

Study on dispersion stability and coating properties from water-thinnable acrylic compositions

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Introduction

The issues regarding environmental protection cause that both the paint and varnish industries as well as consumers appreciate the technologies which reduce or eliminate the application of organic solvents in coat materials [1, 2].

Nowadays, waterborne acrylic coating compositions belong to the most commonly used pro-ecological paints and varnishes. Ease of modification, pigmentation and application as well as an outstanding chemical resistance, resistance to atmospheric conditions and diversified mechanical features make them so popular [2÷6]. Such types of coat compositions already harden at room temperature. Drying time of a coat is dependent upon humidity and air temperature [7].

Nowadays, the application range of products based on acrylic dispersions is very wide, starting from paints and plasters for the construction industry, through wood paints and varnishes to paints for metal protection and decorative systems [8, 9]. It is the wood that is popular and highly valued construction material, due to the ease of processing, strength and natural features. Wood, being the material of organic origin, is susceptible to harmful effects of factors resulting in its destruction. To prevent the loss of aesthetic values and mechanical properties of wood, surface protection in the form of varnish products is applied. The selection of a proper varnish composition is dependent upon the operational conditions [10, 11].

The varnish products applied on the wood are both solvent and waterborne compositions. Contrary to the common opinion, the strength of the varnish coat is determined by a type of used resin, and not the solvent. Therefore, there are tendencies towards using acrylic varnishes that do not change wood tint, do not require a prime coat and are very well spread on the surface. Considered as harmless, they enable the product to be used immediately after drying and hardening of the coat [12].

A field of waterborne varnish products is currently characterised by continuous changes. The tendencies towards using ecological resources in varnish products are strong. Advanced auxiliary varnish products are necessary for avoiding difficulties in developing new waterborne products. That is why demanding requirements have to be met. In case of wood varnish products, their role, that is, the protection of painted construction elements and the maintenance of their properties is primary. An adequate selection of wood varnish composition is likely to guarantee an increasing share of waterborne products on the environmentally friendly market [13÷15].

This paper presents the results from testing waterborne acrylic dispersion and finished coats of waterborne acrylic resins formed with a nonionic surfactant and an organic thickener. The obtained varnish compositions were tested for their stability and application properties; and their resistance to water (leaching) and fluids was evaluated. The characteristics of tested waterborne acrylic paints and the mechanical strength of the obtained coats were described in this paper.

Experimental part

Materials and auxiliary substances

- acrylic dispersion Alberdingk AC 2523, core-shell type, chemical composition: acrylic copolymer, content of solid parts 47-49% wt., pH 7.5 – 8.5, viscosity at a temperature of 23°C 500-4000 mPas, density at a temperature of 23°C 1.04 g/cm³, (manufacturer Alberdingk Boley GmbH)
- nonionic surfactant (SAA), ethoxylated unsaturated fatty alcohol, Rokanol O-18, chemical formula C₁₇H₃₄O(CH₂CH₂O)₁₈H, ethoxylate number 18, molecular weight 1013.0 g/mol (manufacturer Chemical Plant Rokita S.A. in Brzeg Dolny).

The physical and chemical properties of the applied surfactant in an aqueous solution: critical micelle concentration, c_{CMC} 0.3·10⁻³ mol/dm³, surface activity G 84.3·10⁻³ mNm·cm³/mol, size of SAA micelles in water, (IV) 0.0017 cm³/g.

- organic polyurethane thickener, Acrysol RM 825, modified polyurethane block copolymer-based hydrophobic emulsion, components: water 75%, content of solid substances 25%, viscosity at a temperature of 23°C 1000-2500 mPas, specific gravity 1040 kg/cm³, (manufacturer Brenntag Polska Sp. z o.o.).

Preparing waterborne acrylic compositions

Waterborne acrylic compositions consisted of 100 cm³ of acrylic dispersion solution AC 2523 containing 2, 4, 5 and 10 % wt. of surfactant in relation to the quantity of polymer and constant quantity of thickener - 0.3 g. Also, the compositions without the thickener were prepared.

The aqueous compositions were prepared at room temperature by introducing the constant quantity of acrylic dispersion with a specified quantity of surfactant and the constant quantity of thickener as well as without it. Dispergation was conducted using a laboratory homogeniser of IKA-ULTR-TURRAX T-25 type with IKA-S25N-18G stirrer at a rotary velocity of ca. 2000 rpm. The obtained compositions were poured out into sedimentation cylinders and left there for 7 days. They were also applied on metal, wood and glass substrates, and then hardened at a temperature of 20°C. The obtained coats were subjected to further tests.

Methods of testing waterborne compositions and acrylic coats

The properties of SAA aqueous solution and aqueous acrylic dispersions were tested using the methods of sedimentation, tensiometry and viscosimetry.

The properties of an applied surfactant were calculated or experimentally determined. The chemical name, formula and molecular weight of SAA were provided by its manufacturer. The following measurements were made: critical micelle concentration, surface activity, intrinsic viscosity. These values were determined in accordance with the methods presented in the paper [16]. The

properties of five nonionic and ionic surfactants were determined to choose a suitable agent for the tests. The determination of critical micelle concentration (c_{CMC}) was a decisive parameter. The lower its value is, the higher ability to reduce the surface tension of solution is and at the same time, the higher activity. The application of Rokanol O-18 exhibiting the lowest value of c_{CMC} from the tested group was to provide the satisfactory stability of the dispersion system.

Polymer content in the disperse phase was determined by the gravimetric method [17]. The surface tension was measured using du Nouy tensiometer manufactured by the Kruss company. The Ubbelohde type viscometer was used to measure the viscosity of the stable phase of dispersion. The measurement consisted in determining the time of liquid outflow from the tank through a capillary tube. The outflow time for the pure continuous phase (water) and dispersion were determined prior to viscosity calculation. On the basis of the Poiseuille equation and on the assumption that the density of diluted stable phase of dispersion equals water density, the value of specific viscosity was calculated. It allowed us to calculate the reduced viscosity. Intrinsic viscosity (IV), which is expressed by the Huggins' equation, was determined from the graphical relationship between the reduced viscosity and polymer concentration in the tested dispersion. The Huggins constant describing the range of influence of polymer molecule surface and the dispersion medium in a given system was calculated. The detailed methodology of measuring viscosity and calculating the discussed quantities is presented in the papers [18, 19]. The test results are shown in Table I.

Testing functional properties of hardened coats

The obtained coating systems were subjected to physical and chemical as well as physical and mechanical tests. The coat compositions were applied with a brush (acc. to PN-79/C-81514) on glass, wood and degreased steel plates. Then, they were seasoned for 7 days until the film was completely dry at room temperature. The following properties of coats of varnish compositions were tested:

- thickness using the magnetic method (magnetic pen tool measuring magnet detachment force from the steel surface of plate μm) [20]
- pendulum relative hardness of films on steel plates (acc. to PN-EN ISO 1522)
- adhesion to steel substrates (acc. to PN-EN ISO 2409) using cross-cut test with Peters' circular knife
- scratch resistance of varnish films (acc. to PN-EN ISO 1518),
- resistance of varnish coats to cupping (acc. to PN-EN ISO 1520), coat gloss (acc. to PN-EN ISO 2813)
- varnish films resistance to impact acc. to Du Point (PN-EN ISO 6272),
- film resistance to water by calculating percentage change in a sample mass after the exposure in distilled water [18]
- resistance of varnish coats to fluids (acc. to PN-EN ISO 2812).

The preparation of samples for testing coat resistance to fluids was based on applying two coats of the tested varnish on a dark coloured form (beech veneer) or beech fabric. Such a prepared material was subjected to drying at room temperature for 7 days and then to physical and chemical testing involving the impact of various fluids for fixed periods. The coat destruction was evaluated according to the valid scale [21].

- softening point of varnish coats using the Höppler consistometer [18].

The measurement consisted in a uniform heating of the sample and reading the temperature increase and swing of the sensor pointer every specific range, corresponding to the indentation depth of the indenter into the tested sample. The reading was carried out every 2°C. The measurement results were expressed as a graph of deformation dependence on the temperature increase from which the softening point of a given sample or its range was determined using the graphical method. For this purpose, tangents over the first inflexion

point of curve were drawn. The intersection point of both tangents was the point which, projected on the temperature axis, determined the softening point. The range of elastic and viscous flow, whose overflow is related to the flow of individual polymer chains, is also determined from deformation curves.

Test results and their review

Testing sedimentation stability of acrylic compositions

SAA and polyurethane thickener-stabilised aqueous acrylic compositions were subjected to sedimentation tests. These tests were used to determine the size of acrylic dispersion particles expressed by intrinsic viscosity (IV), the Huggins constant and surface tension. Stable and durable systems were tested. Adding the thickener to the systems caused neither the formation of sediment nor dispersion separation. The bigger the sizes of polymer molecules in the solution are, the higher the intrinsic viscosity is [22].

Table I

Results from sedimentation, viscometry and tensometry testing of Alberdingk AC 2523 varnish composition with an additive of ethoxylated unsaturated fatty alcohol Rokanol O-18 and the use of Acrysol MR 825 organic thickener and of compositions without the thickener

No.	Type of acrylic composition	Polymer content in disperse phase g/cm^3	Intrinsic viscosity $IV, \text{cm}^3/\text{g}$		Huggins constant k_H		Dispersion surface tension, mN/m	
			New dispersion	Stable dispersion phase	New dispersion	Stable dispersion phase	New dispersion	Stable dispersion phase
1	AC 2523 100 cm^3 /Rokanol O-18 2% wt.	0.26	0.0261	0.026	16.1	16.1	41	39
2	AC 2523 100 cm^3 /Rokanol O-18 4% wt.	0.24	0.0245	0.0275	14.5	15	40	41
3	AC 2523 100 cm^3 /Rokanol O-18 5% wt.	0.23	0.026	0.028	13.2	13.2	38	41
4	AC 2523 100 cm^3 /Rokanol O-18 10% wt.	0.19	0.0275	0.028	11	12	39	41
5	AC 2523 100 cm^3 /RM 825 0.3g/Rokanol O-18 2% wt.	0.26	0.0285	0.0294	9.8	11.5	42	45
6	AC 2523 100 cm^3 /RM 825 0.3g/Rokanol O-18 4% wt.	0.24	0.029	0.0299	8.7	10.5	44	46
7	AC 2523 100 cm^3 /RM 825 0.3g/Rokanol O-18 5% wt.	0.23	0.0367	0.034	5	5.8	46	49
8	AC 2523 100 cm^3 /RM 825 0.3g/Rokanol O-18 10% wt.	0.20	0.0408	0.041	3.5	4.2	49	50

Data presented in Table I demonstrate that intrinsic viscosity is increasing along with adding the thickener to the aqueous dispersion. On the other hand, the analysis of the Huggins constant indicates the presence of hydrophilic interactions in the system between the surface of dispersion particles and the medium. Such interactions are testified by the positive value of the Huggins constant. A lower Huggins constant

corresponds to the systems with a higher content of SAA. The durability of the dispersion system can be increased by adding the thickener. The test results of thickening aqueous dispersions using polyurethane thickener, presented in Table 1, show unequivocal increase in intrinsic viscosity of the tested systems. The surface properties of tested systems without the thickener are the same because the surface tension values are very close to each other. After adding the thickener, a significant increase in surface tension was observed in the systems. Such systems contain dispersion particles coated with the adsorption layer of the surfactant and separated from each other by the thickener macro-chains. It can be generally stated that the size of dispersion particles and lyophilisation of their surfaces increased when the thickener was added to the dispersion system, which was shown by the increase in the Huggins interaction constant value. The phenomenon of lyophilisation is advantageous and favourable for improving the system stability. It provides the prolongation of a finished product life time.

Functional properties of obtained coats

The subsequent tests concerned the determination of properties of coats obtained from the acrylic compositions. All obtained coatings were characterised by a smooth surface and did not show any surface defects. They were transparent. Due to the application method, the thickness of coats was within the range from 30 to 35 μm .

Coat adhesion to the steel substrate was very good. All coats were characterised by first - the highest, degree of adhesion to the substrate, which was demonstrated by relatively smooth edges of cuts without any chips in the corners of formed squares.

Also, very good scratch resistance of coats was found. The value of a parameter, at which a surface scratch or a scratch to the substrate depth is made, is changing along with adding the thickener to the tested varnish composition (Fig. 1). The coat resistance to scratching is relatively greater in the systems containing polyurethane thickener in comparison with the compositions without the thickener. Therefore, minor loss in coat resistance to scratching was only observed for coats containing 4.5 and 10% wt. of the surfactant only.

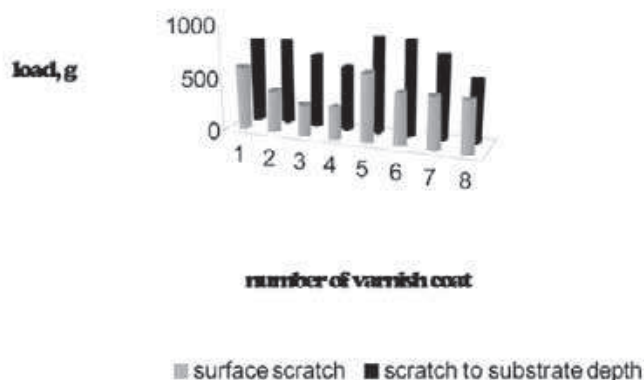


Fig. 1. Acrylic coat resistance to scratching (loading value) determined acc. to PN-EN ISO 1518, sample no. as shown in Table 1

Varnish coat resistance to deformation was checked by conducting drawability testing of these coats. For all tested samples, at first the substrate fracture was observed at the piston depth of ca. 6 mm, and then the fracture of varnish coat at the depth of ca. 7 up to 8 mm. On the grounds of the above observations, the obtained films were stated to have satisfactory elasticity.

Acrylic composition-based coats indicated the satisfactory resistance to water. It was confirmed by results from testing the percentage loss in a film after its extraction in water. The presence of thickener did not have any influence on the film loss after water extraction. The increase in decrement could be observed at increasing content of the surfactant. Therefore, only surfactant is supposed to be removed. The thickener, which is embodied into the coat structure, is not removed (Fig. 2).

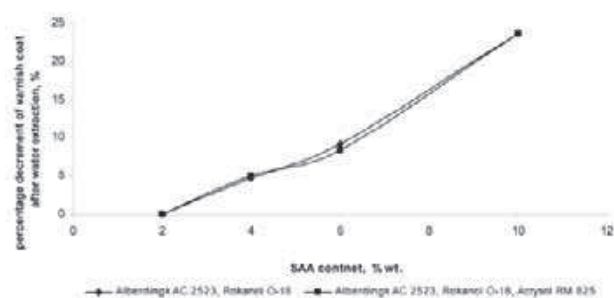


Fig. 2. Percentage decrement of varnish coat after water extraction dependent on SAA content in varnish composition

The tests of coat resistance to impact were very positive. For all tested samples, this type of resistance was the highest (50 cm).

The obtained coats were characterised by very high gloss. The addition of thickener did not have a significant impact on the gloss change of the coats. The gloss value was decreasing along with the increased content of the surfactant in the system (Fig. 3).

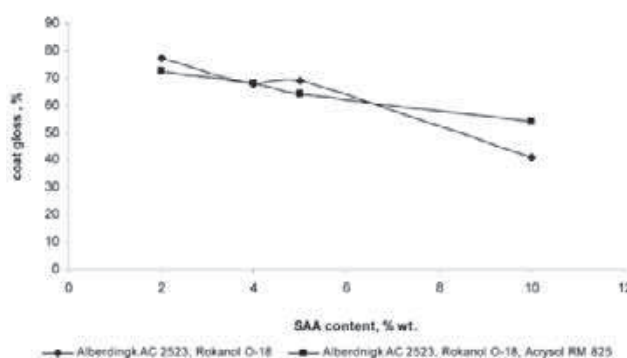


Fig. 3. Gloss of acrylic coats depending on SAA content in varnish composition

All coats on the wood substrate were characterised by high resistance in tests on coat resistance to fluids (Tab. 2). By analysing the level of coat changes, neither acetone nor hot water was found to induce any changes in the appearance of coats. The coats stabilised with a surfactant in a quantity of 2, 4, 5 and 10% wt. demonstrated satisfactory resistance to spirit, wine and oil. Minor changes in gloss and colour, only visible from a certain angle of viewing, were observed on the coats of these compositions. They were also resistant to cleaning milk products such as Cif. The tests on resistance to tea and coffee were the poorest. A significant change in gloss and colour was observed on the coats with higher content of SAA in the varnish composition. The addition of polyurethane thickener into the tested compositions reduced the resistance of obtained coats to the cleaning milk products such as Cif, coffee, tea and edible oil. Nearly none of the tested samples exhibited visible damage of the varnish coat.

Table 2

Resistance of varnish coat to fluids

Fluid type	Exposure time	Degree of coat changes							
		AC 2523, Rokanol O-18,				AC 2523, Rokanol O-18, Acrysol RM 825,			
		2%	4%	5%	10%	2%	4%	5%	10%
Acetone	10"	5	5	5	5	5	5	5	5
Acetone	10'	5	5	5	5	5	5	5	5
Cif	16 h	5	5	5	5	4	2	2	2
Spirit	16 h	4	4	4	4	4	3	3	3
Boiling water	(poured)	5	5	5	5	5	5	5	5
Wine	16 h	5	5	4	4	5	4	4	2
Coffee	16 h	3	3	2	2	5	4	3	3
Tea	16 h	4	4	3	3	5	5	3	3
Water	24 h	5	5	5	5	5	4	3	3
Edible oil	24 h	5	4	4	4	5	3	3	3

The dependence of sample deformation on the temperature was determined from thermal and mechanical tests conducted on obtained films (Fig. 4). The film softening point was determined from the obtained curves. The temperature range of film formation was dependent on the thickener presence and SAA content in dispersion.

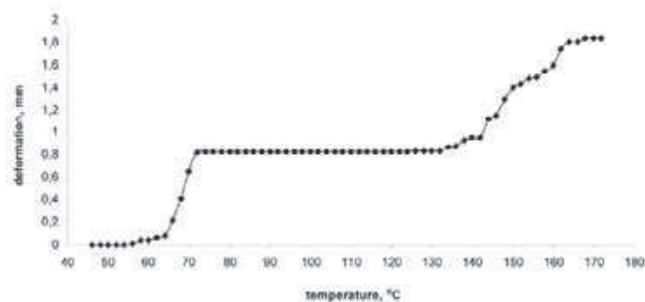


Fig. 4. Dependence of Alberdingk AC 2523 film sample with the additive of SAA, 10% of Rokanol O-18 and Acrysol RM 825 thickener on temperature

A softening point demonstrates the mobility of polymer segments, that is, the displacement of these segments towards the flow. The decrease in softening point in the polymer-thickener system indicated that the thickener particles were smaller than polymer molecules and were entering between polymer chains causing plasticisation. The increase in softening point was caused by polymer chain stiffening; then, the chain was blocked by the thickener particles. It is an undesirable effect. Big macro-particles of the thickener are probably inhibiting the displacement of its chains under the impact of film softening.

The perfect system is the one, in which the thickener enters between the polymer segments and separates them. Such a thickener acts as a lubricant facilitating the flow of the polymer chain. Thus, it should be emphasized that the increase in softening point indicates the presence of an immiscible system. The thickener is occluding on the surface of polymer molecules and makes the displacement of polymer chains difficult. The drop in softening point shows that the thickener is entering between the segment layers of polymer chains and facilitates the flow.

Table 3

Properties of coats obtained from AC 2523 acrylic composition with Rokanol O-18 and polyurethane thickener additives

No. of acrylic composition	Range of coat softening points °C	Description of coat appearance and observations
1	64-176	colourless, elastic, glossy, even
2	42-150	transparent, smooth, glossy, even
3	38-152	transparent, even, smooth
4	52-130	colourless, uneven
5	66-166	transparent, smooth, glossy, hard, with significant sheen
6	38-138	transparent, smooth, with quite low sheen
7	26-146	colourless, smooth, uneven
8	62-176	transparent, smooth, hard, uneven

The conducted tests demonstrate that the range of deformation curve is dependent on the content of surfactant and thickener in dispersion. In case of aqueous acrylic dispersion AC 2523 stabilised with Rokanol O-18, the temperature initiating the softening process of the film is higher for SAA-stabilised systems without the thickener. It ranges from 38 to 66 °C. The addition of thickener to dispersion results in clear drop in film softening point. It ranges from 26 to 52 °C and decreases along with the increase of SAA content in the system. For Rokanol O-18 content of 4 and 6% wt., this temperature is 38 and 26 °C, respectively. For 10% content of SAA in the system with the thickener, the increase in softening point by 26 °C was observed. Such rapid increase in plasticisation indicates the formation of associated molecules strongly bounded to each other or the formation of crystalline structures (Tab. 3).

Summary

The presented results show that it is easy to compose stable and waterborne acrylic paints using nonionic surfactant Rokanol O-18 and polyurethane thickener Acrysol RM 825. The presence of non-ionic SAA influences the reduction in surface tension and lyophilisation of the systems without significant changes in other properties. Lyophilisation of the dispersion system and formation of bigger particles is caused by adding polyurethane thickener. This indicates the presence of associated molecules and probable formation of thick adsorption layers on their surfaces.

The coats of described waterborne acrylic compositions are characterised by satisfactory adhesion to metal, glass and wood substrates. The tests on coats of acrylic compositions indicate that the coats with the thickener have better physical and mechanical properties, particularly manifested in positive test results for scratching, in comparison with the coats without the thickener. For applications requiring significant resistance to fluids, the composition with a lower content of SAA and the thickener are preferred as they exhibit much greater resistance to coffee or tea. The composition with 4, 5 and 10% content of SAA with the thickener had insufficient resistance to cleaning milk product Cif, and simultaneously the coats containing only SAA had very good mechanical properties. Coat resistance to leaching is dependent on SAA content in the compositions. From the tested systems, the lowest softening point was observed for compositions with 5% content of SAA and the thickener.

The obtained results will be the base for selecting parameters to develop a varnish composition suitable for coats protecting steel and wood substrates.

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BASF Science in Action Industry visit

Bradford, United Kingdom, 3th October 2012

This is a ChemNet event for 16-18 year old students. See science in action at BASF, the chemical company. BASF began manufacturing at the Bradford site over 50 years ago and today boasts one of the largest and most productive single site chemical plants in the UK. The session will start with an introduction which includes company history, product demonstrations and a taster of polymer chemistry and how they are so important for the products BASF manufacture. Health, safety and the environment are of great importance for BASF staff and they will be showing you how they minimise risk and their environmental impact on society. You will hear from a range of people working at the BASF site who will talk about their career routes and show how diverse working in the chemical industry can be. Finally, we will go on a site tour and follow the formulation and chemistry of liquid dispersion polymers and see how they are incorporated into BASF's personal care products.

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