



Properties of calcined kaolins in anticorrosion paints depending on PVC, chemical composition and shape of particles

David Vesely, Andrea Kalendova*, Miguel Victor Manso

Faculty of Chemical Technology, University of Pardubice, 532 10 Pardubice, Czech Republic

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ABSTRACT

The properties of calcined kaolins in paints were identified in relation to their pigment volume concentration (PVC). The study involved three kinds of kaolin exhibiting different development of the lamellar structure of primary particles as well as different distribution of particle size. Kaolins also displayed varying contents of a glassy phase – mullite and quartz. Prepared model paints were formulated with the increasing content of kaolins, with PVC ranging from 5 to 40% up to a maximum critical pigment concentration (CPVC), i.e. up to a point where $PVC = CPVC$. The properties of paints were tested by means of laboratory corrosion tests in the environments of neutral salt mist and of condensed humidity. It was the aim of the study to identify the effect of kaolin's PVC on the mechanical and corrosion resistance of coatings based on water-borne epoxy resin.

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1. Theoretical

Kaolin and kaolinite belong to the best known and most important minerals. They are ranked among clay minerals. When calcinated at 600 °C, kaolin loses crystal water and changes to amorphous metakaolin [2]. Controlled kaolin calcination results in a product with a porous structure [1]. At a temperature over 900 °C, a glassy phase is created. At a temperature over 1400 °C, the final products of glassy phase calcination are quartz, cristoballite (SiO₂) and mullite (Al₆Si₂O₂₃).

The applications of kaolin in polymeric matrixes exploit its properties, namely a white shade, fire resistance, plasticity, chemical inertness, low abrasion, and low thermal and electrical conductivity [2]. The kaolin contained in paints is used in its natural or calcined form. It is employed in primer coats based on synthetic solvent resins and in water-borne paints [3]. Kaolin is also exploited as an additive preventing the settlement of pigments. The lamellar shape of the kaolin particles enhances the strength of the coating [4]. Particles with a greater diameter exhibit better abrasive properties and abrasion resistance in paints, whereas they do not substantially reduce the whiteness of the product. The finer granularity of kaolin improves the opacity, whiteness, viscosity and thixotropy of paints [5]. Certain kinds of kaolin that meet size requirements [6] may

partially replace TiO₂ and simultaneously maintain the required coverage power of the coating [7].

2. Experimental

2.1. Determination of physical–chemical properties of calcined kaolins

The specific density of calcined kaolins was identified by means of an Autopycnometer Micromeritics 1320 gas density bottle (Micromeritics Instrument Corp., USA). The value of critical volume concentration (CVC) was estimated for tested kaolins.

Sorption of linseed oil on pigment/filler particles was made by means of a pestle-mortar method [8]. Based on our experience as well as literature data, this method (exploiting so called consumption of oil) is one of the most suitable methods for the estimation of CPVC [9]. The consumption or absorption of linseed oil is defined as the weight of oil (in g) needed to reach paste consistency for 100 g of pigment. The estimation of oil consumption by this method consists in formation of the closest groupement of pigment particles with adsorbing layer of a binder; in this particular case the pigment particles are just covered by the thin film of linseed oil. The evaluation is precise as the transition between powder through paste to liquid suspension is sharp. The linseed oil is used due to its suitable viscosity and because it does not contain any solvent.

Determination of pH of aqueous extracts according to standard ISO 789-9 was carried out by means of a WTW pH 320

* Corresponding author.

E-mail address: andrea.kalendova@upce.cz (A. Kalendova).

Set-2 multi-processor pH-meter with a glass measuring electrode (WTW Wissenschaftliche Werkstätten, Germany). Specific electrical conductivity χ ($\mu\text{S cm}^{-1}$) of aqueous extracts was determined in a conductometry way for 10%-aqueous suspension powdery kaolins in redistilled water ($\chi = 4 \mu\text{S cm}^{-1}$). The determination according to standard ISO 787/14 was carried out by a Handylab LF1 (Schott-Geräte GmbH, Germany) conductometer with a Pt conductivity measuring cell. After 28-day exposure, the aqueous 10%-powder kaolin suspensions were filtered and their final pH value (pH_{28}) and specific electrical conductivity value (χ_{28}) were measured. The so prepared filtrate (extract) was then used to determine corrosion loss (X_{corr}).

Determination of particle size and the distribution of kaolin particle sizes was identified by means of Mastersizer 2000 (Malvern, Instruments Ltd., UK) that is able to measure the distribution of particles sized from 0.01 to 2000 μm .

The specific surface of powdery kaolin particles BET (g m^{-2}) was performed on Micromeritics ASAP 2000 Physic/Chemisorption unit (Micromeritics Instrument Corp., USA).

Annealing-induced weight loss in kaolins at 1000 °C was determined from the difference between the weight of an annealed sample and of a dried sample; the weight loss caused by annealing at 1000 °C is a percentile value representing the ratio of annealing-induced weight loss to the weight of a dry sample.

The content of water-borne substances (i.e. water-extractable substance) was performed on the gravimetric determination of the substance that can be extracted cold (W_{20}) or hot (W_{100}). The content of acid-soluble substances ($A_{\text{C}20}$) was performed on the gravimetric determination of the substance that can be extracted cold at a laboratory temperature in diluted hydrochloric acid (1 wt% HCl). The content of alkali-soluble substances ($A_{\text{L}20}$) was performed on the gravimetric determination of the substances that can be extracted cold in the diluted solution of sodium hydroxide (1 wt% NaOH). The content of soluble substances in acids and alkali environment was calculated in the same manner as the content of water-soluble substances.

The shape of pigment particles was investigated by means of a Jeol-JSM 5600 LV scanning electron microscope (Jeol, Japan).

2.2. Structure identification

X-ray diffraction spectra were measured on an X'pert PRO MPD 1880 X-ray diffractometer (PANalytical, The Netherlands). The diffraction data were evaluated by means of the X'Pert programs (X'Pert HighScore Plus Software version 2.1b and X'Pert Industry Software version 1.1 g); phases are identified with the aid of an ICDD PDF2 diffraction data database.

X-ray fluorescent analysis was carried out on a Philips PW 1404 X-ray spectrometer (Pinhills Microsystems, UK) equipped with a UniQuant program that facilitates semi-quantitative determination of the contents of 74 elements.

2.3. Determination of steel corrosion loss of calcinated kaolins

Determination of steel corrosion loss in the aqueous extracts of powdery kaolins belongs was performed on used 20 mm \times 50 mm \times 0.5 mm steel panels that were weighed with an accuracy of ± 0.001 g. The test panels were immersed in 10%-aqueous extracts of the tested kaolins; exposure time was 28 days; steel corrosion loss in the aqueous extracts of kaolins (K_m) was related to steel weight loss in pure water X_{corr} [10].

2.4. Preparation of paints containing calcinated kaolins

2.4.1. Formulation and preparation of model paints containing the tested kaolins

The calcinated kaolin-based fillers in concern were applied to the solution of medium-molecular, water-borne epoxy resin *ChS Epoxy® 200 V 55* (Spolchemie, Ústí nad Labem, Czech Republic) hardened with polyamine hardener *ChS Telalit® 180* (Spolchemie, Ústí nad Labem, Czech Republic). This binder is designed for the paints ensuring protection of metal materials.

The paints were formulated in relation to PVC so that the kaolin content in the paints was 5, 10, 20, 30 and 40 vol.% (until reaching a value where $\text{PVC} = \text{CPVC}$). The following kaolins were tested: *Alumo-Fill KK 80* (kaolin type-A), *Alumo-Fill KK 15* (kaolin type-B), and *Dorka-Fill Pand* (kaoline type-C) – manufactured by Benefit GmbH, Germany. The paints were dispersed in a Dispermat CV pearl mill (WMA Getzmann GmbH, Verfahrenstechnik, Germany) filled with glass ballotini of 2 mm in diameter. After dispersing, glass balls were separated out of pigmented paint by filtration using stainless sieve. To reduce the formation of foam, an antifoaming additive *EFKA 2526* (Ciba Specialty, Switzerland) was added in an amount of 0.5% along with *EFKA 4550* (Ciba Specialty, Switzerland) in an amount of 0.5%, whereas the latter improves the soaking and dispersion of kaolins. Immediately before the application of coatings on test panels, the pigmented paints were homogenized with polyamine hardener *ChS Telalit® 180* at a ratio of 100:27 units of the hardener.

2.5. Preparation of test panels

To carry out the corrosion tests, resins were coated by an applicator on 152 mm \times 102 mm \times 0.8 mm steel panels (Q-Panel, UK). Mechanical tests of the paints were performed on 152 mm \times 75 mm \times 0.6 mm panels. The thickness of dry paints (DFT) was measured with a Minitest 110 magnetic thickness gauge with a F16 type probe (Elektrophysik, Germany) in accordance with ISO 2808. A 7 mm long test cut was made on the bottom of all steel panels for the purpose of completing corrosion tests. To determine the surface hardness of the paint film, paints were also applied onto 180 mm \times 100 mm \times 0.5 mm glass panels. The drying and conditioning of samples on the test panels took place under standard conditions in an air-conditioned laboratory (temperature: 20 °C, relative humidity: 50%) and lasted 6 weeks.

2.6. Determination of the physical–mechanical properties of the paints containing calcinated kaolins

Determination of the impact resistance of the coating according to ISO 6272 was performed on the reverse of the test panel, using Impactester Elcometer 1615 (Elcometer, Germany). The result of the test of the impact resistance of paints identifies the height (cm) at which a free falling 1000 g weight dropped on a test panel still resists damage. Determination of the resistance of the coating against cupping according to ISO 1520 was performed with a Cupping tester (Pragolab a.s., Czech Republic). The result of the test of the resistance of the coating against cupping is the distance of the cupping of the test panel applied with a coating (in mm) at which the painted panel was first deformed with a steel ball (20 mm in diameter). Determination of the resistance of the coating during bending (Cylindrical bend test) ISO 1519 was carried out with Cylindrical Mandrels Elcometer 1506 (Elcometer, Germany). The result of this test of the resistance of the coating during bending indicates the diameter of the cylindrical mandrel (in mm) on which the cohesion of the paint film was not disturbed during the bending of the painted steel panel. Determination of the degree of coating adhesion by a lattice method according to ISO 2409 was carried out

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