



RADIANTLY COLOURFUL

Solvent-free wetting and dispersing additive for the wood and furniture industry. By Selma Örü, Holger Wach, Marcel Krohnen, Byk-Chemie.

Novel wetting and dispersing additive sets high standards for the dispersion and stabilisation of inorganic and organic pigments in solvent-free radiation-curable wood and furniture coatings. A study across the pigment range has shown the polymer to increase gloss and improve colour strength in the formulated pigment dispersion.

Solvent-free, radiation-curable coating systems have been used in industry since the 1970s, however they are gaining considerable importance globally as an alternative to conventional solvent-borne coating systems for the wood and furniture industry. Initially, radiation-curable coating systems were primarily formulated as high-gloss clear coat systems, but for some time now their development has tended towards deep-matt or pigmented formulations. In particular, the relatively high processing and application viscosity, the lack of volatile organic solvents, the sometimes challenging application methods, and not least the virtually immediate curing

of the solvent-free radiation-curable coating systems present exceptional demands on the choice of suitable wetting and dispersing additives. In addition to broad compatibility with all commonly used monomers and oligomers as well as the user requirement for supplies to be solvent-free, the employed wetting and dispersing additives must fulfil the highest demands when it comes to dispersing and stabilising the most varied pigments. The following application technology study presents a novel wetting and dispersing additive that sets the highest industrial pigment stabilisation standards for solvent-free, radiation-curable coating systems.

SYNTHETIC POLYMER

Controlled polymerisation technologies (CPT) such as group transfer polymerisation (GTP) [1] and radical polymerisation technologies such as atom transfer radical polymerisation (ATRP) [2], nitroxyl-mediated polymerisation (NMP) [3] and reversible addition-fragmentation chain transfer process (RAFT) [4] are

industrial synthesis pathways to manufacture block copolymers. Block copolymers produced in this way are primarily used as wetting and dispersing additives [5-7]. The use of these techniques to manufacture the main chain of graft copolymers for a grafting approach is not necessarily obvious. Perfectly adjusting the main polymer chain using various (meth)acrylates and other unsaturated monomers can influence the position of pigment-affinic and other reactive groups for bonding to side chains along the main chain so as to control the density of the side chains and the homogeneity of their distribution. The molecular weight and polarity of the side chains have a considerable influence on the compatibility and product viscosity of the additive. Increased uniformity of the polymer chains is a positive impact. The quantity of undesirable polymeric by-products that contain neither side chains nor pigment-affinic groups is reduced. As a result of its more precise structure, these comb copolymers are excellent wetting and dispersing additives for numerous applications.

RESULTS AT A GLANCE

- A novel wetting and dispersing additive was developed to stabilise pigments in solvent-free radiation-curable wood and furniture coatings.
- Both inorganic and organic pigments and carbon blacks can be dispersed and stabilised using this wetting and dispersing additive to give high-quality results.
- In addition to the pronounced reduction in viscosity, the improved development in colour strength throughout the dispersion process and the high-quality colouristic properties also comply with the highest technical requirements.
- The solvent-free delivery form of the additive does not add any volatile components to the formulation of radiation-curable coating systems.

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This synthetic approach was used to develop a solvent-free liquid comb copolymer (see *Figure 1*) that has broad compatibility with all commonly used monomers and oligomers, and outstanding pigment stabilisation results for solvent-free radiation-curable coating systems such as:

- > Highly effective reduction in pigment concentrate viscosity
- > Improved colour strength development throughout the dispersion process
- > Highest quality colouristic properties (es-

pecially transparency, colour strength and gloss development), and

- > Long-lasting storage stability of the finished pigment concentrates and the formulated coating systems.

The novel high molecular weight wetting and dispersing additive based on CPT (polymer 2) was tested within an extensive application technology study and compared with both a low molecular weight block copolymer (polymer 1), which was specifically suited to dispersing inorganic pigments, and three differ-

ent high molecular weight, highly branched polymers (polymer 3, 4, 5) that proved very effective at stabilising organic pigments and carbon blacks. *Table 1* compares these five tested polymers.

EFFECTIVE REDUCTION IN VISCOSITY AT LOW DOSAGE

In the first stage of the study, multiple pigment concentrates were prepared using polymers 1 to 5. The study covered the en- ➤

Figure 1: Compatibility study of the novel wetting and dispersing additive (10 parts) in 90 parts dipropylene glycol diacrylate (DPGDA), hexanediol diacrylate (HDDA), isobornyl acrylate (IBOA), trimethylolpropane triacrylate (TMPTA) and tripropylene glycol diacrylate (TPGDA).

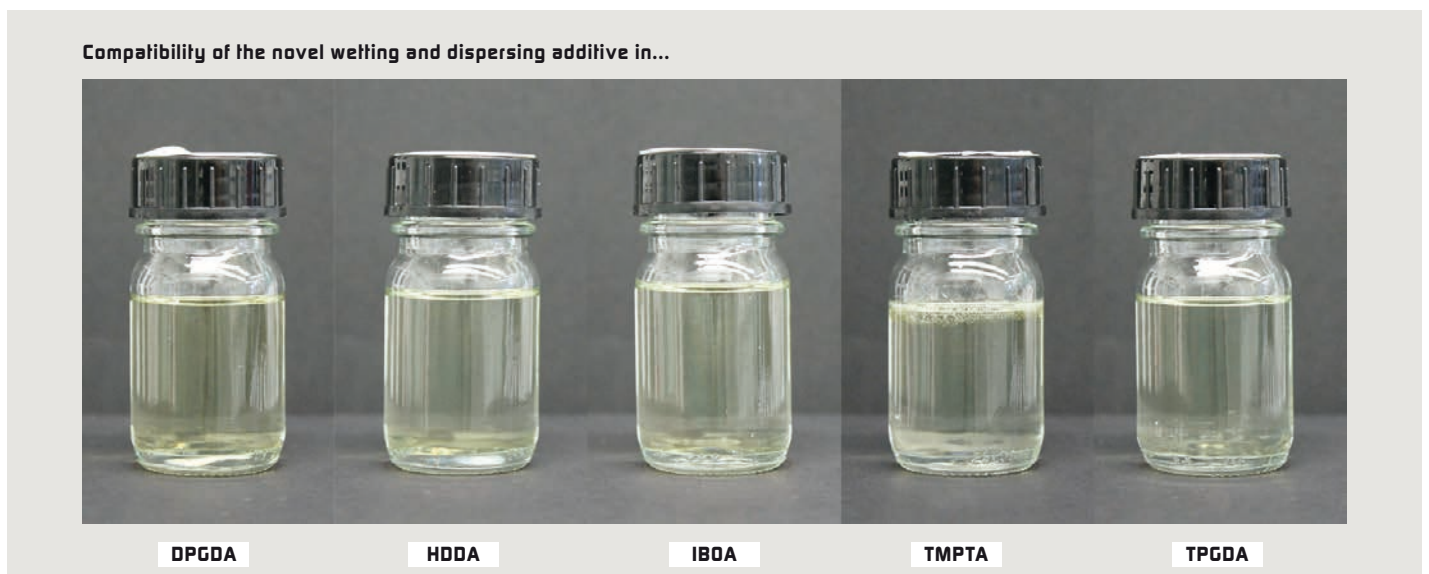


Figure 2: Pigment concentrate viscosity as a function of the polymer dosage used to disperse 75 % TiO₂ (P. W. 6) in dipropylene glycol diacrylate (DPGDA).

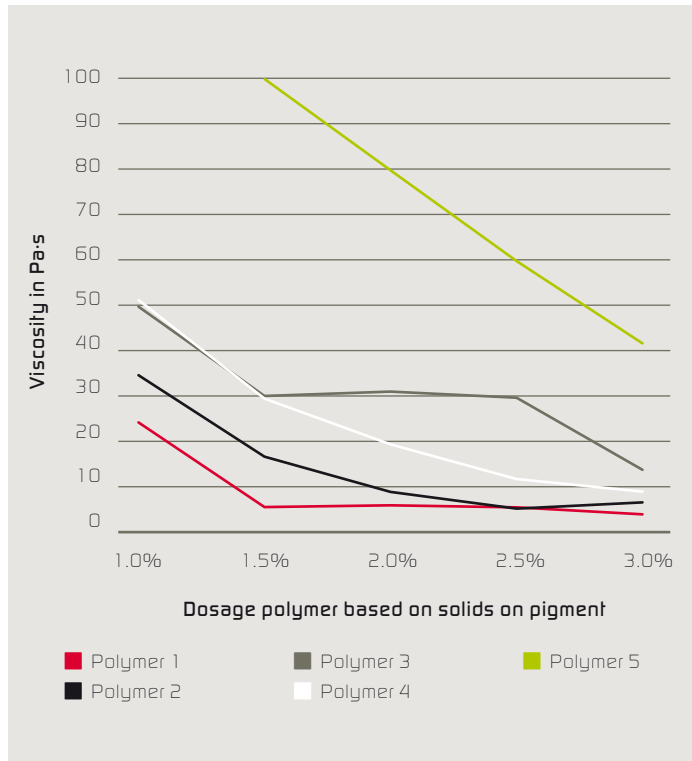


Figure 3: Pigment concentrate viscosity as a function of the polymer dosage used to disperse 35 % diketopyrrolopyrrole pigment (P. R. 254) in dipropylene glycol diacrylate (DPGDA).

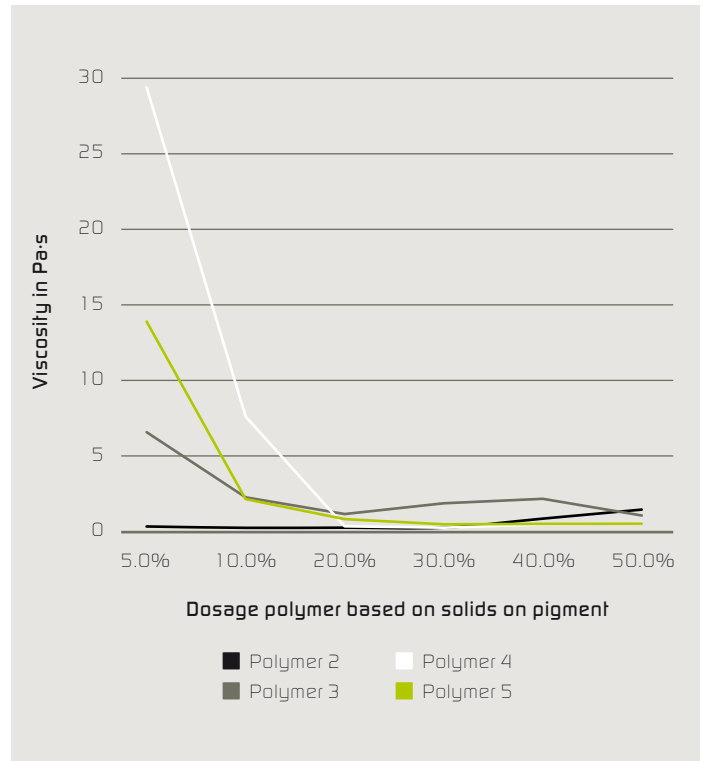


Figure 4: Pigment concentrate viscosity as a function of the polymer dosage used to disperse 75 % TiO₂ (P. W. 6) in dipropylene glycol diacrylate (before and after storage for four weeks at room temperature).

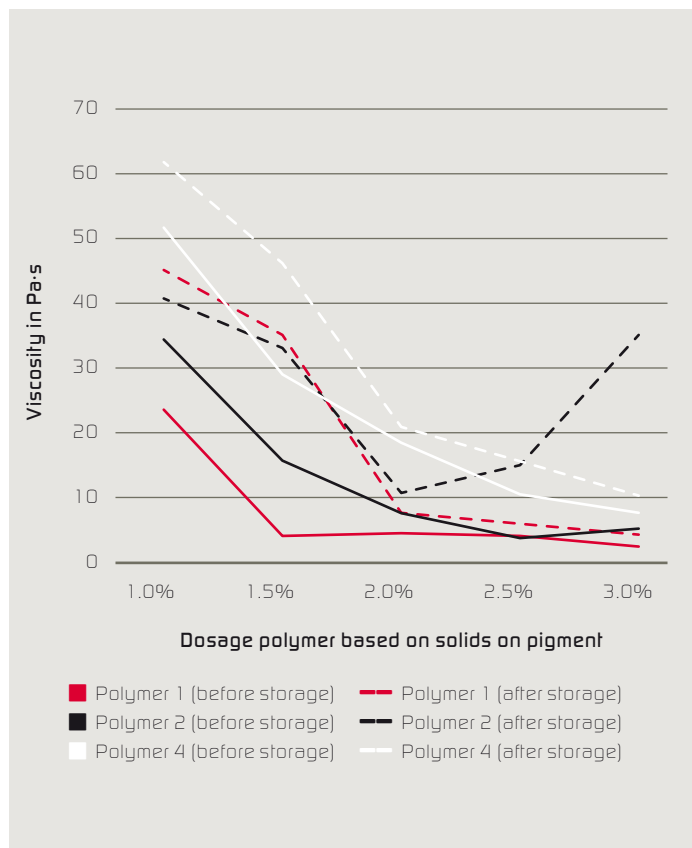
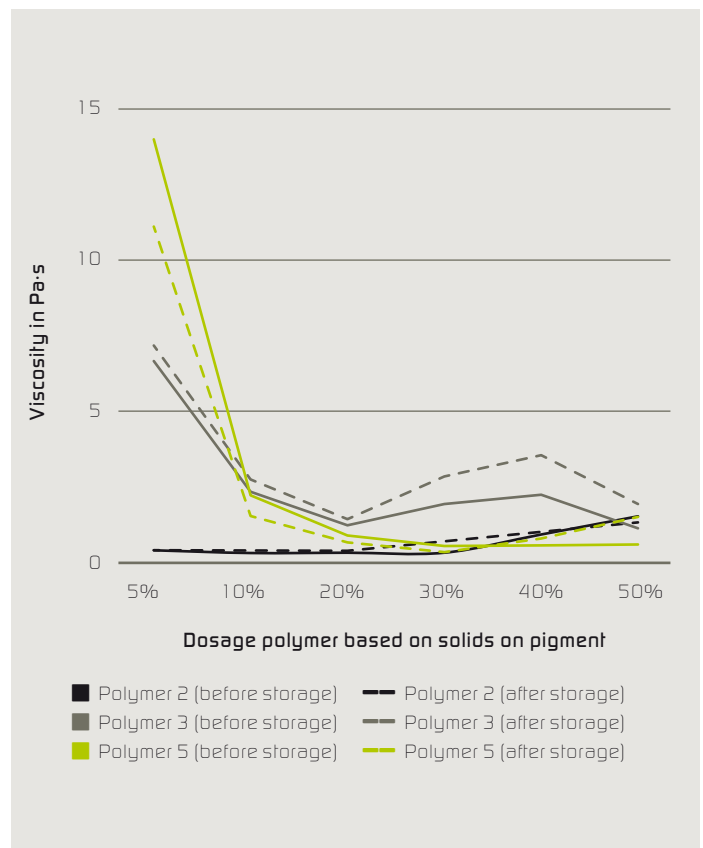


Figure 5: Pigment concentrate viscosity as a function of the polymer dosage used to disperse 35 % diketopyrrolopyrrole pigment (P. R. 254) in dipropylene glycol diacrylate (before and after storage for four weeks at room temperature).



◉ titre colouristic spectrum by using both inorganic and organic pigments as well as carbon blacks. Various dispersion units were used to disperse pigments and produce the pigment concentrates, and the discussed results were all achieved using a mixer that operates with a dispersion principle aligned with the requirements of a dual asymmetric centrifuge. The resulting pigment concentrates were rheologically measured using a rheometer (shear rate $1s^{-1}$, 25 °C), and compared. Figures 2 and 3 illustrate the results for

the dispersion of a titanium dioxide and a diketopyrrolopyrrole pigment, dispersed in dipropylene glycol diacrylate as a continuous phase, and using various quantities of polymer based on the pigment content. In the case of the titanium dioxide pigment, polymer 1 (specifically for inorganic pigments) displayed pronounced viscosity reduction even at a very low polymer dosage of 1.5 % based on the pigment content. Using this as a benchmark, only the novel wetting and dispersing additive (polymer 2) displayed a com-

parably good viscosity reduction (however, with a higher polymer dosage). Further trials using organic pigments and carbon blacks supported the broad application of polymer 2. Consequently, even when using very low dosages of 5–10 % polymer based on pigment content, polymer 2 achieved an unparalleled viscosity reduction compared with polymer 3, 4, and 5 (see Figure 3). As a result of its specific chemical structure, polymer 1 proved to be unsuitable in terms of dispersing the diketopyrrolopyrrole pigment.

LONG-TERM STORAGE STABILITY – A MUST FOR PIGMENT DISPERSIONS

The rheological measurements were repeated for all formulated pigment concentrates following storage at room temperature for four weeks, to make further statements on the long-term storage stability.

For greater clarity, Figure 4 only shows the results for polymers 1, 2 and 4 for the titanium dioxide pigment. Polymer 3 and polymer 5 displayed insufficient viscosity reduction as well as a lack of long-term storage stability, which is highlighted by a substantial increase in viscosity over the storage period. In accordance with Figure 4, we can now discuss the results for polymers 1, 2 and 4 more specifically. For all three of the presented polymers, a polymer dosage of 2.0 % based on the pigment content gave the best compromise between applied dosage, viscosity reduction, and storage stability. Nonetheless, in comparison with specialised polymer 1, the novel wetting and dispersing additive (polymer 2) is significantly more impressive than polymer 4.

For the organic diketopyrrolopyrrole pigment (Figure 5), the considerably improved perfor-



Figure 6: Gloss development (at 20° and 60° angle) for the dispersion of 75 % titanium dioxide (P. W. 6) in dipropylene glycol diacrylate, and application in a solvent-borne radiation-curable coating system based on a polyester/epoxy acrylate.

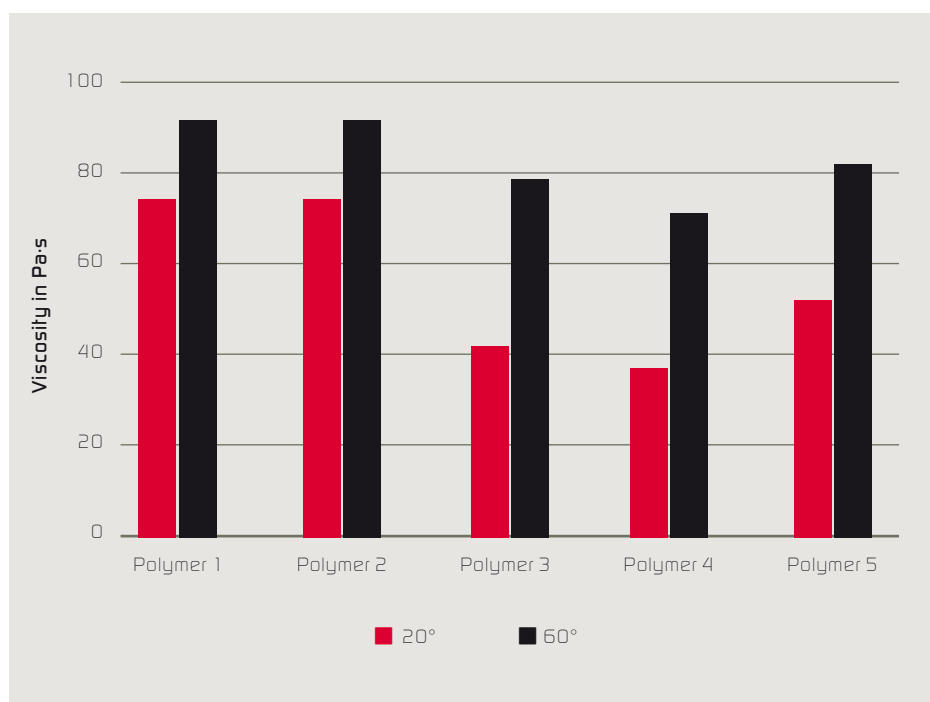


Table 1: Comparison of tested wetting and dispersing additives.

Polymer	Chemical basis	Developed for		Amine value [mg KOH/g]	Acid value [mg KOH/g]	Supplied as
		Solvent-borne systems	Solvent-free systems			
1	Block copolymer based on phosphoric acid ester, low molecular weight	x	x	-	129	100 %, liquid
2	Comb copolymer based on CPT, high molecular weight	x	x	18	8	100 %, liquid
3	Highly branched polymer, high molecular weight	x	x	48	-	100 %, liquid
4	Highly branched polymer, high molecular weight	x		48	36	100 %, liquid
5	Highly branched polymer, high molecular weight	x	x	41	27	100 %, liquid

▶ mance of polymer 2 is significant in terms of viscosity reduction and storage stability. For each selected polymer dosage, polymer 2 not only displays excellent viscosity reduction, it also displays a marked viscosity stability over the four-week storage period at room temperature. Polymers 3 and 5 achieve similar to virtually comparable application results but from a dosage of 20 % or 30 % respectively based on the pigment content. Polymer 4 failed to impress during the storage trials.

NOVEL POLYMER BOOSTS GLOSS AND PIGMENT STABILISATION

In addition to the rheological properties, the colouristic results also play a major role when selecting a wetting and dispersing additive. The pigment concentrations were therefore not only measured rheologically and investigated in terms of storage stability, they were also let down in various solvent-free radiation-curable coating systems and tested for

their colouristic results. *Figures 6 and 7* present two application examples that illustrate the broad range of tested pigments.

Figure 6 compares the gloss development of the different polymers in terms of stabilising the titanium dioxide pigment. The prepared pigment concentrates were let down in a solvent-free radiation-curable coating system based on polyester acrylate/epoxy acrylate, applied, cured using UV radiation and measured at 20° and 60° angle with regard to gloss using a micro-gloss gloss meter. In addition to the highly effective viscosity reduction (see *Figure 2*), polymer 2 also achieves exceptional gloss development at a comparably high level to the inorganic pigment-specialised polymer 1. Especially at a high-gloss angle of 20°, polymer 2 reveals significantly better results than polymers 3, 4, and 5, which were tested for comparison.

A further application technology test investigated the impact of different polymers on stabilising an opaque iron oxide pigment, especially in terms of the impact on the colour shade. The prepared pigment concentrates were again let down in a solvent-free radiation-curable coating system based on polyester acrylate/epoxy acrylate, applied, cured using UV radiation and the colour measured in the $L^*a^*b^*$ colour space. The absolute measurements for the a^* and b^* axes of all the tested polymers were compared with the measurements of polymer 2 as a reference and presented in a coordinate system in accordance with *Figure 7*. It appears that all the tested polymers experience a negative shift in the colour coordinate both for the a^* axis and the b^* axis compared with polymer 2. In all cases, the negative shift along the a^* axis indicates a reduced colour strength (chroma) compared with polymer 2. The negative shift along the b^* axis (colour coordinate shift in the blue undertone) proves poorer pigment stabilisation (tendency to flocculate) for all the tested polymers compared with polymer 2.

Figure 7: Relative colour coordinate shift along the a^* and b^* axes for polymers 1, 3, 4, and 5 compared with polymer 2 when dispersing an opaque iron oxide pigment.

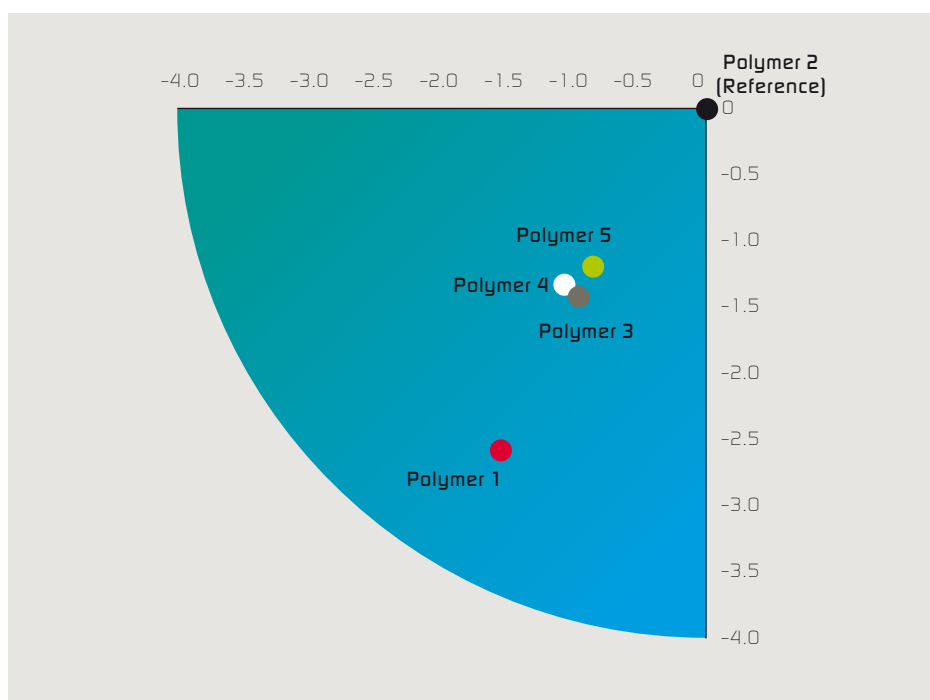
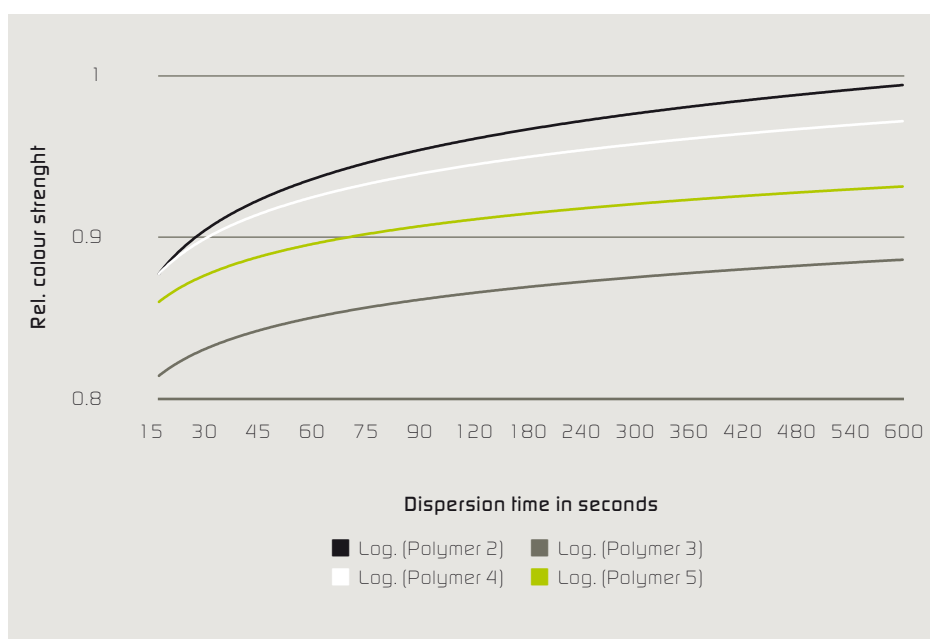


Figure 8: Relative colour strength as a function of the dispersion time for dispersing 25 % carbon black (P. Bl. 7) in dipropylene glycol diacrylate.



IMPROVED COLOUR STRENGTH DEVELOPMENT THROUGHOUT DISPERSION

From economic and plant-specific perspectives, it is not only of great interest to achieve the best possible rheological and colouristic results, but to achieve these in the shortest possible dispersion time with minor mechanical input. For these reasons, further work evaluated the relative colour strength development of different pigments as a function of the dispersion time and compared the effect of the polymers used. In accordance with *Figure 8*, for example, a carbon black concentrate was prepared with a pigment content of 25 % and assessed in terms of its colour strength development over the dispersion

“The additive can be used in all common indoor and outdoor wood and furniture coating applications.”

3 questions for Marcel Krohnen

Did you receive different results for organic and inorganic pigments with regard to dispersion and stabilisation? Fundamentally speaking, it can be stated that many conventional wetting and dispersing additives are either very specially suited to use with inorganic pigments, or their dispersing and stabilising effect is limited to organic pigments, a fact that can represent a technical advantage for selected applications. Thanks to its novel chemical structure and the use of various functional units as pigment-affinic groups, the wetting and dispersing additive under discussion here does however stabilise both inorganic as well as organic pigments in a very high quality, a fact that is an advantage in particular when formulating pigment concentrate systems

Is the new additive suitable for both indoor and outdoor wood and furniture applications? The additive can be used without exception in all common indoor and outdoor wood and furniture coating applications, as it does not have a negative impact on either the water sensitivity or on the UV long-term stability of the coating systems.

How significant are low-VOC UV-curing systems for wood and furniture today? Within the framework of the general global efforts to achieve an ever greater reduction in the share of volatile organic compounds in coating systems, including in industrial use, the significance of solvent-free radiation-curing coating systems in selected wood and furniture coating applications is rising considerably, and is intensifying the development of customised additive solutions.



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process. The individual pigment dispersions were stopped at different dispersion times, a sample taken of the respective pigment concentrate, and it was let down in a white-pigmented radiation-curable base coat, applied, and its colour strength measured. The absolute colour strength measured in this way was considered in relation to the absolute colour strength of polymer 2 at a dispersion time of 600 seconds, which then gave the logarithmic dispersion curves shown in *Figure 8* (relative colour strength development as a function of time). It is clearly visible from the four dispersion curves presented (polymer 1 was not included) that polymers 2 and 4 had the greatest relative colour strength at the start of dispersion, however this developed differently over the course of dispersion. Following a total dispersion period of 600 seconds, polymer 2 displayed the greatest relative

colour strength, followed by polymer 4 with a relative colour strength of ~97 %. Polymers 3 and 5 were less impressive and only achieved a relative colour strength of ~89 % and ~93 % respectively. This illustrative result could be confirmed for various organic pigments.

NEW APPROACH HAS BROAD APPLICATION

The use of controlled polymerisation technology enables the selective development of a novel solvent-free wetting and dispersing additive that fulfils the highest technical requirements in terms of formulating pigmented solvent-free, radiation-curable wood and furniture coatings. Its broad application for inorganic and organic pigments and carbon blacks is equally striking along with its marked rheological and colouristic perfor-

mance capacity, achieved when formulating pigment dispersions based on the novel wetting and dispersing additive. 

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