Application Leaflet

# THIXATROL® AS 8053 and THIXATROL® PM 8054

Organic thixotropes for silylated PU based sealants



# Key Benefits

- Superior extrudability
- Excellent sag stability
- Strongly shear thinning flow

# Chemical and physical data

	THIXATROL® AS 8053	THIXATROL® PM 8054
Composition	Proprietary organic	
Appearance	Fine off white powder	
Bulk density	0.25 g/cm <sup>3</sup>	
Melting point	120 - 130 °C	
Mean particle size	max. 5 µm	max. 6 μm

## Introduction

Silylated polyurethane based polymers (SPUR), are hybrid systems that are used in the formulation of modern adhesives and sealants for the construction, automotive industry and others. They create durable bonds with a high amount of flexibility after application and curing.

The main benefits of SPUR based adhesives and sealants are:

- Easy handling and workability
- High elasticity
- No staining, even on porous substrates
- Excellent durability
- Good aging properties
- Can be painted

# Technical background and activation

Organic rheological additives based on castor wax and diamides have been used in non-aqueous systems because of their outstanding sag and slump control as well as the highly thixotropic structure they provide.

Historically, diamide and castor wax based rheology modifiers were developed for solventborne applications in which a high amount of rheological structure was needed. The selection of a suitable organic thixotrope depends on the polarity of the solvents used and the ability to control the processing temperature.

Higher polarity solvents have the highest solubility, aliphatic solvents have the lowest solubility.

Processing at elevated temperatures increases the solvency of organic thixotropes. The higher the solubility the lower the required processing temperature. In case of solvent free systems, where solubility is low, higher activation temperatures are necessary.

Castor wax based organic thixotropes can be used for low to medium polarity solvent based systems such as aromatic and aliphatic systems, as well as solvent free systems. They are not suitable for ketones, esters and other high polarity solvents, due to a too low activation temperature, sometimes equal to or below room temperature. In solvent free systems the activation temperature can be easily reached.

Castor derivatives at sometimes show undesirable side effects, such as seeding and false body, especially when not produced within the optimum processing window. Seeding and false body can be prevented by constantly monitoring the process conditions. To prevent seeding the maximum solubility temperature should not be exceeded.

False body effects can be eliminated by cooling down under stirring after the activation process. Typically castor wax based materials cannot be packed out hot. Exposure to heat after packing out can cause an increase in viscosity and should be avoided.

Diamide based organic thixotropes are more robust, the side effects occurring with castor waxes are much less prevalent. The activation temperature of diamides is on average higher than with castor waxes depending on the solvent used.

Diamide grades are suitable for high polarity solvent based systems where they can be activated at moderate temperatures. In solvent free applications the activation temperature can be too high, warranting the use of heated jacketed vessels to be able to either actively cool or heat. Nevertheless, most producers prefer using just the friction heat generated during dispersion.

The new organic thixotropes THIXATROL® AS 8053 and THIXATROL® PM 8054 have markedly lower activation temperatures than traditional diamide technology, while having none of the negative side effects of the castor wax derivatives like seeding or false body. They can be packed out hot. A lower activation temperature means that they can be processed at regular mixing temperatures generated in production, this results in a significant cost benefit for the manufacturer.

A major focus of the development was to generate organic thixotropes that act independently from variations in processing temperature. They should provide a stable viscosity build over a wider temperature range. This results in increased process robustness under real life conditions.

## Products tested

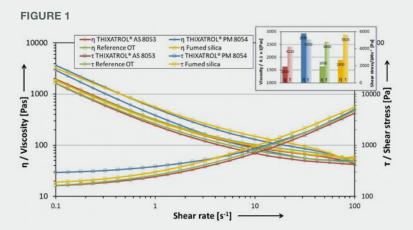
#### **SPUR** polymer sealant - Formulation

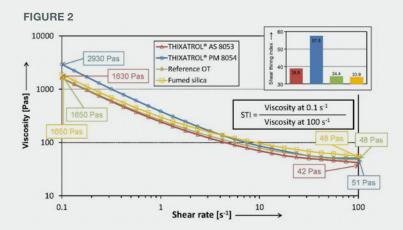
All tested rheology modifiers, including THIXATROL® AS 8053 and THIXATROL® PM 8054, were incorporated and activated without active temperature control. The entire mixing time in a vacuum enabled double planetary mixer was 75 minutes. The maximum temperature reached in all cases was in an ambient range of below 50 °C.

#### **Process instruction**

- 1. Dry all used fillers and pigments at 105 °C for at least 4 hours prior to the use to minimize.
- 2. Place SPUR pre-polymer, plasticizer and moisture scavenger (1st portion) in the mixing vessel and homogenize at low speed using a vacuum enabled double planetary mixer.
- 3. Add the individual portion of rheology modifier under low shear.
- 4. Add pigments and fillers in 2 3 charges under constant agitation.
- 5. Deaerate and mix at highest speed for 75 minutes.
- 6. Reduce mixing speed and break the vacuum to add moisture scavenger (2nd portion), adhesion promoter and catalyst.
- 7. Continue mixing for another 5 minutes under vacuum.
- 8. Fill under nitrogen (N2) blanket into cartridges.

Component	Concentration [%]
Silylated polyurethane prepolymer (SPUR)	22.94
Plasticizer	18.35
Calcium carbonate extender	55.28
Titanium dioxide	0.68
Moisture scavenger	0.34
Adhesion promoter	0.57
Rheological additive	1.81
Catalyst	0.03
Total	100.00





#### Rheological character

The **FIGURE 1** shows the flow characteristics of the SPUR polymer based sealant formulated with equal amounts of 1.81 % rheology modifier. In the small figure, placed in the upper right corner of the diagram, the low shear viscosities at 0.1 s<sup>-1</sup> and the shear stress values at higher shear forces are displayed.

THIXATROL® AS 8053 and THIXATROL® PM 8054 are tested in comparison to a market reference organic thixotrope and fumed silica.

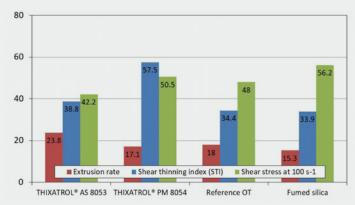
The **FIGURE 1** the direct correlation between shear rate and shear stress is displayed. The viscosity at low shear rates correlates to practical properties such as sag and stability. Shear stress at high shear rate displays the real force values at these conditions and can be helpful to estimate the extruding behavior of a sealant.

In terms of shear thinning characteristics, THIXATROL® AS 8053 performs very similar to the market reference organic thixotrope, however, the shear stress at high shear conditions is somewhat higher than the reference. The fumed silica performs similar at low shear viscosity as well, but the shear stress detected at high shear forces was the highest of all tested samples. The sample with THIXATROL® PM 8054 shows the highest low shear viscosity, but the shear stress observed at higher shear was slightly lower than with fumed silica.

#### **Shear thinning index**

The **FIGURE 2** shows the shear thinning index. This is the factor of the viscosity at low shear divided by the viscosity at high shear. The industry uses the shear thinning index as an indication of the ability to extrude a sealant. The bigger the factor, the better the extrudability is intended to be. The data shown is identical to the one displayed in the previous figure, here it is plotted on a double logarithmic scale. The calculation is based on the viscosities at a shear rate of 0.1 s<sup>-1</sup> and 100 s<sup>-1</sup>.

FIGURE 3



The sample formulated with THIXATROL® PM 8054 displays the highest shear thinning index followed by the one formulated with THIXATROL® AS 8053. The values of the sample with the fumed silica demonstrates very similar results as the one containing the reference organic thixotrope. Both values are only slightly lower than the sample modified with THIXATROL® AS 8053.

#### Extrusion rates in comparison to rheological data

The **FIGURE 3** shows the extrusion rates in grams sealant per minute in comparison to the shear thinning index and the shear stress values determined at higher shear rates of 100 s<sup>-1</sup>. The rheology data was extracted from the previous figure.

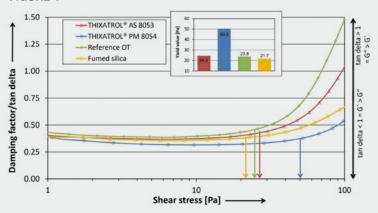
The sample with THIXATROL® AS 8053 provides the best practical extrusion rates. THIXATROL® PM 8054 gives slightly lower extrusion data.

A comparison of the rheological data shows that the shear thinning index for THIXATROL® PM 8054 is somewhat higher than for THIXATROL® AS 8053. It is often assumed that high shear thinning indices show improved extrusion rates. That is not the case here with THIXATROL® PM 8054. The samples formulated with the reference organic thixotrope, the fumed silica and THIXATROL® AS 8053 do show better extrudability for a higher shear thinning index.

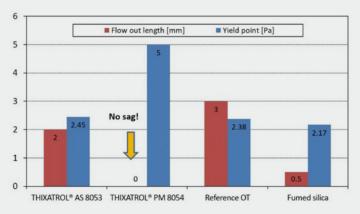
When the shear stress data are compared at high shear rates, it shows a different picture: the lower the shear stress values at high shear rates are in all four cases, the better the extrusion rates are.

In conclusion, the shear stress at high shear rates is a good indicator to estimate the extrudability: the data indicates the relevant energy at the moment of application. The shear thinning index on the other hand only shows a viscosity gradient depending on the applied shear rate.

FIGURE 4



#### FIGURE 5



#### **Determination of the yield value**

The **FIGURE 4** displays the yield points of the SPUR sealants. The yield point is the shear stress at the end of the linear viscoelastic range (LVE). The data is plotted separately in the small figure in the upper part of the diagram.

The sample formulated with THIXATROL® PM 8054 displays by far the highest yield values. Material formulated with the other rheology modifiers performs very similarly to each other.

#### Slump stability in relation to the yield value

The **FIGURE 5** shows the slump control as the length of material flowing out of a vertically positioned gap in relation to the yield point data plotted in the previous figure.

The sample containing THIXATROL® PM 8054 performs the best in terms of slump control. All other samples display weaker performance as the length of the material flowing out of the vertically positioned gap is longer. The sample formulated with THIXATROL® PM 8054, shows the best sag control and has by far the highest yield point. All other samples demonstrate very similar yield points.



# Conclusion

SPUR based sealants formulated with THIXATROL® AS 8053 and THIXATROL® PM 8054, display remarkable improvements over traditionally used organic thixotropes and fumed silica. They are recommended for use in these systems.

#### Samples containing THIXATROL® AS 8053 show:

- Excellent extrudability, visible as the lowest shear stress at the moment of extruding.
- Good sag control and yield point

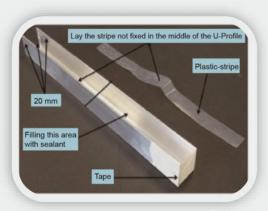
#### THIXATROL® PM 8054 shows:

- The same extrudability as fumed silica and the reference organic thixotrope
- Best slump control and the highest yield point
- Highest shear thinning index

The rheological data support the practical results.

We feel confident that we can help you improve your products. Please contact our technical department if you have any additional questions, or if you would like to try our products.

#### FIGURE 6



#### **APPENDIX**

# Test methods

- The rheograms and viscoelasticity/oscillation curves were determined using the Anton-Paar MCR 301 rheometer, equipped with measuring geometry PP 25 (serrated surface), at a gap width of 1 mm and at a temperature of 23 °C.
- 2. The shear thinning index (STI) indicates the factor of the viscosity at 0.1 s-1 divided by the viscosity at 100 s<sup>-1</sup>.
- 3. Extrusion rates were tested in accordance with ASTM C 603; Extruding the sealant from a Semco cartridge with a 5 mm nozzle; the pneumatic gun used a pressure of 4.1 bar; the amount of sealant extruded at ambient temperature of 22 23 °C in one minute was plotted.
- 4. Slump was tested using below described method: (FIGURE 6)
- Aluminum U-Profile 20 × 20 mm
- 25 cm long
- Close one end of the profile with a tape
- Lay the plastik-stripe not fixed in the middle of the U-Profile
- Fill 12 cm of the profile with sealant (horizontal position)
- Remove the tape
- Hanging the U-Profile in a vertical position

# **Abbreviations**

- OT: Organic Thixotrope.
- LVE: Linear Viscoelastic Range.
- SPUR: Silylated PolyUrethane Resin

#### NOTE:

The information herein is currently believed to be accurate. We do not guarantee its accuracy. Purchasers shall not rely on statements herein when purchasing any products. Purchasers should make their own investigations to determine if such products are suitable for a particular use. The products discussed are sold without warranty, express or implied, including a warranty of merchantability and fitness for use. Purchasers will be subject to a separate agreement which will not incorporate this document.

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For more details please contact:

#### **North America**

Elementis 469 Old Trenton Road East Windsor, NJ 08512, USA Tel: +1 609 443 2500

#### **Europe**

Elementis UK Ltd c/o Porto Business Plaza Santos Pousada Street, 290 4300-189, Porto, Portugal

#### Asia

Deuchem (Shanghai) Chemical Co., Ltd. 99, Lianyang Road Songjiang Industrial Zone Shanghai, China 201613 Tel: +86 21 577 40348

elementis.com

