Comparative evaluation of quality of hard and special paving grade bitumens’

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Abstract. The application of hard paving grade bitumen or polymer modified bitumen is one of the solutions to prevent a creep of road pavement. The implementation of special hard bitumen and multigrade bitumen starts at 80-th of the last century in a purpose to construct high module asphalt pavement. The results of comparative evaluation of conventional and fundamental (cohesion, adhesion, viscosity) quality indicators for hard paving grade bitumen and multigrade bitumen are given in the article. Low value of temperature susceptibility and a wide plasticity interval are obtained as characteristics of multigrade bitumen. The same is inherent to the air-blown bitumen, with a “gel” type structure. Low ductility and a significant change in softening point temperature after hardening can be considered as disadvantages of multigrade bitumen.

1. Introduction

The early failure of flexible road pavements is stipulated by steady increasing in amount of traffic load and axial loads on a pavement all over the world. Early failure reduces the terms between overhauls as a common durability of a road pavement. Permanent deformation (as a rutting, waves etc.) initiated by a decline in quality of a road pavement as by a residual deformation of a base layers of a road construction is a wide spread reason of low durability of road pavement.

The implementation of low penetration bitumen binders (hard paving grade bitumen, polymer modified bitumen and bitumen with warm mix additives) is one of ways to reach a high quality of road pavements and reduce permanent deformations. Mastic asphalts with natural bitumen are the first road pavements in a history. The obtained pavements were characterized by high strength and durability because the low penetration (0 … 40 × 0.1 mm) and extremely high softening point (≥ 90 … 100 ºС) of the binder in their composition. [1]. Road bitumen with penetration range 40…70 × 0.1 mm was implemented in a road building industry in a result of increasing in an oil refining capacity and chemistry production of bitumen at the beginning of the 20-th century [1–2]. The further development of road industry and designing of new types of asphalt concrete led to the common use of bitumen with penetration range from 100…200 × 0.1 mm to 200…300 × 0.1 mm.

The returning of hard bitumen in road mixtures design got in trend in 50-th 70-th last century. This was a consequence of a few main factors [3]. At first, using of dense Middle Eastern oil (as a result of oil crisis) led to decreasing in penetration of bitumen as a product of oil refinery. At second, the increasing in traffic load caused significant residual deformations of existed road pavements that made a choice of bitumen with high penetration doubtful. At third, the development of oil refining technology allowed producing of high-quality road bitumen with lower penetration. Intention to
improve the durability of the road pavements raised more severe demanding for the bitumen temperatures of softening point and breaking point to extend the plasticity interval. It was found that the most common ways to increase softening point temperature and to decrease breaking point temperature are a polymer modification and an improvement in technological processes of bitumen production [3].

In 1984 Royal Dutch Shell PLC introduced the bitumen “multigrade” with lower temperature susceptibility (higher Penetration Index value) and wider plasticity range correspondingly. This bitumen was branded as “Shell Multiphalte” [4]. Since 1985 a global testing of the pavements with multigrade bitumen has been started in different countries [5]. The results of testing of a few years old pavements in field proved an absence of the residual deformations, such as rutting, and a good durability of the pavements. The gathered experience helped to implement this type of bitumen in road industry for other countries [6].

The low temperature susceptibility of Shell Multiphalte bitumen is proved by comparative analysis with conventional road bitumen and polymer modified bitumen, presented in a research report [6]. However for the multigrade bitumen significant changes of the standard properties after hardening by RTFOT are registered. In compare with conventional road bitumen the average value of softening point temperature for the multigrade bitumen is higher than 3.5 °C, average residual penetration for the multigrade bitumen is lower on 5.5 %. The viscosity changes with temperature are similar for both Shell Multiphalte and polymer modified bitumen, which causes the higher temperatures for the multigrade bitumen at mixing with aggregates, and higher temperatures at the compaction (round 19-27 °C more than for the paving grade bitumen) [5, 6]. Due this multigrade bitumen are taking an intermediate position between conventional bitumen and polymer modified bitumen [7].

At the end of 90-th an extended research of multigrade bitumen has been carried out in Poland [8, 9]. The obtained data prove the efficiency of the multigrade bitumen as a component of the High Stiffness Module Asphalt concrete.

Nowadays the multigrade bitumen properties are normalized with European EN 13924-2:2014 [10] and Australian AS 2008-2013 [11] standards. Multigrade bitumen applies as a component of the thin layers of asphalt concrete and in a composition of High Stiffness Module Asphalt concrete.

The process of harmonization of the European standards for test methods and quality of the pavement and special bitumen has been started in Ukraine since 2016. Considering this the finding differences and peculiarities of these binders technology in contrast with conventional air blown road bitumen is a high priority research topic.

2. Materials and methodology

Special bitumen multigrade Unibit 35/50, low penetration bitumen 20/30 and 35/50 supplied by Polish “LOTOS Asphalt” are chosen for the research. For comparative analysis bitumen (Bo) is obtained in a laboratory with a petroleum binder of low viscosity (penetration at 25 °C is 214 × 0.1 mm, softening point temperature is 37.2 °C). To obtain bitumen Bo petroleum binder of low viscosity was blown by air flow with a rate 5 l/min at temperature 200 ± 5 °C in compressor oxidizer until its penetration got close to the value of multigrade bitumen, chosen for the research.

The common quality indexes are obtained for this bitumen (penetration, softening point temperature, Fraass breaking point temperature, ductility and properties changing after hardening). In addition, a number of specific tests are performed: cohesion test, adhesion test, temperature susceptibility and viscosity tests, which can provide more comprehensive evaluation of advantages and disadvantages of multigrade bitumen.

Cohesion test is performed in a planar shear condition [12] with finding of the maximal stress at deformation rate of 1 s⁻¹ at 25 °C. For this purpose, the binder sample in a layer of 200 μm. is placed between two parallel polymer tapes, making a bound. Stretching of this bound with a constant speed initiates sample deformation with a shear rate that can be set in a range 0.2 s⁻¹ – 4.2 s⁻¹. Maximal stress is determined as a maximal value of the resisting force at deformation, divided on a binder sample (spot) area.
Adhesion is estimated by the amount of binder that stays on a surface after exposing in water medium at specific conditions [13]. For this purpose glass surface $70 \times 25$ mm is covered with 0.35 g of bitumen (at this condition the thickness of the bitumen layer is round 200 um). The samples (glass plate with a bitumen layer) are heating at 85 °C higher than a softening point temperature during 30 minutes in a horizontal position (bitumen surface on a top). After cooling samples are placing in a water bath with distilled water at a temperature 75 °C during 25 min. Adhesion is calculated as a percent of the surface covered with bitumen after the test.

Temperature susceptibility of bitumen is evaluated by the Penetration Index, which is calculated with the conventional equation (1) and equation modified by a softening point temperature replacing. In a conventional equation the softening point temperature is defined by Ring and Ball test. In a modified equation the temperature at which penetration reaches $800 \times 0.1$ mm value ($T_{800}$) is set as a softening point temperature.

$$IP = \frac{(20 \times T) + (500 \times \lg P_{25}) - 1952}{T - (50 \times \lg P_{25}) + 120}$$ (1)

In the equation $P_{25}$ is a penetration at 25 °C, $T$ is a softening point temperature by Ring and Ball test ($T_{R&B}$), or a temperature at $800 \times 0.1$ mm penetration ($T_{800}$). $T_{800}$ temperature is found by graph of penetration changing within a test temperature set 5 °C, 15 °C, 25 °C and 35 °C [14].

Evaluation of the colloidal structure type of bitumen is performed with “Coefficient of Standard Properties” ($C_{STD}$) by equation (2).

$$C_{STD} = \frac{(T_{R&B} - T_{Fraass}) \cdot L}{25 \cdot D}$$ (2)

In the equation $T_{R&B}$ is a softening point temperature by Ring and Ball test, $T_{Fraass}$ is a breaking point temperature by Fraass, $L$ is a length of a “neck” part of the dumbbell form for the ductility test (3 cm), 25 is a ductility test temperature, $D$ is a ductility. When the value of $C_{STD}$ is higher than 0.13 bitumen is classified as a “gel” type. Value of $C_{STD}$ is lower than 0.08 points on a “sol” type of bitumen. Intermediate values of $C_{STD}$ refer to a “sol-gel” bitumen type.

Binder viscosity obtained with a rotary rheometer in a wide range of temperatures (from 90 °C to 170 °C) and shear rates (from 0.005 s$^{-1}$ to 300 s$^{-1}$).

The indicators of quality of binders, chosen for the analysis, displayed in Table 1.

### 3. Results and Discussion

The experimentally obtained data points on both convincing advantages and disadvantages of multigrade bitumen. As an advantage can be considered lower temperature susceptibility, that is a result of high softening point temperature, and low Fraass breaking point temperature. Due this the plasticity interval of Unibit 35/50 is significantly wider than the one of hard bitumen 20/30 and 35/50 (on 15.9 °C and 18.3 °C respectively).

The change of the penetration with a temperature was obtained to estimate the temperature susceptibility of the chosen bitumens (Figure 1). The shown data prove the low temperature susceptibility of the multigrade bitumen thus temperature dependency of its penetration flatter than others. Such type of dependency characterizes a “gel” structural type of bitumen (in this research work it is bitumen Bo) and a polymer modified bitumen. Wherein the softening point temperature and a temperature at $800 \times 0.1$ mm penetration for the multigrade bitumen are close, that is inherent to the “sol” type bitumen.

By the Penetration Index value, obtained with softening point temperature and with a temperature at $800 \times 0.1$ mm penetration, multigrade bitumen can be characterized as a “sol-gel” type, but with a Coefficient of Standard Properties, that with softening and breaking point temperatures taking in a consideration ductility at 25 °C [15], multigrade bitumen is a typical sample of the “gel” structure binders ($C_{STD} = 1.91$). This can explain the close values of the softening point temperature and Fraass
breaking point temperature for both multigrade bitumen and Bo bitumen. And the last one can be classified as a “gel” type bitumen only, with C_{STD} = 0.83.

| Table 1. Bitumen properties. |
|-----------------------------|
| Index                      | Value          |
| Penetration at 25 °C, × 0.1 mm | 20/30 35/50 Unibit 35/50 Bo |
| Penetration at 0 °C, × 0.1 mm | 10 15 21 18 |
| Softening point temperature by Ring and Ball test (T_{R&B}), °C | 62.1 53.2 63.5 63.7 |
| Fraass breaking temperature (T_{Fraass}), °C | -6.5 -13.0 -21.0 -19.0 |
| Ductility at 25 °C, cm | 25 > 150 5.3 12 |
| Penetration Index, calculated with a softening point temperature (PI_{TR&B}) | -0.02 -0.72 0.85 1.13 |
| Plasticity Interval (T_{R&B} – T_{Fraass}), °C | 68.6 66.2 84.5 82.7 |
| Coefficient of Standard Properties (C_{STD}) | 0.33 < 0.05 1.91 0.83 |
| Temperature at 800 × 0.1 mm penetration (T_{800}), °C | 67.0 57.0 63.0 69.5 |
| Temperature difference ∆T = T_{R&B} – T_{800}, °C | -4.9 -3.8 0.5 -5.8 |
| Penetration Index, calculated with a T_{800} temperature (PI_{T800}) | 0.82 0.11 0.76 2.13 |
| Hardening at 163 °C during 5 hours | Retained penetration, % 88.5 81.8 94.0 87.0 |
| Increase in softening point, °C | 4.3 4.8 7.3 4.7 |
| Change of mass, % | 0.36 0.36 0.44 -2.4 |
| Adhesion to the glass surface at 75 °C, % | 88.8 62.0 73.0 74.7 |
| Cohesion at 25 °C and at a shear rate 1 s^{-1}, MPa | - - 0.248 0.271 |

Figure 1. Temperature penetration dependencies of bitumens.

One more evidence that colloidal structure of multigrade bitumen even closer to a “gel” type than the one of specially prepared in laboratory bitumen Bo is speed-cohesion dependencies of these binders (Figure 2). With comparison of the properties of Unibit 35/50 and Bo bitumens that have the same penetration differ in cohesion. For multigrade bitumen cohesion is lower in 1.09 times than for
Bo bitumen. This is in agreement with data, obtained by Kolbanovskaya in [16], who noticed that cohesion values decreasing with changing of the bitumen structure from “sol” to “gel” type.

![Figure 2. Influence of the shear rate on a cohesion of bitumen at 25 °C.](image)

In comparison with hard distilled bitumen, Unibit 35/50 is characterized by a higher quality at low temperature. The penetration at 0 °C of multigrade bitumen is in 2.1 and 1.4 times higher than the one of hard bitumen 20/30 and 35/50 correspondingly and Fraass breaking point temperature is lower (on 14.5 and 8.0 °C correspondingly). This may lead to conclusion that asphalt concrete with Unibit 35/50 bitumen may be more flexible at 0 °C and may have higher crack resistance at winter temperatures. Low temperature performance of the multigrade bitumen is similar to the one of the high polymer modified bitumen [15].

Contra verse the low ductility (5.3 cm) at 25 °C can be considered as an obvious disadvantage of the multigrade bitumen. This ductility is much lower than the one for the hard bitumen 20/30 (25 cm) and air-blown bitumen Bo (12 cm). Taking into account the fact that ductility is stipulated by a chemical composition of the bitumen (concentration of resins and aromatic hydrocarbons) and indicates the cracking resistance of the asphalt concrete, it can be concluded that asphalt concrete with multigrade bitumen may have low cracking resistance.

Ductility is not included as a quality indicator in the European standards on paving grade bitumens (EN 12591 и EN 13924-2). This is grounded on a high ductility (more than 100 – 150 cm) [17] that inherent to the European distilled bitumens, but can’t be spread on the multigrade bitumen. The last one differs in a technology of producing and can’t reach the same ductility, so the normalizing of ductility values can be reasonable in this case.

The change of the bitumen quality indicators after hardening is the important technological parameter that characterizes the changes in bitumen in a process of asphalt concrete production. The data, obtained for the multigrade bitumen after hardening with TFOT method (5 hours storage at 163 °C) have a dual character. On the one hand the penetration of the multigrade bitumen stays on the same level. Such a high value of the retained penetration (94 %) is inherent to the sol type bitumen. For the multigrade bitumen it may caused by a low initial penetration of the binder. On the other hand the softening point temperature for the multigrade bitumen after hardening rises up significantly (on a 7.3 °C). Sharply change in a softening point temperature indicates a gel type of the bitumen.
The instability of the multigrade bitumen can be an explanation of obtained data. Instability can be confirmed by an increasing in mass after hardening (on 0.44 %) and by an Oliensis spot test. The last method used to evaluate the homogeneity of the binder and consists in a visualization of the structure of the spot that formed by a drop of the binder dissolved in gas on a filter paper (Figure 3). With this test method the multigrade bitumen in spite of the air-blown bitumen shows few halos that point on heterogeneity of its composition.

Certainly with the increasing in a softening point temperature the technological temperatures of the mixing and compaction of asphalt concrete with multigrade bitumen must be increased. The evaluation of the technological temperatures of the multigrade bitumen is performed with the obtained temperature dependencies of the viscosity (Figure 4).

The 0.5 Pa·s viscosity is set as criteria of the heating temperature for bitumen. Trough the all considered bitumen the highest heating temperatures are required for the air-blown bitumen Bo (168 ºC) and 20/30 bitumen (165 ºC). The technology temperature of the multigrade bitumen (155 ºC) is close to the one of the hard 35/50 bitumen (153 ºC). Because this required technological temperatures of multigrade bitumen can’t be taken as its disadvantage.

The suppliers of multigrade bitumen positioning it as a binder with lower temperature susceptibility that allows producing of asphalt concrete with higher cracking resistance at low temperature, higher shear resistance at high summer temperature and high resistance to hardening. These properties are reaching by a specific composition of the primary products and schemes of its producing.
The estimation of the multigrade bitumen as a component of the asphalt concrete is provided with mastic asphalts. According to the properties shown in Table 2 the mastic asphalt with multigrade bitumen is characterized by the lowest depth of the indenter immersion, which can guarantee the high resistance to the residual deformations. The similar depth of the indenter immersion for the mastic asphalt is gained with using polymer modified bitumen as a binder. With this the compressive strength at 20 °C of the mastic asphalt with the multigrade bitumen is the lowest.

**Table 2. Mastic asphalt properties**

| Bitumen   | Bitumen content, % | Residual porosity, % | Depth of the indenter (2 cm²) immersion after 30 min of loading at 40 °C, mm | Increasing in the depth of the indenter (2 cm²) immersion after 30 min of loading at 40 °C, mm | Compressive strength at 20 °C, MPa |
|-----------|--------------------|----------------------|--------------------------------------------------------------------------------|--------------------------------------------------------------------------------|----------------------------------|
| 20/30     | 10                 | 1.89                 | 3.06                                                                            | 0.52                                                                            | 4.52                             |
| 35/50     | 10                 | 1.73                 | 7.38                                                                            | 1.98                                                                            | 3.16                             |
| Unibit    | 10                 | 1.93                 | 2.73                                                                            | 0.38                                                                            | 2.30                             |
| 35/50     |                    |                      |                                                                                 |                                                                                 |                                  |
| Bo        | 10                 | 1.47                 | 3.36                                                                            | 0.56                                                                            | 4.20                             |

Such a contradiction in obtained results can be caused by a low temperature susceptibility of the multigrade bitumen. The test temperature of the indenter immersion is 40 °C, while the compressive strength is measuring at 20 °C. Despite a low strength at 20 °C of the asphalt concrete with multigrade bitumen, its strength at higher and lower temperatures can be higher than the asphalt concrete with conventional bitumen. The strength at 0 °C and 50 °C is obtained for two mastic asphalts with Unibit 35/50 and hard bitumen 35/50 to prove this assumption. The temperature dependencies of the strength for this asphalt concretes are shown in Figure 5.

**Figure 5. Temperature dependencies of the strength of asphalt concretes.**

The asphalt concrete with multigrade bitumen demonstrates higher performance in a range of high temperatures in terms of compressive strength then the asphalt concrete with distilled bitumen. Despite the difference in penetration of these bitumens (35 for Unibit 35/50 and 44 for hard 35/50) the fact of switching their strength dependencies with a temperature proves the advantages of the multigrade bitumen at high environmental temperatures, other words at conditions when you can obtain significant residual deformations of the asphalt concrete pavement.

The low temperature characteristics in this research work can’t be validated. The lower strength at
0 °C of the asphalt concrete with multigrade bitumen points on possibly higher cracking resistance, but this requires further research.

4. Conclusion
Multigrade bitumen is characterized by a wider Plasticity Interval in relation to air-blown and distillated paving grade bitumens according the obtained data on evaluation of their conventional and fundamental (adhesion, cohesion, viscosity) properties. The increasing in plasticity range is reached by both softening point temperature increasing and Fraass breaking point decreasing. The advantages of the gel type bitumen (higher softening point temperature, wider Plasticity Interval) as the advantages of the sol type bitumen (higher retained penetration, high cohesion) are both inherent to the multigrade bitumen. As a disadvantage of the multigrade bitumen can be considered its low ductility at 25 °C, significant change in softening point temperature after hardening and a low homogeneity.

The lowest depth of the indenter immersion for the asphalt concrete with multigrade bitumen as a high compressive strength at 50 °C proves its high performance at high environmental temperatures, which can results in low residual deformations or their absence on the pavement with multigrade bitumen.

5. References
[1] Petroleum, Asphalt and Natural Gas 1918 (Kansas City Testing Laboratory) 248
[2] Shiperovich V L 1929 Oil bitumen and their application in the road business 40
[3] Korte J-F 2002 Tverdye bitumy i wysokomoduľnye asfal'tobetony na ih osnove ďka i tehniky v dorozhnoj otrasi. Science and Engineering for Highways 3 30–31
[4] Read J, Whiteoak D, Hunter R N 2003 The shell bitumen handbook 460
[5] Koole R C, Valkering C P, Lancon D J 1992 Development of a multigrade bitumen to alleviate permanent deformation Local Government Engineers Association of Queensland Journal 10(1) 8
[6] Nicholls J C 1994 Assessment of multiphalte, the Shell multigrade bitumen. TRL Project Report (PR 61) 23
[7] Zolotarev V A, Bratchun V I 2003 Modificirovannye bitumnye vjazhushhie, special'nye bitumy s dobavkami v dorozhnom stroitel'stve. Vsemirnaja dorozhnaja assciacija. Tehnicheskij komitet «Nezhhestkie dorogi» (S8) 229
[8] Sybilski D, Mularzuk R, Palys M, Kaczycka J 2000 The First Application of Multigrade Bitumen in Poland In Proceedings of the papers submitted for review at 2nd Eurasphalt and Eurobitume congress (Held 20-22 September 2000, Barcelona, Spain) Book 2 Session 2 599-605
[9] Sybilski D 2011 Prace badawcze laboratoryjne i w pejnej skali nad zastosowaniem betonu asfaltowego o wysokim module sztywnosci w niewierzchniej drodzej Drogi i m osty 1-2 81-119
[10] EN 13924-2:2014 Bitumen and bituminous binders – Specification framework for special paving grade bitumen. Multigrade paving grade bitumens Brussels, European committee for standardization 34
[11] AS 2008 – 2013 Australian Standard. Bitumen for pavements Sydney, NSW 2001, Australia 8
[12] Zolotarev V A, Pyrig Ja I, Galkin A V 2013 Opredelenie kozhezi bitumnyh vjazhushhii na kogeziometre KHD-1 Vestnik DonNAS 1 71–77
[13] Pyrig J I, Galkin A V 2019 Sravnitel'naja ocenka adgezii bitumnyh vjazhushhii, opredelennoj po metodam, normirovannyh nacional'nymi sandartami Ukrainy. Bulletin of Kharkov National Automobile and Highway University 86
[14] Pyrig J I 2015 O pokazatele temperaturnoj chuvcvitel'nosti bitumov. Bulletin of Kharkov National Automobile and Highway University 69 128 – 133
[15] Zolotarev V A 2009 Bitumy, modificirovannye polimerami i asfal'topolimerbetony. Road Building equipment 16–23.
[16] Kolbanovskaja A S, Mihajlov V V 1973 Dorozhnije bitumy 264
[17] Hoiberg A J 1965 Bituminous materials: Asphalts, tars and pitches 604