultra-high strength steel, aluminum alloys or metals to fiber reinforced composite parts. Furthermore the adhesives need to perform on oily substrates, as metals in automotive construction are always contaminated with deep-draw oil.

Of course high strength, high toughness and excellent fatigue performance are required. Ageing resistance during the whole life of the automobile and resistance against fuels, oils and coolants are essential, of course. If the bodies of BMW i3 and i8 are considered, they consist of various adhesively bonded CFRC components – and they have to withstand harsh crash tests.

In order to fulfill these tasks, these epoxy adhesives are quite complex formulations and do always contain a component to increase toughness. Here the technologies of the business line Interface & Performance of Evonik Nutrition & Care are applied: depending on the end application products of the ALBIDUR® or ALBIPOX® range are used to improve toughness and fatigue performance. Likewise in the formulation of assembly and repair adhesives. These are in contrast to the hem flange adhesives two component adhesives and cure at room temperature or slightly increased temperatures (e.g. 60 °C). The performance requirements are comparable, however. Specialties like structural adhesive tapes or foamable inserts are modified in a similar way.

In aircraft construction many structural adhesives are used: in the tailplane rudder, the wings and many other areas. If adhesives are used in interior applications, they have to comply with strict fire safety regulations.



Figure 2: structural adhesive application at the interior of the wing of an Airbus A 380

For the formulation of high performance aerospace adhesives ALBIDUR® and ALBIPOX® products are essential. In some applications, where high mechanical performance and excellent fatigue behavior together with low adhesive viscosity are required, products of the NANOPOX® range are the raw material of choice to obtain exactly this property profile and performance level.

Another market for structural adhesives is the construction of rotor blades for wind energy installations. The two half shells of the blade are bonded together using a structural adhesives. As rotor blades become longer and longer, the adhesive performance requirements are increasing constantly as the forces to

bear grow constantly as well. Of course formulations of such two component epoxy adhesives do contain a component to increase toughness, e.g. products of the ALBIDUR® range. The requirements regarding fatigue for adhesives used in rotor blade construction for offshore wind installations are even more drastic. To change a damaged rotor blade at sea a special ship, a helicopter and a crew are necessary for several days – with enormous costs involved. Due to their length the rotor blades of the latest generation are transported in two parts to the construction site of the wind mill. They are bolted together on site – again an application for structural adhesives.

Structural adhesives formulated using NANOPOX®, ALBIPOX® or ALBIDUR® are used in diverse applications: in machine construction, in shipbuilding and in adhesives for fasteners in tunnel construction (so-called „chemical anchoring“).



Figure 3: adhesive bonding of glass fiber-reinforced composite rods in a wooden beam for restoration

A relatively unknown, nearly exotic application is the restoration of ancient wooden bridges or ancient buildings with wooden beams: wholes are drilled over the whole length of the beam, GFRP rods are inserted and fixed using an adhesive. Thus even rotten beams can be fully stressed again. The structural adhesive used has not only to provide a high strength but needs to be able to compensate the different behavior of wood and GFRP during heat or moisture exposure.

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# VESTOPLAST®: Improved hot melts through innovation

In today’s world, innovation is an essential driver for growth in the industry. With new technologies, shorter product life cycles and fast changing trends, it is most crucial to stay up to date and to develop solutions for future challenges. Therefore, the experts for amorphous poly-alpha-olefins of Evonik have joined the forces of R&D, Applied Technology, Marketing and Production to uncover the needs of their customer and transform them into the products they need.

## Transformation of customer needs into innovative solutions

Talking to the customer regularly is part of daily work; it can be either technical discussions or commercial topics. This way, Evonik ensures to keep up with the latest developments in the market and at the customer. It also marked the starting point for the recent innovation. After an intensive market research and various discussions with customers, Evonik defined hygiene and packaging as relevant target applications. Once settled, the team went into a more detailed discussion with customers regarding requirements of the raw materials used in these

applications. The target properties of the new polymers were identified based on the results and the experimental phase started immediately. In order to be efficient and fast, the Evonik team established a high throughput method in cooperation with a producer of lab equipment. A small-scale multi-reactor polymerization device for the lab was build, which enabled the team to evaluate and screen different polymer compositions within a short time frame. This unique way of innovation uses the concept of DoE (Design of Experiment). It allows for a systematical study of the properties as well as a derivation of structure-property correlations. In the end, the team was able to design two new polymers with low viscosities aimed at hygiene and packaging applications.

The two new grades VESTOPLAST® EP V2094 and VESTOPLAST® EP V2103 complement the current portfolio perfectly. Besides the well-known performance of VESTOPLAST®, which provides for example excellent hot tack and adhesion combined with a light color, those two new grades experience a high tensile strength of 1 MPa and 2 MPa respectively although their viscosity remain in the level of 2,500 ± 500 mPas at 190°C and provide a rather low softening point of ~ 94 – 103 °C. Further,



## Designed Polymers: VESTOPLAST® EP V2094 and VESTOPLAST® EP V2103

when looking closely at the softening point and the shear adhesion failure temperature (SAFT), the delta between both are very narrow (~10°C), which creates a major benefit for the applicator (see figure 1). In use, the hot melt can be applied at rather low temperatures, thus saving energy and still exhibiting excellent thermal stability at rather high temperatures, which prevents the adhesive from discoloring and cracking. More technical details are shown in figure 2. When looking at the physical appearance of the new grades, a major advantage is the improved

flowability of the granules, which leads to better handling and processing of the product for the hot melt producer. Additionally, the new grades have an excellent compatibility with hydrogenated tackifiers as well as FT-Hard Waxes such as Shell GTL Sarawax SX 80 and Shell GTL Sarawax SX 105, which increases the flexibility of raw material usage in formulations. With this broad spectrum of properties, various applications are accessible; however, these grades are specially designed for packaging and hygiene applications.

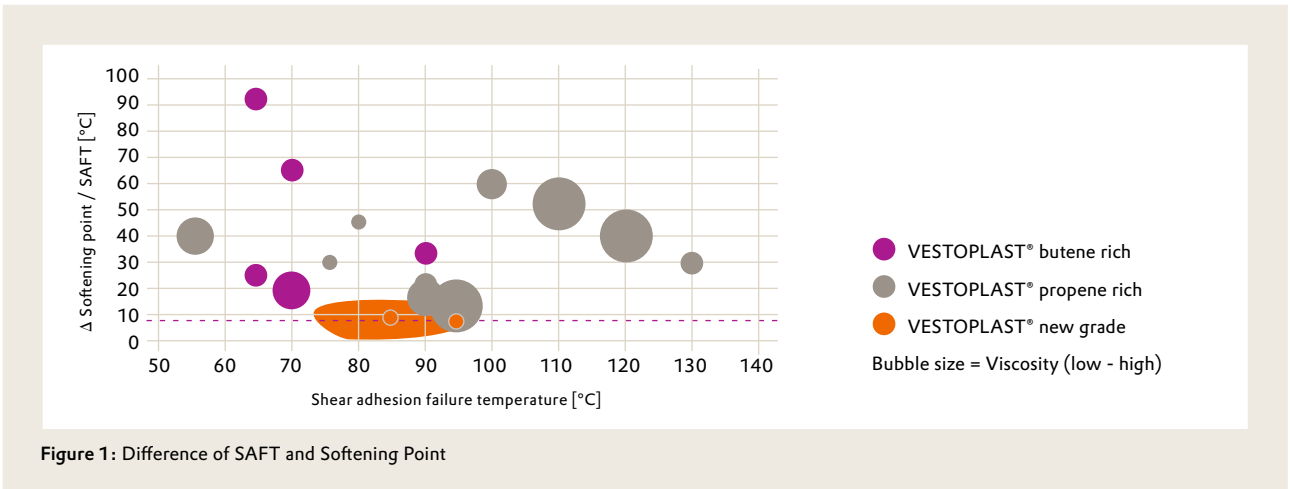


Figure 1: Difference of SAFT and Softening Point

| Technical Data                            |          | VESTOPLAST® EP V2094 | VESTOPLAST® EP V2103 |
|---|----------|----------------------|----------------------|
| Softening Point (R&B)                     | [°C]     | 94 +/- 4             | 103 +/- 3            |
| Needle penetration                        | [0.1 mm] | 20 +/- 4             | 12 +/- 3             |
| Melt viscosity @190 °C                    | [mPas]   | 2,500 +/- 500        | 2,500 +/- 500        |
| S.A.F.T. acc to WPS 68                    | [°C]     | 80-85                | 90-95                |
| Open time                                 | [min]    | > 10                 | ~ 5                  |
| Tensile strength                          | [MPa]    | 1                    | 2                    |
| Elongation at break                       | [%]      | 70                   | 40                   |
| Tensile shear strength wood/wood specimen | [MPa]    | 1                    | 1.5                  |
| Tensile shear strength PP/PP specimen     | [MPa]    | 1                    | 1.5                  |
| Tg (acc to DSC method)                    | [°C]     | -38                  | -33                  |
| Density                                   | [g/cm³]  | 0.86                 | 0.86                 |

Figure 2: Technical data of the two new grades

## Sprayable APAO for hygiene applications

The R&D team has developed VESTOPLAST® EP V2094 to primarily target hygiene applications such as diapers, fem care or incontinence pads. In order to fulfill the requirements in this application perfectly, the team identified three major polymer properties, which are crucial for the formulator and the end producer. The thermal stability is very important for the adhesive as it needs to keep the performance even at high temperatures and over a certain period. Further, customers are often concerned about cost performance ratios, which can be improved by e.g. saving costs when applying the adhesive at low temperatures or by using less adhesive to achieve the same production output. Lastly, end consumers do not want to have any chemical smell in their hygiene product, thus a low odor of the raw materials is important as well. All the above-mentioned requirements can be fulfilled by VESTOPLAST® EP V2094 and at the same time give the polymer an advantage over the commonly used rubber-based hot melts. Additionally, VESTOPLAST® EP V2094 has a low density of 0.86 g/cm<sup>3</sup>, which turns into a mileage advantage during the production process.

In today's hygiene market, a large part of the used adhesives are still based on SBS/SIS, as prices are low and it shows a relatively stable performance as well as a fair processability. However, as standards rise and end consumers become more concerned about quality and health issues, alternative raw materials have started to gain market share, amongst others VESTOPLAST®.

In comparison with the traditional rubber-based systems, VESTOPLAST® possesses exactly those properties, which are crucial for hygiene applications. As shown in figure 3, VESTOPLAST® based formulations and rubber-based formulations were tested for their thermal stability with storage at 150°C under air atmosphere for a time period up to 168 hours. As the viscosity curve shows, the VESTOPLAST® formulation stays relatively stable with a viscosity of ~3,300 mPas, while the rubber based formulations show a significant decrease from an initial viscosity of ~3,000 mPas down to ~900 mPas in the end. This means VESTOPLAST® remains within a certain viscosity range during the formulation at the adhesive producer and during application at the production lines, giving the user a stable processing window.

In terms of color stability, the rubber based formulation turns yellow already after 24 hours and exhibits a deep brown by the end of 168 hours, whereas the VESTOPLAST® formulation remains white throughout the whole period with only a slight change in color. As the adhesive in most cases should not be visible in the end product, this is another major benefit for VESTOPLAST® based formulations.

see page 26 ►

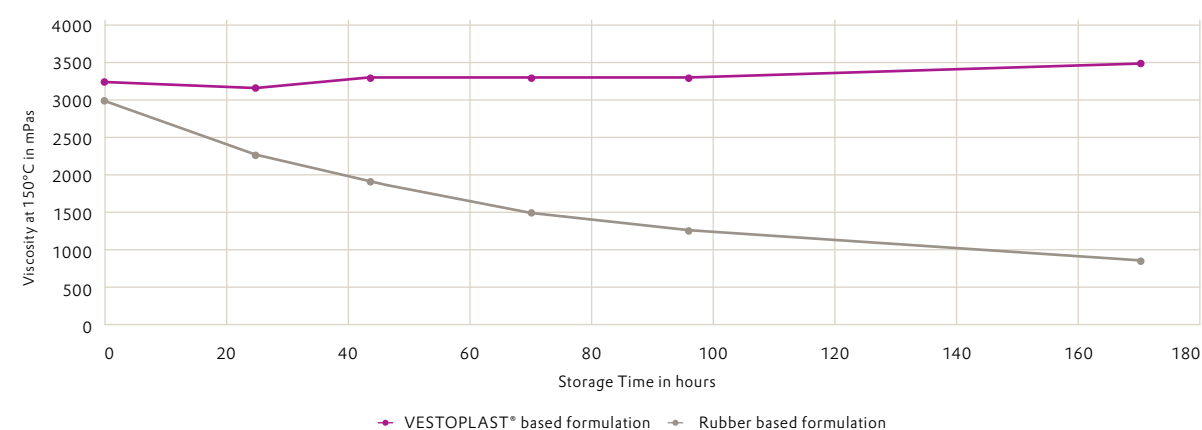


Figure 3: Melt viscosity of VESTOPLAST® vs rubber

## Technical know how for perfect results

The new VESTOPLAST® EP V2094 was specifically developed as an adhesive raw material for hot melts in the hygiene industry such as diapers, fem care, or medical pads. Figure A illustrates the complexity of manufacturing these items, using the example of a diaper. The deep purple colored areas show the layers where the adhesive is applied to.

To get a detailed picture of the requirements for hygiene hot melts, the production process was analyzed and examined for the factors impacting the sprayability of the final adhesive. As amorphous poly-alpha-olefins (APAO) differ substantially from conventional block copolymer-based systems, a detailed assessment and analysis is crucial.

Usually, different non woven substrates and polyethylene films are used for the bonding of hygiene products. Those have to be very thin, breathable, and durable and at the same time prevent the leakage of fluids. Large coating machines are used to spray the hot melt onto the films. During this process speeds of up to 800m/minute can be reached. The coating process is shown schematically in Figure B. The adhesive, ranging from 1 to 5g/m<sup>2</sup> is applied to the substrate by the usage of a spray nozzle. The second substrate is coated from the opposite direction from the top shortly after the adhesive application.

Several spraying method with different machine parameters can be used to apply the adhesive on the substrate. VESTOPLAST® EP V2094 was tested for its sprayability under industrial production premises and high line speeds with excellent spray results. Some of them are shown in figure 6 to 8 on page 27. Due to the intensive sprayability tests, the hot melt applied technology team gained in-depth knowledge about the parameters, which are of significant importance for a good sprayability. One learning for example, is the impact of the pressure which interferes with the polymer in the nozzle in combination with the flow rate per minute on the spray result. If the used pressure and the flow rate are too low, the shear-thinning behavior will not be strong enough to disperse the adhesive threads and create a satisfying spray picture through the air flow. Additionally, the application window, which is given by the nozzle producer is often not considered as well. In the Evonik lab, further tests and analysis are done at a smaller lab coater in order to gain more knowledge about the parameters and also to offer more support to the customers. For more extensive tests, Evonik has the possibility to cooperate with machine accounts which serve the hygiene industry to verify the results at machines used in the hygiene production.

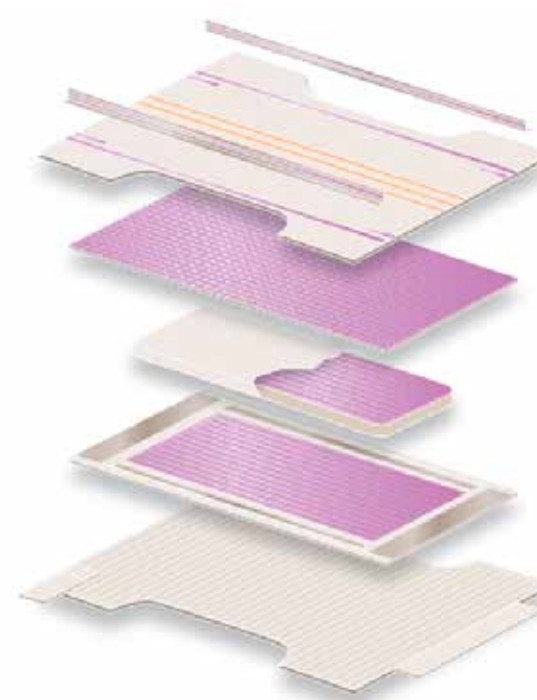


Figure A: Different layers of a diaper

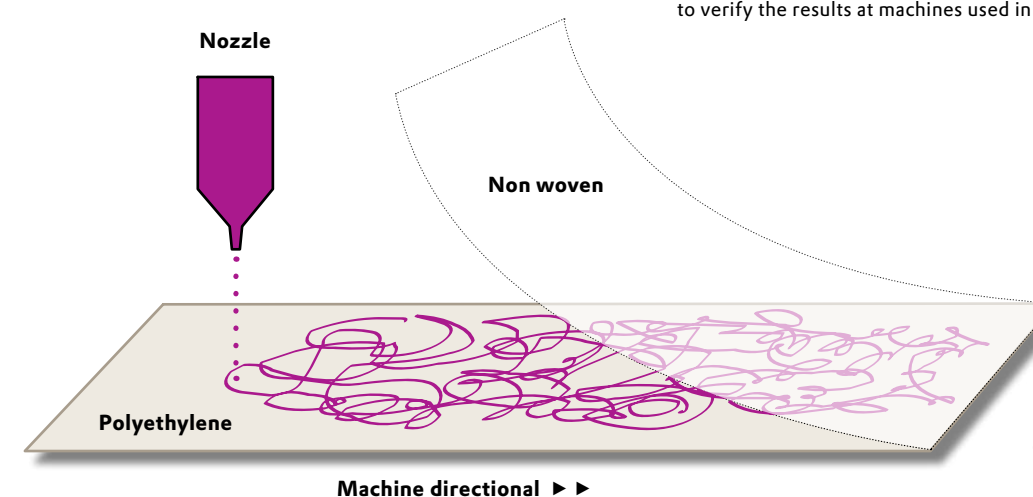


Figure B: Schematic description of the coating process

Figure 4 shows the cracking behavior of formulations with VESTOPLAST® and SBS/SIS. The adhesive is applied on a steel plate and stored at 150°C under air. The results after 1 week show a color change and cracking for the rubber-based formulation, while VESTOPLAST® remains white with no signs of cracking at all. This means less contamination in the machinery and thus also less maintenance during the production process.

The APAO team developed various formulations with different polymer contents varying from high polymer content ones to low polymer content ones as shown in figure 5. Depending on the process parameters and the needed properties, VESTOPLAST® EP V2094 provides a flexibility in adjusting the formulation according to the



Figure 4: Cracking behavior of VESTOPLAST® vs rubber

customer needs and keep its independency from tackifiers at the same time.

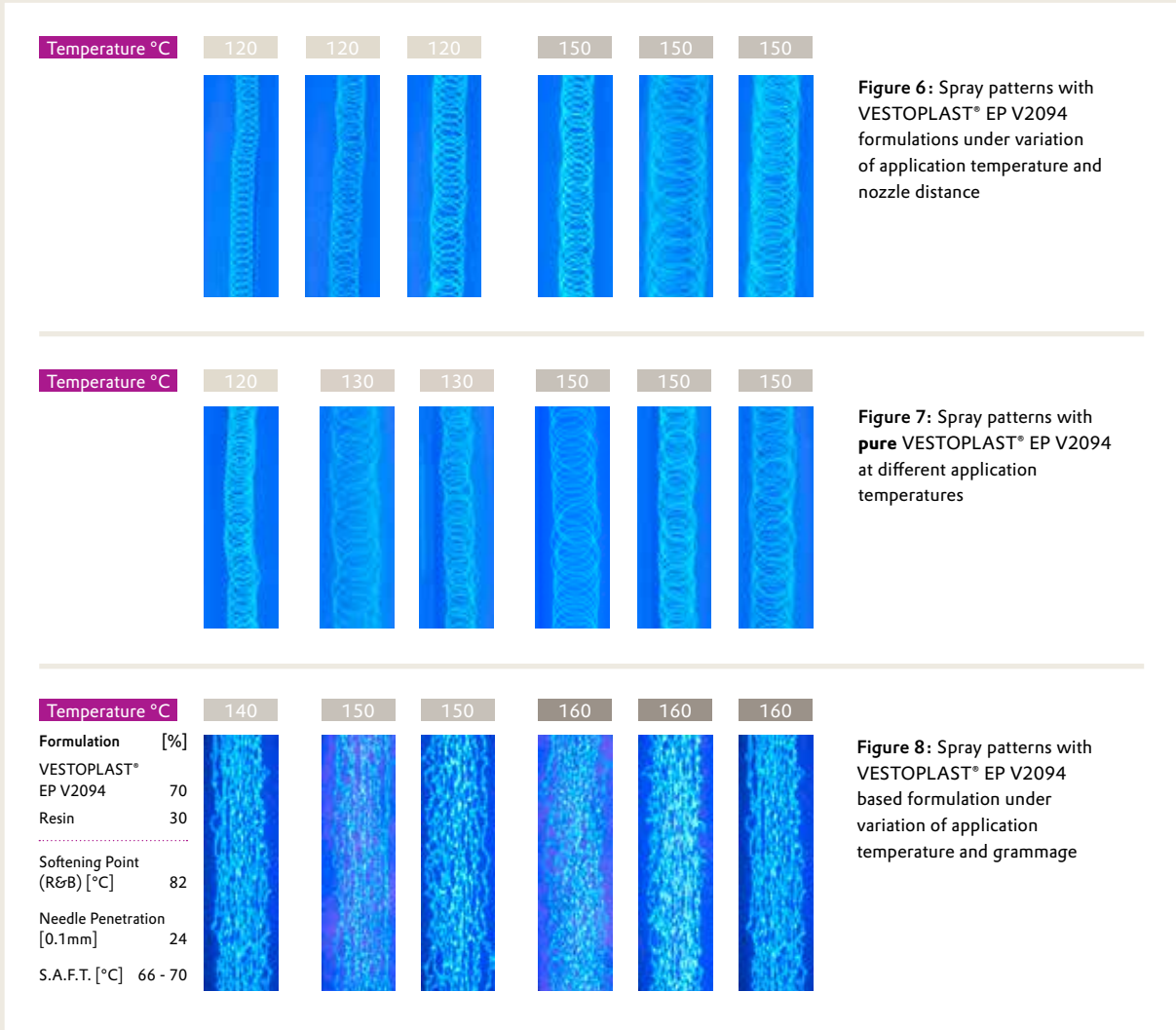
In the hygiene industry, the final adhesive is usually sprayed onto the substrate, e.g. on PE films and non-woven in different ways. Nowadays the most common spray systems are spiral spray and fiberization spray. Several tests were conducted with VESTOPLAST® EP V2094 under industrial production conditions at machine accounts serving the hygiene industry. With these tests, outstanding results were achieved. Amongst all different formulations that have been investigated two are highlighted below. A formulation containing 70% of polymer and the non-formulated pure VESTOPLAST® EP V2094. Both samples have been applied by spiral spray under variation of application speed, pressure and temperature. All results showed very regular spray patterns even at low application temperatures down to 120°C (see figure 6 and 7).

The parameters for the fiberization spray application were similar with application temperatures ranging from 140 – 160 °C, high speed and low coating weight. The realistic and common coating weights for construction in the hygiene market are between 1g/m² and 5g/m². The resulting patterns were as requested by the industry randomly sprayed, with no overspray and no angel’s hair. In addition, the edge stability and the bleeding through the film are also important parameters for a good spray image (see figure 8). The high line speeds were no problem with formulations based on VESTOPLAST® EP V2094, as line speeds up to 800m/min are common in the industry.

### Flexibility in formulation

| Formulation                            |         | Very high polymer content | High polymer content | Low polymer content |
|--|---------|---------------------------|----------------------|---------------------|
| VESTOPLAST® EP V2094                   | [%]     | 85                        | 70                   | 41                  |
| Tackifier resin                        | [%]     | 15                        | 30                   | 49                  |
| Plasticizer                            |         |                           |                      | 10                  |
| Stabilizer                             | [%]     | 0.2                       | 0.2                  | 0.2                 |
| Softening point (R&B)                  | [°C]    | 86                        | 82                   | 71                  |
| Needle penetration 100/25/5            | [0.1mm] | 28                        | 24                   | 28                  |
| Thermal stability under load (S.A.F.T) | [°C]    | 70-75                     | 65-70                | < 50                |

Figure 5: Guide formulation with different polymer content



### VESTOPLAST® EP V2103 for the packaging industry

VESTOPLAST® EP V2103 has been developed to primarily target packaging applications. Besides the common cardboard packaging, several other packaging variations exists, which often involves difficult to bond substrates such as coated cardboards or PP and PE laminates. As the adhesive is also often visible to the end consumer e.g. with straw attachments, the optical appearance of the adhesive plays a major role. In the best case, it is white or even transparent. For the operator of packaging lines fast processes are of utmost importance, which means the adhesive needs to bond fast. VESTOPLAST® EP V2103 addresses all these challenges. By variation of the tackifier and wax content for example very low viscosities down to

960 mPas at 160°C can be achieved with fast setting times in the range of one second and good thermal stability when exposed to heat and air. All the tested formulations showed a very good adhesion on different substrates, especially on PP. The color and the viscosity remain stable even when the hot melt is exposed to air at elevated temperatures (see Figure 9). Due to the variety and flexibility of the formulations, the formulator has several possibilities to steer the properties of the final adhesive and therefore allows for a wide range of possible applications.



Further applications

The technical properties of the new grades are suitable for further hot melt applications, either used as the main polymer or as an additive to reduce the melt viscosity, application temperature etc.

Due to its long open time up to 5 minutes and high tensile strength VESTOPLAST® EP V2103 for example can also be used for the bonding of bigger substrates such as mattresses by spray or glue line application, especially application by roller coater. Another advantage is possible mixtures with other VESTOPLAST® grades to achieve the exact needed properties, which gives the formulator a greater flexibility. In mattress applications for example, a mixture with of VESTOPLAST® EP V2103 and VESTOPLAST® 750 can provide higher elongation values and optimized viscosity. As shown in figure 10, the tensile strength improves significantly to 2.3 MPa with an elongation at break of 200% by adding 30% of VESTOPLAST® 750, while the material remains soft. The high molecular weight of VESTOPLAST® 750 increases the green strength that is required for bonding foams.

The open time is of major importance in this application to provide the operator enough time between applying the adhesive and bonding the large substrates. Depending on the production line, it needs to be adjustable from long to short open times. As shown in Figure 11, the mixture of VESTOPLAST® EP V2103 and VESTOPLAST® 750 can

provide exactly the needed flexibility. By varying the parts of VESTOPLAST® 750, the initial open time of pure EP V2103 of ~5 min can be reduced down to ~1.5 min, this gives the applicator greater flexibility to run the production lines according to his needs. The same goes for the melt viscosity, which can be increased significantly. However, the softening point still remains on a low level of ~100 °C, allowing for a low application temperature, leading to energy cost savings.

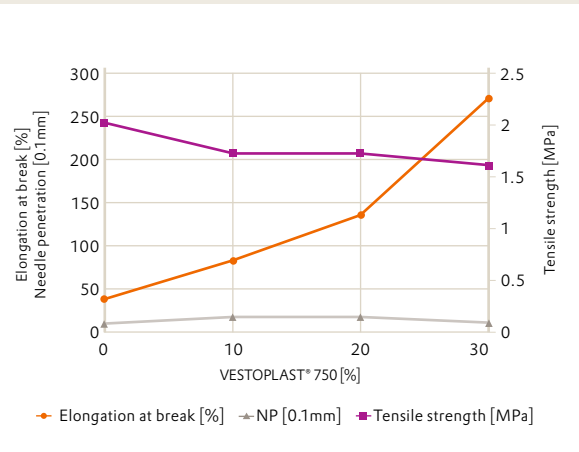


Figure 10: Tensile strength, elongation at break and needle penetration behavior of mixture of VESTOPLAST® EP V2103 and VESTOPLAST® 750

SIS based adhesives are also often used for mattress applications as well. However, depending on the raw material situation, VESTOPLAST® enables for high polymer content formulations thus reducing the dependency on tackifiers, whereas SIS formulations are very tackifier intensive. Additionally, VESTOPLAST® based adhesives are much easier to produce as they do not require any shear mixing. As APAO based formulations do not require any oil there is no risk of oil emigrating onto the substrates.

The main polymer in the formulation is VESTOPLAST® EP V2103. By formulating with VESTOPLAST® 750 and VESTOPLAST® 408 the initial cohesion and green strength is increased, while keeping a low viscosity and a long open time. Tackifiers are added to further increase the adhesion, while a smaller portion of SEBS rubber can be added to get the needed flexibility and add to the cohesion. Polybutene might be used to fine tune the viscosity and softness. As an example, a guide formulation is provided in Figure 12.

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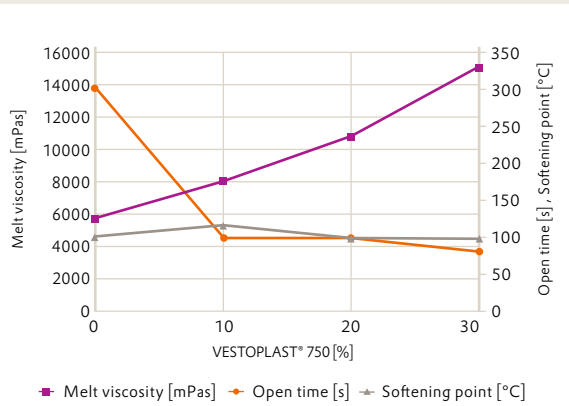


Figure 11: Viscosity, open time and softening point behavior of mixtures of VESTOPLAST® EP V2103 and VESTOPLAST® 750.

Guide formulations for packaging applications:  
Fast setting with high polymer content

| Formulation                 | A             | [%] | B               | [%] |
|-----------------------------|---------------|-----|-----------------|-----|
| VESTOPLAST®                 | EP V2103      | 90  | EP V2103        | 70  |
|                             |               |     | Tackifier resin | 15  |
| Wax                         | SARAWAX SX 80 | 10  | SARAWAX SX 80   | 15  |
|                             | Stabilizer    | 0.2 | Stabilizer      | 0.2 |
| Viscosity @ 160 °C [mPa s]  | 2,600         |     | 960             |     |
| Softening Point [°C]        | 101           |     | 95              |     |
| Needle penetration [0.1 mm] | 17            |     | 14              |     |
| Setting time [s]            | 1             |     | < 1             |     |
| S.A.F.T [°C]                | 85 - 90       |     | 70 - 75         |     |

Figure 9:  
Exemplary formulation

Guide Formulation

|           |                      |
|-----------|----------------------|
| 45-60 %   | VESTOPLAST® EP V2103 |
| 5-15 %    | VESTOPLAST® 750      |
| 5-10 %    | VESTOPLAST® 408      |
| 20-30 %   | Tackifier resins     |
| 0-8 %     | SEBS rubber          |
| 0-10 %    | Polybutene           |
| 0.2-0.5 % | Stabilizer           |

Technical Properties

|                                       |                |
|---------------------------------------|----------------|
| Softening point (R&B) [°C]            | 100 - 110      |
| Melt viscosity at 160 °C [mPas]       | 3,000 - 10,000 |
| Needle penetration (100/25/5) [0.1mm] | 15 - 25        |
| Open time [min]                       | 2 - 6          |
| Elongation at break [%]               | > 250          |

Figure 12: Guide formulation for mattress application and its technical properties

# DEGALAN®:

## Evonik expands portfolio for binders for heat sealing applications

The binders for heat sealing applications sold by Evonik Resource Efficiency GmbH under the brand name DEGALAN® offer unique options for formulating high performance heat sealing lacquers. They are primarily used for sealing dairy packaging such as yogurt cups and pharmaceutical blister packs.

The requirements for heat sealing lacquers keep becoming more demanding. Among other factors, this is reflected by a wide range of materials to be sealed. The lids of yogurt cups, for example, can be made of aluminum, plastics such as PET, or paper-PET composite systems, while cup materials consists of polypropylene (PP), polystyrene (PS) or polyethylene terephthalate (PET). Here, DEGALAN® binders ensure a reliable and strong seal and at the same time are convenient and easy to open. Furthermore,

binders and heat seal lacquers are expected to contribute to sustainability and environmental compatibility and increase the productivity of the packaging industry.

Evonik Resource Efficiency GmbH recently launched DEGALAN® VP 4311 E, which was specifically developed for paper-PET composite systems that are sealed with polystyrene (PS) or polylactic acid (PLA) - a biodegradable plastic made from corn starch. The product is PVC-free and thus meets the requirements of consumers as well as retailers. DEGALAN® VP 4311 E can be applied in a single production process, since a common precursor step to apply an adhesion promoter is no longer necessary.

With the newly developed DEGALAN® VP 4322 E, Evonik Resource Efficiency GmbH offers another innovative

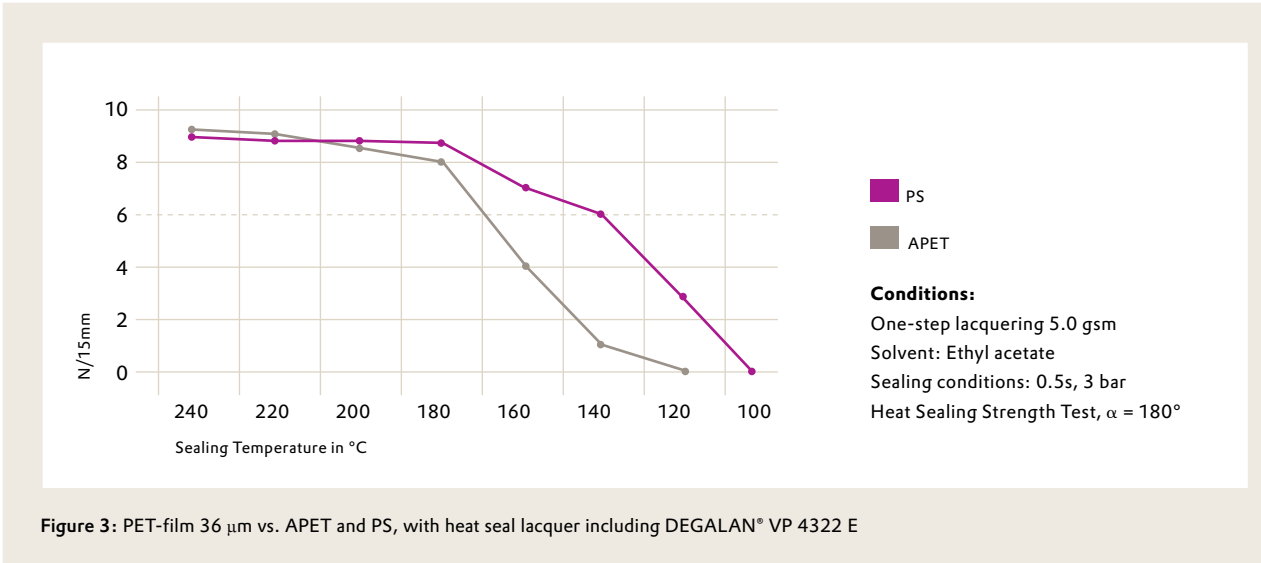
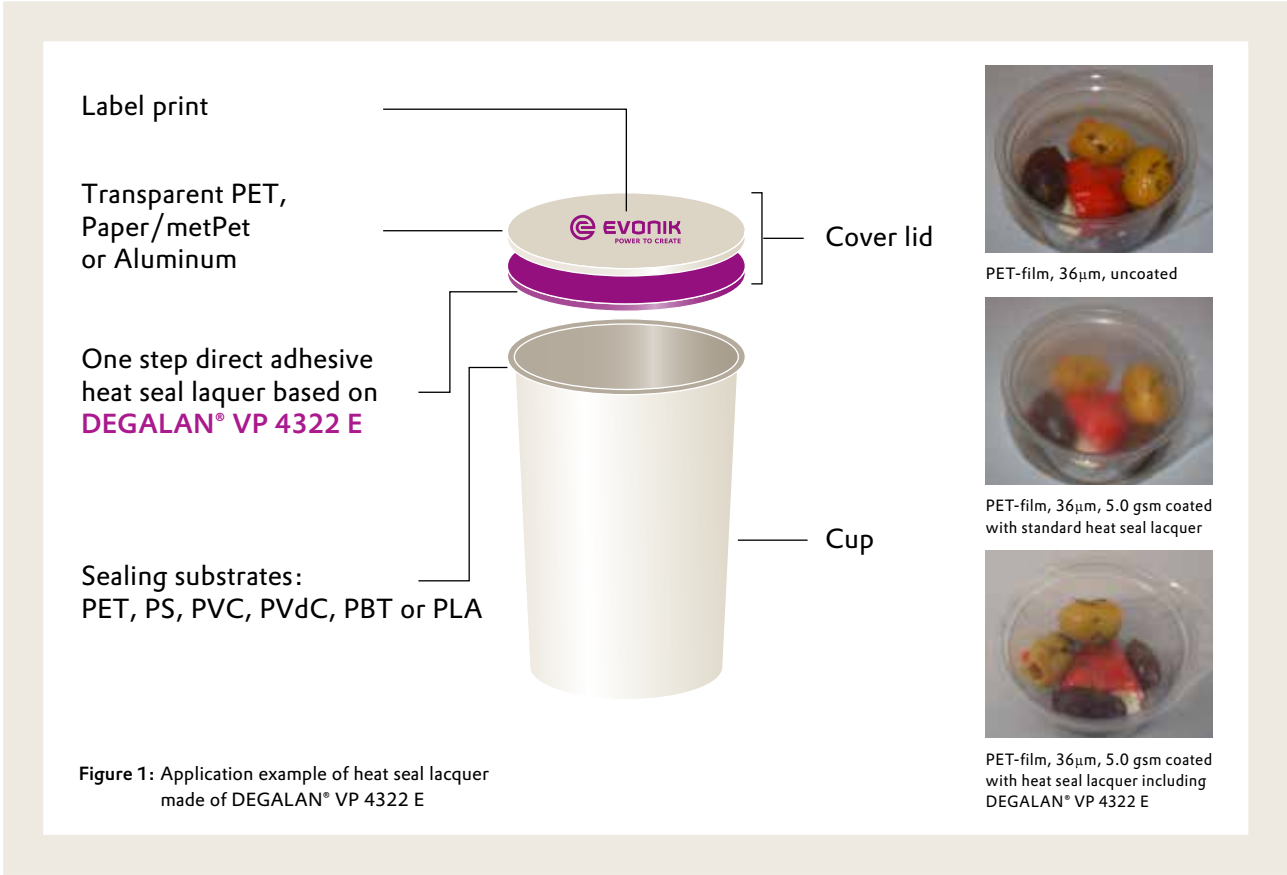
heat sealing binder that is specifically designed for fully transparent packaging applications. The trend toward transparent packaging becomes increasingly evident in retail refrigeration cases. Premium product manufacturers utilize transparent packaging elements like lids to differentiate themselves from competition while packaging suppliers benefit as well: DEGALAN® VP 4322 E is applied in a single coating process, which does not require or contain any PVC. The formulation of heat sealing lacquers for transparent packaging can be greatly simplified by using DEGALAN® VP 4322 E, which directly results in reducing complexity and increasing efficiency. The product can be sealed against the most common materials such as polyethylene terephthalate (PET), polystyrene (PS), polyvinyl chloride (PVC), polyvinylidene chloride (PVdC), and polylactic acid (PLA). A pure PET seal offers additional advantages such as high recyclability, and therefore represents a true and cost-effective alternative to PET-PE composites, which are still primarily used in transparent composite packaging. At the same time, DEGALAN® VP 4322 E also offers the benefit of a high seal strength of more than 8 N/15 mm. Its haze value is below 10, measured on a PET film with a dry coating weight of 5.0 gsm, to give consumers a clear view of the packaged items.

### Technical Properties

|                      |   |
|----------------------|---|
| Heat Seal Strength   | HSS > 8 N/15mm  |
| Lid materials        | Transparent PET and Paper/PET-laminates                 |
| Substrates           | PET, PS, PVC, PVdC, PLA, PBT                            |
| FDA §175.300         | ✓   |
| Solid content        | 40 %  |
| Viscosity            | < 8.000 mPas  |
| Haze at 5.0 gsm      | Bare Film: 4 / Reference: 60 / DEGALAN® VP 4322 E: < 10 |
| Blocking Temperature | > 60°C  |

Figure 2: Technical Properties of DEGALAN® VP 4322 E

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# NEW DEGALAN® 42-SERIES

## Evonik Heat Sealing provides upgraded products to meet trends of PVC-free packaging

Evonik heat seal binders are designed to provide secure sealing and a clean and smooth peel. In yoghurt packaging, cups sealed with DEGALAN® heat seal based lidding open without chattering.

Since 2007 PVC-free DEGALAN® binders have become state of the art in dairy packaging applications. In solvent based lacquer formulations, DEGALAN® PM 666 is meanwhile market standard with regard to primer less heat seal lacquers. DEGALAN® PM 666 is established as an universal binder in PVC-free heat seal lacquers for sealing aluminum foil versus polypropylene (PP), polystyrene (PS), polyethylene terephthalate (PET) or polyvinyl chloride (PVC).

As a consequence of the market request for improved products, Evonik Heat Sealing is now offering a full range of upgraded DEGALAN® binders called the 42-Series. The new 42-Series is based on copolymers containing no ethylidene norbornene, a narrower molecular weight distribution and a lower viscosity. As a result, the new DEGALAN® Series has no restrictions for any filling good and shows less tendency causing rub-off deposits on customer rollers during printing and sealing processes. Besides, most of the new products have a higher solid content.



DEGALAN® PM 666 improved reference is named **DEGALAN® VP 4221 E** and provides best heat seal performance at a typical dry coating weight of 6 gsm. Typically a sealing strength between 7 and 9 N/15 mm is observed with PP and PET substrates, whereas a sealing strength between 8 and 10 N/15 mm is observed with PS substrate.

DEGALAN® 4151 E improved reference is named **DEGALAN® VP 4251 E** and provides best heat seal performance at the same dry coating weight of 6 gsm. The solvent composition has not changed while the solid content was increased from 40 % to 45 % without influence on the viscosity.

The most universal applicable product is the new **DEGALAN® VP 4294 E** which is unique in its polymeric structure. The organic dispersion consists of copolymers of methacrylic esters grafted on olefin copolymers (OCP) and polyesters. DEGALAN® VP 4294 E shows direct adhesion to aluminum and PET. Due to its new polymer and solvent composition, the product has a high solid content of 52 % and is able to be dried fast. It is most commonly used in heat seal lacquers for sealing PET film versus polypropylene (PP), polystyrene (PS), polyethylene terephthalate (PET) or polyvinyl chloride (PVC). Even polylactic acid (PLA) substrates can now be sealed.

**DEGALAN® VP 4294 E** will give best heat seal performance at a typical dry coating weight of 6 gsm. The quality of the PET film has a significant influence on the sealing strength when DEGALAN® VP 4294 E is used. Typically a sealing strength between 4 and 7 N/15 mm is observed on PP substrates, whereas a sealing strength between 5 and 8 N/15 mm is observed with PS substrate. Like all DEGALAN® heat seal binders, DEGALAN® 42-Series fulfills the requirements according to FDA 21 CFR § 175.300.

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Solid content of DEGALAN® 42-Series products

| DEGALAN®           | Solid content [%] | DEGALAN® 42er Serie | Solid content [%] |
|--------------------|-------------------|---------------------|-------------------|
| DEGALAN® PM 555    | 45                | DEGALAN® VP 4220 E  | 45                |
| DEGALAN® PM 666    | 45                | DEGALAN® VP 4221 E  | 45                |
| DEGALAN® 4150 E    | 43                | DEGALAN® VP 4250 E  | 45                |
| DEGALAN® 4151 E    | 40                | DEGALAN® VP 4251 E  | 45                |
| DEGALAN® VP 4174 E | 47                | -                   | -                 |
| -                  | -                 | DEGALAN® VP 4294 E  | 52                |

Figure 1



Samples of all DEGALAN® 42-Series products are now available. Please contact our Technical Service by Phone: +49 6151 18 4960 or +49 6151 18 4753, email: [degalan@evonik.com](mailto:degalan@evonik.com)





# DYNACOLL®: DYNACOLL®-polyesters supports resource efficiency in vehicles

An innovative approach for (semi-) structural lightweight bonding with novel 2C PU hot melts.



Evonik is constantly investing in innovation to support its customers and provide solutions to fulfill the requirements of the markets and their trend of using the resources more efficiently.

Weight reduction is an effective way to improve automobile fuel economy, to decrease the combustion emissions or to extend the cruising range of e-cars. Lighter materials such as composites, which are carbon or glass fibers enclosed in a bonding polymer, started to replace steel as the preferred main material in automobile parts. The use of composites allow reducing up to 75% of the initial weight with the same or higher strength and stiffness ratio. These reflected in fuel economy would produce a decrease of up to 25% in the fuel consumption. The use of alternative substrates brought along the need to develop different joining technologies. DYNACOLL® polyesters, used as key raw material in the production of reactive PU hot melt adhesives, contribute to achieve the bonding of alternative and lighter substrates in the automobile industry.

As shown in figure 1., there are different bonding systems in the industry. One-component epoxy systems cured at high temperatures were the state of the art for structural bonding of steel, but they do not provide sufficient elongation to work with composites, having low resistance to temperature changes.

In order to overcome this, the use of polyurethane systems was widely spread. Two-component polyurethane liquid systems result on bonds with high shear strength and elongation, but low initial strength. The use of heat or catalysts to accelerate and build up sufficient handling strength is effective but results on processes that are more complex.

Another option is to use one-component polyurethane hot melts that provide high initial strength, due to their hot melt character, and temperature resistance by crosslinking with atmospheric moisture.

In 1C PU hot melts, the crosslinking is enabled by the reaction with moisture from the air. Therefore, the water diffusion has an important influence on the final properties

see page 36 ►

## Unique properties of DYNACOLL® for 2C PU hot melts

- Modular system of amorphous, liquid and crystalline DYNACOLL®-polyesters polyols
- Tailor-made linear and branched grades
- Good adhesion to various substrates
- Physical bonding due to hot melt character
- Moisture independent crosslinking reaction
- High elongation and cohesive strength

## Your benefit

- High handling strength and short cycle times
- Robust procedure as no acceleration by catalysis is needed
- Broad application area for mixed materials
- No primers needed
- Complete curing of thick bond lines
- Supports temperature changes ( $\Delta a$ )

### New modified polyesters for 2C PU hot melts

| Polyester properties | Tg [°C] | Tm [°C] | F | OH-number [mg KOH/g] |
|----------------------|---------|---------|---|----------------------|
| DYNACOLL® EP 415.08  | 45      | -       | 2 | 70                   |
| DYNACOLL® EP 412.01  | 25      | -       | 2 | 50                   |
| DYNACOLL® EP 423.02  | -30     | -       | 3 | 100                  |
| DYNACOLL® 7360       | -60     | 55      | 2 | 30                   |
| DYNACOLL® 7362       | -60     | 55      | 2 | 50                   |

Tg: Glass transition temperature  
Tm: Melting temperature  
F: Functionality

### Properties of adhesive formulations

| Adhesive properties               | PU-HM 1 | PU-HM 2 |
|-----------------------------------|---------|---------|
| Tensile strength* [MPa]           | 15      | 22      |
| Elongation at break* [%]          | 240     | 160     |
| Open time [sek]                   | 510     | 190     |
| Handling strength (0.3 MPa) [min] | 30      | ≤ 5     |
| Shear adhesion CFRP** [MPa.s]     | 11      | 14      |

Composition of model formulations  
Coating thickness 2 mm  
PU-HM 1: DYNACOLL® EP 412.01, EP 423.02 and 7362, MDI, Fillers  
PU-HM 2: DYNACOLL® EP 415.08, EP 423.02 and 7360, MDI, Fillers  
\* after 7 days    \*\* Carbon fiber reinforced polymer

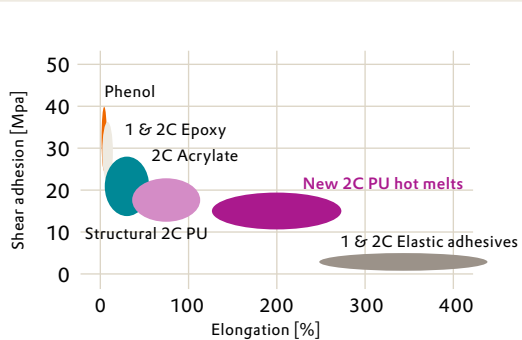
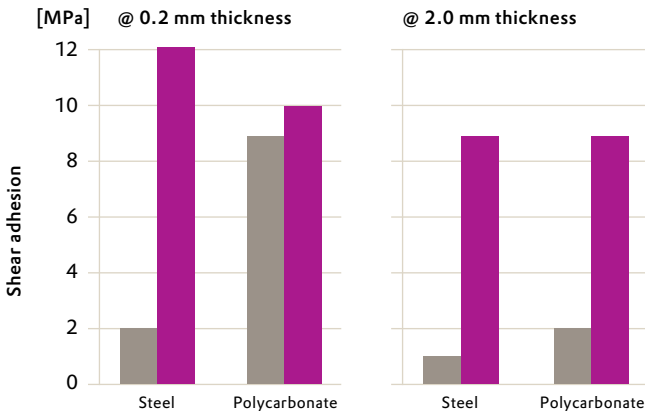


Figure 1: Positioning of new 2C PU hot melt systems



One component polyurethane hot melt (1C PU HM)  
Two component polyurethane hot melt (2C PU HM)

**2C PU - HM:**  
Model formulation PU-HM1  
Shear adhesion after 7 days  
**1C PU - HM:**  
Similar morphology to PU-HM1  
Shear adhesion after 7 days moisture curing @ 20 °C + 65 % rh

Figure 2:  
Comparison of performance between 1C and 2C PU hot melts in steel and PC with different thicknesses

like cohesive strength and processing speed. Depending on the nature of the substrate, the morphology and thickness of the adhesive, the availability of water for the crosslinking varies resulting on different performance and final properties, as shown in figure 2. As for composite bonding relatively high coating weights are applied for gap filling, moisture independent curing adhesive systems are preferred.

Evonik has recently developed a new and complementing approach to support the industry in the formulation of adhesives with high initial and cohesive strength and fast curing without requiring a catalyst. It consists of a two-component (A and B) polyurethane hot melt where the physical bonding occurs by solidification before the

chemical crosslinking; A is a polyester polyol mixture with fillers and B is a polyester prepolymer with fillers. By using new linear and branched DYNACOLL®-polyester polyols it is possible to reduce the cycle time. Thus, improving the productivity in the automobile assembly.

2C PU hot melts provide an additional advantage since catalysts, heat or primers are not required to accelerate or support the adhesion.

Evonik has filed a patent application for this new adhesive system.

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Technological properties of competitive systems

|   | 2C-PU hot melt | 2C-PU liquid | 1C-Epoxy |
|---|----------------|--------------|----------|
| Initial strength built up                   | +              | 0            | -        |
| Robustness of system in production          | +              | -            | 0        |
| Supports temperature changes ( $\Delta a$ ) | +              | +            | 0        |
| Elongation                                  | +              | +            | -        |
| No heat treatment required                  | +              | +            | -        |
| Shear adhesion                              | 0              | 0            | +        |
| Adhesives handling in application           | -              | -            | +        |

New 2C- PU hot melt adhesive formulation:

Component A: Mixture of polyols (polyesters and others) and fillers

Component B: Isocyanate terminated prepolymer and fillers

VESTOPLAST®

Polymers for hot melt adhesives in comparison

There are optimized adhesive systems for every application. Depending on the application different properties can be crucial; these can relate to the chemical-physical properties of the adhesive on the one hand, but on the other hand also to sensory properties such as outer appearance and odor. Further it can also include handling of the product during the production process, and of course, commercial aspects.

The selection of the base polymer has a significant impact on the properties of the final formulation. However, the base polymer also determines the choice of additional raw materials that are required in the formulation, such as tackifier resins, waxes or oils and the needed quantities (or proportions). A number of typical systems are described in the following article.

Metallocene polyolefin (mPO)

Common metallocene-catalyzed polyolefins include copolymers based on ethene and octene or ethene and propene. These monomers have different effects on the polymer: Propene provides hardness or crystallinity as well as a high softening point and reduces the open time. These are all properties which are often required in many applications. However, it has a certain negative impact on the adhesion and the material shows some inflexibility. Ethene, in contrast, makes the polymer chains more flexible and contributes to the amorphous character of the polymer, while butene or octene create more crystalline structures. With the increasing length of the co-monomer, steric effects, which extend the time required for crystallization and accordingly, the open time and the development of cohesion, are also more pronounced. At the same time, melting temperatures are reduced in comparison to pure polypropylene (PP) (see Fig. 1). These effects have to be offset in a formulation sometimes substantially.

VESTOPLAST® is an amorphous and partly crystalline terpolymer based on ethene, propene, and butene. Butene plays a major role in keeping the balance between open time and crystallinity as well as hardness and cohesion. This is clearly evident in the melting temperature of homo-butene, which lies between the values of PP and polyoctene. This is accompanied by an increased thermal stability and good adhesion.

The VESTOPLAST® portfolio offers different grades with higher softening points and shear adhesion failure temperatures (SAFT). This is achieved by the optimal integration of propene and butene.

The unique composition of VESTOPLAST® has a major influence on the formulation for hot melt adhesives. VESTOPLAST® allows for adhesives with a high polymer content, which in turn enables for the perfect adhesive for every application with little formulation effort. In addition, the dependency on additional raw materials such as resins and waxes decreases.

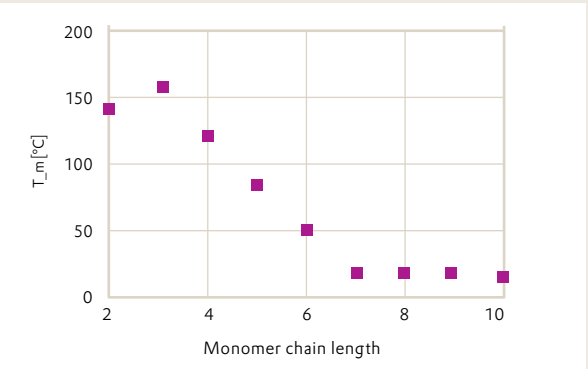


Figure 1: Development of melting temperatures in different (homo) poly-alpha-olefins. The maximum is reached with polypropylene and decreases continuously until it reaches the lowest point at polyoctene.



### APPLICATION EXAMPLE: PACKAGING

Figure 2 shows the comparison between a typical mPO based formulation with a VESTOPLAST® based formulation.

Formulations based on VESTOPLAST® require on the one hand less tackifier resins to achieve the required adhesion and on the other hand less wax to achieve a nucleating effect on the crystalline parts.

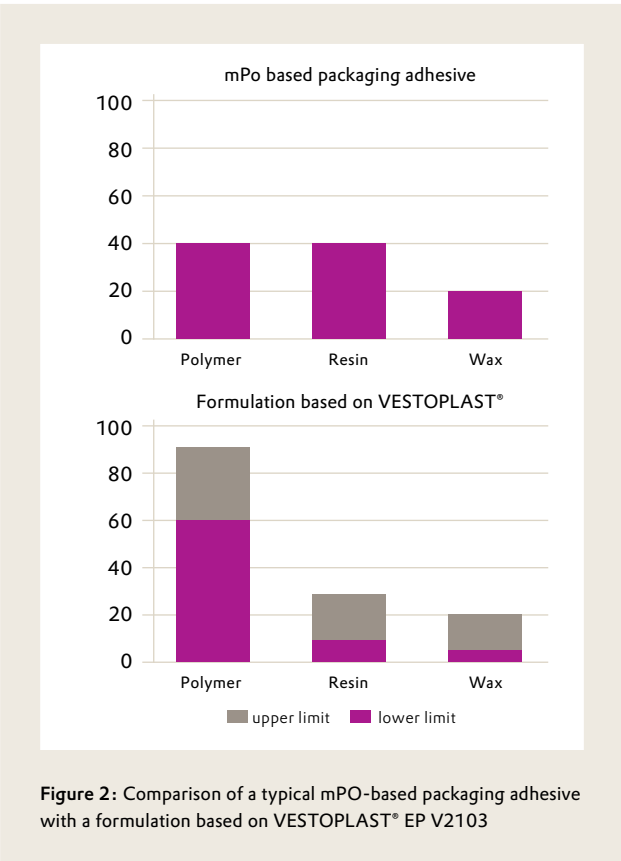


Figure 2: Comparison of a typical mPO-based packaging adhesive with a formulation based on VESTOPLAST® EP V2103

### Ethylene-vinyl acetate copolymers (EVA)

Ethylene-vinyl acetate copolymers are widely used and can be found in basically every hot melt application. They are known for their broad application spectrum and low cost. A limiting effect is the moderate softening point of the polymer, which restricts the thermal stability of EVA-based formulations to temperatures around 80°C. The density of the base polymer is approximately 0.95 g/cm³. Furthermore, fillers are often added to EVA adhesives, which further increases their density and reduces their productivity (also called mileage).

On the contrary, VESTOPLAST®-based hot melts offer high mileage due to the amorphous character of the base polymer and its low density of 0.86 g/cm³, which can exceed the mileage of filled EVA adhesives up to one third. The high thermal stability is another advantage and gives the formulator the possibility to achieve values exceeding 100°C.

### APPLICATION EXAMPLE: PROFILE WRAPPING

Polyolefin hot melt adhesives are suitable for all types of profiles, particularly untreated veneers, paper sheets, melamine resin coated films (also known as continuous pressure laminate or CPL) and of course, thermoplastic films. Figure 3 highlights the essential differences between EVA and VESTOPLAST®.

As the table shows, VESTOPLAST® combines high thermal stability with low to medium viscosity. The formulations result in high joint strength and are resistant to water. In processing, formulations based on VESTOPLAST® show excellent color stability and low cracking or gelling tendency. This contributes to the high quality of the end

product and reduces the cleaning and maintenance efforts in the production machinery. Another major advantage, in particular of unfilled VESTOPLAST®-based hot melt

adhesives, lies in their increased productivity during the application process. The low density results in a 30% higher productivity of the used adhesive.

|             | Density of base polymer [g/cm³] | Typical formulation density [g/cm³] | Productivity [%] | Softening point (R&B) of base polymer [°C] | SAFT     | Water resistance | Odor and color |
|-------------|---------------------------------|-------------------------------------|------------------|--|----------|------------------|----------------|
| VESTOPLAST® | 0.86                            | 0.9                                 | 133              | 85 - 160                                   | > 100 °C | +                | +              |
| EVA         | 0.95                            | 1.0                                 | 120              | approx. 85                                 | 80 °C    | o                | o              |
| EVA filled  | -                               | 1.2                                 | 100              | approx. 85                                 | < 80 °C  | o                | o              |

Figure 3: Comparison of VESTOPLAST® and EVA

### Block copolymers

Styrenic block copolymers (SBC) are the basis for hot melt pressure sensitive adhesives (HMPSA). These are widely used in all types of adhesive tapes, labels, laminates or textile bonding, as well as in sealing applications. The properties of SBC are determined by the styrene end-block and various monomers in the middle block, which can consist of butadiene, isoprene, ethylene/butene or ethylene/propylene.

SBC-based formulations are permanently tacky, making them particularly suitable for pressure sensitive adhesives and low temperature applications. A special feature of SBC is the existence of two different glass transition temperatures, one in the middle block in the range of -50°C to -90°C, and the other in the styrene part, at approximately 100°C. Processing SBC-based formulations therefore requires temperatures of at least 100°C, which means that lower application temperatures cannot be achieved due to the limitation of the styrene glass transition temperature.

The formulations typically contain large quantities of tackifier resins, which counts for the major part of the formulation, along with oils and in part, waxes and fillers. The polymer content is typically only 15-30%. Increasing the proportion of low molecular components imposes limits on properties such as maximum thermal stability or shear resistance.

### APPLICATION EXAMPLE: HYGIENE

SBC-based hot melt adhesives are commonly used for bonding diapers either in elastic bonding or in construction. Over the past few years, however, alternatives such as APAOs have gained more and more popularity as raw materials. The new VESTOPLAST® EP V2094 is tailored to fulfill the needs of the hygiene sector exactly. For more detailed information as well as test results, please refer to the article „VESTOPLAST®: Improved hot melts through innovation“ on page 22 in this magazine.

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