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Interaction process on the phases interface “bitumen – dispersed phase from cement stone”

Процессы взаимодействия на границе раздела фаз «битум – дисперсная фаза из цементного камня»

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Key words: phase interface; bitumen; filler; adsorption; adsorption-solvate layer; thickness; interaction

Ключевые слова: граница раздела фаз; битум; наполнитель; адсорбция; адсорбционно-сольватный слой; толщина; взаимодействие

Abstract. The results of the study of the effect of the main properties of mineral fillers from cement stone with different degrees of hydration, phase composition and pore volume characteristics on the properties of the bitumen-mineral binder are presented. Dependencies of the influence of mineral filler on the basis of cement stone on the basic parameter of the structure of bitumen-mineral binders-the thickness of the adsorption-solvate layer of bitumen, which was established using the rheological method. Analysis of the empirical data obtained shows that bitumen chemically interacts with mineral fillers obtained from cement stone. It is proved that during the interaction of bitumen with the waste of cement concrete, processes of physical and chemical adsorption take place. Maltenes of bitumen penetrate the pores of cement concrete during physical adsorption. Calcium hydroxide $Ca(OH)_2$ and bitumen active functional groups -OH, -COH, -COOH, C = C-, etc. interact with the formation of new bonds that are reflected in IR spectra in the form of peaks of 820 cm^{-1}, 944 cm^{-1}, 947 cm^{-1} or 1430 cm^{-1}. The intensity of the given processes and the degree of the structuring of the bituminous films will depend on the degree of hydration of the cement in the fragments of the concrete present in construction waste. When the degree of hydration increases, the thickness of the adsorption-solvate coating of bitumen increases from 1.5 to 2 times. During the interaction of bitumen with the waste of the building cement of concrete, more heat-resistant bitumen films are formed. When the degree of hydration is an increment, the resistance to high-temperature exposure of structured bitumen increases from 1.2 to 2.4 times.

Аннотация. Представлены результаты исследования влияния основных свойств минеральных порошков из цементного камня с различной степенью гидратации, фазового состава и характеристиками порового пространства на свойства битумоминерального связующего. Установлены зависимости влияния минерального порошка на основе цементного камня на базовый параметр структуры битумоминеральных связующих – толщину адсорбционно-сольватного слоя битума, которую устанавливали с применением реологического метода. Анализ полученных эмпирических данных показывает, что битум химически взаимодействует с минеральными порошками, полученными из цементного камня. Доказано, что при взаимодействии битума с отходами цементобетона происходят процессы физической и химической адсорбции. Малтены битума проникают в поры цементобетона во время физической адсорбции. Гидроксид кальция $Ca(OH)_2$ взаимодействует с активные функциональными группами битума -OH, -COH, -COOH, C = C- и т.д. Реакция взаимодействия происходит с образованием новых связей, которые отражаются в ИК-спектрах в виде пиков 820 $см^{-1}$, 944 $см^{-1}$, 947 $см^{-1}$ или 1430 $см^{-1}$. Интенсивность данных процессов и степень структурирования битумных пленок будут зависеть от степени гидратации цемента во фрагментах бетона, содержащегося в строительных отходах. С увеличением степени гидратации, толщина адсорбционно-сольватного слоя битума увеличивается в 1,5...2 раза. Во время взаимодействия битума с отходами цементобетона образуются более теплостойкие битумные пленки. При увеличении степени гидратации, сопротивление высокотемпературному воздействию структурированного битума увеличивается от 1,2 до 2,4 раз.

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1. Introduction

The use of recycled materials in the production of building materials is a traditional solution for the construction industry. Technogenic raw materials are successfully used in the production of general construction and special materials, as well as road construction materials, including asphalt concrete. Processing of waste in raw materials for the production of asphalt concretes is not only the solution of the ecological issue of recycling but the economic benefit due to the reduction in the cost of raw materials or the increase in the service life of the product, for example, road asphalt concrete. Millions of tons of the waste construction industry (concrete battle based on Portland cement) produced annually in the world, which is stored in landfills and dumps.

A modern production model tends to be involved with the production and accumulation of various wastes, which are often used in other industries as secondary raw materials. The construction industry is also a source of waste, especially in the areas where the production of construction materials, products, and structures is carried out, as well as in construction and renovation areas. Such waste can also be used as a secondary raw material. The use of waste generated after crushing and sorting of concrete and reinforced concrete products and structures in bituminous concrete technology is considered in this study.

Experience in the use of such raw materials suggests high variability. So, for example, the authors [1–4] state that the water resistance of bituminous concrete with the use of crushed stone from building construction waste increases, in other works [5–11] it is stated that the content of such stones should be limited to an amount that does not have a detrimental effect on water resistance. Also, some researchers have obtained results that indicate an increase in the rigidity and deformability of bituminous concretes with the use of recycled stones [1–4, 11–13], and a number of other studies give the data indicating the opposite effect [9, 14].

One of the reasons for obtaining contradictory results by various researchers may be that the distinctive feature of used construction waste for processing and obtaining a large aggregate is the presence on the surface of its grains of fragments of cement stone, which affects the interaction with bitumen. The degree of influence of these fragments on the processes of interfacial interaction will depend both on age and on the type and quality of concrete in the composition of construction waste.

The purpose of this work is to determine the effect of cement stone properties in construction waste products on the processes of their interaction with bitumen.

2. Method and materials

To solve the assigned task, mineral fillers from cement stone were used, which were obtained on the basis of the plain cement PC-500 (C) produced by the Production Company "Rusean" (Russia) with a different water-cement ratio W/C = 0.1; 0.2 and 0.4 (C_1, C_2 and C_3, respectively), which after natural hardening was subjected to grinding.

Table 1. Phase composition of mineral powders

Samples	Content, %									
	Phases of clinker				Hydrated phases		Amorphous phase	Sulphate minerals		$CaCO_3$
	C_3S	β-C_2S	C_3A	C_4AF	Ettringite	$Ca(OH)_2$		Gypsum	Semihydrate	
C	56.4	18.2	4.6	16.3	---	0.5	---	1.5	2.5	---
C_1	41.9	17.9	2.6	15.3	3.0	4.3	15	---	---	---
C_2	27.3	16.5	---	15.0	2.5	12.2	25	---	---	1.5
C_3	13.7	14.7	---	12.3	2.9	22.8	30	---	---	3.6

Construction bitumen "BND 60/90" produced by LLC "Moscow Oil Refinery", with a softening temperature of 51 ^{o}C and a brittle temperature of -20 ^{o}C was used in the study.

The interaction of cement stone with bitumen was evaluated by the thickness of the adsorption-solvate layer on the surface of grains of mineral filler in accordance with the rheological method [15], which is based on a change in the properties of the bituminous film when structuring grains of mineral material.

The research of the interaction of fillers with bitumen was carried out using the Cary 630 FTIR IR-Fourier spectrometer. The phase composition of mineral fillers was determined by the method of qualitative X-ray phase analysis using X-ray diffractometer ARL X'TRA.

Determination of the specific surface area and volume of micro- and mesopores was carried out using the methods of nitrogen porosimetry BET (Brunauer-Emmett-Teller) and DFT (Barret-Joyner-Halenda), respectively, on Nova 2200e, Quantachrome.

The determination of the parameters of phase transformations during heat treatment was carried out by the method of scanning differential calorimetry on the HDSC PT1600 Linseis device.

The bitumen content capacity index of mineral filler is determined by the amount of oil at which its mixture with 100 cm^3 of mineral material has a given consistency.

The average density of mineral filler was determined by the mass of the powder with a volume of 100 cm^3, compacted under a load of 40 MPa for 3 minutes.

The true density of mineral filler was determined by pycnometric methods using an organic solvent.

The porosity of the mineral filler was determined by the calculation method based on the values of the average density and true density.

Swelling was defined as an increment in the volume of samples (cylinder) from bitumen and mineral powder after their water saturation in vacuum and subsequent exposure in water at 60 ^oC for 4 hours. The ratio of bitumen and mineral powder was used at which the water saturation of the samples was 4 ... 5%.

The water resistance was determined by the level of drop in the strength of the sample cylinders after water saturation in a vacuum.

The water saturation was determined by the amount of water, absorbed by the sample when saturated with water in a vacuum for 1 hour.

3. Results and Discussion

Utilization efficiency of new mineral materials acting as fillers in the bitumen-concrete mixture is determined by the physical and chemical features of the grain surface of the material in question and the intensity of the interaction processes with bitumen components.

As a result of processes at the phases interface "bitumen - mineral filler", the group composition of bitumen is redistributed and the bituminous film is structured on the surface of the filler grains, the thickness of which characterizes the intensity of the interaction.

It is known that as the dispersion of the material increases, its activity increases as well, which corresponds to an increase in the volume of structured bitumen in the interface layers. The viscosity of structured bitumen increases excessively in comparison with the viscosity of free bitumen [15]. Taking this fact into account, with the use of the rheological method, studies were conducted to evaluate the degree of physical and chemical activity of the mineral materials in question with respect to bitumen and the thickness of the adsorption-solvate layer of bitumen on the surface of grains for cement stone with different degrees of hydration and at different temperatures was determined.

To assess the influence of the conditions for the preparation of a bitumen-concrete mixture containing components based on cement-containing materials or concrete waste, the effect of temperature on the basic properties of the mineral materials under consideration was researched (Figure 1).

It is obvious that at the temperature of preparation of bitumen-concrete mixture from 120 ^oC to 160 ^oC depending on the type of bitumen-concrete the products of hydration hardening will undergo some structural changes. The results of differential scanning calorimetry showed (Figure1) that at the temperature of up to 100 ^oC there is a release of unbound water, and at a temperature of up to 200 ^oC there is a loss of crystallization water that is characteristic of $C_3A{\cdot}CaSO_4{\cdot}12H_2O$ compounds (at 110-130 ^oC), $3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}31H_2O$ (at 140-170 ^oC), $C_3A{\cdot}CaCO_3{\cdot}11H_2O$ (at 150-500 ^oC), $CaSO_4{\cdot}2H_2O$ (at 140–170 ^oC). It is shown that the intensity of the proceeding processes is directly proportionate to the water-cement ratio and the amount of the hydration reaction products formed. The referred processes naturally lead to a change in the properties of the mineral materials in question (Table 2).

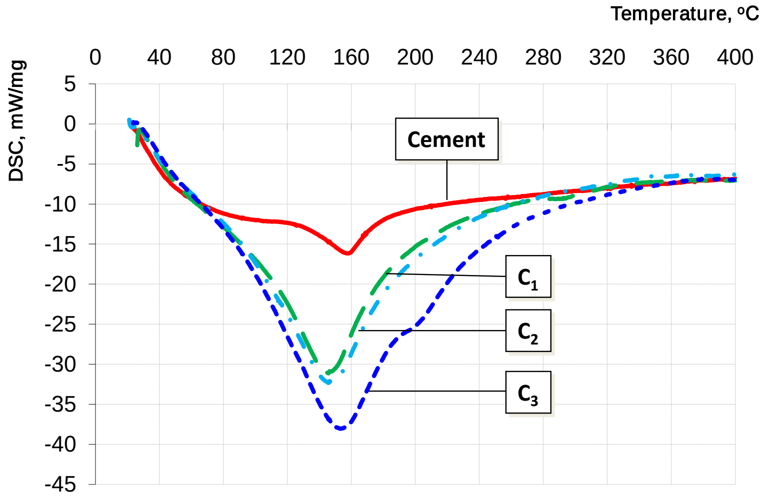

Figure 1. DSC curve of mineral materials

Table 2. Change in the index of mineral properties after warm-up

Indicator name	Change in the index after warm-up at 160 °C, %			
	C	C_1	C_2	C_3
Average density	–	–0.6	–3.3	–12.7
True density	+1.7	+3.8	+11.2	+12.3
Porosity	+2.4	+7.5	+21.6	+63.0
Bitumen capacity index	+1.0	+1.3	+3.4	+18.6

Thus, during the heating of the mineral materials in question to 160 °C, the porosity varies from 2 to 63 %, which naturally leads to an increase in the bitumen capacity index to 18.6 %. In addition, the weight loss after warm-up can amount to 15 %. Therefore, when using construction waste products containing concrete fragments of varying degrees of hydration in the bitumen-concrete mixture, it is necessary to carry out their preliminary drying at a temperature of not less than 160 °C.

Further studies were carried out on mineral fillers from cement stone after heating at a temperature of 160 °C for at least 4 hours, their properties are presented in Table 3.

Table 3. Properties of mineral materials

Indicator name	Indicator value				
	C	C_1	C_2	C_3	Limestone
Average density, g/cm^3	1.76	1.57	1.48	1.44	1.91
True density, g/cm^3	3.08	2.75	2.68	2.55	2.79
Porosity, %	43	43	45	44	32
Specific surface area (BET), m^2/g	9.5	14.9	19.8	27.2	26.2
Volume of micro- and mesopores (DFT), cm^3/g	0.012	0.021	0.025	0.042	0.035
Bitumen capacity index	98	151	122	102	90

As the amount of mixing water increases (C → C_3), the pore structure and morphology of the surface will change due to the proportional increase of hydration reaction products, calcium hydrosilicates possessing a needle-shaped highly dispersed structure. This explains the increase in the roughness of grains of mineral materials due to the increase in the content of calcium hydrosilicates, which is confirmed by an increase in the specific surface area of the filler grains: for example, for C_3 in 2.8 times.

The activity of the mineral materials in question with respect to bitumen, determined by the thickness of the bituminous film, formed during the interaction at the phases interface "bitumen - mineral material" will be determined by the processes of both chemical and physical adsorption [16–18], determined by the features of their surface and intra-port space (Figure 2).

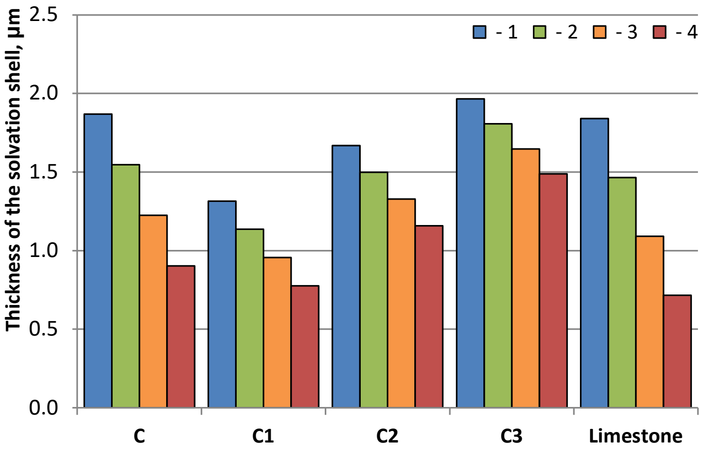

Figure 2. Thickness of the adsorption-solvate layer of bitumen on the surface of mineral materials at the temperature of: 1 – 120 ºC; 2 – 130 ºC; 3 – 140 ºC; 4 – 150 ºC

Analysis of Figure 2 shows that among the mineral materials in question, a larger thickness of the adsorption-solvate layer of bitumen is formed on the surface of grains of filler made from cement stone C_3, in which the amount of hydration products is maximized. It should be pointed out that as the temperature increases, the rate of change in the thickness of the adsorption-solvate layer of bitumen decreases with an increase in the amount of hydrated cement in the mineral filler ($C \rightarrow C_3$) and for C_3 is minimal. Thus, the hydration reaction products, in the process of interaction with bitumen, form an interlayer at the phases interface, which has a higher temperature stability, which will positively affect the high-temperature properties of organic-mineral mixtures with their application (shear-resistance, strength). The intensification of the structuring process is determined by the increase in the specific surface area and the interstitial space, which cause an increase in the volume of diffusion of the bitumen components into the grains of the mineral material and the number of tarry asphaltene complexes concentrated on the surface in the form of an adsorption layer.

However, simultaneously with the processes of physical absorption of maltenes of bitumen, the formation of an adsorption-solvate layer can be determined by reactions of chemical interaction at the phases interface "bitumen-mineral material".

In order to determine the interaction of mineral fillers from cement stone with different degrees of hydration with bitumen, studies were carried out using IR-spectroscopy (Figure 3).

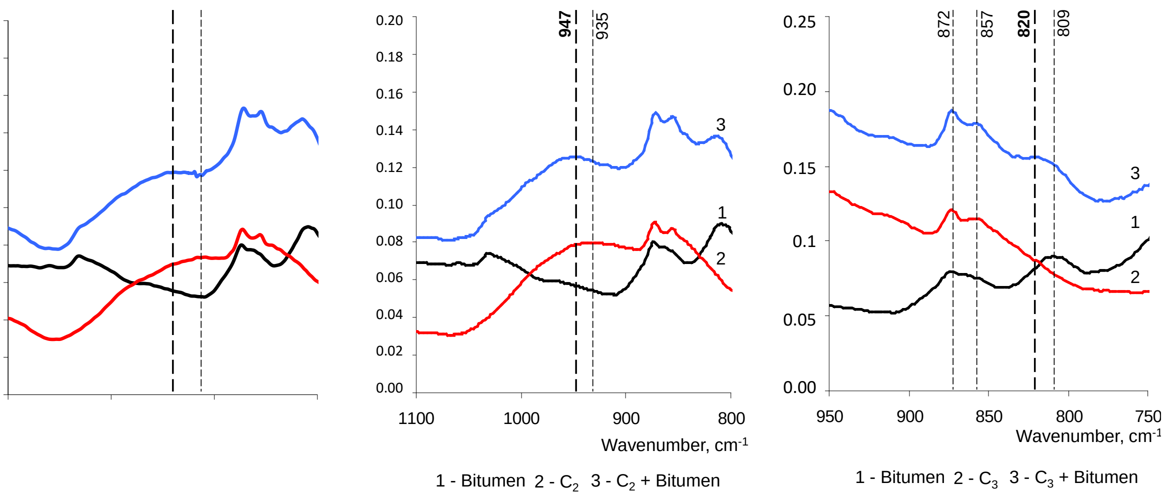

Figure 3. IR-spectra of mineral fillers, bitumen and mixtures of mineral fillers with bitumen

Analysis of the spectra obtained shows that the interaction of all the mineral fillers in question with bitumen is a physical and chemical process leading to the formation of new compounds at the interface. This is indicated by the shifts in the peaks on the spectrograms: in the mixture of bitumen and mineral filler, a peak displacement of 1419 cm^{-1} is observed in the area with a large wave number of 1430 cm^{-1}; for filler C_3, a peak shift from 809 cm^{-1} to 820 cm^{-1} is observed; for fillers C_1 and C_2, – the peak shifts to the left along the axis from 908 to 944 cm^{-1} and from 935 to 947 cm^{-1}, respectively. Depending on the chemical and group composition of bitumen and mineralogical composition during the interaction of bitumen with cement stone, chemisorption processes take place with the formation of chemical types of bonds [19–21]:

$$Ca^{2+} + 2RCOOH^{-} \rightarrow Ca(RCOO)_2$$

On the one hand, the most active product among hydration products are calcium hydroxide $Ca(OH)_2$, on the other hand, bitumen contains active functional groups –OH, –COH, –COOH, C=C–, etc., which provoke chemical interaction with cement appositions [20, 21].

Thus, the use of construction waste in the bitumen-concrete mixture as a large aggregate, a fine aggregate or a filler should be carried out taking into account that, when combined with bitumen melt, concrete fragments will provoke physical and chemical reaction with the formation of heat-resistant bitumen layers at the phases interface. Moreover, the thickness of the bitumen interlayers will increase with the degree of hydration of the cement in the waste used, which actualizes the preliminary assessment of the activity of such materials, both with the use of independent methods for determining the geometric characteristics of the adsorption-solvate layer of bitumen, and in combination with auxiliary methods that allow assessing the degree of hydration cement [23–28].

The results of the study can be used to develop a method for designing asphalt mixes using recycled concrete aggregate [29]. With an increase in the degree of hydration of cement in concrete waste, the amount of bitumen increases in the composition of asphalt concrete. Recycled concrete aggregate must be dried when preparing the asphalt mix, since moisture will degrade the adhesion of the bitumen.

Samples from a mixture of construction waste and bitumen with a water saturation of 4 ... 5 % were prepared for the swell resistance test and water resistance. It has been established that water resistance and swelling to swelling of samples increases with the use of mineral powders, in which the degree of hydration is higher. This indicates the formation of a stronger and water-resistant structure in the interaction of bitumen and mineral material from construction waste.

4. Conclusion

Analysis of the results obtained allows us to draw the following conclusions:

1. It is proved that during the interaction of bitumen with the waste of cement concrete, processes of physical and chemical adsorption take place. Maltenes of bitumen penetrate the pores of cement concrete during physical adsorption. Calcium hydroxide $Ca(OH)_2$ and bitumen active functional groups -OH, -COH, -COOH, C = C-, etc. interact with the formation of new bonds that are reflected in IR spectra in the form of peaks of 820 cm^{-1}, 944 cm^{-1}, 947 cm^{-1} or 1430 cm^{-1}.

2. The intensity of the given processes and the degree of the structuring of the bituminous films will depend on the degree of hydration of the cement in the fragments of the concrete present in construction waste. When the degree of hydration increases, the thickness of the adsorption-solvate coating of bitumen increases from 1.5 to 2 times.

3. During the interaction of bitumen with the waste of the building cement of concrete, more heat-resistant bitumen films are formed. When the degree of hydration is an increment, the resistance to high-temperature exposure of structured bitumen increases from 1.2 to 2.4 times.

4. With an increase in the degree of hydration of cement in concrete waste, the amount of bitumen increases in the composition of asphalt concrete. Recycled concrete aggregate must be dried when preparing the asphalt mix, since moisture will degrade the adhesion of the bitumen.

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