The Method of Integral Assessment of the Quality of Oil Oxidized Bitumen as a Method for Selecting the Parameters of the Two-Stage Oxidation Process

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Abstract. Based on previous research, we used a multicriteria (integral) quality assessment to determine the priority oxidation temperatures for obtaining oil road bitumen. This method allows one to select the process parameters and ensure the production of high-quality products from paraffin raw materials. Moreover, the method allows for evaluating the effectiveness of the two-stage process at different temperatures. The paper shows the results of calculating standardized coefficients and weight coefficients for each quality indicator, as well as the integral quality indicators showing the quality of road bitumen samples. The research results showed that the priority in quality is given to a bitumen sample obtained at the oxidation temperature of 200 °C at the first stage and 260 °C at the second stage. This bitumen showed the best quality compared to other samples corresponding to GOST 33133-2014, which further confirms the previously obtained results. It is established that the multicriteria integral method of quality assessment can be used in bituminous production to identify the priority quality of bitumen and determine the process parameters depending on the quality of raw materials.

Keywords: Bitumen · Two-phasic oxidation · Quality index · Standardized coefficient · Weight ratio · Integral quality index

1. Introduction

Road construction is an integral part of economic development. Improving the quality of roads, their durability, and safety is one of the urgent tasks. Several bitumen varieties are used as a binder in creating asphalt concrete coatings depending on the climatic conditions of the region [7, 9]. Specific physicochemical properties of bitumen determine its irreplaceability as an engineering and construction material for creating paved roads. However, their quality leaves much to be desired. Due to sharp cyclic temperature differences, the service life of asphalt concrete coatings does not exceed ten years in most Russian regions. Moreover, the layers operated in heavy traffic are destroyed in 3–4 years.

In Russia, oil bitumen production is mainly carried out by the oxidation technology of fatty oil residues (tar). This technology does not always allow us to get bitumen meeting the modern requirements of GOST 33133-2014 [2] in some quality indicators. One of the reasons for this is the low quality of the raw materials. The increased content of paraffin substances causes low stretchability and insufficient elasticity of the binder. The reduced range of resinous compounds and naphthenic compounds does not provide the required values for frost resistance and parameters of thermal-oxidative aging of bitumen. The oxidation of paraffin raw materials in many bitumen plants at sufficiently high temperatures (270–280 °C) contributes to the polycondensation of aromatic compounds and resins and
partial cracking without noticeable conversion of paraffin-naphthenic base compounds [2, 7, 13]. It was found that the technological conditions of oxidation, which affect the composition and physicochemical properties of commercial bitumen, need to be selected depending on the quality of the raw materials.

Under these conditions, research aimed at improving technologies for the production of bitumen with increased durability from various raw materials is very relevant.

The studies conducted by N. G. Evdokimova et al. [3, 4] show the results of using a two-stage process of paraffin tar oxidation. This implies the possibility of regulating the physicochemical properties of oxidized bitumen to obtain products in accordance with GOST 33133-2014. In order to select process parameters and ensure the production of high-quality products from paraffinic raw materials, special methods are needed to objectively evaluate the research results to recommend them for implementation in the production. Therefore, our research aims to assess the quality of bitumen and select the parameters of the two-stage oxidation process of paraffinic raw materials based on the integrated estimation method.

2. Materials and Methods

The research object is paraffin tar, a bitumen raw material produced at Gazprom Neftekhim Salavat LLC (Salavat, Russia). Its properties are presented in table 1. The regulatory framework for analyzing the qualitative features of bitumen samples includes the current requirements of the new interstate standard for road bitumen “Bitumen Oil Viscous. Technical requirements” GOST 33133-2014 [6]. Requirements for BND 70/100 road bitumen, specified in GOST R 33133-2014, are given in table 2.

| Indicator                              | Value |
|----------------------------------------|-------|
| Conditional viscosity at 80 °C, s      | 87    |
| Outbreak temperature in an open crucible, OS | 332   |
| The softening point, °C                | 24    |
| Density at 15 °C, kg/m³                | 1,007 |

*Source:* Compiled by the authors.

Several works [3, 6, 8] approved the oxidation of raw materials in two stages. The first stage was carried out at temperatures of 180, 200, and 220 °C. When the softening temperature of bitumen reached 40 °C, the second stage of oxidation was carried out at 250, 260, and 270 °C (other conditions being equal). As a result, samples of bitumen with a softening temperature of 47 °C were obtained.

| Indicator name                        | GOST Norm 33133-2014               |
|---------------------------------------|------------------------------------|
| The depth of needle penetration, × 0.1 mm: |
| at 25 °C                              | 71–90, not less than 20            |
| at 0 °C                               |                                    |
| Ring and ball softening temperature, °C | not lower than 47 °C               |
| Extensibility, cm                     | not less than:                     |
| at 25 °C                              | 62                                 |
| at 0 °C                               | 3.7                                |
| Brittleness temperature               | not exceeding -15 °C               |
| Softening temperature change after heating, °C | not exceeding 7 °C     |
| Adhesion, wt %                        | not less than 94                   |

*Source:* [14].

A multicriteria (integral) quality assessment method was used to determine priority oxidation temperatures for the two-stage oxidation process, taking into account the best quality indicators of the
obtained bitumen [1, 10, 12]. This method allows us to consider a significant number of heterogeneous indicators in relative and absolute values.

The method of comprehensive and integrated evaluation consists of calculating the final quality indicator considering the significance (weight) of each property indicator of the evaluated and basic (reference) samples. The method was tested by N. G. Evdokimova, N. N. Luneva, N. A. Egorova, et al. [5]. When choosing quality indicators, we established a list of quantitative features of the product properties that are a part of its quality and assess the quality of products.

This method allowed us to evaluate the quality of the obtained road bitumen samples according to the GOST 33133-2014 (BND 70/100) standard and determine the priority parameters of the two-stage tar oxidation.

3. Results

After the two-step oxidation, we obtained 12 bitumen samples (table 3). It was found that the samples obtained at temperatures of 180/270 °C, 200/270 °C, and 220/270 °C did not comply with the current GOST 33133-2014 standard. Therefore, these samples were not used in the integral quality assessment. Table 4 shows the physicochemical properties of the selected samples.

Qualitative indicators were evaluated by the weight coefficient \( k_i \), which shows the degree of their importance. It is determined by the Fishburn scale [11]:

\[ k_i = 2(m - i + 1) / [m(m + 1)] \]

where \( m \) – the number of qualitative indicators;
\( i \) – the number of the current quality key figure.

When calculating, the condition that \( \sum k_i = 1 \) at \( k_i \geq 0 \) was met. Table 5 shows the calculation of standardized coefficients and weighting coefficients for each quality indicator. Table 6 shows the integral quality indicators of road bitumen samples.

| Table 3. Physicochemical properties of bitumen obtained during two-stage oxidation. |
|------------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Indicator                          | Viscose petroleum road bitumens 70/100 | Oxidation temperature of the first stage, °C | 180 | 200 | 220 | Oxidation temperature of the second stage, °C | 250 | 260 | 270 |
| Viscous petroleum road bitumens 70/100 | 21 | 39 | 47 | 48 | 43 | 56 | 46 | 42 | 50 | 46 |
| Softening temperature by ring and ball, °C | 47 | 47 | 47 | 47 | 47 | 47 | 47 | 47 | 47 |
| The depth of needle penetration at 25 °C, × 0,1 mm | 70–100 | 64 | 84 | 70 | 64 | 78 | 72 | 67 | 74 |
| Extensibility at 25 °C, mm | 620 | >1,500 | >1,500 | >1,500 | >1,500 | >1,500 | >1,500 | >1,500 | >1,500 |
| Maximum tensile force, H | – | 1.50 | 1.44 | 1.06 | 1.33 | 1.30 | 0.78 | 1.15 | 1.19 |
| The temperature of softening after heating, °C | – | 50 | 54 | 52 | 50 | 51 | 50 | 51 | 51 | 54 |
| The temperature of softening after warming up, °C | 7 | 3 | 3 | 5.5 | 3 | 3.5 | 6 | 4 | 4.5 | 7 |
| The change in sample weight after heating, % | 0.6 | 0.01 | 0.1 | 1.00 | 0.01 | 0.15 | 1.25 | 0.02 | 0.2 | 1.70 |
| Brittleness temperature, °C | -18 | below | below | below | below | below | below | below | below | below |

Source: [3, 4].
Table 4. Physicochemical properties of the bitumen used for the study.

| Indicator                                      | BND 70/100 | Temperature, °C | 180 | 200 | 220 | 250 | 260 | 250 | 260 | 250 | 260 |
|------------------------------------------------|------------|-----------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Softening temperature by ring and ball, °C     |            |                 | 47  | 47  | 47  | 47  | 47  | 47  | 47  | 47  | 47  |
| The depth of needle penetration at 25 °C, × 0.1 mm | 70         |                 | 64  | 84  | 64  | 78  | 67  | 74  |     |     |     |
| The depth of needle penetration at 0 °C, × 0.1 mm | 21         |                 | 39  | 47  | 43  | 56  | 42  | 50  |     |     |     |
| The change of softening temperature after heating, °C | 7          |                 | 3   | 3   | 3   | 3.5 | 4   | 4.5 |     |     |     |
| The change of sample weight after heating, %    | 0.6        |                 | 0.01| 0.1 | 0.01| 0.15| 0.02| 0.2 |     |     |     |
| Adhesion, % of masses.                          |            |                 | 94  | 98  | 97  | 96  | 94  | 94  | 94  |     |     |

Source: Compiled by the authors.

Table 5. The results of the calculation of standardized coefficients and weighting coefficients for each quality indicator.

| Sample number | Coefficient ponderability, $k_i$ |
|---------------|----------------------------------|
|               | The standardized coefficient, $r_i$ |
| 1             | 0.9143 1.2000 0.9143 1.1143 0.9571 1.0571 0.333 |
| 2             | 1.8571 2.2381 2.0476 2.6667 2.0000 2.3810 0.267 |
| 3             | 0.4286 0.4286 0.4286 0.5000 0.5714 0.6429 0.200 |
| 4             | 0.0167 0.1667 0.0167 0.2500 0.0333 0.3333 0.133 |
| 5             | 1.0889 1.0778 1.0667 1.0444 1.0444 1.0111 0.067 |

Source: Compiled by the authors.

Table 6. Integral quality indicators of the samples of road bitumen.

| Sample number | Integral quality indicator, K | Place |
|---------------|-------------------------------|-------|
| 4             | 1.5624                        | 1     |
| 6             | 1.4317                        | 2     |
| 2             | 1.3906                        | 3     |
| 5             | 1.2289                        | 4     |
| 3             | 1.2285                        | 5     |
| 1             | 1.1464                        | 6     |

Source: Compiled by the authors.
4. Discussion
The analysis of the obtained results shows that sample No. 4 has the best quality. It is obtained according to a two-stage oxidation scheme, where the temperature of the first stage is 200 °C, and the temperature of the second stage is 260 °C. According to GOST 33133-2014, this sample has the best quality indicators compared to other samples.

5. Conclusion
The multicritical (integral) method of quality assessment can be used in the production of bitumen. It allows identifying the bitumen of the best quality and determining the process parameters depending on the quality of raw materials.

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