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Investigation of the Bitumen Modification Process Regime Parameters Influence on Polymer-Bitumen Bonding Qualitative Indicators

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Abstract. The objects of this study are petroleum road bitumen and polymeric bituminous binder for road surfaces obtained by polymer materials. The subject of the study is monitoring the polymer-bitumen binder quality changes as a result of varying the bitumen modification process. The purpose of the work is to identify the patterns of the modification process and build a mathematical model that provides the ability to calculate and select technological equipment. It is shown that the polymer-bitumen binder production with specified quality parameters can be ensured in apparatuses with agitators in turbulent mode without the colloidal mills use. Bitumen mix and modifying additives limiting indicators which can be used as restrictions in the form of mathematical model inequalities are defined. A mathematical model for the polymer-bitumen binder preparation has been developed and its adequacy has been confirmed.

1. Introduction
Modern requirements to the quality and durability of highways in conditions of constant increase in traffic intensity and growing loads on the vehicles axis necessitate the use of new higher quality building materials. Road bitumen, which in a relatively recent past satisfied the needs of the road industry, today does not meet modern requirements and is increasingly replaced with polymer bituminous binders (PBB) [1 - 3]. For the PBB production, oil bitumen is subjected to the modification process by polymeric materials [4 - 7]. The most widespread among them were thermoplastic styrene-butadiene-styrene [4, 8 - 11], which have a relatively high cost. The bitumen modification process was carried out using expensive colloid mills, the failure of which is often cause temporary and financial losses. In [12], the studies results of bitumen modification by thermoplastic elastomer, low density polyethylene (LDPE) and an adhesive additive with a mathematical model for the dependence of the PBB indexes on the mixture components content were presented. Bitumen modification was carried out on the developed laboratory blade mixers of various volumes. Studies have shown a significant quality dependence of the obtained PBB from the regime variables of the bitumen modification process. To solve the optimal equipment design problem for the PBB production with the proposed composition, it is necessary to develop a mathematical model for the process of combining bitumen with the complex modifier components in the presented type apparatus.
2. Formulation of the problem
The task was to investigate hydrodynamic regimes of combining bitumen with modifiers in a paddle mixer, to determine the influence of regime variables of the process on the qualitative indices of the obtained PBB, to investigate the features of the state of the components distributed in the bitumen, to compile a mathematical model for the process of obtaining the PBB and to verify its adequacy.

3. Theory
To obtain the PBB, it was suggested to use a traditional kind of mixing equipment. This is a vertical, cylindrical mixer, the rotation axis of which coincides with the axis of the apparatus. Due to the fact that the thermoplastic elastomer and adhesion additives dissolve in the bitumen [9] and the polyethylene does not dissolve, the apparatus was calculated from the viewpoint of mixing the mutually insoluble liquids: bitumen and LDPE. It was hypothesized that in order to obtain the specified qualitative parameters of the PBB, it is necessary to provide such a hydrodynamic regime in the apparatus so that during the dispersal of the LDPE in the bitumen, the size of the thermoplastic droplets does not exceed the specified values.

Experimental studies were carried out on geometrically similar laboratory mixers with paddle stirrers [8], capable to create a meridional (axial) circulation flow and characterized by the ratio

$$G_d = \frac{D}{d_m} ;$$  \hspace{1cm} (1)

$D$ is the apparatus diameter; $d_m$ is the stirrer diameter.

The range of hydrodynamic regimes is determined by the Reynolds number $Re$, calculated from the flow velocity, the apparatus dimensions and the medium viscosity:

$$Re = \frac{n \cdot d_m^2 \cdot \rho}{\mu}$$  \hspace{1cm} (2)

where $\rho$ is the mixture density; $n$ is the mixer speed; $\mu$ is the mixture dynamic viscosity.

The density $\rho$ and the dynamic viscosity $\mu$ of the mixture are calculated by the following relationships [13]:

$$\rho = \rho_d \times \phi + \rho_c \times (1 - \phi) ;$$  \hspace{1cm} (3)

$$\mu = \frac{\mu_c}{1 - \phi} \left( 1 + 1.5 \cdot \phi \cdot \frac{\mu_d}{\mu_c + \mu_d} \right) ;$$  \hspace{1cm} (4)

where $\rho_d$ is the disperse phase density; $\rho_c$ is the continuous phase density of; $\phi$ is the dispersed phase concentration; $\mu_c$ is the continuous phase dynamic viscosity; $\mu_d$ is the dispersed phase dynamic viscosity.

In apparatus with internal devices, the circumferential flow characteristics must satisfy the moment equilibrium requirement:

$$M_r = M_d + M_i ,$$  \hspace{1cm} (5)

where $M_r$ is the torque; $M_d = M_w + M_b$ is the moment of body device resistance; $M_w$ and $M_b$ are the moment of resistance on the walls and bottom of the apparatus; $M_i$ is the moment of resistance forces arising on internal devices.

To equalize the circumferential velocity values along the apparatus radius, we used reflective partitions satisfying the condition:

$$\sum_{z_{on}} \zeta_{on} f_{on} > 0.1 \cdot D \cdot H ,$$  \hspace{1cm} (6)
where \( z_{on} \) is the internal partitions number; \( \zeta_{on} \) is the coefficient of the baffle plate hydraulic resistance; \( f_{on} = h_{on} \cdot b_{on} \) is the projection area of the partition on the meridian plane; \( h_{on} \) is the immersion height of reflecting partitions in the medium; \( b_{on} \) is the baffles width; \( H \) is the fill height of the device.

In this case, under turbulent conditions \( (Re > 1000) \), the differences between the circumferential velocity local values in the main part of the apparatus volume do not exceed 10 - 15\% [14] and we can assume that the velocity is constant and equal \( v_{cp} \). Then, to calculate the resistance forces moment arising on the walls of the apparatus body, we can take the expression [15]:

\[
M_{ropa} = \frac{\pi \cdot \zeta}{2.2} \cdot \frac{\lambda}{Re^{0.25}} \cdot \gamma \cdot G_{d}^{2.75} \cdot \rho^{1.75} \cdot \nu_{cp}
\]

(7)

where \( \lambda \) is the coefficient of resistance of the device body; \( \gamma \) is parameter of the filling height of the device.

When \( G_{d} \geq 2 \) the value \( \lambda \) is constant and equal to 0.095 [15], at lower values \( G_{d} \) it increases with decreasing distance between the blades of the stirrer and the wall of the apparatus and can be calculated from the approximation equation [15]:

\[
\lambda = \frac{G_{d}}{(20.35 \cdot G_{d} - 19.1)};
\]

(8)

1.05 \leq G_{d} \leq 2.

The filling height parameter \( \gamma \) for devices with free liquid level is calculated by the formula [15]:

\[
\gamma = 4 \frac{H}{D} + 1.
\]

(9)

The resistance forces moment arising on internal devices can be found by the formula [15]:

\[
M = G_{d} \cdot \zeta_{on} \cdot f_{on} \cdot \left( \frac{\nu_{cp}^{2}}{(0.5 \cdot D)^{3}} \right) \cdot \rho \cdot \nu_{cp}.
\]

(10)

where \( r_{on} = 0.5 \cdot (D - b_{on}) \) is the partitions arrangement radius.

Torque transmitted to the liquid during rotation of the agitator blades [15]:

\[
M = z_{m} \cdot \zeta_{m} \cdot k_{N}
\]

(11)

where \( z_{m} \) is the number of stirrers on the shaft; \( \zeta_{m} \) is the coefficient of mixer resistance; \( k_{N} \) is the binding power coefficient with the characteristics of the fluid circumferential flow in the apparatus.

The relationship between the power expended and the mixing conditions has the form [15]:

\[
N = 3.87 \cdot \zeta_{m} \cdot z_{m} \cdot k_{N} \cdot \rho \cdot n^{3} \cdot d^{3}. \]

(12)

With full geometric similarity of industrial apparatus and laboratory mixers, as well as in accordance with the values of the criterion \( Re \), the calculating the power, method using formula (12), is completely reliable and provides the required accuracy [15].

The average droplet diameter of the disperse phase \( d_{k} \) can be calculated by the formula [15]:

\[
d_{k} \approx 0.13 \cdot \left( \frac{\sigma}{\rho_{l}} \right)^{0.6} \cdot \left( \frac{N}{\rho_{l} \cdot V} \right)^{0.4},
\]

(13)

where \( \sigma \) is the interfacial tension; \( V = 0.25 \pi D^{3} H \) is the stirred medium volume.

The condition for a disperse phase uniform distribution along the height of the apparatus is formulated as a restriction on the Peclet number [14]:

\[
P_{e} = \frac{\omega_{mf} \cdot H}{D_{f}} \leq 0.3,
\]

(14)
where $\omega_{nc}$ is the rate of particles or droplets deposition (emergence); $D_T$ is the macroscale turbulent transport coefficient (turbulent diffusion).

The coefficient of turbulent diffusion $D_T$ with respect to apparatus with baffles is determined by the equation [15]:

$$
D_T = 0.435 \cdot n \cdot d_m \cdot D \cdot \sqrt{\frac{z_m \cdot \zeta_m}{G_d \cdot \gamma}}. \tag{15}
$$

To calculate the rate of deposition (emergence) of small droplets, we use the Hadamard-Rybczynski equation [14]:

$$
\omega_{nc} = \left( \frac{d_c^2 \cdot |\rho_d - \rho_c| \cdot g}{18 \cdot \mu_c} \right) \left( \frac{3 \cdot (\mu_c + \mu_d)}{2 \cdot \mu_d + 3 \cdot \mu_c} \right), \tag{16}
$$

where $g$ is the acceleration of gravity.

4. Experimental results

Investigation the regime variables influence of the oil bitumen BND 90/130 modification process with a complex modifier [12] was carried out in laboratory mixers with a volume of $3.0 \times 10^{-3}$ and $5.0 \times 10^{-3} \text{ m}^3$ [8]. The process temperature was maintained at 160° C, mixing time 60 minutes [12].

Investigation the influence of the hydrodynamic mixing regime on the produced PBB properties was carried out by changing the rotational speed of the mixer working elements. Table 1 presents the results of changes in the penetration, softening temperature, ductility and elasticity of the PBB, depending on the rotational mixers speed.

| Rotatio frequency (min$^{-1}$) | Penetration at 25 °C, (× 10 mm) | Ductility, (mm) | Softening point, (°C) | Elasticity, (%) |
|-------------------------------|---------------------------------|-----------------|------------------------|----------------|
| 200                           | 39                              | 740             | 59                     | 57             |
| 400                           | 55                              | 500             | 62                     | 64             |
| 800                           | 62                              | 705             | 73                     | 83             |
| 1200                          | 64                              | 730             | 72                     | 84             |
| Initial bitumen               | 114                             | 765             | 46                     | -              |

With an increase in the rotation speed of the working bodies, an increase in the penetration index occurs, which stabilizes at a frequency of 800 rpm. This is due to the decrease in the LDPE particles size due to a more intensive dispersion in the bitumen. The softening point reaches its maximum values at a speed of 800 rpm. The bitumen elasticity reaches its maximum value at a frequency of 1200 rpm, but has a slight increase in comparison with the frequency of 800 rpm. The highest ductility values were observed at a rotation speed of 200 rpm. The samples were stretched into a filament, which is typical for the initial road bitumen. However, these results were not stable: often the filament broke at ductility below 200 ... 300 mm, which was caused by the presence of inclusions from the LDPE, which at the given rotation frequency were not dispersed to the required level. Thus, the most preferred was the hydrodynamic regime, at which the rotational speed of the working bodies was 800 rpm. Further increase in the rotational speed of the working bodies was not deemed expedient.

On the basis of the mathematical model equations (1) - (16) presented above, calculations were made of the average LDPE particle size reached at 800 rpm and the conditions for the uniform distribution of the dispersed phase along the apparatus height. The following initial data for calculation was used: bitumen density and dynamic viscosity $\rho_c = 1000 \text{ kg} \cdot \text{m}^3$ [16] and $\mu_c = 9 \cdot 10^{-2}$
Pars [17]; LDPE density and dynamic viscosity, respectively: \( \rho_d = 920 \text{ kg} \cdot \text{m}^{-3} \), \( \mu_d = 7.9 \cdot 10^{-2} \text{ Pas} \) [18].

From (3) and (4), the calculated mixture density \( \rho = 996.8 \text{ kg} \cdot \text{m}^{-3} \) and the dynamic viscosity \( \mu = 9.9 \cdot 10^{-2} \text{ Pas} \) were obtained. Substituting the obtained values into equation (2), taking into account that \( n = 13.3 \text{ s}^{-1} \) and \( d_m = 0.095 \text{ m} \), we get the criterion value \( Re = 1177 \) corresponding to the turbulent regime in the mixer [13 - 15].

If inequality (6) is fulfilled, and taking into account that the baffle hydraulic resistance coefficient \( \zeta_m = 0.88 \) and the apparatus body \( \lambda = 0.125 \), we obtain \( v_{cp} = 0.29 \text{ m s}^{-1} \).

The power consumption for mixing is found from equation (12): \( N = 16 \text{ W} \), and the average LDPE droplet diameter in the PBB from equation (13): \( d_k \approx 9 \cdot 10^{-5} \text{ m} \).

The turbulent diffusion coefficient for apparatus with baffles is determined by equation (15) \( D_r = 0.05 \text{ m}^2 \cdot \text{s}^{-1} \), and the average velocity of droplet precipitation from equation (16) \( \omega_p = 6.7 \cdot 10^{-6} \text{ m s}^{-1} \). Substituting these values into equation (14), we find the Peclet number \( Pe = 1.6 \cdot 10^{-5} < 0.3 \), which satisfies the uniform distribution of the disperse phase along the apparatus height.

In Figure 1 there is LDPE distribution photo of the in bitumen obtained on a stereoscopic microscope MBS-10 with an increase of \( \times 300 \). The average LDPE particle diameter on microphotographs is of the order \( d_k \approx 1.1 \cdot 10^{-4} \text{ m} \), which is in agreement with the calculated values.

![Figure 1. Photo of LDPE distribution in bitumen.](image)

Verification the hypothesis on the determining effect of the LDPE particle size in achieving the specified qualitative PBB parameters was carried out by setting similar studies on a laboratory mixer with a volume of \( 5.0 \cdot 10^{-3} \text{ m}^3 \) with a full geometric similarity to a \( 3.0 \cdot 10^{-3} \text{ m}^3 \) mixer [8]. The conducted studies showed that the required PBB quality indicators are achieved at a rational value of the mixer rotational speed 450 rpm. Table 2 shows a comparison of the PBB quality index obtained in both mixers with a rational agitator blades speed value for each of them.

The LDPE average particle diameter, measured by using a stereoscopic microscope MBS-10, was of the order \( d_k \approx 1.7 \cdot 10^{-4} \text{ m} \). Using the presented mathematical model equations of LDPE dispersion in bitumen, we calculated the average droplet diameter: \( d_k \approx 1.49 \cdot 10^{-4} \text{ m} \). In this case the Reynolds number is equal to \( Re = 1161 \), which corresponds to the turbulent regime in the mixer. Thus, the presented data indicate the validity of using our mathematical model.
Table 2. PBB physical and mechanical indicators obtained in various volume mixtures at rational values of rotation frequency.

| Mixer volume (× 10$^3$ m$^3$) | Rotation frequency (min$^{-1}$) | Penetration at 25 °C, (× 10 mm) | Ductility, (mm) | Softening point, (°C) | Elasticity, (%) |
|-------------------------------|-------------------------------|---------------------------------|-----------------|----------------------|----------------|
| 3.0                           | 800                           | 62                             | 705             | 73                   | 83             |
| 5.0                           | 450                           | 65                             | 745             | 71                   | 80             |
| initial bitumen               | -                             | 114                            | 765             | 46                   | -              |

5. Discussion of the results

As a result of the conducted experimental studies we established that polymer-bitumen binder samples, obtained in different volume mixers with the regime parameters of mixing calculated according to the proposed mathematical model, have comparable quality and size indicators of the LDPE drops. Thus, the obtained results that testify the proposed mathematical model adequacy of the PBB producing process in which we combine petroleum bitumen with a complex modifier: thermoplastic elastomer, low density polyethylene and adhesive additive in devices with paddle stirrers. A confirmation of the hypothesis about the LDPE particle size determining influence in achieving the specified PBB qualitative parameters has been obtained.

6. Conclusions

Petroleum bitumen modification with a complex modifier composition (the thermoplastic elastomer, low density polyethylene and adhesive additive) can be carried out in vertical mixers with paddle stirrers at a temperature of 160 °C. To obtain a polymer-bitumen binder with the required parameters of penetration, softening point, ductility and elasticity, it is necessary to provide a turbulent mixing regime with a Reynolds number $Re > 1000$ and also have in the modifying process the average droplets of low density polyethylene not higher than 1.7 × 10$^{-4}$ m. The Peclet number has to be $Pe < 0.3$. A mathematical model is presented for the process of obtaining a polymer-bitumen binder with a given composition, which allows to do optimal mixers design.

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