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Increasing the level of aging stability of bitumes modified by kaolinite

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Abstract. During the literature review analysis, the main causes of bitumen binders aging were identified and the kaolinite modifier was proposed that have increased the bitumen aging resistance. The physical and mechanical properties of bitumen modified by kaolinite for the following indicators were studied: penetration at 25 °C, softening point, ductility at 0 °C, Fraas brittle point, dynamic viscosity at 105 °C, 135 °C and 165 °C. The bituminous binder aging was simulated in an RTFOT oven. The aging quality was determined by the softening point change, after aging in the RTFOT oven. The possibility of short-term aging reducing of modified bitumen at a kaolinite content of 2 % to 8 % by 12.5–28.6 % has been established. The characteristic peaks of bituminous binder aging: 1738, 1640, 1654, 1217, 1033 cm^{-1} were revealed by the IR spectroscopy method. It was established that the kaolinite introduction into bitumen has caused a significant decrease of aging processes, which has confirmed by the IR spectroscopy data: the peaks at 1640, 1564 and 1217 cm^{-1} almost completely have disappeared in the difference spectrum, and the intensity of carbonyl absorption band at 1738 cm^{-1} has decreased. The obtained experimental data confirmed the similarity of the processes described in previously published studies of other authors and the continuing research prospects in the direction of increasing the bitumen binders aging resistance at searching for new modifiers types and determining them optimal quantities.

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1. *Introduction*

Asphalt concrete consists of various types of coarse-grained and fine-grained aggregates, connected by bitumen. In asphalt mix the bitumen makes up only 3.5–7 % by weight or approximately 11–18 % by volume. The bitumen makes up only 3.5–7 % by weight or approximately 11–18 % by volume of asphalt concrete mixture and provides the significant effect on the road surfaces durability, exhibiting its elastic properties at negative ambient temperatures and viscous properties at high air temperatures [1, 2].

However, due to the constant increase of traffic intensity and axial loads on asphalt concrete pavements prepared from ordinary bitumen, various types of defects are formed: plastic rut, fatigue cracks, low-temperature cracking, wear, spalling, aging, etc. [3, 4].

Bitumen, being one of the main building materials in road construction, is exposed to air for a long time, solar radiation, wind, rain and vehicles [5, 6]. Road bitumen is the organic binder that constantly changes its properties, group and chemical composition as part of the road surface [7]. One of the main factors affecting the defects formation in asphalt concrete is the bitumen aging [8]. The bitumen aging is a complex process, the consequence of which is the rigidity increase and the fragility increase, both during the asphalt concrete mixtures (short-term aging) preparation and during the asphalt concrete (long-term aging) operation. The aging process mainly involves material oxidation and manifests itself in physical changes such as volatile fractions loss or bitumen hardening [7, 9].

Bitumen aging results to increase its cohesive strength and softening temperature. In addition, oxygen-containing functional groups are formed (determined by IR spectroscopy) as a result of interaction with oxygen. There are changes in the four main bitumen fractions, namely, saturated and aromatic hydrocarbons, resins and asphaltenes, in the form of components transition from less to more polar fractions. Since the bitumen fractions have different reactivity during oxidation, the result is the decrease of naphthenic aromatic compounds content with a corresponding increase of asphaltenes proportion [10].

One of the main factors is thermal oxidative bitumen aging, which is an irreversible chemical reaction between bitumen and atmospheric oxygen [10]. Bitumen oxidation can occur during mixing with hot aggregates, during transport, laying and compaction of mixture. In addition, bitumen aging is possible when it was modified by polymers due to increased preparation temperature (more than 170 °C) and high shear rate for a long time (2–4 hours) [11, 12]. Slow oxidation processes occur during the entire service life in road pavements [13].

There are number of publications confirming the effectiveness of studies about bitumen modification by nanoparticles of titanium dioxide, copper, zinc, silicon, etc., as well as nanoparticles of clay minerals (kaolinite, montmorillonite, etc.) [14–16]. It has been established that these additives have improved the bitumen quality, including increasing the binder aging resistance [17–19]. It should be noted that the studies performed with these modifiers were considered on residual bitumen, and in Russia, oxidized bitumen was mainly used. These bitumens are obtained by oxidizing oil residues with atmospheric oxygen in various types of oxidizing reactors. Residual bitumen is mainly obtained by two technologies: with a sufficiently high vacuum of the oil distillation residue or by compounding oil residues [20]. There is the fundamental difference in the road bitumen quality produced by domestic oil refineries from bitumen produced abroad, where only oils of a certain group and chemical composition, in which there is practically no solid paraffin, are used as raw materials. The number of oils suitable for the residual bitumen production is limited in Russia. This explains the fact that almost the entire volume of consumed bitumen is produced by the oxidation of various oil residues [21].

In this regard, there are practically no studies on improving the bitumen aging resistance in Russia.

Studies [22, 23] describe the possibility of clay minerals using as modifiers. One of these minerals is kaolinite [19, 24, 25].

In this regard, the purpose of the work is to establish the degree of properties change and the possibility of aging resistance increasing of Russian bitumen modified by kaolinite.

To achieve this goal, the following tasks were solved:

- study the kaolinite influence on basic physical and mechanical properties of modified bitumen;
- study the effect of kaolinite on the change of softening temperature of modified bitumen after aging in the RTFOT furnace;
- study by IR spectroscopy the main absorption bands as a result of bitumen aging in the RTFOT furnace;
- establishment by the method of IR spectroscopy of the kaolinite influence on the modified bitumen aging resistance.

2. Materials and Methods

The study used bitumen grade BND 100/130 produced by Novokuibyshevsk Oil Refinery JSC in accordance with Russian State Standard GOST 33133-2014 (Table 1).

Kaolinite clay is produced in Samara region, LLC NPP Industrial Minerals. In accordance with Russian State Standard GOST 9169-75, according to mineral composition, it refers to kaolinite clay with a kaolinite mineral content up to 95 %.

The preparation of modified bitumens was carried out on a SILVERSON L5M laboratory mixer (Great Britain). The packing used was a standard high shear mixing head with square holes. Mixing of the modified bitumen was carried out in tin cans with a volume of 1 liter. An oil bath Memmert ONE 22 (Germany) was used to heat and maintain the required operating temperature during the preparation and ripening process. Silicone oil PMS-100 (LLC Penta Junior, Russia) was used as a heat carrier. A tin with a measured amount of bitumen was immersed in an oil bath (the coolant liquid did not reach the top of the tin by 2–3 cm).

The modified bitumen preparation was carried out as follows: a pre-weighed amount of bitumen in can was immersed in an oil bath. The bitumen was heated up to operating temperature of preparation –

160 °C. The mixer head was immersed in bitumen and a mixing speed of 2500 rpm was created. Kaolinite clay, previously dried to constant weight, was introduced at a rate of 5 g/min. After the introduction of additives, mixing was carried out for 10 minutes at a temperature of 160 °C and a rotation speed of 2500 rpm. It was previously established [19, 22, 23] that significant changes occurred in the range of 2–6% kaolinite content in bitumen. Taking these data into account, research estimates range from 2 to 8%.

Table 1. Physical and technical properties of bitumen grade BND 100/130.

Indicator name	Unit	Actual values	Requirements Russian State Standard GOST 33133-2014	Test Methods
2	3	4	5	6
Penetration at 25 °C	0.1 mm	107	101–130	Russian State Standard GOST 33136
Softening point	°C	46	not lower than 45	Russian State Standard GOST 33142
Ductility at 0 °C	cm	4.6	not less than 4.0	Russian State Standard GOST 33138
Softening point change after warming up	°C	5,6	No more than 7.0	Russian State Standard GOST 33140 Russian State Standard GOST 33142
Fraas brittle point	°C	-21	Not higher than -20	Russian State Standard GOST 33143
Flash point, not below	ºC	293	230	Russian State Standard GOST 33141
Sample mass change after aging, %, no more	%	0.21	0.6	Russian State Standard GOST 33140

Modified bitumen tests were carried out in accordance to Russian State Standard GOST 33133-2014 requirements for the following indicators:

- Penetration at 25 °C. The test method consists in measuring the depth to which the penetrometer needle is immersed in the test bitumen sample under certain conditions (temperature, load and duration of load application), which is expressed in units corresponding to tenths of a millimeter (0.1 mm). The LinteL PN-20 penetrometer, produced by JSC Bashkir Special Design Bureau Neftekhimavtomatika, was used as a device.
- Softening point. The test method consists in determining the temperature at which bitumen, poured and cooled inside rings of given sizes, softens under test conditions and, moving under the weight of a steel ball, touches the bottom plate. As a device, an automatic apparatus for determining the softening temperature of petroleum bitumen Lintel KISH-20, manufactured by JSC Bashkir Special Design Bureau Neftekhimavtomatika, was used.
- Ductility at 0 °C. The method consists of stretching of bitumen sample at a constant rate, at a given temperature, to determine the maximum tensile force and bitumen extensibility. As a device, an automatic apparatus for determining the extensibility of oil bitumen LinteL DB-20-100, manufactured by JSC Bashkir Special Design Bureau Neftekhimavtomatika, was used.
- Fraas brittle point. The test method consists of cooling at a uniform rate and periodically bending the bitumen sample and determining the temperature at which cracks appear or the bitumen sample breaks. As a device, an automatic apparatus for determining the temperature of oil bitumen LinteL ATX-20, manufactured by JSC Bashkir Special Design Bureau Neftekhimavtomatika, was used.
- Sample weight change and softening point change after aging in the RTFOT oven. The method consists of exposing a moving thin film of bitumen to high temperature (163 °C) and air and determining the effect of this exposure on the bitumen by comparing the physical and chemical parameters of the bitumen obtained before and after exposure. Rolling thin-film B066N1 equipment (Matest, Italy) was used.
- Dynamic viscosity at 105 °C, 135 °C and 165 °C, respectively, according to Russian State Standard GOST 33137-2014. The test method is to measure the relative flow resistance caused by shearing bitumen by rotating configuration elements. Dynamic viscosity is calculated as the ratio between applied shear stress and shear rate. A Brookfield DV2T viscometer (Brookfield, USA) was used.

The IR spectroscopy method was used to identify functional groups, as well as to determine the effect of aging after the RTFOT oven. The registration and processing of IR spectra was carried out using a Miracle ATR for FT-IR Spectrometer ATR attachment (ZnSe crystal) in the region of 4000-650 cm^{-1} on a Perkin Elmer FT-IR Spectrometer model Spectrum 65 spectrophotometer under standard recording conditions and the supplied software.

3. Results and Discussion

It can be seen (Figure 1) that kaolinite introduction in an amount of 2–8% into bitumen, the softening point, Fraas brittle point, dynamic viscosity at 105 °C, 135 °C and 165 °C increased by 1.1–2.6 %, 9.5–28.6 %, 9.2–33.1 %, 6.9–24.1 %, 11.1–55.6 %, respectively. The following indicators decreased: penetration at 25 °C by 8.4–22.4 %; ductility at 0 °C by 4.3–15.2 %; sample weight change and softening point change after aging in the RTFOT oven by 12.5–28.6 %.

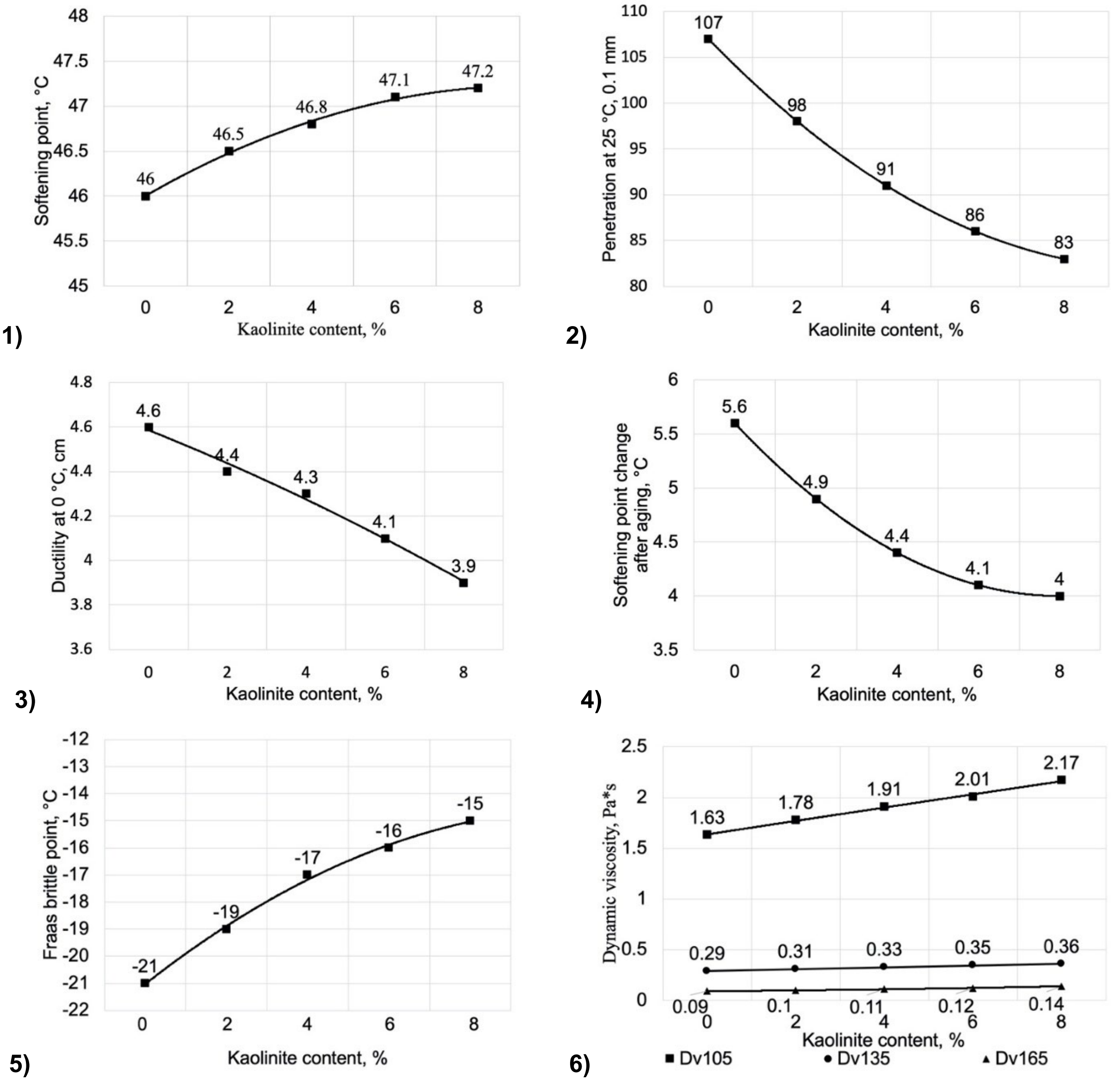

Figure 1. The dependence of bitumen physical and mechanical characteristics from the kaolinite content:
1 – softening point; 2 – penetration at 25 °C; 3 – ductility at 0 °C; 4 – softening point change after aging; 5 – Fraas brittle point; 6 – dynamic viscosity at 105 °C, 135 °C and 165 °C.

It should be noted that the effect increased with the increasing of kaolinite amount and it was observed in seven studied dependencies (Figure 1). The exception is the parameter that determines the softening temperature change after RTFOT oven aging. The effectiveness of the kaolinite influence on bitumen with a content of more than 6 % for the softening temperature change after RTFOT oven aging is sharply reduced.

In our opinion, the increase of dynamic viscosity and softening point, as well as a decrease of penetration at 25 °C and ductility at 0 °C, are possibly associated with the solid clay particles presence in bitumen, which cause less mobility of bitumen matrix and, consequently, higher flow resistance. In addition, the following assumption is interesting [26, 27]: due to the dual nature (polar and nonpolar), clay plates can be located at the interface between asphaltenes and maltenes, and aromatic compounds, due to their lower molecular weight, can intercalate between clay plates. Clay plates, partially exfoliating, can be distributed in aromatic compounds and resins, located mainly at the interface between them. In the intercalation process, the clay mineral plays the surfactant role, which provides and enhances the interaction between the asphaltene and maltenic phases. The consequence of this process is a decrease in asphaltene micelles size. As a result, the amount and mobility of the "free" aromatic fraction decreases. This, obviously, is the reason for the resulting increase of modified bitumen viscosity.

The established dependencies (Figure 1) show that the short-term aging reduction with the kaolinite introduction in amount from 2 % to 8 % into the bitumen composition was 12.5–28.6 %.

The given IR spectra of the original bitumen and bitumen after aging in the RTFOT furnace and their difference spectrum (Figures 2 and 3) allow us to judge the changes that occur after aging in the middle spectrum part with following peaks: 1738, 1640, 1654, 1217, 1033 cm^{-1} absent in the original bitumen. The band at 1738 cm^{-1} corresponds to stretching vibrations of C=O carbonyl groups formed during the thermal-oxidative degradation, and the 1217 cm^{-1} peak corresponds to C-O stretching vibrations, which are part of the ester groups. The 1033 cm^{-1} peak corresponds to S=O bonds of sulfoxide groups, which are also formed during aging. These newly formed groups intensity in the bitumen spectrum (after aging in the RTFOT furnace) is quite high, which indicates intensive processes of bitumen thermal degradation during aging in the RTFOT furnace.

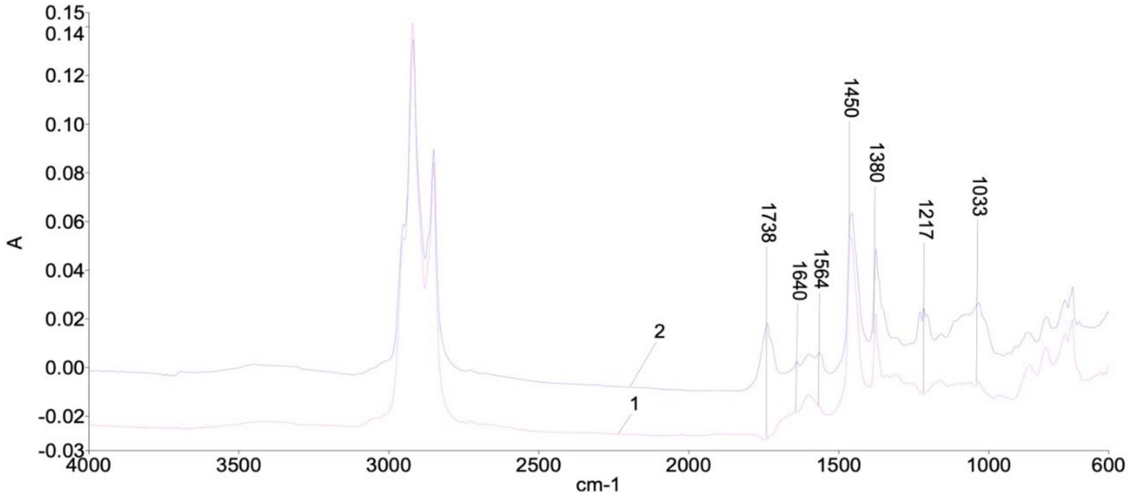

Figure 2. The original bitumen IR spectra and bitumen after aging in the RTFOT furnace: 1 – original bitumen; 2 – bitumen after aging in the RTFOT furnace.

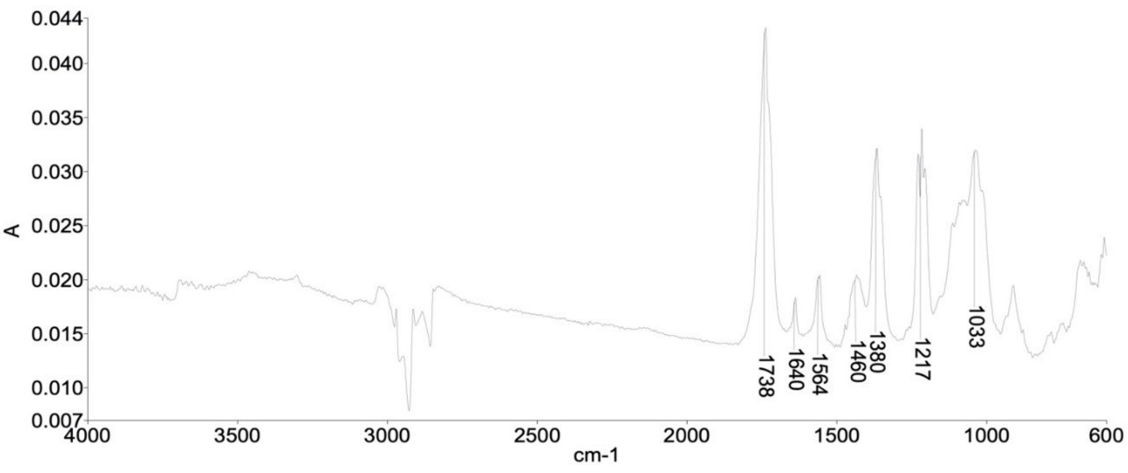

Figure 3. Difference IR spectrum of the original bitumen and bitumen after aging in the RTFOT furnace.

In the middle spectrum part the methylene CH_2 groups bending vibrations correspond to a peak at 1460 cm^{-1}, and to methyl CH_3 groups - a peak at 1380 cm^{-1} (Figure 3). In the middle spectrum part these vibrations are separated, while in the high-frequency part, at 2800-3000 cm^{-1} stretching vibrations, they are strongly superimposed.

Along with the fact that all the "aging" bands at 1738, 1217, 1033 cm^{-1} are clearly manifested in the difference spectrum, it is noteworthy that the 1380 cm^{-1} band becomes noticeably more intense than the 1460 cm^{-1} peak, while in the original spectrum the dependence is inverse. This means that the CH_3 groups concentration increases during aging significantly. Methyl groups are usually the end groups of chain linear or slightly branched organic molecules, and methylene CH_2 groups form the chain backbone. A significant increase of end groups concentration should mean a general significant molecules "shortening" as a result of thermal degradation and, as a result, a decrease of weighted average molecular weight of bitumen macromolecules after aging by the RTFOT method.

Bitumen IR spectra before and after aging in the RTFOT furnace (Figure 4, curves 1 and 2, respectively), the spectrum of bitumen modified by kaolinite after aging in the RTFOT furnace (curve 3), as well as the difference spectrum of bitumen modified by kaolinite after aging in RTFOT furnace and original bitumen (Figure 5) shows a significant increase the bitumen aging resistance level. As can be seen from the spectra, the kaolinite introduction has a very significant effect on the aging processes: the “aging” peaks at 1640, 1564 and 1217 cm^{-1} almost completely disappear in the difference spectrum, and the carbonyl absorption band at 1738 cm^{-1} intensity significantly decreases. It is difficult to judge the sulfoxide groups behavior in this case, since the 1033 cm^{-1} S=O groups absorption peak in this case is superimposed on a very intense 1030 cm^{-1} Si-O kaolinite peak, which is the reason of sharp increase of peak intensity in the modified by kaolinite bitumen spectrum, after aging in the RTFOT furnace and the original bitumen. However, it can be assumed that the kaolinite introduction inhibits the sulfoxide groups formation as well as carbonyl ones. We add that the peaks intensities ratio at 1460 cm^{-1} and 1380 cm^{-1} of the CH_2 and CH_3 groups deformation vibrations in the modified by kaolinite bitumen spectrum, after aging in the RTFOT furnace, becomes equally comparable (Figure 5). The above facts quite convincingly testify to the unambiguous kaolinite influence on the thermal and mechanical bitumen degradation processes.

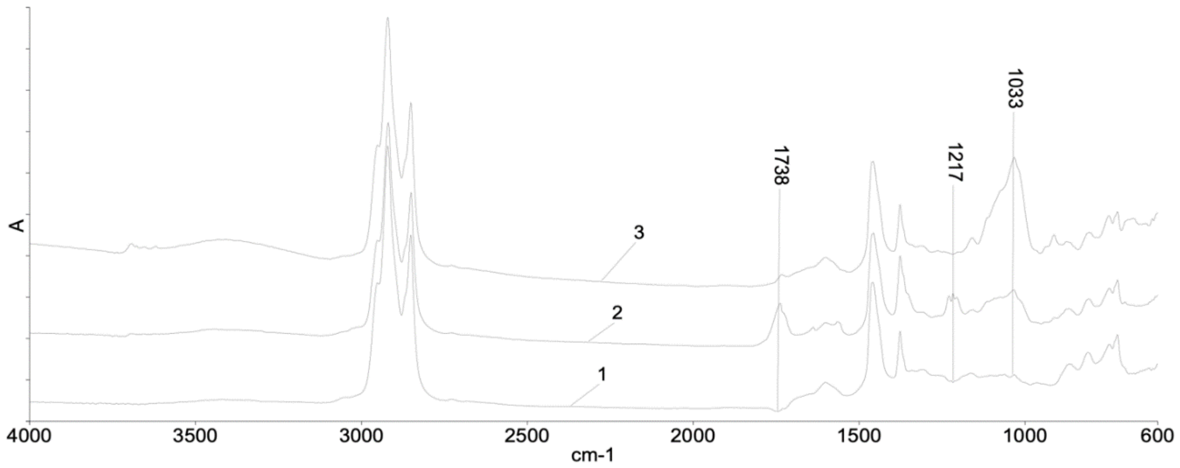

Figure 4. IR spectra:
1 – original bitumen; 2 – bitumen after aging in the RTFOT furnace;
3 – bitumen modified by kaolinite, after aging in the RTFOT furnace.

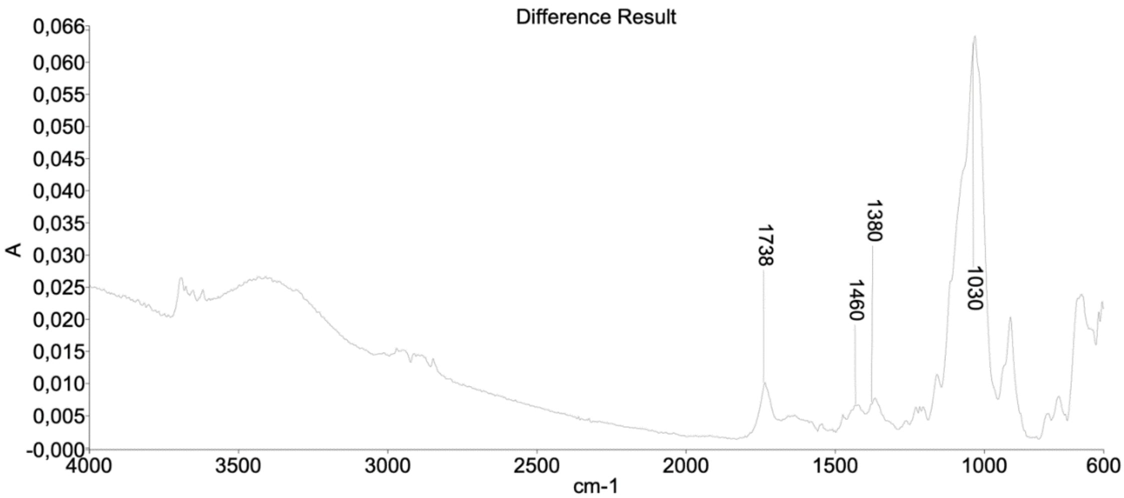

Figure 5. Difference spectrum of bitumen modified by kaolinite after aging in the RTFOT furnace and the original bitumen.

It was noted in [23] that in bitumen, which is in an intermediate state between a sol and a gel, some asphaltenes combine to form open-packed irregular micelles, which are peptized by resins. In our opinion, in a binary mixture of bitumen modified by kaolinite, due to clay plates located mainly at the asphaltene interface, solvation processes with a bitumen structure close to sol proceed more actively. This raises the possibility that these processes result clay plates inhibit the bitumen molecules oxidation and the light fraction from the binder volatilization. As a consequence of these processes, as noted in [26, 27], as a result of the clay plates introduction the paths for the oxygen influx and the light fraction molecules exit from the bitumen matrix become more elongated, which significantly affects the bitumen binder aging mechanism.

The effect of mass change after RTFOT oven aging was studied. When kaolinite is introduced in bitumen from 2 to 8 %, the mass changes were 0.6, 0.32, 0.21, 0.11, 0.09 %, respectively, which confirmed the decrease the light fraction molecules yield as a result of binder thermal oxidation [27, 28].

The experimental data obtained in this work confirm the similarity of the processes described in the studies [19, 22–28].

4. Conclusions

It was found that the kaolinite introduction in amount of 2–8 %, the softening point, Fraas brittle point, dynamic viscosity at 105 °C, 135 °C and 165 °C increased by 1.1–2.6 %, 9.5–28.6 %, 9.2–33.1 %, 6.9–24.1 %, 11.1–55.6 %, respectively. The following indicators decreased: penetration at 25 °C by 8.4–22.4 %; ductility at 0 °C by 4.3–15.2 %. The increase of dynamic viscosity and softening point, as well as the decrease of penetration at 25 °C and ductility at 0 °C are obviously associated with the solid clay particles presence in bitumen, which causes bitumen matrix less mobility and, consequently, higher flow resistance. In addition, the following assumption is interesting: due to the dual nature (polar and nonpolar), clay plates can be located at the interface between asphaltenes and maltenes, and aromatic compounds, due to their lower molecular weight, can intercalate between clay plates. Clay plates, partially exfoliating, can be distributed in aromatic compounds and resins, located mainly at the interface between them. In the intercalation process, the clay mineral plays the surfactant role, which provides and enhances the interaction between the asphaltene and maltenic phases. This process consequence is a decrease the asphaltene micelles size. As a result, the amount and mobility of the "free" aromatic fraction decreases. This, obviously, is the resulting reason of increasing the modified bitumen viscosity.

Based on the analysis of changes the physical and mechanical properties and structure by IR spectroscopy, it was established that the short-term aging reducing on 12.5–28.6 % of modified bitumen with a kaolinite content of 2 % to 8 % have been possible. It was established that the kaolinite introduction into bitumen have caused a significant decrease of aging processes, which has been confirmed by the IR spectroscopy data: the peaks at 1640, 1564 and 1217 cm^{-1} almost completely have disappeared in the difference spectrum, and the intensity of carbonyl absorption band at 1738 cm^{-1} has decreased. In our opinion, in the binary bitumen mixture modified by kaolinite, due to clay plates located mainly at the asphaltene interface, solvation processes with a bitumen structure close to sol proceed more actively. This suggests the possibility that clay plates prevent the oxidation processes of bitumen molecules and volatilization the light fraction from the binder. As a result of clay plates introduction, the oxygen inflow paths and the light fraction molecules exit from the bitumen matrix become more remote, which significantly affects the aging mechanism of bituminous binder.

The obtained experimental data confirmed the similarity of the processes described in previously published studies of other authors and the continuing research prospects in the direction of increasing the bitumen binders aging resistance at searching for new modifiers types and determining them optimal quantities.

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