ELEMENTIS

A global specialty chemicals company

Application Leaflet

THIXATROL® AS 8053

Alternative to fumed silica in SPUR based adhesives and sealants

Key Benefits

- Easier, less dusty, incorporation and safer handling
- Significantly higher efficiency compared to fumed silica
- Wide activation temperature window starting already at a very low range

Introduction

Silyl-terminated polyurethanes (SPUR) are the basis for numerous adhesives and sealants used worldwide. The high-performance of this hybrid technology is a result of the synergy between the silane-curing mechanism and polyurethane backbone. The SPUR polymers generally cures at room temperature and offer good durability. The curing mechanism of SPUR polymers is shown in *Figure 1.*

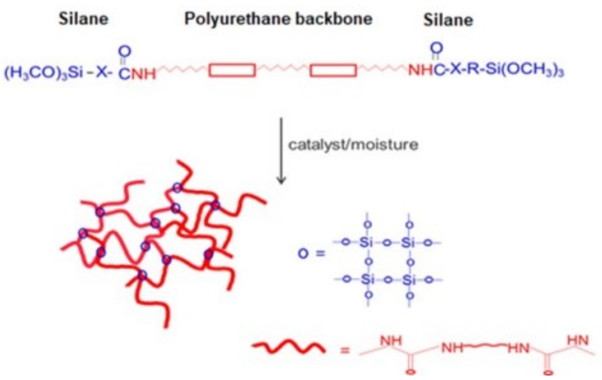

Figure 1: Curing mechanism of SPUR polymer

One of the major requirement of the sealants is to have a certain thixotropic behaviour resulting in sag specific properties for successful practical application. The thixotropic agent predominantly used in adhesive and sealants industry is fumed silica. In addition to thixotropy fumed silica provides reinforcement and mechanical properties. Although fumed silica contribute to the required structure-imparting properties, there are still a number of limitations associated with it. One of the major disadvantage is the very fine dust nature of the material, which makes it unpleasant and difficult to handle. Another drawback is the requirement for high shear, capital intensive dispersion equipment for adequate incorporation. Consequently, an alternative solutions have been sought for many years. Despite the efforts invested in replacing fumed silica, it still remains as a predominant thixotropic agent in adhesive and sealants industry.

To overcome these factors, Elementis is offering a product based on a diamide based wax technology under the commercial name THIXATROL® AS 8053.

Appearance	Fine off white powder
Composition	Proprietary organic
Buld density [g/ml]	0.25
Melting point [°C]	120-130
Main particle size [µm]	max. 5

THIXATROL® AS 8053, offers following benefits against traditional rheological additives (fumed silica, organic thixotropes etc.)

- Lower activation temperature allowing higher throughput and energy savings
- Higher efficiency compared to market reference resulting in excellent slump/sag resistance
- Wider application window in activation temperature leading to more robust production processes
- Easier (less dusty) incorporation and safer handling
- Compared to fumed silica, 20 times higher density allowing less volume in transport and storage space
- THIXATROL® AS 8053 is labelling-free and based on renewable raw material sources

In this study, we evaluated the performance of THIXATROL® AS 8053 against fumed silica in both pigmented and non-pigmented system. SPUR based prepolymer was selected as the base polymer. Various loading levels of THIXATROL® AS 8053 and fumed silica were considered in order to understand the influence on rheological, practical as well as mechanical properties.

Activation and mechanism

THIXATROL® AS 8053 needs to be activated in the system. It has been noticed that THIXATROL® AS 8053 starts activating around 50^0C. As the temperature increases more rheological performance is achieved. In the current system, good rheological performance was achieved in a range of 65-70^0C. Depending on the polarity of the system, solvent used one has to carry out their own temperature ladder study to realise the best performance.

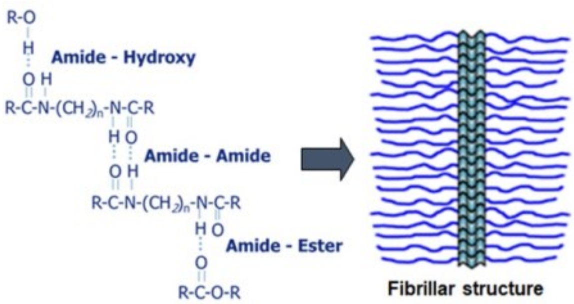

Figure 2: Diamide structure build

THIXATROL® AS 8053 function by forming a three dimensional network after proper activation. Once fully activated, the molecules orientate themselves towards each other, forming a fibrillar structure due to the nature of the amide functionality.

Intermolecular interactions such as hydrogen bonding and van-der-Waal forces are responsible for this formation backbone *(Figure 2)*.

THIXATROL® AS 8053 is non-associative by nature, it does not require the presence of pigments or fillers to function hence can be used in pigmented or clear systems. Scanning electron microscope (SEM) image of a fully activated diamide powder is shown in *Figure 3*.

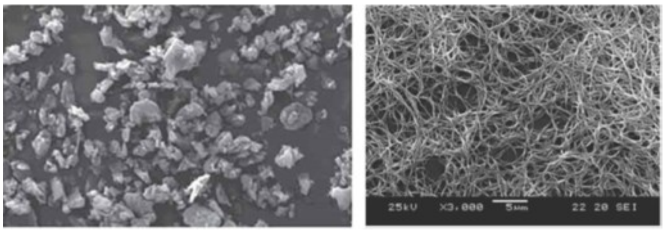

Figure 3: SEM fully activated diamide wax

Pigmented SPUR system

Both rheological modifiers, THIXATROL® AS 8053 and fumed silica were incorporated along with all other ingredients in vacuum controlled speed mixer for up to 5 mins at 2200 rpm. The temperature achieved for all samples were recorded between 64-70°C.

Figure 4a&b show the rheological performance (return flow curve) of SPUR polymer based sealant formulated with various amount of fumed silica and THIXATROL® AS 8053.

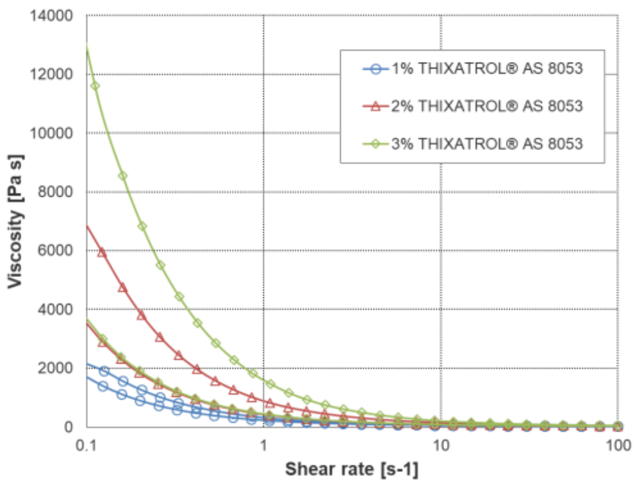

Figure 4a: THIXATROL AS 8053 in SPUR

All samples showed shear thinning behaviour which is important for the ease of practical application such as processing, extrusion etc.

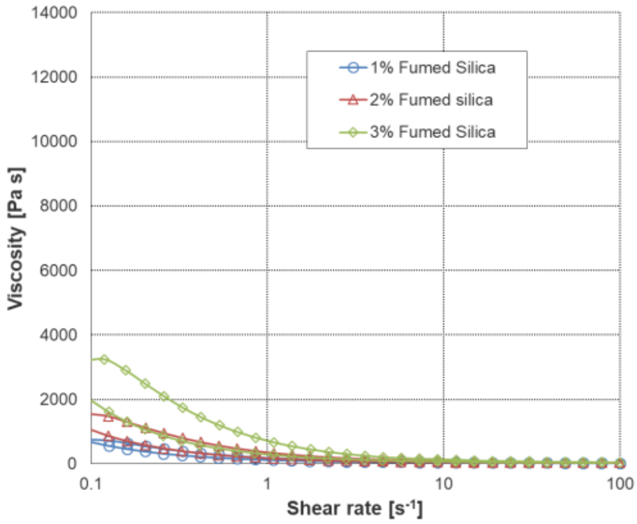

Figure 4b: Fumed silica in SPUR

Figure 5 extracted from the flow curve compares the low shear (0.1 s^{-1}) and high shear rate (100 s^{-1}) viscosity of both THIXATROL®AS 8053 and Fumed Silica. It is clearly visible that THIXATROL® AS 8053 outperforms fumed silica at all loading levels and provide significantly more performance as compared to fumed silica.

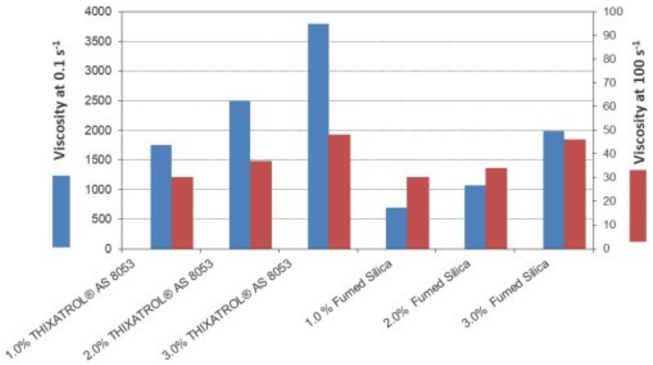

Figure 5: Viscosity comparison

A sealant's yield point is a direct gauge of its practical applicability. Measuring the yield point is a direct measure of how much force is needed to initiate movement and start the material flowing. If the concentration of rheological additive is too high, the compound can only be processed to a certain extent because of its high viscosity. Hence it is important to keep the concentration to optimum level in order to have a better extrudability.

Figure 6 shows that there is direct correlation between yield point and extrusion rate. The higher the yield point, the lower the extrudability.

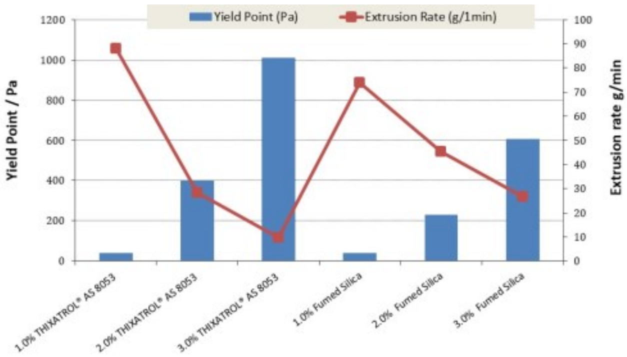

Figure 6: Yield value vs extrusion rate

As the loading level of both THIXATROL® AS 8053 and fumed silica increases, the thickening effect increases and the sealants extrudability drops as a result of the higher viscosity and higher yield point. At 1.0% loading both THIXATROL® AS 8053 and fumed silica showed a higher extrusion rate i.e. 88 and 74 g/min respectively which is not considered ideal from a practical point of view. An extrusion rate around 30-35 g/min is considered ideal for application. As the loading level of THIXATROL® AS 8053 was increased to 2.0%, an ideal extrusion rate of ca. 30 g/min is achieved while to achieve the similar extrusion rate 3.0% of fumed silica is required. Moreover, the yield point achieved with 3.0% fumed silica is higher than 2.0% THIXATROL® AS 8053 at almost equal extrusion rate which implies that one needs to apply 30% more force to extrude the same amount of material.

Structural recovery tests (ORO) provide the application -specific assessment of a sealant after it is applied. The most interesting part in this test is the step from structure decomposition at high shear $100 s^{-1}$ (interval 2) to small amplitude oscillation (interval 3) as shown in *Figure 7*. This is a direct simulation of the movement due to the pressing out of the cartridge and the behaviour directly after the force is removed.

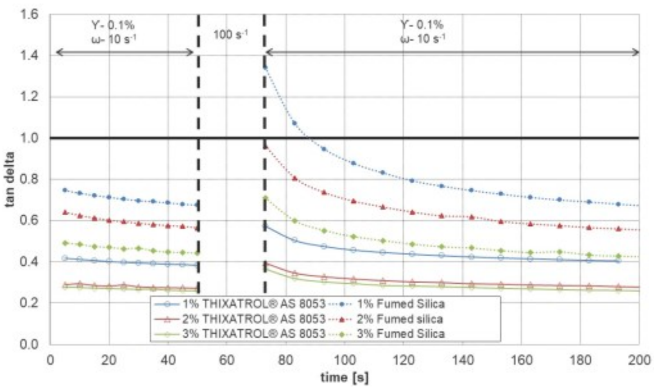

Figure 7: Structure recovery

Tan delta higher than one indicate the viscous modulus dominating over storage modulus which means the sample will start flowing.

Figure 7 compares the Tan delta of THIXATROL® AS 8053 and fumed silica at various sag behaviour measurement values. In addition to Tan delta, Flow point is another parameter related to sag stability of the material. Flow point (G'=G'') was extracted from the amplitude sweep and compared with the sag values in *Table 1*.

Sample	Tan Delta	Flow Point [Pa]	Sag Performance
1.0% THIXATROL® AS 8053	0.57	2342	No Sag
1.0% Fumed Silica	1.30	2000	100% Sag (complete run-down)
2.0% THIXATROL® AS 8053	0.40	3028	No Sag
2.0% Fumed Silica	0.96	2625	No Sag
3.0% THIXATROL® AS 8053	0.37	4728	No Sag
3.0% Fumed Silica	0.70	3199	No Sag

Table 1: Flow point versus sag stability

Fumed Silica with 1.0% loading showed higher Tan delta (1.30) and also the lowest flow point and hence no sag stability was observed, the sample completely run out of the channel (*Figure 8*). However at the similar loading level, THIXATROL® AS 8053 showed lower Tan Delta and higher flow point hence no sagging was observed with THIXATROL® AS 8053. Subsequent increase in the loading level of both THIXATROL® AS 8053 and fumed silica has improved the rheological as well as sagging stability.

Figure 8: Sag stability

Table 2 indicates the mechanical properties of cured THIXATROL® AS 8053 and fumed silica based SPUR sealants. THIXATROL® AS 8053 showed very similar mechanical performance in comparison to fumed silica.

Sample	Shore- A- Hardness	Elongation at break [%]	Elastic recovery [%]
2.0% THIXATROL® AS 8053	34	412	50
2.0% Fumed Silica	36	423	50
3.0% THIXATROL® AS 8053	35	406	50
3.0% Fumed Silica	40	391	50

Table 2: Mechanical properties

1.0% loading level of THIXATROL® AS 8053 and fumed silica was not considered for mechanical properties due to insufficient application properties. Increasing the loading level showed no significant effect on the mechanical properties in this formulation. Elongation at break, Shore A hardness were only marginally affected in the chosen formulation.

Clear SPUR sealant

To describe the influence of THIXATROL® AS 8053 and fumed silica on the properties of non-pigmented SPUR sealants, the loading level was varied. All samples were prepared with the speed mixer for up to 4 min at 2200 rpm. The temperature achieved for all samples were recorded between 65-70 ^{0}C.

Figure 9 shows the differences in the rheological performance (return flow curves) obtained with various loading level of THIXATROL® AS 8053 and fumed silica.

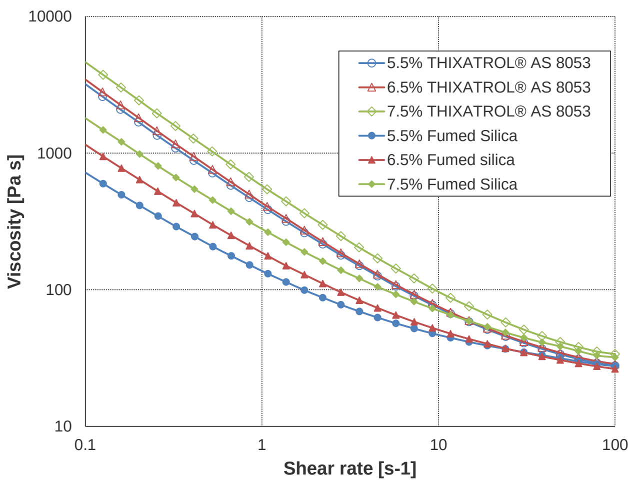

Figure 9: Rheology clear SPUR sealant

As the loading level of THIXATROL® AS 8053 and fumed silica increases, the viscosity increase considerably. This is clearly evident from the flow curves. Moreover, THIXATROL® AS 8053 showed more efficiency in comparison to fumed silica at all loading levels proving the higher rheological effectiveness of THIXATROL® AS 8053 against fumed silica in non-pigmented system as well.

Figure 10 compares the rheological efficiency at low (0.1 s^{-1}) and high shear rates (100s^{-1}) as extracted from the flow curves.

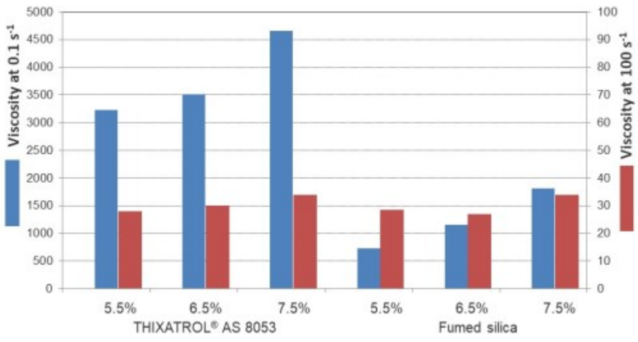

Figure 10: Viscosity comparison

Comparing the rheological efficiency at all loading levels THIXATROL® AS 8053 requires less amount to obtain the desired rheological behaviour, hence is more effective than fumed silica.

Figure 11 compares the yield Point of all samples with extrusion rate values. It is clear from the graph that yield point correlates well with the extrusion rate. Hence it is possible to use the yield point as a parameter to quantify extrusion rate.

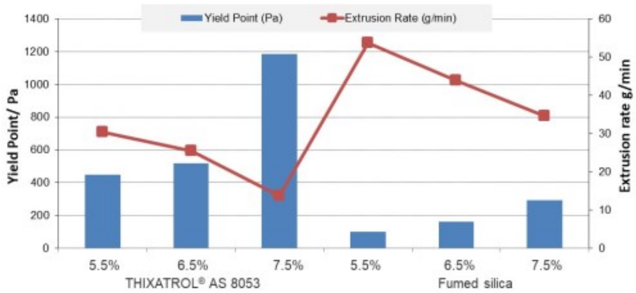

Figure 11: Yield value vs extrusion rate

Yield point increases as the concentration of both THIXATROL® AS 8053 and Fumed Silica was increased. THIXATROL® AS 8053 showed significantly higher yield point at all the loadings and consequently lower extrusion rate. With its higher efficiency in comparison to fumed silica THIXATROL® AS 8053 offers the formulators to adjust the extrusion rate accordingly but at significantly lower loading level than fumed silica.

Table 3 indicates all the application and mechanical properties of SPUR sealant with THIXATROL® 8053 and Fumed Silica.

Sample	Tan delta	Sag resistance	Shore-A-Hardness	Elongation at break [%]	Elastic recovery [%]
5.5% THIXATROL® AS 8053	0.4	No Sag	24	261	80
5.5% Fumed Silica	1.0	complete run down	31	267	80
6.5% THIXATROL® AS 8053	0.4	No Sag	26	258	80
6.5% Fumed Silica	0.6	No Sag	31	255	80
7.5% THIXATROL® AS 8053	0.4	No Sag	26	261	80
7.5% Fumed Silica	0.5	No Sag	32	255	80

Table 3: Mechanical properties

All samples with THIXATROL® AS 8053 showed lower Tan delta after the structure decomposition rheological tests. On the contrary, Fumed silica with 5.5% loading displayed higher tan delta after the structural decomposition depicting its fluid behaviour which was also evident from the application test as it completely run out of the vertical channel. As the loading level of Fumed silica increased to 6.5 and 7.5% lower Tan delta values were obtained representing its elastic response after the structural decomposition which also correlates well with the application behaviour.

A point of interest is that in all tested samples with various loadings, mechanical properties of relatively even quality are obtained.

Conclusion

THIXATROL® AS 8053 showed excellent rheological performance and outperformed fumed silica in both pigmented and non-pigmented SPUR based sealants. Influence on mechanical properties is marginal. It is possible to adjust the rheology of SPUR based sealant with THIXATROL® 8053 for the required application properties.

Transparency in non-pigmented systems needs to be checked.

Appendix

Formulation SPUR sealant pigmented

Component	Concentration [%]
Silyated polyurethane prepolymer (SPUR)	23.0
Plasticizer	18.0
Calcium Carbonate	54.3
Titanium Dioxide	0.7
Moisture Scavenger	0.3
Rheological Additive	Various*
Adhesion Promoter	0.6
Catalyst	0.1
Total:	100

* Concentration of fumed silica and THIXATROL® AS 8053 was varied at 1.0, 2.0 and 3.0% by weight.

Formulation SPUR sealant clear

Component	Concentration [%]
Silyated polyurethane prepolymer (SPUR)	58.0
Plasticizer	31.5
Moisture Scavenger	1.5
Rheological Additive	Various*
Adhesion Promoter	1.0
Catalyst	0.5
Total:	100.0

* Concentration of fumed silica and THIXATROL® AS 8053 was varied at 5.5, 6.5 and 7.5% by weight.

Experimental

Extrusion Rate were tested in accordance with ASTM C 1183 – 04. The test consists of extruding the sealant from cartridge equipped with a 3 mm orifice nozzle using a pneumatic gun at 40 psi, measuring the grams extruded in 1 min at ambient temperature.

For the sag tests, Aluminium U profile channel were filled with sealants up to 12cm and placed in vertical position. Sag was measured in millimetres from channel after one hour at ambient temperature.

Rheology-Flow curves, amplitude sweeps, Yield stress, and Oscillatory-Rotation-Oscillatory (O-R-O) structural recovery tests were performed on Anton-Paar MCR 300 series rheometer utilizing the 25 mm parallel plate (PP25) serrated geometry with a 1.0 mm gap at 25° C.

Shore A hardness was measured on cured samples after 7 days with Durometer. For the elastic recovery and elongation at break test, fully cured samples were casted in dumbbell shape with 3mm thickness. For the elastic recovery tests, dumbbell shape samples were stretched twice the length of the material for 24 h and left for 1 hour to recover. Recovery was then measured and converted into %age.

Literature

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