

THIXATROL[®] PM 8054 & THIXATROL[®] PM 8056

Highly efficient organic thixotropes for protective and marine coating systems



Results on one view

- ❖ Provide wider process window compared to conventional organic thixotropes
- ❖ Allows high flexibility in production
- ❖ Higher efficiency in viscosity build and providing sag resistance

Introduction

Several industrial, maintenance and marine spray coatings 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 limit the selection of suitable rheology modifiers.

Organic thixotropes provide are typically used as they provide the properties to fulfill this specific need. However, this class of rheology modifiers has 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.

Various processing methods are used to produce coating systems. In case of the most commonly used batch-by-batch processing technique, the necessary energy to obtain and adjust the activation temperature is 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.

External heating or cooling equipment is often not available so that active temperature control during processing is either not possible or only available on a lab scale. In such cases, rheology modifiers which are suitable for use over a wider temperature range show significant benefits.

Post correction procedures currently play a minor role under real life production conditions as most commercially available organic thixotrope grades require higher shear forces for proper activation than usually available in the letdown stage.

Thickener class comparison

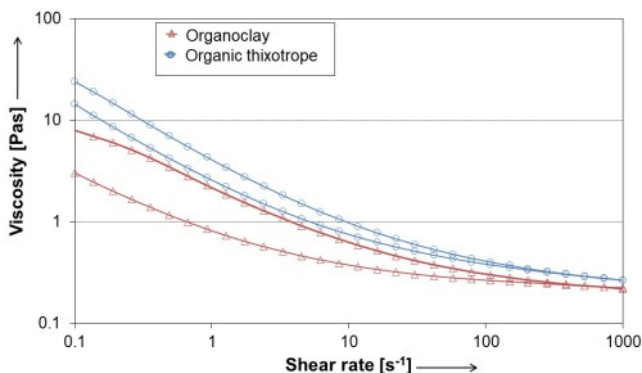


Figure 1: Comparison thickener classes

Organic rheological additives provide outstandingly high low shear viscosity in combination with a strong thixotropic flow character in non-aqueous systems. As described in *figure 1*, in comparison to organoclays, 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. Organoclays are more beneficial in terms of levelling combined with well-balanced anti sag performance.

Chemical background

The first generation of organic thixotropes is based on ricinus oil derivatives. These products are excellent organic thixotropes or “rheological additives”, providing good performance when incorporated and activated in a specific manner.

Pure castor waxes are typically limited for use in aliphatic solvent based- and solvent-free systems. To generate a rheology modifier suitable for solvents of higher polarity, such as aromatics, it is necessary to shift the melting point of the castor wax upwards by the addition of higher melting point components. This broadens the range of compatible solvents, as well as raises the general activation temperature. However, none of the ricinus oil derivatives are suitable for systems formulated with high polarity solvents, such as oxygenated solvents e.g. ketones and esters.

The second class of rheology modifiers for the mentioned spectrum of applications is based on diamide waxes, which consists of a different general molecular structure, as described in *Figure 2*.

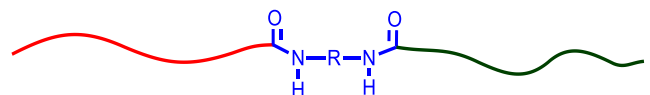


Figure 2: Molecular structure diamide

Once properly activated, the molecules orientate themselves towards each other forming a fibrillar structure (*Figure 3*) due to the nature of the amide functionality. The driving forces for this formation are intermolecular interactions such as hydrogen bonding- and van-der-Waals forces.

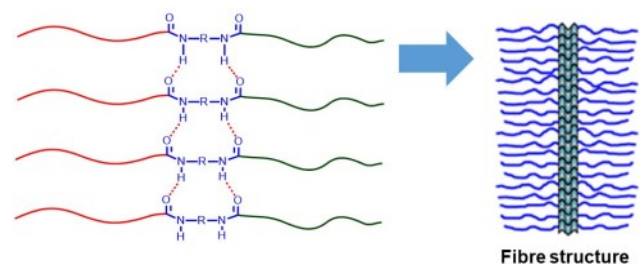


Figure 3: Molecular orientation diamide

The visualized intermolecular interactions become relevant after activation of the additive, which is similar to castor waxes. Mechanical forces applied along with specific temperature, which have to be in line with the relevant solvency forces, over a defined period of time need to be applied to the system for proper activation. The rheological structure is then built up by a redistribution of crystalline phases of the rheological additive accompanied by cooperative self-assembly of certain molecules producing the described microstructure.

The melting point of the diamide waxes is generally higher than of castor oil derivatives. This enables this class of rheology modifiers to be used in the largest range of solvent polarities. However, the potential activation temperatures are usually higher than with ricinus oil derivatives.

This new class of diamide rheology modifiers has been developed to be provide proper activation 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 cost saving in the manufacturing process. The process can be carried out without external heating and is faster.

Another major focus of the development was, to generate organic thixotropes which provide stable activation over a wider temperature range. This results in increased process robustness under real life conditions where temperature variations often occurring, e.g. as differences between summer and winter.

THIXATROL® PM 8054 and THIXATROL® PM 8056 are newly developed diamide based rheology modifiers dedicated predominately for non-aqueous industrial, marine and protective coatings. For all organic thixotropes, THIXATROL® PM 8054 and THIXATROL® PM 8056 need specific process temperatures and mixing times in order to achieve their maximum performance. However, in comparison to currently available organic thixotrope grades, both products are less temperature sensitive and provide a broader solvent compatibility.

Chemical and physical data

	THIXATROL® PM 8054	THIXATROL® PM 8056
Appearance	Fine off white powder	
Composition	Proprietary organic	
Buld density [g/ml]	0.25	0.25
Melting point [°C]	120-130	120-130
Main particle size [µm]	max. 6	max. 6

Key benefits

- Designed for low temperature activation, processing and packaging
- 100 % active seed-resistant powder
- Highly efficient already at low loadings
- Provide a wider process window compared to market references
- More effective in comparison to market reference
- Cost efficient due to complexity reduction in production/processing
- Excellent storage stability of the readily formulated system
- Provides outstanding viscosity control, thixotropy and excellent very thick layer sag resistance
- Have a broad compatibility to various solvents
- Show no intercoat adhesion problems when overcoated with a topcoat.
- Have no negative impact on corrosion properties
- Excellent stability of finished paint systems

Industry & marine coatings

The following study identifies the ideal activation conditions to achieve optimum performance and was carried out in an epoxy based marine primer. As visualized in *figure 4*, THIXATROL® PM 8054 and THIXATROL® PM 8056 provide excellent low shear viscosity build over a wide range of activation

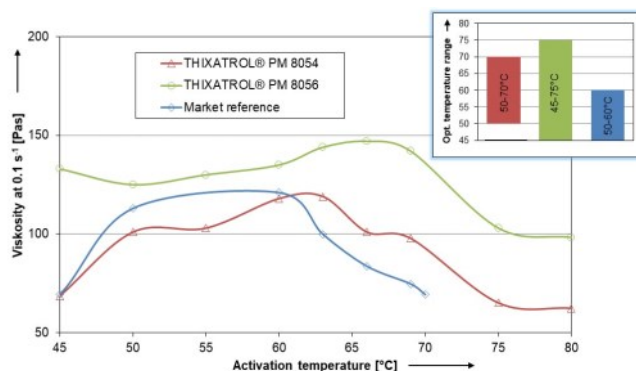


Figure 4: Activation temperature window

Based on these results it can be concluded that THIXATROL® PM 8054 achieves the best results at a temperature range from 50°C to 70°C. THIXATROL® PM 8056 has a much wider temperature window of 45°C to 70°C for activation. In comparison to the application temperature range of 50°C and 60°C for the market reference product, it is clearly indicated that the use of both grades results in markedly more flexibility.

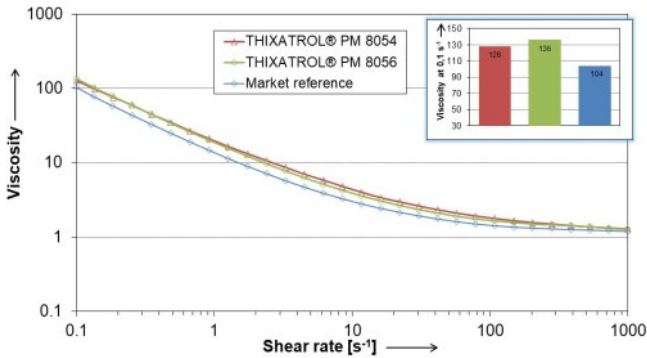


Figure 5: Rheology curves

The rheological activity of THIXATROL® PM 8054 and THIXATROL® PM 8056 after activation at the same activation temperature of 63°C, display only minor differences when compared to each other (figure 5). However, the resulting viscosities at low and mid shear rates are markedly higher than with the market reference. Furthermore, the new organic thixotropes provide clear advantages in terms of the maximum applicable layer thickness after spray application. As displayed in figure 6, layer thickness of up to 1200 µm can be applied in one spray pass.

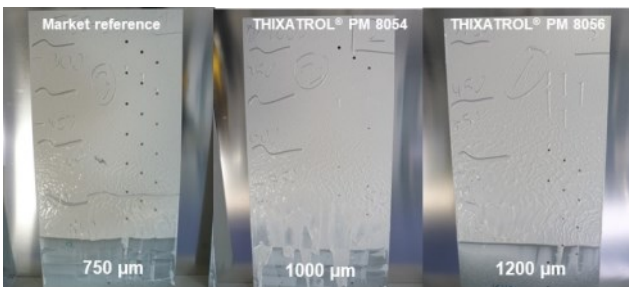


Figure 6: Sag stability

In figure 7, the viscoelastic characteristics of the tested coating, generated in a structure recovery test, are shown. This test simulates the behavior of the system after application using high shear forces, which are relevant during spray application.

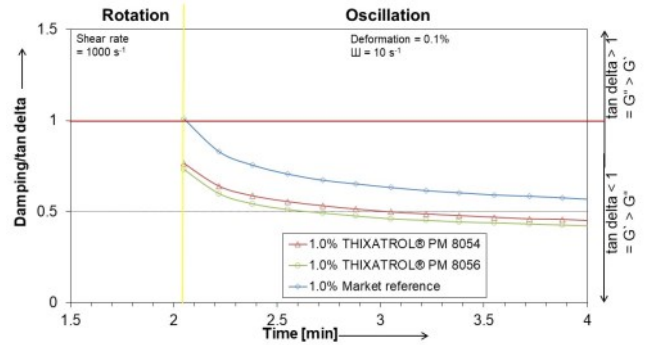


Figure 7: Viscoelastic data

In the first rotational measuring step the structure of the coating was disrupted by high shear forces. In a second oscillatory step, the behavior after the removal of shear, was analyzed. This step visualizes the properties of the coating after impact on the substrate. When the displayed damping factor (tan delta) is below 1, the elastic behavior of the sample is dominant. The smaller this value is, the stronger the samples structure will be. A strong structure indicates excellent sag stability and should consequently correlate with the results generated in practice.

As it can be seen in figure 7, all samples display strong elastic behavior soon after removal of shear. However, both samples with THIXATROL® PM 8054 and THIXATROL® PM 8056 rebuild their structure noticeably quicker than the material formulated with the market reference. This is shown by the first measuring points of lower tan delta values, which indicate a stronger structure. With the market reference product the structure recovery occurs with a slight time delay. Comparing samples with PM grades, slight benefits for THIXATROL® PM 8056 can be noticed. These results strongly correlate with the sag stability data shown previously in figure 6.

Intercoat adhesion

As the tested types of coating is typically applied in multi-layer build-up, intercoat adhesion is of significant importance. Therefore, the influence of rheological additives on the intercoat adhesion is usually tested. In this assessment the epoxy primers equipped with the individual organic thixotropes has been applied on a metal substrate and cured. After re-coating a cross-cut test with a connected tape-peel test based on DIN EN ISO 2409 was performed and the intercoat adhesion judged.

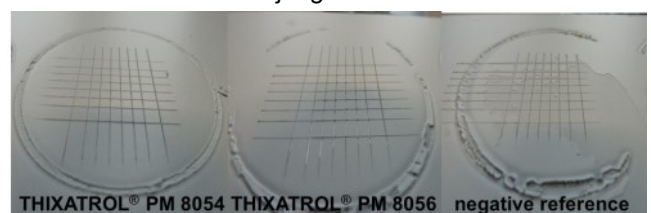


Figure 8: Intercoat adhesion testing

In accordance with our experience high temperature and humidity during curing of the primer could be critical for the intercoat adhesion. Therefore, a temperature of 40°C together with 100% of humidity was chosen.

As it can be seen in *figure 8*, neither the new products showed indications of an intercoat adhesion failure. This was only the case with the negative reference which shows a total loss of the topcoat adhesion after the tape-peel test.

Process variations

As also the method of incorporation has got an influence on the resulting viscosity of the final system, two different activation procedures were performed. In the first case, the additive portion was added before pigments and filler and dispersed with the entire system until the activation temperature of 66°C was reached. Here, the energy to obtain the relevant temperature was generated only by shear. By this method two batches were manufactured using two different tip speed values.

In another process, the THIXATROL® PM 8054 quantity was post-added to an already dispersed system. Due to the markedly lower system viscosity, the dispersion tip speed needed to be markedly lower compared to the first case. This resulted also in lower friction so that an external heat source was necessary to control the temperature during the activation process.

Point of addition	Tip speed	Temperature control	Process	Viscosity@ 0.1 s ⁻¹ [Pas]	Sag as of (Blade) [µm]
millbase	21 m/s	No, up to 66°C		141	1200
millbase	18 m/s	No, up to 66°C		157	1200
post-add	12 m/s	Yes, 63°C		128	1200
Post-add	12 m/s	Yes, 66°C		131	1000

● Addition of diamide

Table 2: Various process conditions

Comparing the results in terms of viscosity at low shear rates and sag stability of material generated by both methods, only rather slight differences could be noticed. The highest viscosity values and the highest achievable layer thicknesses are to be obtained after millbase addition of the organic thixotrope. However, the results of the post-added activated THIXATROL® PM 8054 are also surprisingly good. Viscosity values and maximum applicable layer thicknesses are only slightly lower than with millbase addition.

Consequently, this method is suitable to be used for screening of organic thixotropes or, if the equipment allows, in production scale. In such the activation of the organic thixotrope and the pigment dispersion could be done separately.

Conclusion

- THIXATROL® PM technology provides high rheological efficiency with thixotropic flow
- The application window is enlarged, low temperature activation is possible
- Both grades are more effective than conventional diamide

Appendix

Epoxy test formulation

Component A		
Raw material	Function	Concentration [%]
Epikote 1001x75	Resin (Momentive)	30.0
Methylisobutylketone	Solvent	2.71
Xylene	Solvent	5.00
n-Butanol	Solvent	1.57
Rheological additive	Additive	1.00
Disperse for 5 minutes at 18 m/s		
Antiterra U	Wetting agent (Byk Chemie)	0.16
Kronos 2310	Titanium dioxide (Kronos)	5.47
Blancfixe micro	Filler (Sachtleben Chemie)	28.00
Talkum IT extra	Filler (Elementis Talc)	8.9
Dispersing without temperature control at 18m/s - milling process was stopped at the time where the milling base reached 66°C		
Reduce the temperature of the mill material under stirring to <30°C before adding the let-down.		
Epikote 1001x75	Resin (Momentive)	7.92
Methylisobutylketone	Solvent	2.71
Xylene	Solvent	5.00
n-Butanol	Solvent	1.56
		100.00
Stirred in at 12 m/s for 5 min.		
Component B		
Epicure 3115 x 70	Hardener (Momentive)	14.00
Slow stirring for 5 minutes		

Graphics & Test methods

Figure 1: Schematic representation of the flow characteristics provided by the various classes of rheology modifiers.

Figure 2: Partial chemical formula of a diamide wax

Figure 3: Schematic representation of the acting mechanism of a diamide based rheology modifier after correct activation.

Figure 4: Visualization of the optimum activation temperature range of THIXATROL® PM 8054 and THIXATROL® PM 8056 in solvent based protective and marine coatings; displayed are the low shear viscosity at 0.1s⁻¹ and the maximum applicable layer thicknesses up on blade application in dependence from the relevant activation temperature.

Figure 5: Rheology curves of a solvent based protective and marine coating equipped with 1% of the relevant rheology modifier activated at 63°C; 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; 23°C; Low shear viscosity data extracted and plotted in the upper right corner of the rheogram.

Figure 6: Test cards displaying the maximum applicable layer thickness of a solvent based protective and marine coating equipped with 1% of the relevant rheology modifier activated at 63°C up on spray application (Sata Jet 4000 B RP with 1.4 mm spray nozzle).

Figure 7: Viscoelastic data solvent based protective and marine coating equipped with 1% of the relevant rheology modifier activated at 63°C; 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; tested as a 3 step test splitted in oscillation/rotation/oscillation.

Figure 8: Intercoat adhesion testing solvent based protective and marine coating equipped with 1% of the relevant rheology modifier activated at 63°C; the epoxy primer applied on a metal substrate at layer thicknesses from 170-190 µm and cured at different conditions in terms of humidity and temperature. Afterwards a second layer was applied. After curing a cross-cut test with a connected tape-peel test was performed and the intercoat adhesion judged.

Table 2: Table shows low shear viscosity data at 0.1 s^{-1} and the maximum applicable layer thickness by blade application solvent based protective and marine coating equipped with 1% of THIXATROL® PM 8054 in accordance to the relevantly described method of incorporation and activation.



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