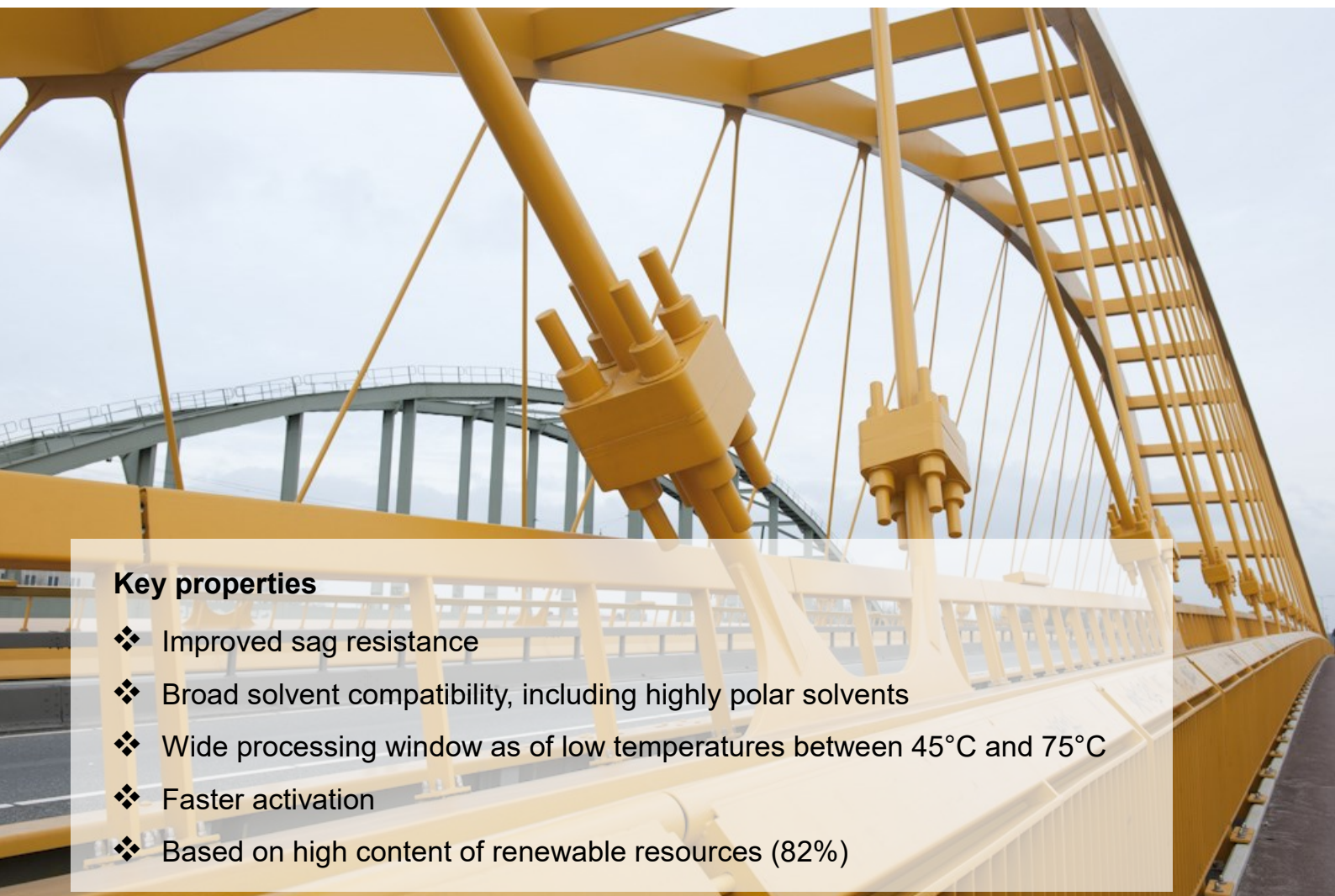


THIXATROL[®] PM 8058

Highly efficient organic thixotrope for non-aqueous industrial coating systems



Key properties

- ❖ Improved sag resistance
- ❖ Broad solvent compatibility, including highly polar solvents
- ❖ Wide processing window as of low temperatures between 45°C and 75°C
- ❖ Faster activation
- ❖ Based on high content of renewable resources (82%)

Introduction

Marine and protective coatings typically have very high requirements in terms of sag stability. The systems are typically spray applied in high layer thickness of around 1000 μm or even more in one pass. This high demand of the systems limits the selection of suitable rheology modifiers.

Organic thixotropes are providing the properties to fulfill this specific need. However, especially older classes of this technology have complex requirements in terms of incorporation and activation. Elevated temperatures, at a specified range, combined with high shear, for a defined period of time, in accordance with the system, which can be based on a manifold of binder chemistries and various solvent composition are required to achieve optimum performance (Figure 1).

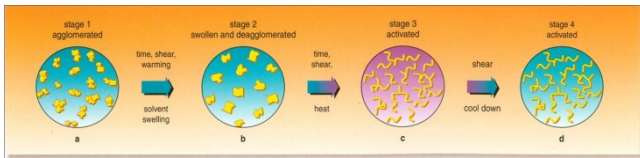


Figure 1: Activation organic thixotropes

As from a processing point of view, usually batch-by-batch technique is used. External heating or cooling equipment is often not available so that active temperature control during processing is either not possible or only available in a lab scale. The necessary energy to obtain and adjust the activation temperature is in such cases normally generated by friction during dispersion. The end temperature is dependent on parameters such as disperser speed and pigment/extender loading level and has a strong influence on the entire processing time. Therefore, rheology modifiers which are suitable for use over a wider temperature range show significant benefits.

Product class comparison

Organic rheological additives provide outstandingly high low shear viscosity in combination with a strong thixotropic flow character in non-aqueous systems. As visualized in Figure 2, in comparison to e.g. fumed silica, the low shear viscosity is much higher. With respect to practical relations this means that organic thixotropes are providing potentially the highest film build properties. On the other hand, fumed silica is providing higher viscosities at high shear rates. This effect often is adversely affecting the sprayability, especially the atomization during the application.

Organoclays, which are used to a lesser extent in this market segment, are more beneficial in terms of levelling combined with well-balanced anti sag performance.

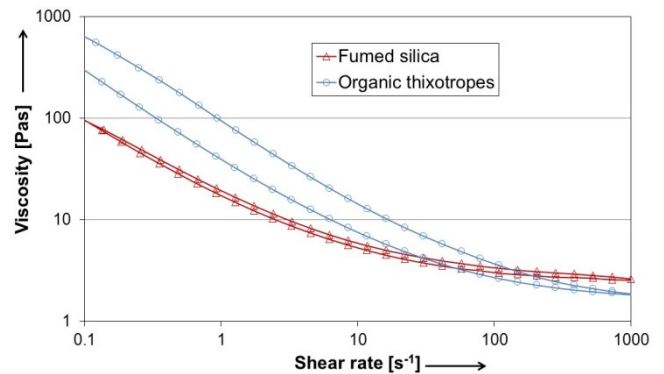


Figure 2: Comparison to other product classes

For activation, the diamide based rheology modifiers need to be exposed to mechanical forces applied alongside with specific temperature over a defined period. The mentioned activation temperature needs to be in line with the relevant solvency forces.

Product chemistry

After the activation, the rheological structure is being built up by the molecules orientation towards each other forming a fibrillar structure due to the nature of the amide functionality as described in Figure 3. The driving forces for this process are intermolecular interactions such as hydrogen bonding- and van-der-Waals forces.

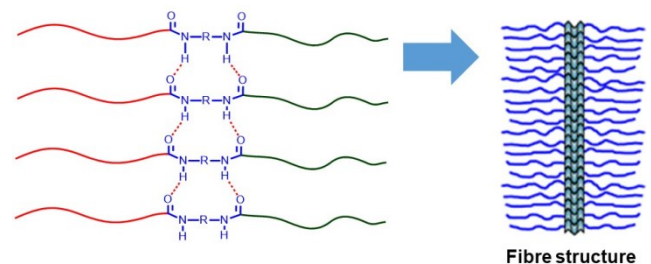


Figure 3: Thickening mechanism

Castor waxes, the oldest class of organic thixotropes, require a similar processing, however, are much more sensitive to changes in the systems solvency forces and processing temperature. Further they do not cover the entire solvent range. Additionally, they are having a much higher risk of certain side effects such as seeding and false body.

The latest class of diamide rheology modifiers out of the THIXATROL[®] PM[™] and THIXATROL[®] AS series allows proper activation over a very wide range of application temperatures in order to allow the formulator robust and predictable processing under real life conditions where temperature variations often occur, e.g. between summer and winter.

Further, these products are applicable at markedly lower temperatures than traditional grades based on similar chemistries. This is especially of great importance in solvent-free systems where the activation temperatures are normally high. The main advantage of such low temperature activation is energy and cost saving in the manufacturing process. The process can be carried out without external heating and is faster.

Renewable resources

Also, from a sustainability point of view these kind of rheology modifiers are very interesting. The new rheology modifiers out of the THIXATROL® product line are produced from renewable resources (>75% renewable content). In case of THIXATROL® PM 8058 the content of bio based materials is ca. 82%.

However, even with these major improvements by the described latest developments not all formulations could be covered. In systems of higher polarity, especially when formulating with various alcohols e.g. benzyl alcohol or butanol, a drop in efficiency in comparison with less polar systems is often the result. Alcohols are becoming more and more famous in the recent and are being formulated often in higher concentration to reduce the amount of aromatic solvent. In addition they are required to stabilize the system and to enhance the adhesion of the coating to the substrate further improvements.

The new THIXATROL® PM 8058 has been developed to provide solutions exactly for this kind of formulations utilizing higher amounts of highly polar solvents. Furthermore THIXATROL® PM 8058 acts significantly higher effective than the other rheology modifiers out of the THIXATROL® PM and THIXATROL® AS series.

These improvements with THIXATROL® PM 8058 become obvious in the following model study in which a high solid epoxy based primer of 83% solid content has been formulated with varying solvent ratios in comparison to commercially available reference organic thixotrope. In all case the concentration was chosen on a very low level of 0.5%. This was done to emphasize the effect of the organic thixotrope at lower viscosity levels and to visualize the effect of the various solvent compositions on the viscosity build.

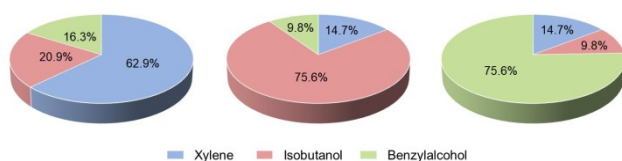


Figure 4: Solvent blend used in test system

As visible in Figure 4, in the standard formulation the solvent part is dominated by the Xylene portion. In case of both other versions either isobutanol or benzyl alcohol is used in the highest concentration. The activation process was performed by a tooth bladed cowles dissolver at 16 m/s. The activation temperature was in all cases adjusted to 66°C for 25 minutes.

A comparison of the rheological characteristics was done 24 hours after manufacturing. The data visualized in Figure 5 are displaying the performance of both organic thixotropes in the systems equipped with described various solvent compositions with a special focus on the low shear viscosity. The small graph in the upper right corner displays the viscosity variations of the alcohol rich samples to the sample in the Xylene dominated system.

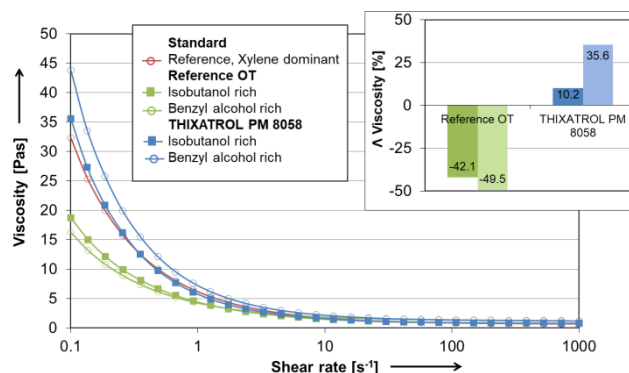


Figure 5: Influence solvent blends in viscosity build

It becomes obvious that the reference thickener is losing significantly in viscosity at low shear rates when formulated in both alcohol rich systems. A different picture can be observed when utilizing THIXATROL® PM 8058 in the same range of systems. Contrary to the market reference product, a viscosity rise in comparison to the reference product has been observed. This is especially the case when benzyl alcohol is dominant in the formulation. In case of the systems dominated by Isobutanol the difference was with a plus of about 10% somewhat lower.

The viscoelastic characteristics of the tested coating, generated in a structure recovery test, are also underlining the benefits of THIXATROL® PM 8058.

To simulate the post application behavior systems, the structure of the coating was disrupted in a rotational measuring step by high shear rates of 1000 s⁻¹. In a second, oscillatory step, the viscoelastic behavior of the coating after the removal of initially applied shear is visualized, to analyze the properties of the coating after impacting the substrate. This has been visualized in Figure 6.

In case the displayed damping factor, also called tan delta value, is above 1, the fluid behavior of the sample is dominant and indicates that practically no structure is the relevant, the system flows. If the damping factor acts below 1, the elastic behavior of the sample is dominant and indicates the presence of an internal structure. A strong structure indicates excellent sag stability and should consequently correlate with the results generated in practice.

However, in this testing also the time behavior plays a major role. The faster the structure recovers, indicated by the crossing a tan delta value of 1 coming from higher, the lower the time window for eventual sagging will be.

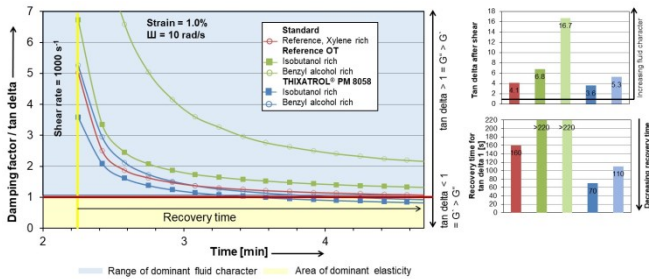


Figure 6: Influence solvent blends in viscoelasticity
 It can be seen that all samples display a dominance of the fluid character, indicated by tan delta values of above 1, directly after shear removal.

With the reference organic thixotrope a strong influence of the various solvent compositions on the viscoelasticity can be observed. Directly after shear removal, both alcohols are shifting the characteristics strongly towards a further fluid dominated behavior. With respect to the time scale, no redomination of the elasticity could be detected. The effects are strongest with a dominant concentration of Benzyl alcohol.

The influence of the various alcohols on the sample with THIXATROL® PM 8058 is different. The absolute damping factors are only slightly differing in comparison to the reference sample in the standard, Xylene dominated system. The main difference is the point in time when tan delta is crossing the line of a value of 1. This structure recovery time is in both alcohols dominated system somewhat shorter.

As a consequence, these data are making potentially higher layer thicknesses after application of the coatings with THIXATROL® PM 8058 than with the reference rheology modifier expectable.

In order to identify the optimum activation temperature window, the Xylene dominated system was chosen (Figure 7). In this study, individual samples were equipped either with THIXATROL® PM 8058 or a reference thickener activated at individual temperature.

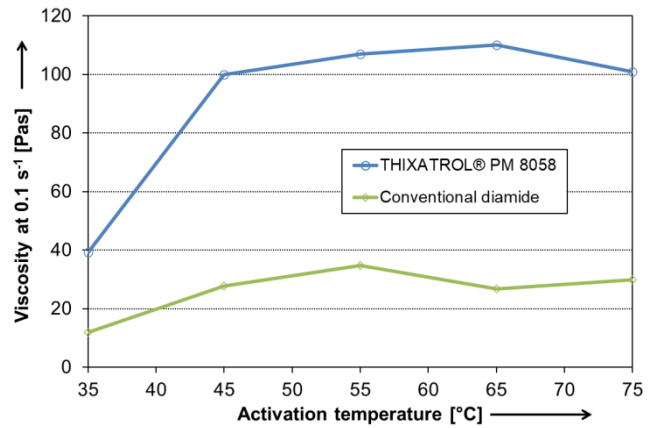


Figure 7: Viscosity build/activation temperature
 It can be observed that THIXATROL® PM 8058 provides significantly higher viscosities at a shear rate of 0.1 s^{-1} than the reference product over the entire tested temperature range from 35°C to 75°C . Stable and predictable viscosity build with THIXATROL® PM 8058 can be obtained in a temperature range between 45°C and 75°C .

The influence on the activation time at various temperature on viscosity build and sag control of THIXATROL® PM 8058 is shown in Figure 8.

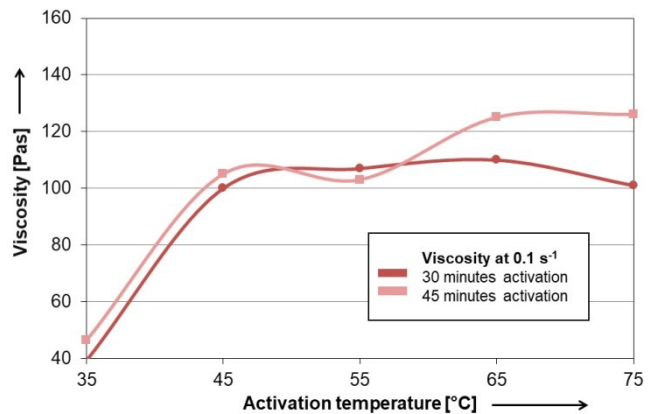


Figure 8: Influence of activation time
 The results are confirming the wide activation temperature window. Already with 30 minutes dispersing time at the lowest activation temperature of 35°C excellent viscosity build at low shear of 0.1 s^{-1} could be detected. The most stable values have been obtained in a temperature range as of 45°C up to until 75°C could be repeated. In this range, an activation time of 30 minutes is sufficient for proper viscosities. An extension of the activation period to 45 minutes is generating a further slight increase of the performance.

Also in terms of stability after 4 weeks of storage at elevated temperature of 50°C, THIXATROL® PM 8058 provides excellent results. In *Figure 9* it is shown that only when activated at 35°C noticeable changes of the viscosity or the sagging stability could be observed. As of an activation temperature of 45°C only slight variation of the viscosity could be detected. In case of the sagging control no changes at all were seen.

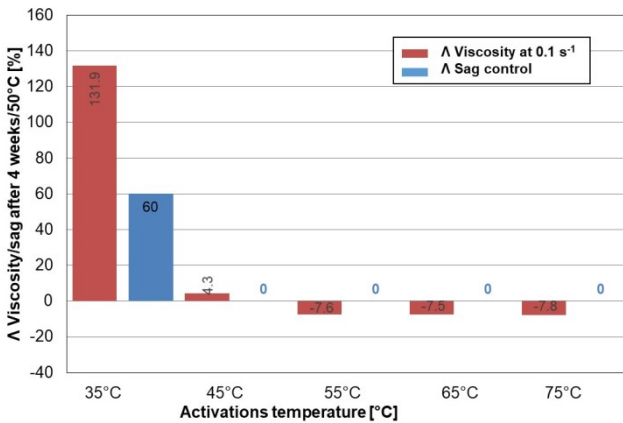


Figure 9: Storage stability

Comparing the achievable maximum applicable layer thicknesses after application by airless spray in *Figure 10*, is showing the THIXATROL® PM 8058 is clearly outperforming the reference rheology modifier clearly.

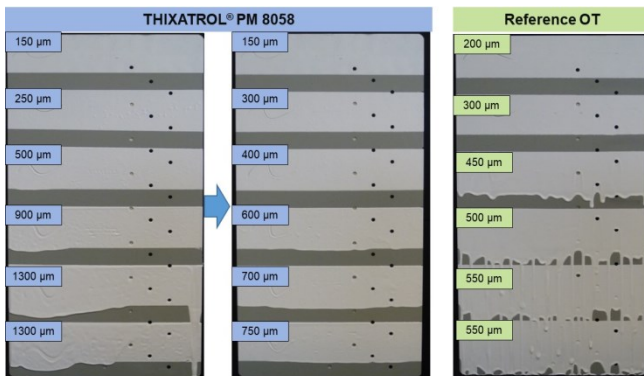


Figure 10: Sag stability after spray application

The market reference organic thixotrope allows a maximum layer thickness of 300 μm. THIXATROL® PM 8058 formulated at equal concentration and activation conditions guarantees sag control up to a range of 500 -700 μm.

The investigation of various processing conditions has shown that the activation of THIXATROL® PM 8058 can successfully be performed by various methods (*Figure 11*). The best results at all were achieved when adding THIXATROL® PM 8058 to the grind process from the begin of manufacturing, or alternatively, in a readily formulated, but unthickened coating system. In the last case, the applied shear can not be equally high as with the grind activation to avoid squirting out material from the vessel.

In this case, a dissolver tip speed of 12 m/s should be used. The relevant activation temperature needs to be controlled externally. In case of the direct addition, 16 m/s or even higher tip speeds can be used. Also, no artificial temperature control needs to be done as the friction is high enough to generate the required energy.

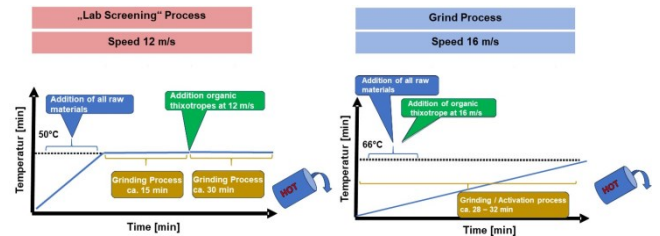
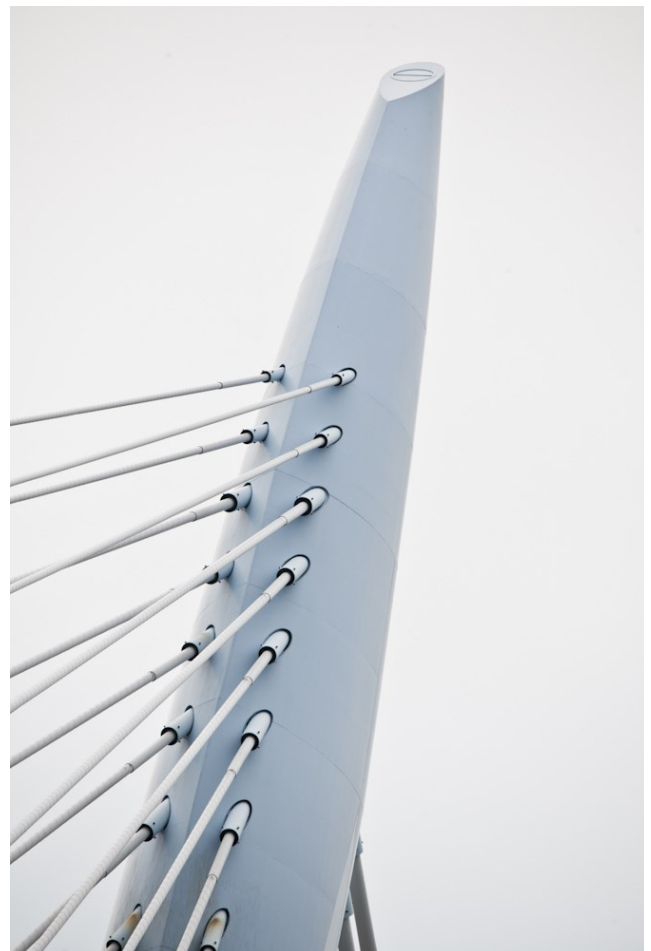


Figure 11: Recommended processing methods

Adding the organic thixotrope to the millbase has been mostly performed in factory based production. Post activation offers additional options, e.g. a time efficient lab screening .

Due to the low shear requirements of THIXATROL® PM 8058 for the activation, it might also possible to create pregels in certain solvents or solvent/resin blends which can then be furtherly used in coatings processing.



Conclusion

The discussed results and properties are showing that THIXATROL® PM 8058 is extending the series of outstandingly efficient organic thixotropes.

THIXATROL® PM 8058 utilizes the latest technology of highly sustainable diamide waxes being based on more ca. 82% renewable raw materials.

THIXATROL® PM 8058 can withstand higher amounts of various alcohols and other highly polar solvents and clearly outperforms other thickener classes. In comparison to other amide based rheology modifiers even higher viscosities and sagging stabilities can be achieved when compared under similar formulatory conditions.

THIXATROL® PM 8058 provides a wide activation temperature window for very robust manufacturing under real life conditions.

It furtherly acts storage stable when processed at lower temperature for limited dispersing times. Short activation periods as of 30 Minutes are providing viscosity stable coatings system. Also the potential activation temperature starts from low levels. As of 45° C a significant and stable viscosity build as was as excellent sagging stability was noticed. A further rise of the activation temperatures up to 75°C at equal dispersion period resulted in only small variations.

In comparison to commercially available reference organic thixotropes, THIXATROL® PM 8058 acts significantly higher in efficiency. This allows the formulator to significantly reduce the loading level whilst keeping viscosity and sag control.

Consequently, THIXATROL® PM 8058 is an ideal rheology modifier in the cost-effective and sustainable formulation of industrial, marine and protective coatings as well as other non-aqueous systems.

Test methods:

Rheology measurements

Determined using the Anton-Paar MCR 301 rheometer, equipped with PP 50 measuring geometry at a gap width of 1 mm, at a temperature of 23°C. In case of the oscillatory, amplitude sweep data shown an fixed angular frequency of 10 rad/s was pre-adjusted.

Conditions spray application

The coating has been applied airless by Grace XForce HD (Nozzle 419; 40° Spray angle; 0,19 mm diameter; 140-160 bar) in stripes (1-6 spray passes) like a wedge in order to obtain various layer thicknesses. Marking strips on the panel have been used to determine the maximum applicable layer thickness without sagging. Curing of the samples has been done in in vertical position.

Formulation:

Component A	
Difunctional bisphenol A/ epichlorohydrin - low mol weight	18.0
Hydrocarbon resin	7.2
Epoxy bisphenol A resin - low molecular weight	3.0
Xylene	2.5
Rheological Additive	0.5
Calciumcarbonate	46.7
Titandioxid	10.0
Solvents (in the mentioned concentration ratio)	12.0
Total:	100.0
Component B	
Hardener - Polyamide	30.7



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