

Neuburg Siliceous Earth

in 2C-polyaspartic coatings

for corrosion protection

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1 Introduction and objectives

Functional fillers are able to improve the performance properties of paints and coatings, and for this reason find an integral place as ingredients in many anti-corrosion formulations. In recent years, more stringent requirements placed in particular on solvent based formulations have led to intensive development projects for conventional protection systems, which until now have been mainly built up from a combination of a basecoat and a topcoat, with optionally in addition an intermediate layer. Such a multi-layer design is necessary in order to ensure long-lasting corrosion protection even under unfavorable exposure conditions, but increasingly goes against the interest to work with more efficient and cost saving coating technologies. In this area, polyaspartic paints offer a first approach towards a solution.

Polyaspartic coatings resemble two-component PUR systems because of a similar curing mechanism. However, in place of a classical polyol component, a sterically hindered aliphatic amine (an aspartic acid ester) reacts with an aliphatic isocyanate under formation of a polyurea network. Coatings on the base of this concept, in comparison with conventional solvent based two-component PUR systems, among others offer the following advantages*:

- lower VOC content
- prolonged pot life
- shorter drying times
- blister-free application also at higher film thickness
- improved weathering resistance

* Source: Bayer MaterialScience

For corrosion protection, polyaspartic coatings which have been traditionally used as topcoats combined with one-component PU basecoats, now also find increasing use in form of straight one-layer systems directly applied onto the metal surface.

The objective of the present work was to evaluate the effects of Neuburg Siliceous Earth on the performance level of such polyaspartic anti-corrosion coatings using established corrosion protection tests as well as optical, mechanical and rheological evaluations. The references used were fillers well established in corrosion protection such as barite, talc and wollastonite. The main focus was directed at suitable filler selection and loading. Additional approaches towards optimum properties were focused on the formulations additives.

2 Experimental

2.1 Base formulation

The composition of the base formulation from Bayer MaterialScience, with a zinc aluminum phosphate as the active anti-corrosion pigment, is given in *Fig. 1*. The high solids content of 89 mass % and the VOC content of about 156 g/l point to the highly reduced solvent status of the formulation. For the tests reported here, the rheological additive included in the formulation was not used throughout, in order to more clearly bring out the rheological effects of the fillers tested.

	Ba	ase Formulatio	Hoffmann Minieral			
LON PASS						
		2C-Polyaspartic for steel	constructions	n buu		
	•	Desmonhen	Polyappartia actor	175		
	A	NU 1520	Polyaspartic ester	1/5		
		Desmonhon	Reactive diluent	55		
		VPIS 2142	blocked cycloalinhatic diamine	55		
			Dowatoring agent zoolito	24		
		MBA (Solvesse (1:1)	Solvont	100		
		BVK-085	Descration and defeaming additive	8		
		B1K-005	Methylalkylpolysiloxane	0		
		Disperbyk 110	Wetting and dispersing additive	7		
		2.000.230				
		Tinuvin 292	Light stabilization additive	5		
	Tronox R-KB-4		Titanium dioxide	125		
		Heucophos ZPA	Zinc-aluminum phosphate	125		
		Barite	Filler	162		
			natural barium sulfate			
		CAB-O-SIL TS 720	Rheological additive	12		
			fumed silica, hydrophobic			
	в	Desmodur	Hardener	202		
		N 3600	low viscosity HDI Polyisocyanurate			
	То	1000				
	Solid Content w/w 1%1 approx 89					
	Pi	ment volume concentration (E	approx. 20			
			approx. 20			
Martin 2- N		,		appiox. 150		
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2.2 Fillers

Fig. 2 gives a summary of the fillers tested in the study, beginning with barite which was used as a reference. Comparisons were made with a commercial talc and wollastonite grade. For Neuburg Siliceous Earth, Sillitin Z 86 and Aktisil PF 777 were included; in some cases, also the results for some other grades are listed.

Apart from untreated fillers, the test program included two modified grades where via functionalization of the filler surface the interactions with the polymer matrix were appropriately enhanced. Aktisil PF 777 is a version of Neuburg Siliceous Earth Sillitin Z 86 surface-modified in direction of hydrophobicity with alkyl-functional groups, while the wollastonite was treated with an aminosilane.

	Fillers MU							FFMANN NERAL	
	Characteristic	CS							
	Filler	Morphology	Particle- size [µm]		Specific surface area	Oil ab- sorption	Density	Surface treatment	
			d ₅₀	d ₉₇	[m²/g]	[g/100g]	[g/cm³]		
	Barite	corpuscular	2.9	14	1	14	4.2	-	
	Talc	lamellar	6.8	18	5	45	2.8	-	
	Sillitin Z 86	corpuscular / lamellar	1.8	8	12	50	2.6	-	
	Aktisil PF 777	corpuscular / Iamellar	1.8	8	8	35	2.6	alkyl- functiona- lized, hydrophobic	
	Wollastonite	blocky / acicular	3.5	13	4	26	2.8	Aminosilane	
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Fig. 2

All results were determined at Hoffmann Mineral, and therefore are directly comparable. For talc, the analysis indicates the highest average grain size, but in view of the lamellar character of the filler the validity of this result has to be considered as limited. Barite, wollastonite and in particular Neuburg Siliceous Earth give evidence of improved fineness. The high density of the barite, together with the compact grain shape goes in line with a lower oil number and specific surface area. Neuburg Siliceous Earth offers a markedly higher surface area compared with the reference fillers. The oil numbers and the density are at level with the talc.

As the naturally existing form of barium sulfate, barite presents a simplified corpuscular grain shape. The magnesium silicate hydrate talc distinguishes itself by pronounced platelike, lamellar primary particles, while the calcium silicate wollastonite is characterized by a blocky-acicular (needle-like) structure. The particular morphological composition of Neuburg Siliceous Earth is illustrated with the SEM image in *Fig. 3*. The Neuburg Siliceous Earth, extracted in the surrounding of Neuburg (Danube), is a natural combination of corpuscular Neuburg silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods. As a result of natural formation, the silica portion exhibits a round grain shape and consists of aggregated, cryptocrystalline primary particles of about 200 nm diameter.



Fig. 3

2.3 Test Design

While eliminating the rheological additive throughout, the filler replacement followed two basically different routes.

In a first step, the filler was replaced at equal weight. In view of the different specific filler densities, the formulation with barite reaches a PVC of 20 %; talc and Neuburg Siliceous Earth arrive at 23 %. Wollastonite was not included in this series. In preliminary tests, apart from rheological effects in general the corrosion protection properties were just moderate and the filler effects almost negligible.

As a consequence, the filler loading was increased. The original barite loading of 162 pbw was firstly raised to a PVC of 30 % (502 pbw), and then the filler was replaced at equal volume (*Fig. 4*).



Fig. 4

Besides a version with straight talc, also a talc / barite 1:1 combination (per volume) was included, as this represents a blend which is commonly used in corrosion protection. As can be seen in the figure, the adjustment of uniform PVC requires differing amounts of the fillers. Apart from the fillers, all other formulation ingredients have been left unchanged. The formulations with a PVC of 30 % in the preliminary test series partly showed significant filler-dependent differences and, therefore, are the base of the following work.

2.4 Preparation, application and conditioning

The preparation of the formulation batches was carried out in a laboratory dissolver with adapted bead mill under cooling with water. The dispersion time chosen was 20 minutes using glass pearls as grinding media. The light stabilizer and half the solvent resp. the deaerating agent were slowly introduced after the milling step. After finally adding the isocyanate, the wet paint was applied in one layer by air spraying (4 bar, nozzle 1.2 mm) onto SA $2^{1/2}$ corundum blasted cold-rolled steel and onto non-blasted cold-rolled steel panels of the type Q-Panel R-48. For the abrasion tests, special Taber panels 10 x 10 cm with a center bore-hole made of DC04 flat steel were used. Finally, the coated panels were stored during 14 days at 23 °C and 50 % relative humidity. The dry coating thickness was about 120 µm.

3 Results

3.1 Producibility

With the exception of talc, all fillers can be incorporated easily to very easily into the binder matrix (*Fig. 5*). In spite of the somewhat more favorable conditions with barite and wollastonite, the best results according to Grindometer tests (DIN EN ISO 1524) are obtained with Neuburg Siliceous Earth.

The batches with straight talc were impossible to prepare, because the addition of the filler caused too strong a viscosity increase to allow effective milling and dispersion. In order to add the complete filler loading, twice the dispersing agent concentration and at least a 50 % higher solvent addition, unfavorable with respect to the VOC balance, would have been required. Already in the blend with barite, the talc portion gave rise to negative effects with regard to incorporation and resulting fineness of grind.

	Preparation	HOFFMANN MINIERAL		
	Producibility A-Com			
	Filler	Incorporation	Fineness of grind [µm]	
	Barite	$\underbrace{\bullet} \\ \underbrace{\bullet} \\ $	10	
	Talc	not producible	-	
	Barite / Talc	••	15	
	Sillitin Z 86		< 10	
	Aktisil PF 777	••	< 10	
	Wollastonite	$\underbrace{\textcircled{\baselineskip}}_{\bullet,\bullet} \underbrace{\textcircled{\baselineskip}}_{\bullet,\bullet} \underbrace{\genfrac{\baselineskip}}_{\bullet,\bullet} \underbrace{baselineskip}}_{\bullet,\bullet} \underbrace{baselineskip}_{\bullet,\bullet} \underbrace$	10	
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3.2 Storage stability

Fig.6 gives a summary of the results after 28 days of storage at 50 °C in closed containers for the A components of the formulations. With the exception of Neuburg Siliceous Earth, all compared fillers caused a lot of very tough sediment which was difficult to re-stir.

The low sedimentation tendency of Neuburg Siliceous Earth effectively works against the formation of a hard sediment, and thus allows a faster rehomogenization of the batches.



3.3 Rheology

The rheological characterization of the samples took place in the cylindrical system of a MCR 300 Rheometer under rotation, according to DIN 53019. As is seen in *Fig.* 7, the properties of a liquid coating are markedly affected by the kind of filler used. Pseudoplasticity is of advantage for the application of higher film thickness. Such a behavior can be obtained with lamellar filler portions as in talc or Sillitin Z 86. With Aktisil PF 777, the effect comes out particularly pronounced.



Fig. 7

When barite or wollastonite are used, in order to reach comparable properties the addition of a rheological additive is absolutely necessary, in particularly also for maintaining a sufficient storage stability.

3.4 Corrosion protection on blasted substrate

The anti-corrosion properties were determined via a salt spray test according to DIN EN ISO 9227 resp. a humidity test with condensation-water atmosphere according to DIN EN ISO 6270-2 CH.

The degradation of the coatings was assessed following ISO 4628.

The substrates were corundum-blasted unalloyed test panels 150 x 100 x 2 mm made from cold-rolled steel. According to DIN EN ISO 12944 ("Corrosion Protection of Steel Structures through Coating Systems"), Part 6, the degree of surface preparation was Sa $2^{1/2}$ at a surface roughness "medium (G)". The rough surface corresponds to what an application worker would find during practical repair efforts after removing the old coating by sandblasting.

Fig. 8 demonstrates the very good results of the cross-cut test on blasted steel for all formulations prior corrosion tests.

	Adhesion	HOFFMANN MINERAL			
	Cross-cut test 2 mm / 3 mm tape tear-off				
	DIN EN ISO 2409				
	Rating for all formulations:	0 - 1			
AS ANY A	VM-5/1207/09.2019				

The corrosion protection tests did basically not show any effect onto the good adhesion properties, with some limitation for the formulation with straight barite. The evaluation of the cross-cut test after exposure to condensation water was impaired by strong blistering and, therefore, gave a poor result. As shown in *Fig. 9*, the stripping gives rise to a heavily corroded substrate surface; with all other filler systems tested a defect-free coating film prevents a corrosion of the substrates.



Fig. 10 gives a representative and comparative summary of the protection efficiency at a scribe which was applied in a defined manner prior to the exposure. As a result of the corrosion and in view of the simultaneously proceeding cathodic side processes, delamination symptoms are evident. The free metal area on both sides of the scribe corresponds to this delaminated region, which is clearly limited and can easily be laid open with a knife operated at a flat angle.



Fig. 10

In the humidity test, a reliable assessment of the rust formation at the scribe for the straight barite formulation runs into difficulties because of the strong surface corrosion. The wollastonite batch tends only to weak rust formation at the scribe, but gives rise to complete delamination. In comparison with barite and wollastonite, the filler blends talc / barite or the Neuburg Siliceous Earth grades offer very good corrosion protection in combination with low delamination tendency. In particular Aktisil PF 777, as a result of the hydrophobization of the filler surface, allows to obtain excellent results.

After the salt spray test, the differences with respect to the delamination at the scribe come off smaller. The surface treated fillers Aktisil PF 777 and especially wollastonite still show the strongest delamination, but the rust formation at the scribe is reduced. The use of barite brings out exactly the contrary effect. Likewise, working in addition with talc does not improve the performance at the scribe. Looking at corrosion and delamination, Sillitin Z 86 is able to offer the most balanced result after salt spray exposure.

Sillitin Z 86 in total proves itself as the best suited filler with good resistance on blasted steel during salt spray and humidity test.

3.5 Corrosion protection on non-blasted substrate

Durable surface protection by coatings requires good adhesion on the substrate. Extensive tests, therefore, were also run on critical substrates like non-blasted cold-rolled steel (Q-Panel type R-48, 200 x 100 x 0.8 mm). This substrate proves to be very critical compared with blasted steel.

In case of deficient adhesion, local damage of the coating without further external influences can lead to large area defects, as shown in *Fig. 11* for the formulation containing 162 pbw barite.



Fig. 11

The increase of the original filler loading to a PVC of 30 % basically works out positively with respect to the adhesion prior to exposure. As shown in *Fig. 12*, the cross-cut test rating for the formulation with barite is impressive improved from 5 to 1, a level also found for the formulation filled with wollastonite. Talc or Neuburg Siliceous Earth already at low loadings give rise to good adhesion, which is further improved when using Neuburg Siliceous Earth at higher PVC.



Fig. 12

After the salt spray test, Sillitin Z 86 offers outstanding corrosion protection properties on non-blasted steel. While the adhesion of the other formulations markedly deteriorates during the exposure, the adhesion of the coating with Sillitin Z 86 remains on a very good level with a cross-cut rating of 1. *Fig. 13* illustrates this with representative test panels after an exposure of 480 hours.



Fig. 13

With similar clarity such results come out via differences at the scribe (test panels upper row), where the Sillitin Z 86 formulation long-lastingly acts against corrosion and shows nearly none tendency towards delamination. All other coatings can be easily removed starting at the scribe with a knife, indicating higher corrosion intensity and high delamination, at least 40 mm due to limited width of the test panel. A surface corrosion under the unscribed and stripped coating (lower row) has not been observed for any of the formulations tested.

Ongoing tests have confirmed the overall positive results with Sillitin Z 86 und showed no change in good adhesion and corrosion protection even after a prolonged exposure time of 1000 hours.

Compared with the salt spray test, the damage picture in the humidity test (*Fig. 14*) is characterized by a strong tendency towards blister formation.

The highest blister density is evident for the barite coating. The combination with talc or the formulation with wollastonite shows a moderate decrease of the amount of blisters. In contrast the size of the blisters comes out bigger. Sillitin Z 86 has to be ranged in between barite and the barite / talc blend.

As the only functional filler, Aktisil PF 777 leads to a coating without any defects. The visible surface texture only gives the wrong impression which goes back to the pseudoplasticity and structure recovery of the wet paint after application, resulting in a limited leveling.



Fig. 14

Despite the strong blister formation, only in the formulation loaded with Sillitin Z 86 noticeable signs of corrosion appear in the substrate surface (*Fig. 15*). The hydrophobization of the filler surface brings out favorable effects with Aktisil PF 777. Apart from very isolated rust points, the steel surface is free from corrosion.



Fig. 15

It is true, however, that with cross cut ratings of 5 without exception all formulations tested proved critical on non-blasted steel. A limited delamination area at the scribe is no longer observed (*Fig. 16*).



3.6 Optimization of both salt spray and humidity performance

In further tests it could be established that there are approaches to improve the adhesion after exposure by formulating without the dispersing additive. In order to continue to ensure good incorporation, the filler loading was taken down to the original level of 162 pbw of the reference formulation without rheological additive. As illustrated in *Fig. 17*, in the batch with Aktisil PF 777 not only the adhesion is enhanced, but also corrosion and delamination at the scribe are pushed back. The negative influence of the dispersing additive is also evident from the comparison of the formulations without respectively with this additive at equal PVC.



Fig. 17

Fig. 18 demonstrates similar effects after exposure to salt spray test.



Fig. 18

The elimination of the dispersing additive also acts favorably on the corrosion resistance at high ionic exposure (salt spray test) for all formulations (*Fig. 19*).



When a well balanced protection under salt spray as well as condensation water exposure is required, the appropriate selection of fillers becomes a deciding element. In direct comparisons, Aktisil PF 777 comes out as the filler of choice. It is true that in the salt spray test along with very good adhesion the largest rust extension at the scribe is observed; but in the humidity test (*Fig. 20*) this filler gives the very best result. Aktisil PF 777 here leads to markedly improved adhesion after exposure and to reduced corrosion and delamination at the scribe.



Fig. 20

4 Further properties

4.1 Effects of the optimization on pot life

The pot life was assessed via the time-dependent viscosity increase after addition of the hardener. A strong viscosity increase indicates a reduced pot life which means a shortened processing time. Repeated viscosity tests were carried out at low shear rate in a Brookfield viscosimeter.

Fig. 21 demonstrates the strong effect of the filler loading and the dispersion additive for the examples of the barite respectively Aktisil 777 formulations.



Fig. 21

From these curves, via linear regression the corresponding rates of the viscosity increase were determined. The resultant reciprocal values are a measurement for pot life and in *Fig.* 22 describe the time period needed for a viscosity increase of 1 Pa*s.



Fig. 22

By eliminating the dispersing additive and simultaneous reduction of the filler loading, the time period is considerably extended with Neuburg Siliceous Earth. Aktisil PF 777 ensures a markedly longer processing time, while Sillitin Z 86 comes out on level with the other fillers. With those, the elimination of the dispersing additive has a distinct negative effect despite the lower filler loading.

5 hours after beginning of the viscosity measurements non-blasted Q-Panels were coated with the wet paints, conditioned for 14 days and then tested for adhesion. Compared with immediately coated specimens the cross-cut rating for the wollastonite formulation showed a critical decrease from 1 to 5, while all other formulations did not show a deterioration of the adhesion strength.

4.2 Drying characteristics

Already in the preliminary tests no significant influence of the fillers on the drying properties could be established. The observed degree of drying T4 according to DIN 53150 with dry film thicknesses of 70 resp. 140 μ m was always in a comparable range of 5 to 6 hours. If for the determination of full drying the wire bow method according to Erichsen is used, one arrives at coating films whose surfaces are no longer damaged by the application and movement of the bow after 4 h.

4.3 Abrasion loss

Abrasion tests were run on a Taber Abraser with sand paper strips type S-42 at 50 rpm on conditioned samples. After the pre-set number of revolutions, the weight loss through the abrasive medium was determined and converted into graphs. According to *Fig. 23*, the fillers can be divided into three groups.

Barite, Sillitin Z 86 and Aktisil PF 777 show a comparable characteristic. Talc leads to higher abrasion loss, as already evident in the blend with barite. The non-linear curve confirms the lower resistance of the coating with increasing penetration.



Fig. 23

The lowest abrasion loss figures are obtained with wollastonite, which can be related to the effect of the reactive surface modification of this filler. A Sillitin Z 86 surface treated in the same way with aminosilane, Aktisil AM, imparts similarly favorable abrasion resistance.

4.4 Gloss

The gloss results under the test geometry of 60° are presented in *Fig. 24*. Quite evidently, in contrast to barite and wollastonite Neuburg Siliceous Earth offers ideal approaches towards matting. Only via eliminating the dispersing additive in the correspondingly less loaded systems, all fillers lead to reduced gloss, while the advantages of Neuburg Siliceous Earth are maintained. Neuburg Siliceous Earth on the other hand is also capable of meeting requirements with respect to highly glossy coating surfaces if in place of Sillitin Z 86 the somewhat coarser grade Sillitin V 85 is used. The results for the highly loaded formulation indicate that a level comparable with the competitive fillers can be attained.



Fig. 24

4.5 Color

The formulations with Sillitin Z 86 and Aktisil PF 777 in comparison with the competitive fillers tend to exhibit a slight yellowish tinge. Against high requirements as to the optical appearance, the use of the more color-neutral Sillitin Z 89 is recommended.

4.6 Hiding power

The hiding power was assessed by coating contrast cardboards with films of different thickness, and testing after drying. The film thickness where the ratio of the standard color value Y over black to white underground reaches 98 % was used as the criterion for defining a coating with high hiding power.

With Neuburg Siliceous Earth, according to *Fig. 25* a good hiding power will be obtained already at lower coating thicknesses. The required material usage for a well-hiding film can, therefore, reduced by 10 to 20 % compared with the other fillers, which offers effective cost savings.



5 Summary

The performance properties of polyaspartic coatings can be optimized with Neuburg Siliceous Earth by appropriate grade selection and concentration. Compared with the conventional fillers barite, talc and wollastonite, in particular Sillitin Z 86 as well as Aktisil PF 777 impart markedly improved corrosion protection:

- on blasted steel distinctly optimum and best balanced properties with Sillitin Z 86
 - » reduced corrosion / delamination at scribe combined with excellent protection and adhesion on unscribed area
- on non-blasted steel and strong ionic exposure (salt spray) benefits with Sillitin Z 86
 - » outstanding adhesion and very low corrosion / delamination at scribe
- on non-blasted substrate and condensation water exposure generally poor adhesion, strong blistering and surface corrosion.
 Significant improvement of anti-corrosion properties with Aktisil PF 777
 - » blister-free coating almost without surface corrosion
- on non blasted substrate considering both humidity test and salt spray test best results with adapted dosage of Aktisil PF 777 in absense of the dispersing additive
 - » markedly increased adhesion and reduced corrosion / delamination at scribe in salt spray as well as humidity test

Further beneficial effects of Neuburg Siliceous Earth round off the excellent property profile:

- excellent adhesion also on non-blasted substrate
- improved storage stability and application in higher film thickness without rheological additive
- possibility to produce both matted (Sillitin Z 86, Aktisil PF 777) or glossy (Sillitin V 85) coating surfaces
- enhanced hiding power
- excellent price / performance ratio

The combination of the properties mentioned for Neuburg Siliceous Earth with the basic performance of polyaspartic coatings offers application areas:

- as one-layer coatings on predominantly non-blasted substrates for construction and agricultural machinery, railway vehicles and industrial coatings
- on blasted substrates as one-layer coatings or primers for medium to heavy corrosion protection of bridges, cranes, pylon and tower constructions as, for instance, in wind power plants.

Starting formulations for working with Neuburg Siliceous Earth are summarized in Fig. 26.

	Starting Formulations				Hoffmann Minieral					
Long Pars	Wit	/ith Neuburg Siliceous Earth								
	[1] f	distant								
	[1]10	[1] for strong ionic exposure on blasted or non-blasted steel								
	[2] fo	or intense humide e	environment particularly on non	-blaste	d stee	əl				
				[4	1	[2]				
					J 0/	[2]				
	A	Desmophen NH 1520	Polyaspartic ester low viscosity, amine-functional resin	175	15.4	175	17.8			
		Desmophen VP LS 2142	Reactive diluent blocked cycloaliphatic diamine	55	4.8	55	5.6			
		UOP-L Powder	Drying agent, zeolite	24	2.1	24	2.4			
		MPA / Solvesso (1:1)	Solvent	100	8.8	100	10.2			
		BYK-085	Deaeration and defoaming additive Methylalkylpolysiloxane	8	0.7	8	0.8			
		Disperbyk 110	Wetting and dispersing additive	7	0.6	-	-			
		Tinuvin 292	Light stabilization additive	5	0.4	5	0.5			
		Tronox R-KB-4	Titanium dioxide	125	11.0	125	12.8			
		Heucophos ZPA	Zinc-aluminum phosphate	125	11.0	125	12.8			
		Sillitin Z 86	Filler	311	27.4	-	-			
		Aktisil PF 777	Filler, surface treated, hydrophobic	-	-	162	16.5			
	В	Desmodur N 3600	Hardener low viscosity HDI Polyisocyanurate	202	17.8	202	20.6			
	Тс	Total				981	100.0			
	Sc	Solid Content w/w [%]			approx. 91		approx. 90			
	Pi	gment volume concentration	on (PVC) [%]	approx. 30		approx. 23				
	V	VOC [g/l]			approx. 141		approx. 148			
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Fig. 26

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