

# Raising the barrier to rust

## Properties of different talcs compared in corrosion testing

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**Talcs with different properties were evaluated as anticorrosive pigments. Differences in performance could be found much more rapidly and clearly by using EIS methods than by salt spray testing. Pure, platy low oil absorption talcs gave excellent barrier properties in heavy duty protective coatings without the use of any other anticorrosive pigment.**

Talc (magnesium silicate) is, after CaCO<sub>3</sub>, the second most widely used extender in coatings of all types. Talc is a soft mineral with a moderate oil absorption (15-50 g/100 g) and density (2.7-2.8 kg/dm<sup>3</sup>), it is inert and has a low price.

Talcs with differing crystal structures affect the film strength of coatings differently. The most common types have a platelet form (macro-crystalline talc) that gives good barrier properties and reinforces the coating film. The particle shape can also be blocky (micro-crystalline talc) which gives a good balance between hiding powder and whiteness.

The platy talcs are preferred for anti-corrosion performance where they presumably act in a manner similar to mica or MIO (micaceous iron oxide). That is, the extender functions by means of its flat, platy particles, which overlap and complicate the path that water must travel to get through the coating.

Based on this knowledge, talc is expected to influence water absorption as well as the formation of conduc-

tive pathways positively and therefore is expected to improve the barrier properties of various coatings. Since the composition can differ between sources, the composition of commercial talc grades varies. The talc can also be pure or may be combined with other minerals such as chlorite (Mg- Al-Fe-silicate) and/or carbonate-based minerals.

In this study, different commercial low oil absorption talcs were evaluated in relation to their corrosion protection, based on barrier behaviour. This behaviour was determined by EIS (Electrochemical Impedance Spectroscopy) and the accelerated salt spray method.

### Raw material selection for test coatings

The anti-corrosive performance of five different talcs with different composition and morphology was tested in epoxy coatings. The talc products differ in their composition, particle form and the production technique used. The macro-crystalline talcs have very platy particles, while the micro-crystalline (small crystal size) talcs have a blocky particle form. Magnesite (MgCO<sub>3</sub>) is a very common impurity in talc, and one talc tested contained about 15 % of this.

An initially high oil absorption (OA) is one problem with talc, which limits its loading levels in paint formulations. To lower the OA, the fines content is normally reduced by extracting the smallest particles from the talc. This process is commonly called de-dusting.

The properties of the talc products used in this study are shown in *Table 1*. A solution of semi-solid epoxy resin



For more information about anticorrosive coatings: European Coatings CONFERENCE "Architectural Coatings" 24.-25.4.2012 [www.european-coatings.com/events](http://www.european-coatings.com/events)

	Talc 1 = Pure, platy talc with de-dusted PSD	Talc 2 = Pure, platy talc with high whiteness & de-dusted PSD	Talc 3 = Pure, platy talc with coarse, standard PSD	Talc 4 = Magnesite rich talc with standard PSD	Talc 5 = Pure, blocky talc with de-dusted PSD
LOI [w%]	6.0	7.0	6.1	12.4	5.4
HCl-solubles [w%]	3.8	7.2	3.4	14.7	5.9
ISO-Brightness [%]	77.6	88.1	76.3	88.8	72.8
DIN-whiteness [%]	79.2	89.1	78.3	90.7	76.4
CIE-values, L*	91.32	95.6	90.88	96.27	89.95
a*	-0.40	-0.07	-0.37	-0.05	-0.45
b*	1.57	0.79	1.8	1.40	3.1
Hegman Fineness [µm]	120	125	160	50	100
Oil absorption [g/100 g]	28	28	22	26	17
BET [m <sup>2</sup> /g]	1.9	5.3	2.2	6.4	7.3
D98 [µm]	44	42	50	29	58
D50 [µm]	15.6	15.4	14.6	6.7	24.7

Table 1: Properties of talc products tested

in xylene ("Araldite GZ 290x90") cured with a polyamidoamine adduct ("Aradur 450") was used as the polymer binder.

"Disperbyk 180" was used as dispersing agent. Titanium dioxide ("Kronos 2315") was also integrated as well as micronised iron oxide black ("Bayferrox 318M").

### How PVC levels were set

The Pigment Volume Concentration (PVC) is one key parameter that describes paints. The critical PVC (CPVC) above which many coating properties change abruptly has to be taken into account. This can be calculated from the oil absorption [1-2]. A relationship between the PVC and the CPVC is defined as "reduced PVC" ( $\Lambda$ ).

At  $\Lambda < 1$  (below CPVC), a dry coating film is a continuous coating, a composite consisting of pigment particles randomly embedded in a continuously connected matrix of polymer. Above the CPVC ( $\Lambda \geq 1$ ), there are void structures in the film due to insufficient polymer, but the pigment particles can still be thought of as being continuously connected. Above the CPVC there is not enough resin to cover the pigment surface and a significant amount of the polymer is absorbed by the pigment [2]. When using talcs with different oil absorption, the CPVC changes. If the PVC were kept constant when using

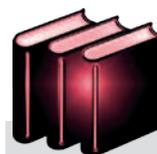
### Results at a glance

» A number of talcs with different properties were evaluated as the sole anticorrosive pigment in an epoxy formulation.

» Blocky (micro-crystalline) talcs may be preferred when seeking to optimise whiteness or opacity, but the EIS results showed that pure, platy (macro-crystalline) and low oil absorption talcs give the best barrier properties in heavy duty protective coatings.

» In salt spray testing, the best of these talcs showed acceptable corrosion protection for the C5-M climate without any other anti-corrosion pigment, which is an outstanding result.

» Differences in performance between the talcs could be found much more rapidly and clearly by EIS methods than with salt spray testing. Coating developments can be improved and accelerated by combining High Throughput Screening with EIS testing.



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	Talc 1	Talc 2	Talc 3	Talc 4	Talc 5	Talc 1	Talc 3	Talc 5
Ingredients	wt %							
Araldite GZ290x90	31.33	31.33	28.99	30.58	26.72	26.06	22.96	23.1
Disperbyk 180	0.64	0.64	0.63	0,63	0.64	0.64	0.65	0.65
Xylene	9.94	9.94	9.82	9.91	9.69	9.65	9.48	9.48
Kronos 2315	6.92	6.92	6.95	6.95	6.99	7.01	7.03	7.02
Bayferrox 318 M	0.87	0.87	0.87	0.87	0.87	0.88	0.88	0.88
Pure, platy talc with de-dusted PSD	40.20					47.37		
Pure, platy talc with coarse, standard PSD			43.40				51.61	
Pure, platy, high whiteness talc with de-dusted PSD		40.20						
Magnesite rich talc normal				41.22				
Pure, blocky talc with de-dusted PSD					46.47			51.42
Aradur 450	10.09	10.09	9.34	9.85	8.61	8.39	7.39	7.44
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Properties								
PVC	3248	32.48	35.75	33.51	39.07	40.10	45.05	44.81
Λ	0,60	0.60	0.60	0.60	0.60	0.74	0.76	0.69
Mix Viscosity at shear rate 50 rpm, mPas	1950	2200	1700	1680	1015	3730	4880	1650

Table 2: Coating formulations (volume solids for all is 75 %)

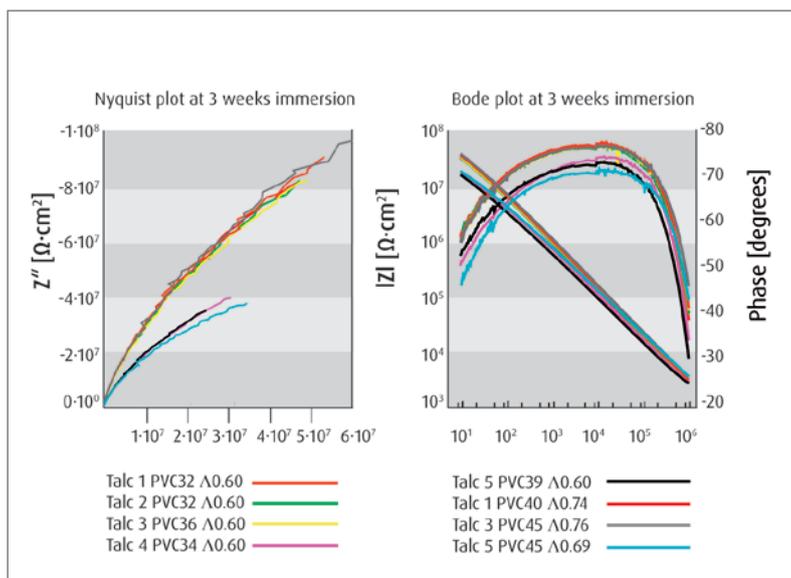


Figure 1: EIS results according to Nyquist and Bode diagrams

Table 3: Electrical parameters for different talc coatings obtained through fitting EIS data results after three weeks' immersion.

	Λ Reduced PVC	R <sub>c</sub> [Ωcm²]	Y <sub>0</sub> [snΩ]	n	Θ <sub>∞</sub>
Talc 1	0.60	1.08E+08	1.49E-10	0.940	0.22
Talc 2	0.60	1.13E+08	1.61E-10	0.929	0.25
Talc 3	0.60	1.04E+08	1.53E-10	0.931	0.23
Talc 4	0.60	4.57E+07	3.00E-10	0.895	0.28
Talc 5	0.60	4.51E+07	3.90E-10	0.888	0.38
<b>Results at higher PVC levels</b>					
Talc 1	0.74	1.09E+08	1.34E-10	0.939	0.21
Talc 3	0.76	1.24E+08	1.28E-10	0.932	0.22
Talc 5	0.69	5.24E+07	3.09E-10	0.877	0.44

higher oil absorption talcs, the coating would be closer to the CPVC, and thus give different barrier properties. As shown in Table 1, different talc products used here do show differences in oil absorption.

In order to control the contribution of each talc properly, a simple coating formulation was set up and the reduced PVC (Λ) of each coating was maintained at a constant 0.6. For comparison, some coatings were also formulated at higher PVC levels. Table 2 contains specific information about each coating studied.

### Initial samples produced by high throughput system

The coating formulations were kept simple and no other anti-corrosion pigment was added to the formulation, in order to see the effect of the talcs most clearly. The coating formulation step in this project was performed by using a High Throughput System, which automatically prepares coatings in parallel using reactors equipped with a dissolver blade.

Raw materials can be automatically added while processing, so the normal coating processing can be applied. When the optimal formulations were obtained, these were scaled up and sprayed for further testing. The coatings were sprayed on Sa 2.5 grit blasted steel panels with a dry film thickness of around 150 μm and were allowed to cure for one week at room temperature before testing.

### Coatings tested by salt spray and EIS

Accelerated testing of coatings is known to formulators to speed up the performance testing before introducing the coating into the market. There is constant discussion of which accelerated test is the best to relate to real life coating performance.

For this study, the accelerated tests Electrochemical Impedance Spectroscopy (EIS) and salt spray were used to determine the anti-corrosion performance of the coatings. EIS is an electrochemical measuring method that quantifies the protective behaviour of coatings. According to many authors [3-5] even a relatively short period of testing with EIS provides reliable data for predicting long-term behaviour.

The salt spray cabinet test is performed according to ISO 9227. In this cabinet, a 5 % NaCl solution is sprayed over the panels at a temperature of  $35 \pm 1$  °C. To classify the protective coatings, salt spray testing was done for C5-M climate, part of ISO 12944, which means 1440 h of testing.

### How the EIS procedure was evaluated

Electrochemical impedance diagrams were obtained by EIS after initial, 24h and one and three weeks of measurements via the EIS cell on the coated steel immersions, containing the different types of talc. The EIS cell contains RIVM 50x concentrated Marine Industrial Rainwater. Nyquist plots and Bode diagrams after three weeks' EIS testing are presented in *Figure 1*.

In general, all impedance spectroscopy data can be fitted exactly to an equivalent circuit when enough parameters (i.e. elements in the equivalent circuit) are used [6]. The results may, however, be physically meaningless. Therefore, it is preferable to fit the data to the most probable impedance equivalent circuits (MPEC) [5].

With respect to this strategy, the circuits are based on the process changes according to a unified degradation mechanism model based on the formation of conductive pathways [7]. The electrical parameters were obtained by fitting the EIS data using the best electric equivalent circuit  $R_c(QR_c)$  for this case. The parameters after three weeks of immersion are listed in *Table 3*.

The coating resistance  $R_c$  expressed in  $\Omega \text{ cm}^2$  is a parameter for the resistance to ion transport through the coating. One of the most important factors of protection against corrosion by barrier coatings is through their resistance to ion transport [8]. As a guideline to the protective behaviour of coatings the following rule is often used [9-10]:

- »  $>10^8 \Omega \text{ cm}^2$  - excellent
- »  $10^7 - 10^8 \Omega \text{ cm}^2$  - adequate
- »  $10^6 - 10^7 \Omega \text{ cm}^2$  - doubtful
- »  $< 10^6 \Omega \text{ cm}^2$  - bad

The constant phase element (Q) allows for small deviations from ideal capacitance behaviour and is characterised by two parameters,  $Y_0$  and  $n$ .  $Y_0$  can be related to coating capacitance if  $n = 1$ , while  $n$  itself represents a deviation from ideal coating behaviour.

Based on the capacitance data, the volume fraction of water absorbed by the coating ( $\phi_f$ ) can also be calculated [5, 7, 11], with  $\phi_\infty$  being the volume fraction of water in the saturated coating. *Figure 2* shows the differences in water uptake for each talc.

It has been proved [12] that the water uptake calculated from EIS is higher than from gravimetric measurements (due to calculation assumptions and the swelling of the supported film). Nevertheless, the water uptake using the EIS method is valid for comparative purposes.



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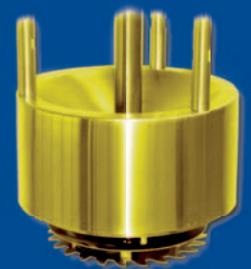
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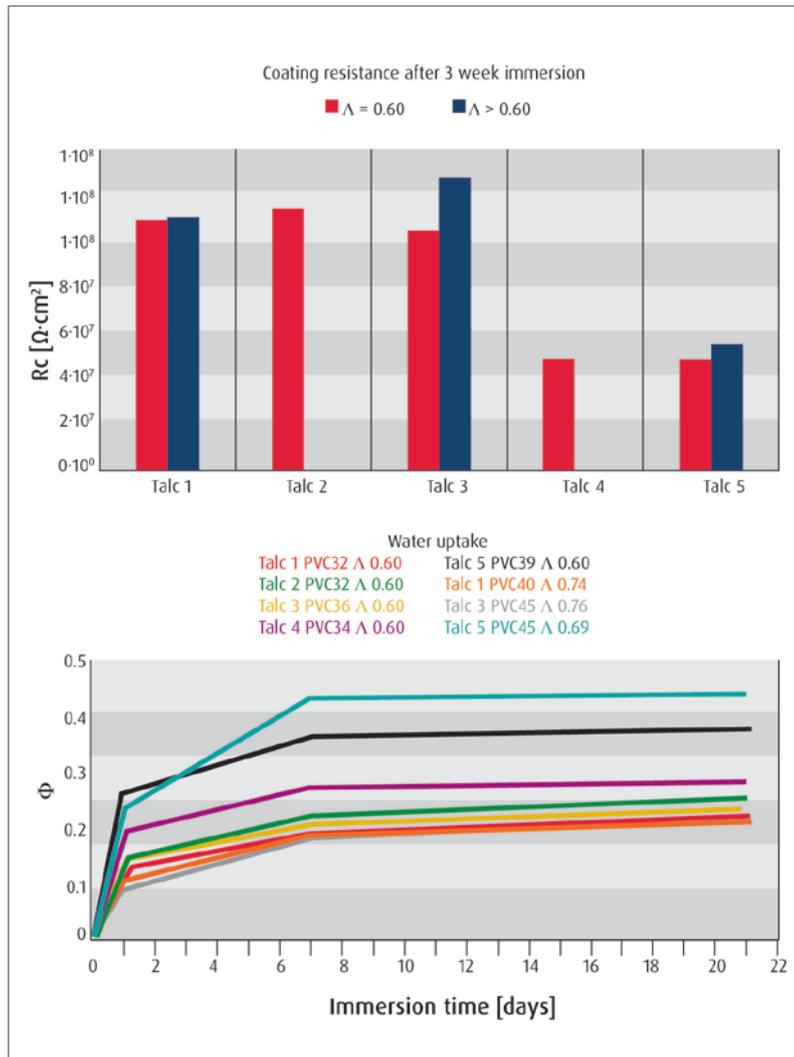


Figure 2: Coating resistance ( $R_c$ ) and water uptake ( $\phi$ ) evaluation

Table 4: Overall ranking of corrosion test results

Talc	$\Delta$ / PVC	EIS	Salt-spray	Overall = combination of EIS and salts spray	Ranking
Talc 1 = Pure, platy talc, de-dusted PSD	0.74/40.0	++	+	++	1
Talc 3 = Pure, platy talc, coarse, standard PSD	0.60/5.8	+	+	+	2
Talc 1 = Pure, platy talc, de-dusted PSD	0.60/32.5	+	+	+	3
Talc 2 = Pure, platy, high whiteness talc, de-dusted PSD	0.60/32.5	+	+	+	4
Talc 3 = Pure, platy talc, coarse, standard PSD	0.76/45.0	++	+/-	+	5
Talc 4 = Magnesite rich talc	0.60/33.5	+/-	+	+/-	6
Talc 5 = Pure, blocky talc, de-dusted PSD	0.60/39.1	--	+	-	7
Talc 5 = Pure, blocky talc, de-dusted PSD	0.69/44.8	--	+/-	-	8

### EIS test detects corrosion problems rapidly

Looking at the absolute values of the EIS measurements, the following can be observed:

- » Overall an increase of talc concentration (at a PVC below CPVC) improves the corrosion protective performance due to increased resistance.
- » The talc with the best overall performance in this EIS test is Talc 1 (pure, platy talc with particle size distribution modified by de-dusting) followed by Talc 3 (pure, platy talc with coarse, standard particle size distribution).
- » Talc 4 (magnesite rich) and Talc 5 (blocky de-dusted talc) provided a standard resistance level but with a high water absorption, high  $Y_0$  and low n-value.

This EIS test shows significant differences between the coatings. A change in PVC often directly influences corrosion protective performance (barrier properties). EIS is a good way to detect differences in corrosion performance even with similar coating ingredients.

The effect of PVC on corrosion performance can be detected using EIS long before visual signs appear. The optimal PVC from a corrosion protection point of view can also be determined using EIS. The coatings using pure, platy talcs are suitable for more severe conditions such as immersion, especially in a de-dusted version.

Blocky or magnesite-rich talcs are limited in their applications for immersion applications, because of their higher water uptake. Based upon this study, it is seen that blocky and magnesite-rich grades are of lower quality from a corrosion protection point of view.

### Salt spray test shows differences less clearly

One scratch was made through the coating to the steel substrate, and panels were placed for 1440 hours in the salt spray test. Classification of the salt spray test panels for C5-M climate, part of ISO 12944, was focused on evaluating creep and blisters.

The first number indicated in relation to blisters is their density, ranging from 1 low to 5 very high, and the number between brackets is the size of the blisters, ranging from S1 small to S5 large. The creep is measured as mm of rust from the 1 mm scratch made on the coating panel. No blisters and a creep value of < 1 mm are considered excellent.

Normally, epoxy coatings are applied in at least two layers of 75-100  $\mu$ m and can achieve these requirements after 1000 hours. In this case, only one layer of around 130  $\mu$ m was applied to see the differences between the coatings more clearly. The ranking is thus now based on the relative performances of the talcs.

No difference was observed in blistering between the talcs tested. The talcs do show a slight difference in creep results, but only the blocky and one pure platy talc at PVC 45 show a slightly higher value. Higher creep values are obtained with pure platy Talc 3 only at  $\Delta$  0.76 (PVC 45) and the blocky de-dusted Talc 5 only at  $\Delta$  0.69. In this salt spray test, it is thus difficult to see differences between the talcs, while EIS yields more obvious differences.

### Platy talcs can be effective anticorrosive pigments

EIS measurements appeared to give faster and more reliable results than salt spray regarding corrosion protection. Coating development can be significantly improved and accelerated by combining High Throughput screening and EIS testing.

In this study, the talc grades with different composition, particle shape and particle size distribution were ranked on their anti-corrosion performance. *Table 4* shows that pure, platy talcs (Talc 1, 2 and 3) gives better barrier properties (based on EIS results) than impure or blocky talcs (Talc 4 & 5).

The corrosion performance with only pure, platy talcs without any anti-corrosion pigments is sufficient to give acceptable results in corrosion protection for the C5-M climate.

The blocky or micro-crystalline talc (Talc 5) is worst in that respect. Pure, platy, de-dusted talc (Talc 1) is overall a very suitable extender for corrosion protective coatings under severe conditions such as C5-M. The optimum PVC level with this talc is 40 vol % in this formulation. ◀

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