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EFFECT OF DISPERSIONS ON THE VAPOUR PERMEATION RESISTANCE OF ACRYLIC SEALANTS

A.A. Shikunova, O.I. Nikolaeva

Anastasia Alekseevna Shikunova, Student; **Olga Ivanovna Nikolaeva**, Candidate of Chemical Sciences, Associate Professor
Ivanovo State University of Chemistry and Technology, Ivanovo, Russia. *shikun-nastya@yandex.ru; olgynja1975@mail.ru*

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Abstract. *Currently, acrylic sealants are widespread due to their environmentally friendly materials, ease of use, and low cost. Their compositions are constantly being improved. It allows ones to improve their quality and reduce the cost by selecting new substances for their manufacture. We obtained acrylic sealants with changing polymer base. The authors used acrylic and styrene acrylic dispersions obtained by emulsion polymerisation. The basis for sealants production is the mechanical mixing of the components and their further dispersion in the Dispermat VLOK dissolver. The main parameter to be determined is the vapour permeability resistance of the material layer. According to research results, the nature of the polymer base, degree of its bonding, type and amount of the filler used affected on vapour permeability of the sealing material. Moreover, increasing in film former temperature, viscosity, and pH causes increasing in tensile strength. By comparing the sealant samples on the basis of dry residue, we found increasing of the Shore hardness in samples with lower % index.*

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Introduction

Nowadays, polymer-based sealants are important in many of industry. The main use is in construction and automotive industry for sealing windows, various seams, etc. Meanwhile, sealants are used for a wide range of household applications, such as sealing wall openings, door frames, etc. Acrylic sealants are highly demanded on the global market [1]. Indeed, they are the most environmentally friendly materials; simple in use and cost-effective ones. It makes them accessible to wider target groups. They are produced on the basis of aqueous dispersions of acrylic (co)polymers, which do not contain solvents and any toxic substances. Therefore, they are not harmful to human health [2-6].

The dispersion is the most productive method for obtaining acrylic sealants. Most of the properties of the finished product depend on the dispersion – physical and mechanical properties, vapour permeability, water absorption, etc. Acrylic dispersions are obtained by emulsion polymerisation [7, 8]. One of the main acrylic sealants parameters is vapour

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permeability. It is used to assess the suitability of the sealant formulation for sealing joints. Vapour permeation resistance indicates how much water vapour passes through the surface of the sealant per unit of time. Any vapour permeability resistance parameters can be obtained by adjusting the thickness of the layer. According to the requirements for the inner layer, the vapour permeability resistance value should be at least 2.0 (m^2·h·Pa)/mg.

Nowadays, there is quite wide range of sealants based on acrylic dispersions on the market. However, vapour permeation resistance tests on known sealant brands showed low values in the range of 0.24-0.25 (m^2·h·Pa)/mg. These results ensured the development of a proprietary product with a given value of vapour permeability resistance, and the study of factors affecting this parameter.

Experimental part

To obtain sealants, we used a basic composition, the variable part was the polymer base (Table 1).

Table 1. General description of the sealant formulation

Component name	Content, %
Dispersions 1-14	38.44
Functional additives	0.85
Filler	54.91
Plasticiser	5.00
Thickening agent	0.80
Total	100.00

Styrene-acrylic (SA) and acrylic (A) dispersions by various domestic manufacturers were chosen as the polymer base. Tables 2 and 3 summarise the characteristics of these dispersions.

Table 2. Characteristics of industrial SA dispersions

Indicator	Number								
	1	2	4	7	9	10	11	12	14
Dry residue, %	50	50	59	57	50	57	50±1	50±1	49-51
MFFT, °C	5-7	0	<0	<0	20	<0	0	0	20
T_{gt}, °C	no	-7	-8	-6	32	-6	-31	-18	23
Brookfield viscosity, mPa·s	100-500	4,000-11,000	<500	150-1,200	8,000-15,000	500-1,500	500-1,500	500-3,000	5,000-16,000
pH	7.5-9.0	8	6.5-7.5	6.0-9.0	7.0-9.0	8	7.5-8.5	5.0-8.0	7.0-9.0
Particle size, nm	130	100	250-400	no	<100	no	150	no	no

Table 3. Characteristics of industrial A dispersions

Indicator	Number				
	3	5	6	8	13
Dry residue, %	60	69	48	65	62±1
MFFT, °C	<0	0-4	17	0-4	5-23
T_{gt}, °C	no	-43	13	-40	7
Brookfield viscosity, mPa·s	550-950	150-500	200-900	no	40-250
pH	7.5-8.5	6.5-8.5	7.5-8.5	4.0-5.0	6.4-7.2
Particle size, nm	200-400	no	no	no	no

The production of both styrene-acrylic and acrylic sealants was based on mechanical mixing of components and their further dispersion in a Dispermat VLOK dissolver with a capacity of 2 litres (motor power – 1000 W; adjustable working speed 20-6000 rpm) [9].

We put the weighed suspensions of dispersion (1-14) of functional additives (defoamer and dispersant) into the bowl; stirred them for 15 min at low speeds of the cutter and the dissolver shaft. We charged a certain amount of filler into the bowl after obtaining a homogeneous mass. At the same time, we kept the shaft speed in the lower range and increased the cutter speed depending on the viscosity of the reaction medium until the formation of a funnel was achieved. We stirred the substance for 30 minutes from the moment the funnel was formed until the sealant was homogeneous. Next, we added the thickener to the bowl at a shaft speed of 25-30 rpm. We switched on the cutter at full power and mixed for 40 minutes. The viscosity of the sealant began to increase strongly during this period. It causes its heating. Indeed, there was necessity to monitor the temperature up to 35 °C. We added plasticiser at a shaft and cutter frequency of 10-15 rpm and further mixing for 15 minutes. We discharged the prepared sealant out of the dissolver and conveyed it to the cartridges for storage.

We determined the vapour permeation resistance of sealants in accordance with GOST 25898-2012; tensile strength properties of sealants were determined according to GOST 21751-76; hardness of sealants was determined by Shore A on a hardness tester according to GOST 263-75.

Results and discussion

We used acrylic and styrene acrylic dispersions to produce sealants. A number of SA dispersions presented in Table 2 differ by their glass transition temperature (T_{gt}). It is an indicator for assessing styrene content in the dispersion: the higher is this value, the more styrene is in the dispersion, respectively. However, dispersions differ in viscosity and particle size.

The series of dispersions A, presented in Table 3, also differ in terms of the minimum film forming temperature (MTP) and T_{gt}. These indices show possible monomers in the dispersion composition and side chains of the polymer macromolecule. As the chain length of the macromolecule increases, the T_{gt} of the polymer increases; the hardness and relative elongation of the films grow due to the increasing degree of crystallinity of poly(meth)acrylates. Products obtained by copolymerisation of 'soft' monomers with low T_{gt} value (butyl and ethylhexylacrylate) with 'hard' monomers with high T_{gt} value (butyl and methyl methacrylate) are usually used for sealants. This combination allows ones to obtain copolymers with T_{gt} 0-40 °C [10-12].

Vapour permeability resistance is one of the main parameters assessing the suitability of the developed formulation of sealing material for joint sealing [13]. The essence of the method of its determination is to develop a stationary flow of water vapour through the sample under study and to determine this flow intensity. Indeed, for this test two samples of material with a square cross-section with a side size of 100 mm and a thickness of 5 mm were prepared. First, the water vapour flux density through the sample was determined for all sealants *g* (Tables 4, 5).

Table 4. Water vapour flux density through the sample for sealants (S) containing SA dispersion

Number	1S	2S	4S	7S	9S	10S	11S	12S	14S
Δτ, h	24								
A, m^2	0.002								
Δm, mg	105.5	125	145	126.5	111	134	246	200	141
g, mg/(h·m^2)	1,850.2	2,192.2	2,543.0	2,218.5	1,946.7	2,350	4,305	3,516	2,472

Table 5. Water vapour flux density through the sample for sealants (S) containing A dispersion

Number	3S	5S	6S	8S	13S
Δτ, h	24				
A, m^2	0.002				
Δm, mg	33.5	190.5	85.5	325.5	90
g, mg/(h·m^2)	587.5	3,340.9	1,499.5	5,708.5	1,578

By Tables 4 and 5, water vapour flux density through the sample *g* for sealant 3S containing acrylic dispersion is the lowest one. According to the requirements for the inner layer, the vapour permeability resistance value should be at least 2.0 (m^2·h·Pa)/mg. Having determined the vapour permeation resistance of sealants with all dispersions, we have established that for sealant 3S this index is the highest – 2.38 (m^2·h·Pa)/mg (Tables 6, 7).

Table 6. Vapour permeation resistance of sealants (S) based on A dispersions

Number	3S	5S	6S	8S	13S
Rv, (m^2·h·Pa)/mg	2.38	0.41	0.78	0.28	0.87

Vapour permeation resistance in a number of SA dispersions grows with increasing T_{gt}, except for sealants 10S, 11S, and 12S (Table 7). This, in turn, is obviously directly related to the styrene content in the dispersion.

Table 7. Vapour permeation resistance of sealants (S) based on SA dispersions

Number	1S	2S	4S	7S	9S	10S	11S	12S	14S
Rv, (m^2·h·Pa)/mg	0.67	0.53	0.48	0.56	0.63	0.58	0.3	0.38	0.55

According to the research results, vapour permeability of the sealant material is influenced by the nature of the polymer base, its degree of bonding, and the type and amount of filler.

The absence of necessary data on physical and mechanical properties of acrylic sealants in the literature caused the conducting of experiments on their determination. The essence of the methods for determining tensile strength at break and relative elongation at break consisted in stretching samples at a constant rate at a given temperature before rupture, measuring the strength, elongation of the sample at break, and calculating the relative residual strain after rupture. Samples were cut in the form of blades 115 mm long, 19 mm wide and 2 mm thick. The Shore hardness was determined by measuring the resistance of the material to indentation. The test sample was a 6 mm thick parallel plane washer. Tables 8 and 9 summarise the results of the physical and mechanical properties of all sealants.

Table 8. Physical and mechanical properties of sealants (S) based on SA dispersions

Indicator	Number								
	1S	2S	4S	7S	9S	10S	11S	12S	14S
Tensile strength, N/mm^2	2.43	4.73	0.18	1.77	4.83	0.6	2.96	1.12	3
Relative elongation at break, %	136	71	103	145	54	148	116	118	94
Shore hardness	61	73	4	52	58	32	54	46	76

Table 9. Physical and mechanical properties of sealants (S) based on A dispersions

Indicator	Number				
	3S	5S	6S	8S	13S
Tensile strength, N/mm^2	1.85	0.67	5.68	2.04	2.6
Relative elongation at break, %	62	39	37	164	82
Shore hardness	66	35	70	48	60

Corresponding to the data obtained (Table 8), an increase in tensile strength was observed with increasing minimum film formation temperature, viscosity and pH. It is probably related to the increase in styrene content in the dispersion. Relative elongation at break compared to the strength values decreases.

Comparing the samples in terms of dry residue, we conclude that the sealants with lower % have better strength properties. Shore hardness also increases for samples with lower % of dry residue in the dispersions.

Conclusions

Hence, since the indicator 'resistance to vapour permeation' is one of the main ones for sealing materials used for sealing seams of various assemblies. Therefore, quality assessment of the developed formulation was determined by this indicator. The impact of dispersion type, their physical and mechanical properties on the vapour permeation resistance of sealants was investigated. As a result, the optimum sealant formulation, 3S, was selected. It had higher vapour permeation resistance of 2.38 (m^2·h·Pa)/mg.

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