

**JOURNAL
OF CHEMICAL TECHNOLOGY
AND METALLURGY**

Journal of Chemical Technology and Metallurgy

The Journal of Chemical Technology and Metallurgy started originally in 1954 as Annual Journal of the former Higher Institute of Chemical Technology. It ran in Bulgarian. In 2000 its name was changed to Journal of the University of Chemical Technology and Metallurgy. It was published quarterly in English. Since 2013 it has run bimonthly as Journal of Chemical Technology and Metallurgy.

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University of Chemical Technology and Metallurgy,
8 Kl. Ohridski blvd., 1756 Sofia, Bulgaria
Tel: (+ 359 2) 8163 302, E-mail: journal@uctm.edu

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Prepress and design: **A. Staneva**; Printing: **S Print Ltd., Sofia**

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DISPERGATION OF ALUMINUM POWDER (ALUMINUM FLAKES) IN SILICON ENAMEL IN PRESENCE OF TROYSERSE™ 98C ADDITIVE

Vitaliy Y. Tyukanko¹, Antonina N. Duryagina²,
Kirill A. Ostrovnoy², Oleg Petukhov², Kristina V. Makievskaya²

¹ "Kazneftegazmash" Ltd., 122 New st., 150000 Petropavlovsk
Republic of Kazakhstan
E-mail: vetal3333@mail.ru

Received 26 November 2017

Accepted 31 May 2018

² M. Kozybaev North Kazakhstan University, Chemistry Department
18 Abayst., 150000 Petropavlovsk, Republic of Kazakhstan

ABSTRACT

The effect of the nitrogen-containing additive Troysperse™ 98C on the aluminum pigment dispersion in industrial silicone enamel KO-814 and the quality of the coating obtained was studied. The results showed that Troysperse™ 98C improved the dispersion of the aluminum pigment and the aggregative stability of the enamel. At an additive concentration of 0.375 % (by weight of the pigment) 25 % decrease of the sedimentation rate and 85 % reduction of the pigment particles average diameter were observed. Compared to the industrial use of KO-184 silicone enamel for a two-layered thermally cured coating, the developed composite containing 0.375 % of Troysperse™ 98C showed: 3.5 times reduction of the corrosion rate in case of a single-layered coated steel substrate; 1.5 times reduction of coatings absorption of petrol; 36 % reduction of roughness; 43% - 52 % reduction of water absorption. The single-layered coating with Troysperse™ 98C showed protective characteristics comparable to the two-layered coatings with no additive. The results presented proved that Troysperse™ 98C added to the silicone enamel KO-184 provided to skip the application of a second layer of a coating improving its protective capacity.

Keywords: nitrogen-containing additive, dispersion, aluminum pigment, silicone enamel, coating quality.

INTRODUCTION

Silicone enamels are widely applied for heat-resistant coatings. They are supposed to be perspective materials for development of novel protective coatings [1-3]. Silicone coatings strength depends mainly on the type of the pigments, their dispersiveness and concentration [4], the fine structure of the coatings [5] and the chemical structure of the resins used [6]. One of the most effective ways of improving the service life of the protective coatings refers to the addition of a surfactant [7]. Traditionally, polyether (PEG-based) and alkyd compounds are used as surfactants in silicone enamels [8]. A thorough study on the effect of nitrogen- and phosphorus-containing compounds in the form of

alkyl phosphates and amines on the characteristics of silicone enamel AS-1117 is published [9]. The reported results prove the effectiveness of their incorporation in silicone enamels. The amines effect on the structural and the morphological changes of the siloxane films both in its initial state and following a thermal treatment at various temperatures is studied [10]. It is found that the addition of amines prevents the coatings loosening by a reduction of the supramolecular structures size. Addition of various siloxane based surfactants (in particular catamine) results in 30 % increased resistance of the composites obtained [11]. It is shown that the addition of polyamide and polyethylenepolyamine to a silicone composition based on an epoxysiloxane resin and an aluminum pigment improves the characteristics of the

coatings [12]. Silicone composites films based on a dimethylsiloxane resin and an aluminum pigment of a homogeneously ordered structure and improved characteristics are obtained using texturing additives [13]. Silicone composites containing nitrogen compounds like polyethylenepolyamine, oligoamide, diamine [14], melamine, ethylenediaminephosphate, polyamidoamine, polyamidoimidazoline [15-17], amines [16,18,19], polyimides [20] are reported in the literature. The main part of the listed nitrogen compounds acts as a surfactant. Their activity is governed not only by the dispersing force [7], but also by the opening of the ring structures of the siloxane molecules and the subsequent formation of additional bonds between them. Composites based on silicone resins and metal pigments such as Zn [21], Sn [22] and Al [1, 23] are offered for development of heat resistant coatings. Aluminum containing composites are the most widely used in the industry as heat resistant coatings. A series of studies on the effect of the content and the type of the surfactant or the type of the silicone and the filler on various substrates wetting with silicone compositions are recently published [24-27]. The barrier properties of silicone coatings applied simultaneously with oxidation films are studied [28]. A strong influence of subsurface stresses on the permeability of the films and their service life is observed. Although a considerable amount of work in the field of chemical modification of silicone resins and composites is done, the potential of

nitrogen-containing surfactants as additives in silicone based coatings is not fully explored. Their application for prolonging service life of silicone coatings seems to be quite promising.

This study is aimed at the development of silicone coatings of increased service life. Composites based on an industrial two component silicone enamel KO-814 and a nitrogen-containing additive Troysperse™ 98C are reported. The effect of Troysperse™ 98C on the dispersion of the aluminum powder in the silicon enamel is studied in details.

EXPERIMENTAL

Materials

Polyphenylsiloxane lacquer KO-85 (Spectr, Russia,) was used (Fig. 1). The lacquer contained 17 % non-volatiles and showed an acid number referring to 2 mg KOH per gram KO-85 (GOST 110066-74). A toluene solvent defined by GOST 14710-78 standard (specific density of 0.866 g/cm³ (at 20°C); toluene content of 99.80 %; nonaromatic hydrocarbon of 0.1 %; benzol of 0.1 %; total sulphur of 0.00005 %) by «Slavneft», Russia was used. The aluminum pigment PAP-2 (GOST 5494-71: active aluminum of 97 %; iron of 0.3 %; silicone of 0.3 %; copper of 0.01 %; manganese of 0.002 %; fatty additive of 2.5 %), a product of «SUAL-PM», Russia was used. Troysperse™ 98C based on a fatty amine (Troy Chemical, USA) was employed as a surfactant.

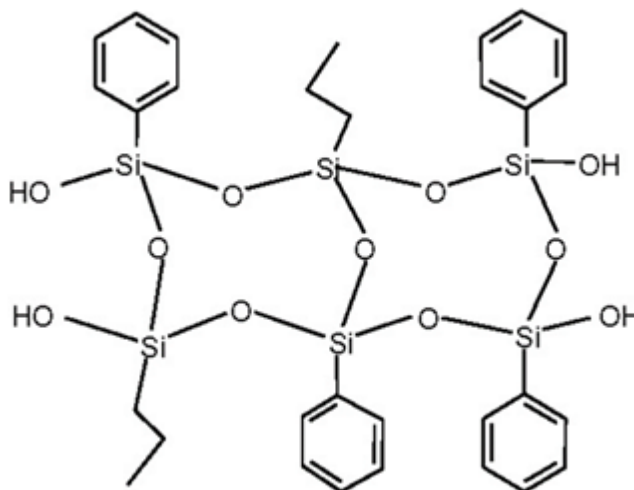


Fig. 1. Chemical structure of the main polymer (silicone): polyphenylsiloxane (lacquer KO-85).

It was known as a highly-effective moistening and dispersing agent, which could be used in a wide area of non-aqueous film-forming systems compatible with aromatic, aliphatic and oxygen-containing solvents.

Procedures

Sample preparation: 8.85 g of enamel (siloxane KO-814) was diluted with 1.0 g toluene. After homogenization, 0.35 g of aluminum pigment were added to the diluted enamel. Troysperse™ 98C was added to the silicone enamel in a quantity corresponding to 0.125 % - 2 % of the weight of the pigment. The enamel suspensions were prepared at (20±2)°C in a hermetic reactor with a volume of (0.2±0.01) L and a filling coefficient of 0.60. The mixture was agitated by a impeller mixer at 300 min⁻¹ for 30 min. After that the samples were taken by pneumodosimeter (a drop volume of 0.02 mL). The samples were kept between object and cover glasses under a static load ($P = 15 \text{ g/cm}^2$) for a minute and left to settle with no load for 72 h, 222 h and 327 h. Then they were characterized by computer-optical microscopy.

Computer-optical microscopy (COM) measurements: A home-made COM-1 device was used. Its calibration was carried out with standard samples. The measurements RSD was less than 1.3 %. The whole surface of the composite films obtained was continuously scanned. The effect of Troysperse™ 98C on the pigment dispersion was estimated on the ground of the changes of fractional composition ($P, \%$) and the average diameter of the pigment particles ($d_{av.}$). The aggregate stability of suspensions was followed by COM scanning at 72 h, 222 h and 327 h following sample preparation.

Determination of the corrosion rate and the petrol absorption: the gravimetric method was used [29]. The coatings were prepared by pouring the composite mixture over defatted steel plates. The latter both sides were covered by the composites developed. Two series of samples were prepared: (1) dried for 24 h at 18°C and (2) exposed to thermal curing for 3 h at 365°C. The steel plates were kept in H_2SO_4 solution (pH 5.5) 96 h at 20°C prior to the corrosion rate determination. The petrol absorption was determined after soaking the samples in gasoline (AI-93) for 168 h at 18°C.

Determination of the coatings fineness: a profilom-

eter TR202 (TIME Group Inc. China) was used. The measurements were made following GB/T3505-2000 standard.

RESULTS AND DISCUSSION

Study on the dispergation effect of Troysperse™ 98C additive

The effect of Troysperse™ 98C additive on the pigment dispersion is illustrated in Fig. 2. The results show that the additive enhances the process of the aluminum powder dispergation in the industrial enamel KO-814. The optimal additive content ranges from 0.25 % to 0.5 %. The further increase of the additive content (from 0.375 % to 2 %) leads to further aggregation of the pigment particles - their average diameter increases up to 7 μm .

The fractional content of the suspensions ($P, \%$) is determined aiming to study the dispergation process mechanism. The results obtained are presented in Fig. 3. The positive effect of Troysperse™ 98C additive on aluminum powder dispergation is verified by the increased content of the fine (a particles diameter < 4 μm) and medium (a particles diameter of 4 μm -20 μm) fractions increases by 22 % and 37 %, respectively, in presence of 0.375 % additive. At the same time the coarse fraction (a particles diameter > 20 μm) decreases by 86 %. Hence, the pigment dis-

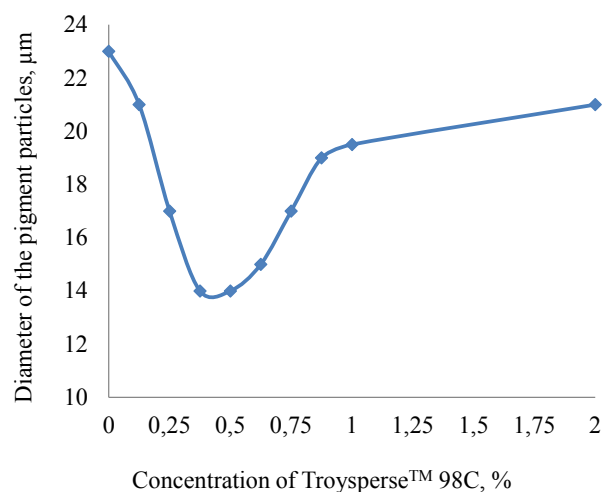


Fig. 2. Influence of Troysperse™ 98C additive content on the average diameter of pigment particles in suspensions of KO-814 enamel.

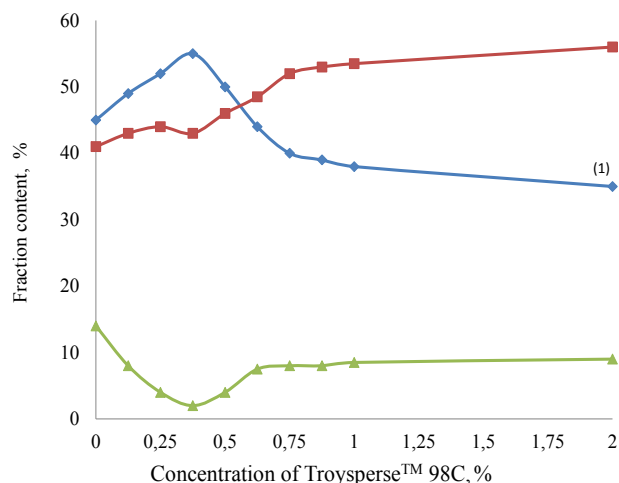


Fig. 3. Influence of Troysperse™ 98C concentration on the fractional content of pigment particles in suspensions of KO-814 enamel: (1) fine fraction (diameter of the particles < 4 μm); (2) medium fraction (diameter of the particles 4 - 20 μm); (3) coarse fraction (diameter of the particles > 20 μm).

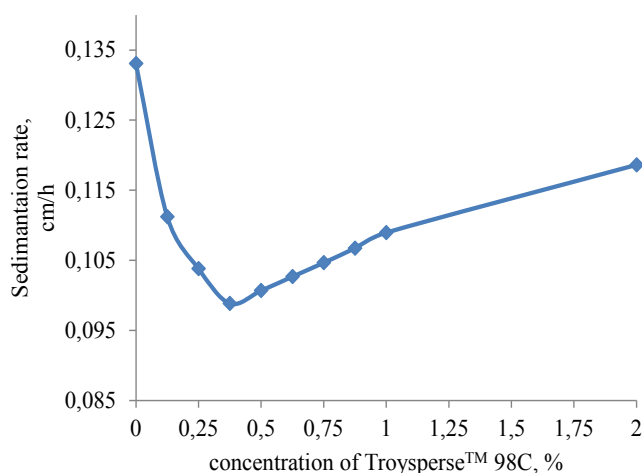


Fig. 4. Influence of Troysperse™ 98C additive on the sedimentation rate of suspensions of KO-814 enamel.

pergation in presence of Troysperse™ 98C additive occurs through destruction of the coarse pigment particles resulting in formation of highly dispersed suspensions.

The sedimentation rate of KO-814 enamel suspensions in the presence of Troysperse™ 98C is shown in Fig. 4. The sedimentation rate and the average diameter of the pigment particles of the studied composite are lower than those of the sample with no additive. As seen

from Fig. 4, the addition of Troysperse™ 98C to silicone enamel increases the aggregative stability of the system by 25 %. The optimum content of the additive amounts to 0.375 %. Its further increase leads to sedimentation rate increase. The observed effect can be explained with the particles size increase as a result of the coagulation. The variation of the additive content from 0.375 % to 2 % results in increase of the sedimentation rate by 20 %. The positive effect of Troysperse™ 98C additive on the suspensions aggregative stability is verified by the results of the computer-optical microscopy study (Fig. 5). They show that the presence of the additive at a strictly defined concentration (0.375 % in respect to the pigment weight) provides not only particles diameter decrease in the course of the dispergation process but also decrease of the pigment particles coagulation, which is a time determined phenomenon. Moreover, the diameter of the pigment particles at the maximum concentration of Troysperse™ 98C additive (2 %) is still lower than that of the particles of the pigment with no additive.

Study of the quality of coatings

The effect of Troysperse™ 98C addition to silicone enamel on the corrosion rate of a steel substrate is studied. Two types of coatings are prepared: (1) by cold curing at 180°C and (2) by thermal curing at 365°C. The obtained results show that 0.625 % of Troysperse™ 98C in silicone enamel decreases the corrosion rate of the steel substrate from 0.648 mm/year (without an additive) to 0.02 mm/year (with an additive). However, relatively high corrosion coefficients are observed for both types of coatings at 10 h holding time. The corrosion rate followed in presence of 0.375 % of the additive is relatively low. It is found equal to 0.05 mm/year - 0.066 mm/year and which is why the concentration pointed above is chosen as the optimal one. The single-layer coatings containing 0.375 % of Troysperse™ 98C show a corrosion rate 3.5 times lower than that of two-layers coatings obtained after thermal curing (the industrial application of the enamel studied). Hence, the studied additive provides an increased protective capacity of the enamel coatings and besides decreases the number of the coating layers. In addition, the effect of the coating thickness and the curing temperature on the corresponding

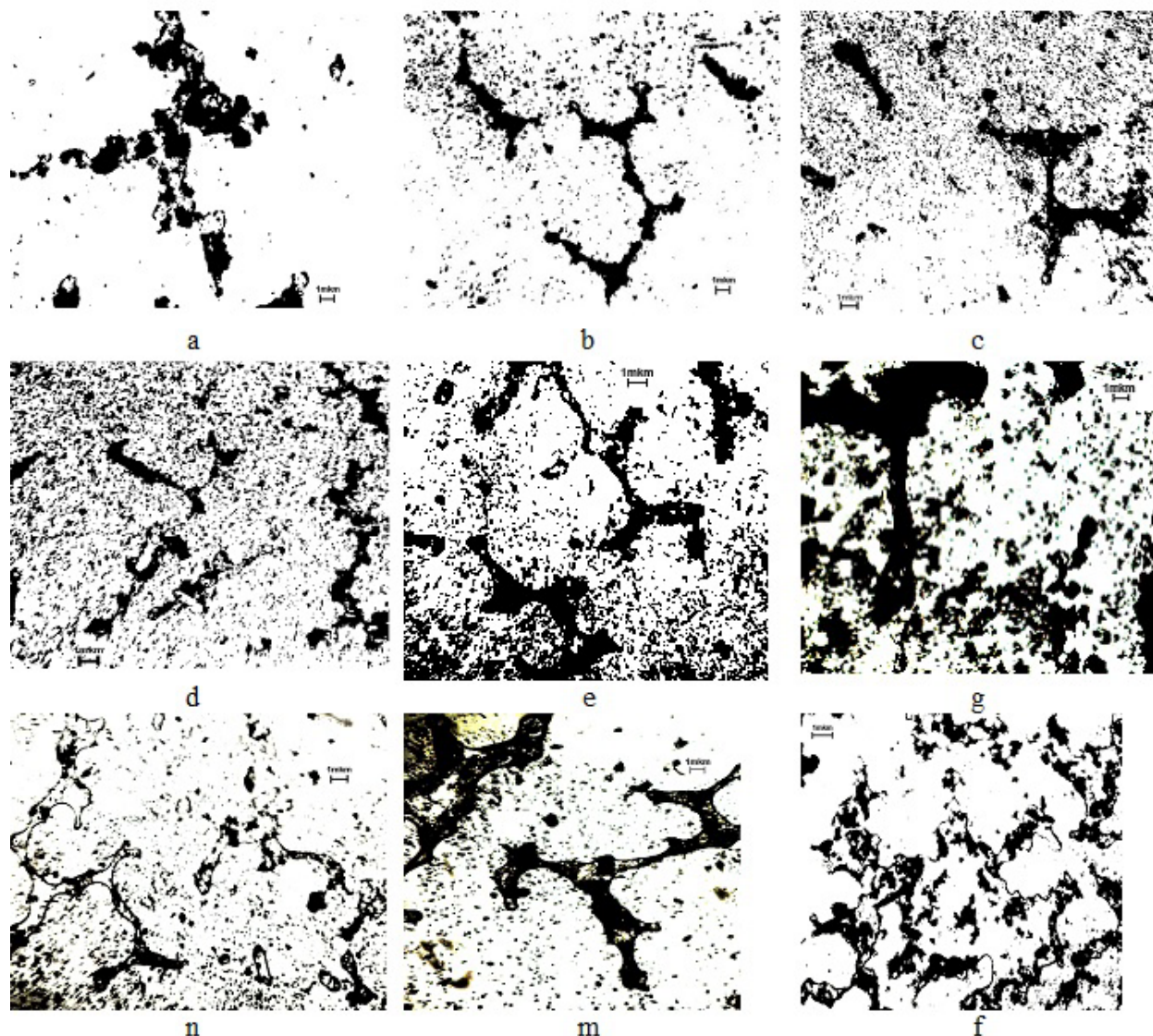


Fig. 5. Micrographics of suspensions with different content of Troysperse™ 98C additive in enamel KO-814: (a) 0 %; (b) 0.125 %; (c) 0.25 %; (d) 0.375 %; (e) 0.5 %; (g) 0.625 %; (n) 0.75 %; (m) 1 % and (f) 2 %.

anticorrosive characteristics is studied. The results show that the coatings treated by hot curing show a smaller rate of corrosion than those prepared by cold curing. The corrosion rate of a single-layer hot cured coating is 0.896 mm/year, while that of two-layered one - 0.233 mm/year. For comparison, the corrosion rates of coatings prepared by cold curing (for 24 h at 18°C) are 0.648 mm/year and 0.173 mm/year for a single-layer and a two-layered coating, respectively. The latter shows better protective properties irrespective of the curing temperature, which is in agreement with the literature data.

Nowadays, the studied enamel KO-184 is often used for staining gasoline cans, tanks, vessels for storage of light hydrocarbons (gasoline, kerosene, etc.). It is interesting to study the effect of Troysperse™ 98C additive on the petrol absorption of the coatings. The results obtained show that the addition of Troysperse™ 98C decreases the petrol absorption of the coatings by approximately 1.5 times from 0.99 g/m² to 0.57 g/m². The values of the petrol absorption of a two-layered hot cured coating with no additive (this enamel industrial use) and a single-layer coating containing 0.375 % of

Table 1. Influence of Troysperse™ 98C additive on the roughness of the coatings (Ra).

Content of the additive, %	R _a , μm
0	0,85
0.125	0,70
0.375	0,54
0.5	0,55
0.75	0,58
1.0	0,60
2.0	0,70

Troysperse™ 98C additive coincide (0.58 g/m²). Therefore, the addition of Troysperse™ 98C to the enamel composition at a concentration of 0.375 % provides skipping of a second enamel layer application.

As stated above KO-814 enamel is usually applied for gasoline and kerosene storage vessels aiming to avoid vigorous heating of the tank walls due to the sunlight and to prevent spontaneous ignition of the stored liquids. To respond to this requirement, the coatings should provide a high degree of light reflection. The gloss of the coatings can be indirectly estimated by their surface roughness. The latter values are listed in Table 1. The results show that the addition of Troysperse™ 98C to the enamel decreases the surface roughness of the coatings by 36% (Ra decreases from 0.85 μm to 0.54μm). The additive effect on the composites water absorption is also studied. It is found that the addition of Troysperse™ 98C in concentrations of 0.25%-0.375% decreases the moisture absorption of the coatings by 43 % (coatings subjected to thermal curing) and 52% (coatings subjected to cold curing).

CONCLUSIONS

The presented results showed that Troysperse™ 98C additive improved the dispergation of the aluminum pigment in the industrial silicone enamel KO-814. The maximum effect was obtained at an addition of 0.375 % of Troysperse™ 98C. The improved dispergation of the pigment in presence of Troysperse™ 98C additive was attributed to the destruction of the coarse particles and the increase of the content of those of a low and a medium size.

The addition of Troysperse™ 98C additive increased the aggregative stability of the suspensions which was manifested by the decrease of the sedimentation rate and the average diameter of the pigment particles. It was demonstrated that at an additive concentration of 0.375 % resulted in decrease of the coagulation of the pigment particles by 85 % and of the sedimentation rate by 25 %.

Compared to the industrial use of KO-184 silicone enamel in the form of a two-layered thermally cured coating, the developed composite containing 0.375 % of Troysperse™ 98C showed: 3.5 times decrease of the corrosion rate of a single-layer coated steel substrate, 1.5 times decrease of the coatings petrol absorption, 36 % decrease of the roughness, 43%–52 % decrease of the water absorption. The results showed that the single-layer coating containing 0.375% of Troysperse™ 98C as an additive showed protective characteristics comparable to those of the two-layer coatings with no additive. Hence, Troysperse™ 98C added to the studied enamel provided to skip the application of a second layer of a coating but with the simultaneous improvement of the protective capacity observed.

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