

Influence of Adhesion Additives on the Grip of Bitumen to Stone Material and Water Resistance of Asphalt Concrete

R.Kh. Salakhov^{1,4*}, A.P. Kenzhegaliyeva¹, M.S. Abdikarimov¹, B.A. Mansurov², A.O. Elshibaev³,
A.U. Nugmanova³, S.Zh. Ashimova³, B.B. Teltaev³, A.R. Khamidi⁵

¹Institute of Combustion Problems, Bogenbay Batyr Str., 172, Almaty, Kazakhstan

²Abai Kazakh National Pedagogical University, Dostyk ave., 13, Almaty, Kazakhstan

³JSC «Kazakhstan Highway Research Institute», 2a, Nurpeisova str., Almaty, Kazakhstan

⁴LLC «In. Prometheus», 20, Toretai str., Almaty, Kazakhstan

⁵Kazan National Research Technological University, 68 Karl Marx str., Kazan, Russia

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Abstract

The primary condition for obtaining high-quality asphalt concrete is good adhesion of bitumen to mineral material, which is ensured by good wetting and chemical interaction of the components. The solution to this problem is achieved by using adhesive additives with surface-active properties that enhance adsorption and chemisorption processes on the surface of the mineral material. This paper presents the results of an experimental study to determine the effect of adhesive additives on the adhesion of bitumen to stone material. For this work, bitumen of the BND 100/130 brand with adhesive additives “Wetfix Be”, Amdor-9 and AlfaDob were used. The elemental and phase compositions of the stone material of 10 quarries of the Republic of Kazakhstan were determined using X-ray fluorescence and X-ray phase analyses. The elemental composition showed the content of oxides: CaO, MgO, Fe₂O₃, Al₂O₃, and oxides: SiO₂, K₂O, Na₂O, CO₂ in minerals. Based on the results of the X-ray phase analysis, the percentage mineralogical composition of stone materials was determined and the total silicon dioxide was calculated based on the chemical formula corresponding to certain minerals.

1. Introduction

Nowadays, the mechanisms of adhesive interaction in “bitumen–filler” systems have not been fully elucidated. A great deal of scientific work has been devoted to this problem. Understanding the adhesion mechanisms between aggregates and bitumen is essential to ensure the strength and durability of asphalt pavements [1–5].

One effective way to improve bitumen adhesion to the stone material is the use of adhesive surfactants, with the dissolution of a small number of which surface tension is greatly reduced. Due to the asymmetric nature of the surfactant molecules and the presence of radicals and polar groups on

both sides, a sufficiently large force field is created with its dipole moment. Based on the charge of the polar group, ionogenic surfactants can be anionic and cationic. As a rule, the stone material can be acidic or alkaline type. The acidic types are stone materials with a high content of silicon, while the alkaline types are those containing carbonates. When wetting the surface of stone materials with bitumen, the surfactant molecules are oriented by the polar functional group to the surface of the mineral material, and the radicals – to the bitumen film. In this case, the interaction of electric charges of the opposite sign occurs, which contributes to the adhesion of bitumen to stone material [1].

The interaction of bitumen film with mineral components in asphalt concrete should be strong and not be peeled off from the surface during physical, chemisorption, and filtration processes [1, 6–8].

*Corresponding author.

E-mail: Salakhov.r.x@gmail.com

Cationic surfactants are often used in asphalt concrete mixtures. This is due to the fact that the groups of substances contained in the bitumen, including asphaltenes, provide sufficiently strong adhesion to the surface of the stone material.

Cationic surfactants work most effectively with stone materials only from acidic rocks, forming chemisorption compounds such as silicatamines. In addition, there are rocks with a mixed surface charge.

The different chemical nature, composition, and mechanism of the adhesive additives allow a flexible selection of surfactants for any asphalt concrete production conditions [2, 4, 8–12].

The purpose of this work is to evaluate the effect of adhesive additives on bitumen by boiling and to determine the water resistance of modified asphalt concrete. The study revealed effective adhesive additives that improve bitumen properties during long-term or periodic humidification of compacted asphalt concrete mixtures by AASHTO T 283-14.

The following tasks were set to achieve the goal: by spectral methods to determine the elemental and phase compositions of the stone material of 10 quarries of the Republic of Kazakhstan, to divide the stone material into acid and basic types, to evaluate the effect of adhesive additives on bitumen by boiling and to determine the water resistance of asphalt concrete with adhesive additives, to identify the optimal percentage composition of adhesive additives from the mass of bitumen.

2. Materials

Three of the most famous additives in Kazakhstan were used for research:

– «Amdor-9» is a cationic surfactant that provides a high degree of adhesion of road bitumen with various mineral materials, including granite gravel and sand, having increased acid properties, production of LLC «Uralchimplast – Amdor» (Russia);

– «Wetfix Be» is a liquid cationic adhesive additive specially developed for hot asphalt concrete mixtures, which require high-temperature stability of the mixture of the cationic surfactant, produced by Akzo Nobel (Sweden);

– «AlfaDob» is a liquid cationic additive in bitumen based on organic esters and vegetable oils to improve the adhesion of bitumen to rock material, both acidic and basic rocks, produced by UnidAs Group LLP (Kazakhstan).

Viscous oil road bitumen grade BND 100/130

produced by Pavlodar Petrochemical Plant LLP and stone materials in fractions of 10–20 mm from 10 different quarries in Kazakhstan were used for the research: crushed stone “Shetpe” quarry in Mangistau region, crushed stone “Mugalzhar” quarry in Aktobe region, crushed stone “Volgodon” quarry in Akmola region, crushed stone “A & Ya service” quarry in Zhambyl region, crushed stone “Bereke Building” quarry in Turkestan region, crushed stone “Vostok-Asphalt” quarry in East Kazakhstan region, Crushed stone of “As-Income” quarry in Zharma district, crushed stone of “Ozen-tas” quarry in Almaty region, Talgar district, Aktas village, crushed stone of “KazTasProm” quarry in Almaty region, Kapchagai district, crushed stone of “Novo-Alekseyevsk” quarry in Almaty region, Talgar district, Avat village.

3. Research methods

3.1. X-ray fluorescence analysis

The elemental composition of samples of mineral materials was determined on the device SRM-25 (X-ray multi-channel spectrometer). For this purpose, the fine powders (particle size not exceeding 100 μm) of each sample were prepared in a quantity of 10 g at the RETSCH MM 301 vibration mill. The elemental analysis results are presented in the oxide form as is generally accepted for rocks and minerals in Table 1.

3.2. X-ray phase analysis

The mineralogical composition of the samples was determined by X-ray phase analysis. The images were taken with D8 Advance (Bruker), $\alpha\text{-Cu}$, tube voltage 40 kV, and current 40 mA. Processing of the obtained diffractogram data and calculation of interplanar distances were carried out using EVA software. Sample interpretation and phase search were carried out by Search/match using the PDF-2 Powder Diffractometer Database (Tables 2 and 3).

3.3. Determination of adhesion of binder to stone material by boiling method according to ST RK 1808

The experiments were carried out in boiling water for 30 min (Table 4). The surface of the crushed stone grains was examined and the quality of the adhesion of the bituminous binder to the crushed

Table 1
Elemental composition of mineral materials from 10 quarries

No	Deposit	Content, %												
		Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	TiO ₂	MnO	Fe ₂ O ₃	SO ₃	*I	Total
1	Berekebuilding	4.49	4.54	14.34	48.26	<0.10	0.20	9.20	1.54	0.18	19.87	0.45	2.93	100.00
2	Novo-Alekseevsk	4.16	0.30	13.65	73.00	<0.10	5.21	0.94	<0.10	<0.10	2.35	<0.10	0.39	100.00
3	Mugalzhar	0.80	0.77	3.11	29.50	<0.10	0.89	34.87	0.13	<0.10	1.74	<0.10	28.19	100.00
4	As-Income	2.73	4.06	14.30	53.52	<0.10	0.58	16.44	0.87	0.17	10.09	1.19	6.05	100.00
5	Ozentas	3.77	1.19	13.51	66.11	<0.10	4.32	2.96	0.47	0.27	6.25	<0.10	1.04	100.00
6	KazTasProm	3.57	0.21	12.22	74.48	<0.10	5.65	0.57	0.52	<0.10	2.29	<0.10	0.49	100.00
7	Vostok-Asfalt Shetpe	2.55	3.84	14.09	44.96	<0.10	0.65	16.11	1.12	<0.10	10.05	1.01	5.62	100.00
8	Shetpe	2.42	1.40	1.11	63.69	<0.10	1.87	7.54	0.25	<0.10	5.94	<0.10	5.78	100.00
9	A & Yaservice	2.61	1.63	9.84	52.50	<0.10	2.65	18.10	0.34	0.33	4.58	<0.10	7.42	100.00
10	Volgodon	2.96	3.00	14.72	58.87	<0.10	0.71	7.45	0.67	0.14	9.50	<0.10	1.98	100.00

* Impurities: K, Ba, Rb, TR, Y, Sr, U, W, Mo, Ga, Li, Zn, Sc.

stone was assessed according to the degree of preservation of the binder film under ST RK1808 “Bitumens and bituminous binders. Methods for determining adhesion, binder”.

3.4 Determination of water resistance according to ASTM D1075-11

The preparation of asphalt concrete mixtures was carried out by weighing the estimated number of raw materials, heating them in a drying cabinet to the required temperature, mixing them in a laboratory paddle mixer, and introducing mineral powder and bitumen with an adhesive additive. As a result, an asphalt concrete mixture was obtained following the requirements of ST RK 1225 type B, which has the following component composition – crushed stone of fraction 10–20 is divided into fractions of 10–15 mm and 15–20 mm: gravel crushed stone of fraction 15–20 mm – 12%; crushed stone from gravel fraction 10–15 mm – 13%; crushed stone from gravel fraction 5–10 mm – 21%; elimination of crushed stone from gravel – 49%; mineral powder – 7%; bitumen BND 100/130 – 4.8%.

The temperature of the finished asphalt mix was 150–155 °C. Asphalt concrete samples with an adhesive additive were compacted at a temperature of 125 °C and a load of 20.7 MPa, kept in an oven at a temperature of 60 °C for 24 h, then immersed in water for 24 h at a temperature of 60 °C, and as a result, the strength was determined compression.

The strength of asphalt concrete is characterized by the tensile strength of standard cylindrical samples tested at temperatures of 20 and 50 °C at a sample deformation rate of 3 mm/min. The essence of the method is to determine the load required to destroy the sample under given conditions.

The compressive strength (R_{str} , MPa), is determined by the formula (1):

$$R_{str} = \frac{N}{A} \cdot 10^{-2} \quad (1)$$

where: N – breaking load, N; A – initial cross-sectional area of the sample, cm²; 10^{-2} – conversion factor in MPa.

The calculation of the water resistance coefficient (K_B) taking into account the loss of durability of asphalt concrete due to wetting, is carried out according to the formula (2):

$$K_B = \frac{R_{str}^B}{R_{str}^{20}} \quad (2)$$

where R_{str}^B is the compressive strength at temperature (20±2) °C, water-saturated samples in vacuum, MPa; R_{str}^{20} is compressive strength at temperature (20±2) °C of specimens before water saturation, MPa.

Table 2
Results of X-ray phase analysis of the studied stone materials

#	Mineral	Compound Name	Formula	S-Q
1	As-incom	Albite	Na(AlSi ₃ O ₈)	30.10%
		Quartz,	SiO ₂	27.20%
		Clinochlore-1MIIB, ferroan	(Mg,Fe) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈	26.30%
		Calcite	Ca(CO ₃)	11.30%
		Pargasite, potassian	(Na,K)Ca ₂ (Mg,Fe) ₄ Al(Si ₆ Al ₂)O ₂₃	5.10%
2	Shetpe	Quartz, syn	SiO ₂	75.60%
		Albite	Na(AlSi ₃ O ₈)	11.00%
		Calcite	CaCO ₃	7.50%
		Clinochlore-1MIIB, ferroan	(Mg,Fe) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈	3.90%
		Muscovite	H ₂ KAl ₃ Si ₃ O ₁₂	2.10%
3	Mugalzhar	Albite	Na(AlSi ₃ O ₈)	42.40%
		Diopside	(Ca _{0.95} Fe _{0.05})(Mg _{0.81} Fe _{0.19})Fe _{0.026} (Si ₂ O ₆)	19.40%
		Clinochlore, ferroan, oriented	(Mg,Fe,Al) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈	17.80%
		Quartz	SiO ₂	9.10%
		Glagolevite	NaMg ₆ (Si ₃ Al)O ₁₀ (OH,O) ₈ ·H ₂ O	6.90%
		Calcite	Ca(CO ₃)	4.40%
4	KazTasProm	Quartz, syn	SiO ₂	56.40%
		Albite	Na(AlSi ₃ O ₈)	28.40%
		Microcline, intermediate	KAlSi ₃ O ₈	8.20%
		Muscovite	H ₂ KAl ₃ Si ₃ O ₁₂	3.10%
		Calcite, syn	CaCO ₃	2.20%
		Clinochlore-IIb-2	Mg ₃ (Mg ₂ Al)((Si ₃ Al)O ₁₀)(OH) ₂ O ₃	1.60%
5	Novo-Alekseevsk	Quartz, syn	SiO ₂	57.50%
		Albite, calcian, ordered	(Na,Ca)Al(Si,Al) ₃ O ₈	23.40%
		Microcline, intermediate	KAlSi ₃ O ₈	11.30%
		Indialite, syn	Mg ₂ (Al _{3.9} Si _{5.1} O ₁₈)	3.20%
		Clinochlore-1MIIB, ferroan	(Mg,Fe) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈	2.50%
		Muscovite-2M1	KAl ₂ (Si,Al) ₄ O ₁₀ (OH) ₂	2.20%
6	Berekebuilding	Calcite	Ca(CO ₃)	52.60%
		Quartz, syn	SiO ₂	38.70%
		Albite	Na(AlSi ₃ O ₈)	6.00%
		Microcline, intermediate	KAlSi ₃ O ₈	2.70%
7	Ozentas	Quartz	SiO ₂	56.30%
		Albite, calcian, ordered	(Na,Ca)Al(Si,Al) ₃ O ₈	18.20%
		Microcline	K(AlSi ₃ O ₈)	15.50%
		Clinochlore-1MIIB, ferroan	(Mg,Fe) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈	4.10%
		Indialite, syn	Mg ₂ (Al _{3.9} Si _{5.1} O ₁₈)	3.50%
		Muscovite-2M1, heated	KAl ₂ (Si,Al) ₄ O ₁₀ (OH) ₂	2.40%
8	Volgodon	Albite, disordered	Na(Si ₃ Al)O ₈	27.10%
		Quartz, syn	SiO ₂	22.40%
		Ferro-tschermakite	Ca ₂ Fe ₃ Al ₂ (Si ₆ Al ₂)O ₂₂ (OH) ₂	21.50%
		Anorthite, sodian	(Na _{0.45} Ca _{0.55})(Al _{1.55} Si _{2.45} O ₈)	18.40%
		Clinochlore-1MIIB, ferroan	Mg,Fe) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈	7.20%
		Muscovite 2M1, syn	KAl ₂ Si ₃ AlO ₁₀ (OH) ₂	3.40%
9	A & Yaservice	Quartz	SiO ₂	69.80%
		Albite	Na(AlSi ₃ O ₈)	12.30%
		Calcite	CaCO ₃	6.60%
		Microcline, intermediate	KAlSi ₃ O ₈	4.90%
		Dolomite	CaMg(CO ₃) ₂	2.60%
		Muscovite-2M1	KAl ₂ (Si,Al) ₄ O ₁₀ (OH) ₂	1.60%
		Clinochlore-1MIIB, ferroan	(Mg,Fe) ₆ (Si,Al) ₄ O ₁₀ (OH) ₈	1.40%
		Indialite, syn	Mg ₂ Al ₄ Si ₅ O ₁₈	0.70%
10	Vostok-Asfalt	Quartz	SiO ₂	32.70%
		Alcite	CaCO ₃	21.10%
		Albite, calcian	(Na _{0.75} Ca _{0.25})Al _{1.26} Si _{2.74} O ₈	15.10%

3.5. Determination of accelerated water resistance of asphalt concrete with adhesive additive according to AASHTO T 283-14

The test was carried out by conditioning compacted specimens by saturation with water, subjecting them to a freezing cycle at -18 ± 3 °C for 24 h and a subsequent soaking cycle in a hot water bath at 60 °C for 2 h, then the specimens were tested for indirect tensile strength by loading specimens at a constant speed and measuring the force required to break the specimen.

Steel load bars are placed between the specimen and the bearing plates. A load is applied to the specimen by compressing the carrier plates together at a constant rate of 50 mm per minute until breaking.

The tensile strength factor is calculated as the ratio of the strength of the sample after freezing to the strength of the sample before freezing. This coefficient is considered as water resistance.

4. Results and discussion

Research on this work was carried out in the laboratory of KazdorNII, where the optimal percentage of adhesive additives from the mass of bitumen was experimentally selected.

Due to differences in the chemical composition and structure of the crystal lattices of minerals, active areas on the surface of the stone material are characterized by a different nature. As a result, the adhesion in the “bitumen-mineral” systems will depend to the greatest extent on the nature of the aggregate. Table 1 shows the elemental composition of minerals to determine the correlation be-

tween cohesion and the chemical composition of the rock material.

A characteristic increase in the content of calcium (CaO), magnesium (MgO), iron (Fe_2O_3), and aluminum (Al_2O_3) oxides in the mineral leads to an increase in adhesion and is in good agreement with the data of other publications [13–15]. The adhesion mechanism is explained by the formation of water-insoluble salts of organic acids of alkali and alkaline earth metals on the interfacial surface. The opposite effect is observed with an increase in the content of oxides: SiO_2 , K_2O , Na_2O , CO_2 and is explained by the formation of water-soluble compounds on the interfacial surface.

Based on the results of X-ray diffraction analysis, a division of the rock material into acid or main type was carried out (Table 2).

Table 2 shows the percentage mineralogical composition of stone materials of 10 quarries, based on which the calculation of the total silicon dioxide was carried out, based on the chemical formula corresponding to certain minerals. The content of quartz and total silicon dioxide in stone materials is given in Table 3.

Increasing the acidity of the mineral, as a rule, leads to a decrease in adhesion to bitumen. Table 3 (depending on the content of total silica) shows that crushed stone of the quarries “Mugalzhar”, “As-Income”, “Bereke Building”, “Vostok-Asphalt” refer to the basic rock and presumably should have the highest coefficient of adhesion.

Table 4 shows comparative data on the adhesion of stone material with bitumen, modified with adhesive surfactants, obtained by boiling according to ST RK1808 (on a 5-point scale).

Table 3
The content of quartz and total silicon dioxide in stone materials

Deposit	Quartz content, %	The content of total silicon dioxide, %	Material type
Berekebuilding	38.7	41.5	ultrabasic
Mugalzhar	9.1	53.0	basic
Vostok-Asfalt	32.70	45.8	basic
As-incom	27.2	55.8	medium
A & Yaservice	69.8	73.8	acidic
Shetpe	75.6	77.9	acidic
Ozentas	56.3	78.1	acidic
Volgodon	45.2	81.3	acidic
KazTasProm	54.6	73.2	acidic
Novo-Alekseevsk	57.50	74.1	acidic

Table 4
Results of testing the adhesion of stone material with bitumen modified with adhesive surfactants, obtained by boiling according to ST RK1808

No	Deposit	Adhesion (score) of the original bitumen	Additive content by weight of bitumen, %	Adhesion, (score) Amdor-9	Adhesion, (score) Alfadop	Adhesion, (score) Wetfix-BE
1	Crushedstone “Shetpe” quarry	0	0.2	3	3	4
			0.4	4	3	3
			0.6	4	3	4
2	Crushedstone “Mugalzhar” quarry	2	0.2	35	3	3
			0.4	5	4	4
			0.6	4	4	5
3	Crushedstone “Volgodon” quarry	0	0.2	4	3	3
			0.4	2	3	4
			0.6	3	3	4
4	Crushed stone “A & Ya service” quarry	0	0.2	2	2	3
			0.4	3	3	4
			0.6	0	3	4
5	Crushed stone “Bereke building” quarry	3	0.2	3	1	3
			0.4	0	2	2
			0.6	2	3	3
6	Crushed stone “Vostok-Asfalt” quarry	0	0.2	4	2	3
			0.4	1	3	3
			0.6	2	4	3
7	Crushed stone “As-Income” quarry	3	0.2	0	1	3
			0.4	0	2	5
			0.6	2	3	5
8	Crushedstone “Ozentas” quarry	0	0.2	2	1	3
			0.4	2	2	4
			0.6	2	3	4
9	Crushedstone “KazTasProm” quarry	0	0.2	2	1	3
			0.4	3	2	3
			0.6	2	0	3
10	Crushed stone “Novo-Alekseevsk” quarry	0	0.2	3	1	3
			0.4	4	2	4
			0.6	2	3	5

Table 4 shows that the crushed stone from 3 quarries “Mugalzhar”, “As-Income”, “Novo-Alekseevsk” have good adhesion – 4 and 5 points, which agrees well with the calculation of the total silica content in the stone material and the mineral belonging to the basic type. Acidic type minerals with bitumen showed an unsatisfactory result of 0 (zero) points.

The adhesive additive “Wetfix-BE” showed universal qualities both with the basic type of mineral material and with the acidic type – from 3 to 5 points (on a 5-point scale).

According to the average index of bitumen adhesion to stone material (percentage of the adhesive additive from the mass of bitumen: 0.2%, 0.4% and 0.6%), we can conclude that the best

results for the adhesive additive “Wetfix-BE” for all formulations. Worst of all, from a concentration of 0.2% by weight of bitumen, the adhesive additive “Alfadop” showed itself. Additive “Amdor-9” showed an average result in all experiments.

In order to improve the physical and chemical properties of asphalt concrete using the adhesive additives “Wetfix-BE”, “Alfadop” and “Amdor-9”, laboratory water resistance tests according to ASTM D1075-11 and AASHTO T283-14 were carried out. The essence of the method is to evaluate the degree of drop in compressive strength of the samples after exposure to water. In accordance with the requirements of these GOSTs the coefficient of water resistance (KB) of asphalt concrete must not be lower than 0.75. Increasing the value above 0.75 increases the water resistance of the asphalt concrete and hence the quality of the pavement. The results of testing asphalt concrete with adhesive additives for water resistance according to ASTM D1075-11 are shown in Fig. 1.

Figure 1 shows that the coefficient of water resistance for type B fine-grained dense asphalt concrete prepared from crushed stone from different quarries according to ASTM 1075-11 should be at least 0.75. It can be seen that the control samples of asphalt concrete without an adhesive additive do not pass the water resistance coefficient determined according to ASTM 1075-11. When adding the adhesive additives Amdor-9, Alfadop and Wetfix-BE in an amount of 0.2 to 0.6% of the bitumen weight, all samples began to meet the requirements of ASTM 1075-11 (0.75-0.85 with a requirement of at least 0.75).

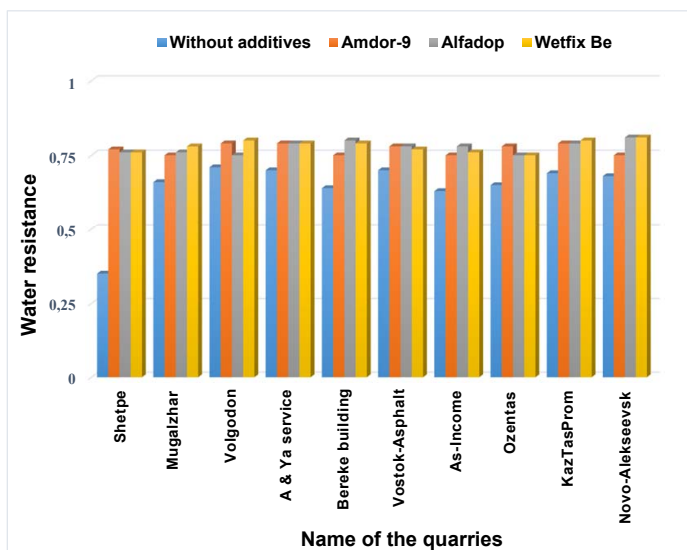


Fig. 1. The coefficient of water resistance of asphalt concrete according to ASTM 1075-11.

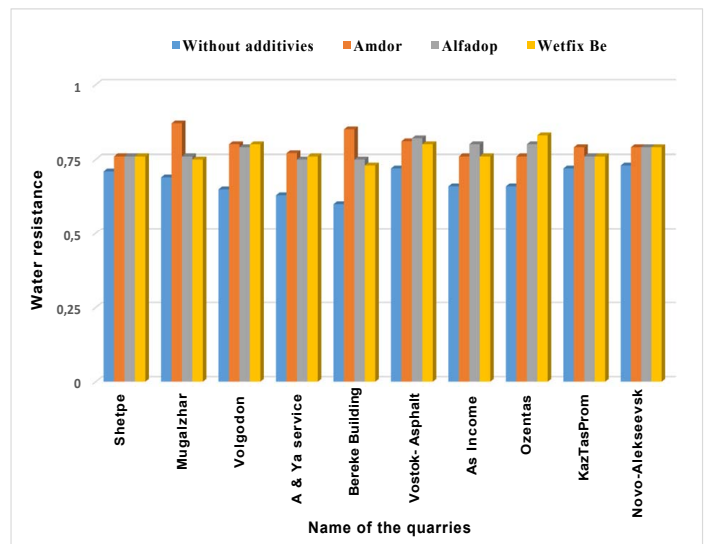


Fig. 2. The coefficient of water resistance of asphalt concrete according to AASHTO T283-14 for different quarries.

The results of the AASHTO T283-14 water resistance tests for asphalt concretes with three adhesive additives are shown in Fig. 2.

Figure 2 shows that the control asphalt concretes without an adhesive additive also do not pass the water resistance coefficient determined by the AASHTO T283-14 method. By adding the adhesive additives “Amdor-9”, “Alfadop” and “Wetfix-BE” in an amount of 0.2 to 0.6% of the bitumen weight, the asphalt concrete became requirements of AASHTO T283-14.

5. Conclusion

The results of the influence of adhesive additives “Amdor-9”, “AlfaDob”, “Wetfix Be” on the adhesion of bituminous binder to stone material from various quarries of the Republic of Kazakhstan are presented.

X-ray fluorescence and X-ray phase methods were used to determine whether minerals in oxide form belonged to the acidic or basic type. Significant contents of oxides CaO, MgO, Fe₂O₃ and Al₂O₃ lead to an increase in adhesion, and oxides SiO₂, K₂O, Na₂O, CO₂ – to a decrease in the adhesion of bitumen to rubble. Such data are explained by the formation of insoluble and water-soluble salts of organic acids of alkali and alkaline earth metals on the interfacial surface.

The results of testing the adhesion of stone material with bitumen modified with adhesive additives obtained by boiling are in good agreement

with the content of silicon dioxide (SiO₂) in stone material and the affiliation of the mineral to the basic or acidic type.

The results of water resistance testing of type B fine-grained dense asphalt concrete with adhesive additives showed an increase in the water resistance coefficient in accordance with the requirements of AASHTO T283-14.

Bitumen modified with the adhesive additive “Wetfix-BE” with a concentration of 0.4% and 0.6% by weight of the bitumen showed the best adhesion to the stone material, showing good chemisorption chemical interaction with the entire surface of the stone material, thereby ensuring long-term water resistance of the bitumen film and consequently the durability of the pavement.

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