Researhe Article
ISSN 2658-5553
Received: February 15, 2022 Accepted: March 21, 2022 Published: March 21, 2022

Bitumen Aging on the Surface of Igneousand Carbonate Rocks of Various Grain Size

Salihov, Muhammet Gabdulhaevich[1]
Veyukov, Evgenij Valerianovich[1*]
Lomakina, Ekaterina Aleksandrovna[1]
Ponedelko, Lyudmila Ivanovna[1]

[1]Volga State University of Technology, Lenin Sq., 3, Yoshkar-Ola, Russia, 424000;
SalihovMG@volgatech.net (S.M.G.); VeukovEV@volgatech.net (V.E.V.);
LomakinaEA@volgatech.net(L.E.A.); PonedelkoLI@volgatech.net (P.L.I.)
Correspondence:* email VeukovEV@volgatech.net; contact phone +79696276745

Keywords:

Limestone; Asphalt; Asphalt mixtures; Durability; Elevated temperature; Binders

Abstract:

The object of research is asphalt concrete mixtures.Experimental study of the aging process of bitumen films in the composition of bitumen-mineral mixtures consisting of dispersed mineral grains of various rocks (granites and limestones) of fractional composition fr. 5...20 mm, 0.315...5.0; 0.05...5.00 and 0.05...0.71 mm and BND 90/130 and BND 70/100. **Method.** The experiments were performed under the method covered by the Russian Federation Patent No. 2654954, proposed at the Volga State University of Technology (Department of Building Technologies and Highways). The assessment of the aging rate of bitumen-mineral mixtures was performed by: 1) establishing the values of the ultimate compressive strength at +50 °C for standard cylindrical samples (height and diameter 50x50 and 71.4x71.4 mm). The samples are formed from mixtures and stoved at +150 °C for 0...7 hours; 2) Calculating the values of the aging factor K_{aging}, the aging rate I_{aging} and analyzing the change in the values depending on the preheating time of the mixture at a high temperature. **Results.** Statistical processing of the data obtained during the experiments was carried out by the one-factor design method in the CurveExpert software environment. After that, mathematical models with a high correlation degree were obtained (S=0.000432…0.13578; r = 0.90498….0.99997). These mathematical models are mainly represented by Sinusoidal Fit-; Harris and Exponential models and can be used to plan the operational behavior of materials in the composition of road structural layers.

1 Introduction

In road construction, aggregates from igneous and carbonate rocks (granite, limestone) are widely used as a part of various bitumen-mineral mixtures. These aggregates can be used in the form of gravel, manufactured sand and mineral filler. The bitumen functions as a binder for discrete materials, but its adhesive ability decreases by the aging process caused by intrinsic and extrinsic variables [1]–[5]. Consequently, the durability and other indicators of bitumen-mineral materials decrease, which, in its turn, leads to lower levels of performance of the road structural layers. Aging of bitumen in the composition of bitumen-mineral materials is the result of an irreversible loss in performance due to high and low temperatures, inimical environment (e.g. water, salt and acid solutions, radiation, biological media, mechanical forces, chemical changes caused by evaporation of light fractions and spontaneous transformations in the binder). The study of the bitumen aging process is needed for predicting and assessing durability and performance level of bitumen-mineral materials in road structures [6]–[12].

The aging process of road structural layers can be evaluated by constant/periodic monitoring of their operating condition for extended periods of time or by artificial aging within a short time during laboratory testing. Since there is no standard method, several methods were proposed by various authors to study this process [13]–[28]. Each method has its advantages and disadvantages. At the Volga State University of Technology (Department of Building Technologies and Highways) a method covered by the Russian Federation Patent was proposed. This method has an advantage of the known ones – an opportunity to study the aging processes of bitumen both in complex and in monomineral bitumen-mineral mixtures, which enables us to investigate the impact of each component on the aging of the whole [29]–[32].

The object of research is asphalt concrete mixtures. **The purpose** of this work is to study of the aging process of bitumen films in the composition of bitumen-mineral mixtures consisting of dispersed mineral grains of various rocks (granites and limestones) and bitumen petroleum viscous of grades BND 90/130 and BND 70/100. To achieve this goal, the following tasks were solved:

1) Establishing the values of the ultimate compressive strength at +50 °C for standard cylindrical samples (height and diameter 50x50 and 71.4x71.4 mm). The samples are formed from mixtures and stoved at +150 °C for 0...7 hours;

2) Calculating the values of the aging factor K_{aging}, the aging rate I_{aging} and analyzing the change in the values depending on the preheating time of the mixture at a high temperature;

3) Statistical processing of the data obtained during the experiments by the one-factor design method in the CurveExpert software environment;

4) Obtaining mathematical models of the aging rate of bitumen-mineral mixtures depending on the holding time at a temperature of 150 °C.

2 Materials and Methods

The following materials are used as the base and surface layers of pavement (I-IV categories): asphalt concrete, stone mastic asphalt and their varieties, black crushed stone. These materials consist of compacted crushed granite or limestone (crush strength grade not lower than 400-600, fr. 5...40 mm), crushed or natural sand (grain size – 0.16...5.0 mm), mineral filler (more often – limestone of MP-1 grade) and other components with petroleum bitumen. Black limestone or granite crushed stone, and sometimes simple and highly-porous asphalt concrete are used in the base layers. Considering the above, the following mixtures were chosen for the experiments: mixtures of bitumen petroleum viscous of grades BND 90/130 and BND 70/100 (the Kstovsky oil refinery in the Nizhny Novgorod region), mixtures of granite crushed stone (M1200 fr. 5...20 mm), screening dust of these rocks (crushed sand) fr. 0.16...5 mm, crushed limestone of the M400 grade fr. 5...20 mm (the Novotoryalsky stone crushing plant, Mari El Republic), crushing waste of limestone (M400 grade; the 2nd crushing stage; fraction 0.16...5mm) and limestone mineral filler (MP-1 grade, fr. 0.05...0.071 mm).

Some characteristics of the studied materials are presented in Table 1-5.

Table 1. Some characteristics of bitumen 70/100

Indicator	Standard requirement	Actual value of the indicator
1. Needle penetration depth at temperature +25 °C, 0.1 mm	71-100	78
2. Softening point, °C, not lower	47	48
3. Tensile at °C, cm, not lower	3.7	3.7
4. Fragility temperature, °C, not lower	-18	-19
5. Flash point, °C, not lower	230	308
6. Change in sample mass after aging,%, not more than	0.6	0.3

Table 2. Some characteristics of bitumen 90/130

Indicator	Standard requirement	Actual value of the indicator
1. Needle penetration depth at temperature +25 °C, 0.1 mm	90-130	102
2. Softening point, °C, not lower	43	46
3. Tensile at °C, cm, not lower	4.0	4.3
4. Fragility temperature, °C, not lower	-17	-19
5. Flash point, °C, not lower	230	310

6. Change in sample mass after aging,%, not more than	0.6	0.4

Table 3. Granulometric composition of crushed stoneM400 grade fr. 0.16...5 mm

Size of sieves, mm	Sieve fraction values								
	10	5	2.5	1.25	0.63	0.315	0.16	0.071	<0.071
Private balances, %	0	1.55	34.83	19.68	19.31	10.78	6.17	6.80	0.89
Total balances, %	0	1.55	36.38	56.06	75.37	86.15	92.31	99.11	100
Full passing, %	100	98.45	63.62	43.94	24.63	13.85	7.69	0.89	0

Table 4. Granulometric composition of crushed stoneM400 grade fr. 5...20 mm

Size of sieves, mm	Sieve fraction values										
	20	15	10	5	2,5	1,25	0,63	0,315	0,16	0,07	<0,07
Private balances, %	0	3.3	51.9	44.99	1.7	0.14	0.05	0.1	0.24	0.83	0.07
Total balances, %	0	3.3	51.9	96.87	98.58	98.71	98.76	98.86	99.1	99.93	100
Full passing, %	100	96.7	48.1	3.13	1.42	1.29	1.24	1.14	0.9	0.07	0

Table 5. Granulometric composition of mineral filler MP-1 grade, fr. 0.05...0.071 mm

Size of sieves, mm	Sieve fraction values					
	1.25	0.63	0.315	0.16	0.07	<0.07
Private balances, %	0	0.05	0.78	21.12	6.16	71.89
Total balances, %	0	0.05	0.83	21.95	28.11	100
Full passing, %	100	99.95	99.17	78.05	71.89	0

Bitumen-mineral mixtures consist of a mixture of petroleum bitumen with mineral materials of crushed granite and limestone, crushed sand, limestone crushing waste and mineral filler of various grain sizes. In the process of preparation, laying in engineering structures and when in service, bitumen-mineral mixtures are subject to the aging process. The following methods were obtained to optimize the study process of the bitumen aging in the composition of mineral materials: samples heating at high temperatures, preliminary sample heating and further thawing and freezing test, studying the changes in the sample properties after prolonged water saturation.

The accuracy of the research results obtained for the aging process of bitumen-mineral materials largely depends on the chosen research methods. To date, there is a standard method for studying bitumen aging, the point of which is to measure the weight loss of bitumen during heating at +163 °C for 5 hours according to Interstate Standard GOST 33140-2014 [33] with free air supply. However, this method does not take into account the effect of base material on the condition of the films. In 2018, a new method for studying the aging processes of bitumen films on the surfaces of individual mineral grains of bitumen-mineral mixtures was developed at the Department of Building Technologies and Highways (Volga State University of Technology) whole [34]. This method has the following significant advantages:

1) allows to study the aging process of bitumen films on the surfaces of any mineral components of bitumen-mineral mixtures and assess their role separately in the aging process;

2) allows to study the dynamic pattern of properties during aging, i.e. at any stages of structure formation, starting from the combining the components in a loose state until the complete loss of mechanical strength in a compacted state;

3) the study of the aging of bitumen-mineral mixtures is carried out under laboratory conditions, which makes it possible to adjust the mixture composition during the technical project development at the design stage;

4) allows to study the bitumen films aging by choosing, as the main, one of the most sensitive to temperature changes indicators according to Interstate Standard GOST 9128-2013 [35] or National Standard of the Russian Federation GOST R58406.2-2020 [36].

Fig. 1 – Standard cylindrical specimens size of 71.4 mm

For the experiment, samples of the crushed limestone and oil bitumen, dried to constant weight and heated to operating temperatures, are taken and mixed in optimal proportion until a homogeneous mixture is obtained. Standard cylindrical specimens were made from these mixtures at a working temperature (+140... +160 °C). These specimens have a diameter size of 71.4 mm, according to Interstate Standard GOST 12801-98 [37]. In 24 hours, they were tested in order to establish the values of the ultimate compressive strength at +50 °C.

Fig. 2 – Bitumen-limestone crushed stone on metal trays in ventilated thermal furnace

The next step is to prepare samples of bitumen-limestone crushed stone, place them in even layers on metal trays to a ventilated thermal furnace, where the set temperature (+150 °C) is automatically maintained. Every half hour, to ensure the processes of volumetric aging, the mixture is mixed with a spatula. After the estimated heating time (0, 1, 3, 5, 7, etc. hours), samples are removed from the furnace and standard cylindrical samples are formed and tested according to Interstate Standard GOST 12801-98 [37] to determine the average density, compressive strength at +50 °C. Further, the aging coefficient $R_C^{+50°C}$ ($K_{aging}^{R_C^{+50°C}}$), aging rate ($I_{aging}^{K_{aging}^{R_C^{+50°C}}}$) are calculated according to formulas:

$$K_{aging}^{R_C^{+50°C}} = \frac{R_C^{+50°C, t_h = t_i}}{R_C^{+50°C, t_h = 0}} \quad (1)$$

where $Ind_i^{T, t_h = t_i}$ and $Ind_i^{T, t_h = 0}$ is values of the indicators of the studied physical and mechanical properties of the bitumen-mineral mixture before and after heating at a high temperature for a time from 0 to t_i, respectively; t_h is heating time of mixtures;

$$I_{aging}^{K_{aging}^{R_C^{+50°C}}} = \frac{\Delta K_{aging}^{R_C^{+50°C}}}{\Delta t_h} \quad (2)$$

where $\Delta K_{aging}^{R_C^{+50°C}}$ is the change in the aging coefficient values during Δt_h.

3 Results and Discussion

The optimal mineral/bitumen weight ratio was preliminarily established. Experiments of strength indicators made it possible to calculate the values of the aging factors and the aging rate (Table 1). In addition, ‘value-holding time’ dependency graphs were established (the holding temperature – 150°C) (Fig. 3).

Table 6. Experimental result and calculation data

Aging indicators for mixtures of bitumen (B) and mineral materials (MM)	Duration time of aging at a temperature of +150 °C t_{dur}, *h*					Mathematical model* and its convergence
	0	1	3	5	7	
1	2	3	4	5	6	7
1. Aging factor (compressive strength at +50 °C $K_{aging}^{R_C^{+50\,°C}}$) for mixtures of bitumen and: a) granite crushed stone in a ratio of 1:25	1.00	0.32	0.15	0.01	0.01	$K_{aging}^{R_C^{+50\,°C}} = a \cdot e^{-b \cdot t_{dur}}$ S=0.0639; r=0.9909
b) stone screening dust (crushed sand) in a ratio of 1:10	1.00	1.02	1.43	2.00	1.07	$K_{aging}^{R_C^{+50\,°C}} = a + b \cdot \cos(c \cdot t_{dur} + d)$ S=0.1281; r=0.9875
c) crushed limestone in a ratio of 1:9	1.00	1.26	1.71	1.23	1.03	$K_{aging}^{R_C^{+50\,°C}} = a + b \cdot \cos(c \cdot t_{dur} - d)$ S=0.00432; r=0.9997
d) crushing waste of limestone in a ratio of 1:6.69	1.00	1.53	1.94	2.32	2.23	$K_{aging}^{R_C^{+50\,°C}} = a + b \cdot \cos(c \cdot t_{dur} - d)$ S=0.00432; r=0.9999
e) limestone mineral filler in a ratio of 1:5.67	1.00	1.00	1.13	1.63	1.38	$K_{aging}^{R_C^{+50\,°C}} = a + b \cdot \cos(c \cdot t_{dur} + d)$ S=0.003230; r=0.99689
2. Aging rate $I_{aging}^{K_{aging}^{R_C^{+50\,°C}}}$ for mixtures of butumen and: a) granite crushed stone	-0.68	-0.08	-0.07	0.00		$I_{aging}^{K_{aging}^{R_C^{+50\,°C}}} = \frac{1}{a + b \cdot e^{c \cdot t_{dur}}}$ S=0.003230; r=0.99689
b) stone screening dust (crushed sand)	0.02	0.20	0.28	-0.47		$I_{aging}^{K_{aging}^{R_C^{+50\,°C}}} = \frac{1}{a + b \cdot t_{dur}^{c}}$ S=0.007965; r=0.98113
c) crushed limestone	0.26	0.23	-0.24	-0.10		$I_{aging}^{K_{aging}^{R_C^{+50\,°C}}} = a + b \cdot \cos(c \cdot t_{dur} + d)$ S=0.00832; r=0.99981
d) crushing waste of limestone	0.53	0.20	0.19	-0.04		$I_{aging}^{K_{aging}^{R_C^{+50\,°C}}} = \frac{1}{a + b \cdot e^{-c \cdot t_{dur}}}$ S=0.135578; r=0.90498
e) limestone mineral filler	0.00	0.07	0.25	-0.12		$I_{aging}^{K_{aging}^{R_C^{+50\,°C}}} = \frac{1}{a + b \cdot t_{dur}^{c}}$ S=0.02456; r=0.99178

*in mathematical models, there are the constant coefficients (*a, b, c, d*) in Column 7 of Table 6.

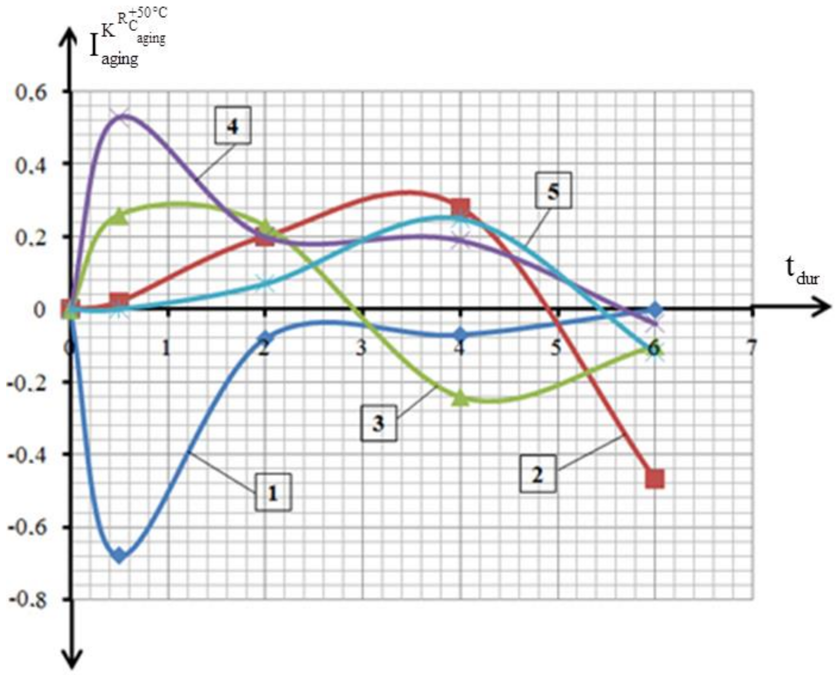

Fig.3 – The dependency graph of the aging rate values of the bitumen mixtures and:1 – granite crushed stone; 2 – stone screening dust; 3 – crushed limestone;4 – crushing waste of limestone; 5 – limestone mineral filler

Analyzing Table 6 and Fig. 3 the following may be noted:

1) the bitumen aging on the surface of granite crushed stone occurs mainly during the 1st hour of aging treatment at 150 °C with an intensity of 0.68 h^{-1}. The factor value decreases 3 times, i.e. the durability of the structural layer made of compacted black crushed stone decreases the same number of times. With further heating, the aging process stabilizes and becomes equal to 0 by the 7th hour;

2) the aging of a mixture of bitumen with stone screening dust (crushed sand) occurs with a gradual acceleration for 5 hours – the value of aging rate varies from 0.02 to 0.28 h^{-1}, then there is an increase in the value of the aging factor and the aging rate (up to $K_{aging}^{R_C^{+50°C}}=1.00...1.71$ and $I_{aging}^{K_{aging}^{R_C^{+50°C}}}=-0.47h^{-1}$);

3) the bitumen aging on the surfaces of crushed limestone is relatively uniform within 3 hours with an average intensity of 0.23...0.26 h^{-1}. Within 3...7 hours, the mechanical strength of the samples increases ($I_{aging}^{K_{aging}^{R_C^{+50°C}}}=-0.11...024h^{-1}$);

4) the bitumen aging on the surfaces of limestone mineral filler and limestone crushing waste is characterized by an increase in mechanical strength with an intensity of 0...0.25 and 0.53...0.19 h^{-1} (at 150 °C in the first 5 hours). After that, there is a decrease in mechanical strength with an intensity of -0.12 and -0.04 h^{-1}. A noteworthy detail is that bitumen mixed with limestone crushing waste ages relatively faster in the first 5 hours of heating than bitumen mixed with mineral filler;

5) when the mixtures of bitumen with granite crushed stone are kept at 150 °C for 0...7 hours, aging process is characterized by a constant decrease in durability. When mixed with crushed limestone there is an increase in the first 3 hours. After that, the reverse process is observed;

6) the bitumen aging at 150 °C in dispersed systems of smaller size, regardless of the geological composition, occurs with a lower intensity over time;

7) the change in the numerical values of the aging rate of bitumen-mineral mixtures overtime occurs according to a decreasing sinusoidal law, logistic and Harris models, regardless of the rock type and grain size;

8) there is the following sequence under the absolute value of the bitumen aging rate on the surfaces of MM (in decreasing order): granite crushed stone – crushing waste of limestone – limestone crushed stone – stone screening dust (crushed sand) – limestone mineral filler.

4 Conclusions

The results of the performed experimental work make it possible to reach the following conclusions: the aging rate of bituminous films on the surface of films of discrete materials depends on the rock type and grain size:

1. On the surface of acidic igneous rocks, the aging process is more intense in the 1st hour of heating, then this process decelerates;
2. On the surface of carbonate rocks in the initial period of aging, the mechanical strength increases, and after 3 hours of heating, the reverse process begins;
3. The aging process is faster in a mixture of bitumen with mineral particles with a larger grain size;
4. Mathematical models of the aging rate of bitumen-mineral mixtures depending on the holding time at a temperature of 150 °C were obtained.

5 Acknowledgements

The authors thank the staff of the Department of construction technologies and highways of the Volga state technological University (Yoshkar-Ola, Russian Federation).

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