



Optimization of a Bitumen Binder Modification Process Under Continuous Operation Conditions

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ABSTRACT

This paper presents an analysis of batch and continuous modification modes of BND 60/90 bitumen binder with petroleum resin. Optimal conditions, namely modification time, stirring rate, and the ratio of components were found. The conditions of continuous operation mode were further optimized.

Keywords: Bitumen, Petroleum resin, Asphalt, Protection coating, Organic solvent, Industrial oil, Continuous mode reactor.

INTRODUCTION

Asphalt concrete is the most practical and widespread paving material. Although it has many benefits, some drawbacks still exist: cracking upon time, pavement scaling, spalling etc. Heavy traffic, overloaded trucks and climatic factors speed up the degradation processes. Therefore, it is important to develop and implement efficient materials and technologies to protect the road pavement from workload and climatic factors in order to prolong their working lifespan.

A common method used for the prevention of asphalt concrete pavement degradation is the application of protective layers to the surface (thin layer coating of the pavement with bitumen emulsion or protective compositions)¹.

Treatment with protective impregnation compositions does not require power-consuming or expensive equipment and large amounts of raw materials.

After the treatment of the surface of asphalt concrete, the impregnation composition forms a

strong thin hydrophobic film on the whole surface of the pavement, providing high protective properties. Consumption of the material per square meter of the pavement is low.

In² we earlier described an optimal ratio of components for a protective impregnation composition based on road bitumen of BND 60/90 grade which is modified by a petroleum resin (PR). Physico-chemical, technological and working parameters of the prepared samples of the compositions were described.

All the samples of the impregnation composition were prepared in batch operation mode: the bitumen binder was modified by the introduction of PR (up to 15 mass %) in a steel reactor with an overhead stirrer. The initial temperature was 160°C, then it was maintained not higher than 180°C for 90 (industrial oil was used as a plasticized when needed). The modified binder was then combined with the solvent by pumping the latter to the reactor.



Fig. 1: Laboratory model of a flow reactor to produce a binder component

To evaluate the transfer of the technology to a pilot scale, it is important to consider the analysis of batch and continuous operation modes of binder modification, as well as to select the optimal conditions: time of modification, stirring rate, and ratio of the components.

The goal of the current research was to optimize the conditions of continuous mode modification of the bitumen binder.

MATERIAL AND METHODS

For comparison, materials from different manufacturers of BND 60/90 grade bitumen were used. The manufacturers were JSC «Slavneft-YANOS» (YANOS), «Gazpromneft – Moscow oil processing plant» LLC (MNPZ) and «JSC Ufa Refinery» (UNPZ).

Viscous road bitumen of the 60/90 grade is the most demanded bitumen for the production of asphalt concrete pavement, because its rheological properties match the requirements for roadwork to the greatest extent. It is used as a binder in the construction and reconstruction of road pavements.

Petroleum resin (PR) «Sibplast» was selected as the modifying agent, while industrial oils I-20 and I-30A were used as plasticizers (Tables 2 and 3).

Petroleum resin as film former described in⁵⁻⁹, and the use of industrial oil as plasticizer in [10].

The modification process under continuous operation mode conditions was performed as follows:

Table 1: Properties of raw bitumen

Bitumen BND 60/90	Penetration 0,1 mm		Softening temperature, °C	Ductility, sm		Fraass breaking point, °C
	25°C	0°C		0°C	25°C	
YANOS	60	17,5	53	4,2	80	-14,5
MNPZ	68,5	20,2	51	5	92	-14
UNPZ	60	16,6	51	4,7	150	-14

The bitumen (1000 g) was heated to 130°C and placed to a steel vessel (2000 mL) equipped with a hot plate (500 °C maximum temperature) with electric heat regulator, an overhead propeller stirrer and an electronic thermometer. The stirrer blade length was 70 mm. The hot plate was turned on and the stirring rate set to 500 rpm. The heat regulator was set to 450°C, when the inner temperature reached 160°C, petroleum resin was quickly added in one portion. When the temperature reached 170°C, the time count was started. When the inner temperature reached 175°C, the hot plate temperature was set to 320 – 330°C. The heating time of the modified bitumen was 30 minutes. The time of modification was shortened by 7.5 min. Samples were taken after 30, 45, 60, and 90 minutes of modification time.

To evaluate the continuous operation mode of modification of the bitumen binder we constructed a laboratory model of a flow reactor for the preparation of the binding component. The summary of the process is in dispensing bitumen and

petroleum resin, heating the mixture up to 180 °C and feeding it into the steady-flow reactor where the modification process occurs. The model has several entrance points for the introduction of industrial oil. Piston pumps were selected for the feeding of PR and bitumen. The technological parameters of the model can be managed either by a computer or from a control panel (Figure 1).

RESULT AND DISCUSSION

To evaluate the transition of the technology to a pilot scale, an analysis of batch and continuous operation modes of modification of the bitumen binder was performed. Laboratory (up to 10 kg) and semi-industrial (up to 200 kg) conditions for the preparation of the product were considered. The parameters analyzed included: loading of the raw material, efficient stirring, heat retention of the reaction mass. The analysis of the modification schemes is given in Table 4.

Table 2: Physical and chemical properties of the PR “Sibplast”

Properties	Index
External	The solid from yellow to brown in pieces
Softening temperature, °C	80-130
Mass fraction of water,%, not more	0,2
Mass fraction of ash content,%, not more	0,4
Acid number, mg KOH / g, not more	1,0
Mass fraction of mechanical impurities,%, not more	0,3

Table 3: Physical and chemical characteristics of industrial oils

Unit	Norm for brend	
	I-20A	I-30A
Kinematic viscosity at 40°C, mm ² /s	29 – 35	41 – 51
Acid number, mg KOH / g, not more	0,03	0,05
Ash content,%, not more	0,005	0,005
Sulfur content of sulfur in petroleum oils,%, not more	1,0	
Content of mechanical impurities	non	
Water content	traces	
Density at 20°C, kg/m ³ , no more	890	
Pour Point°C, not higher	–15	
Flash point in open crucible, °C, not lower	200	210

The most important difference of the continuous mode from the batch mode is the stable power consumption without any peak loads. The finish warming of the mixture is performed by a pipe furnace in less than 1 min prior to feeding into the reactor. Heating from 160 to 180°C in batch mode conditions should require not more than 30 min. For the 10 kg and especially 200 kg scale it would require heating equipment of increased power capacity. Due to the high heat retention of the reaction mass, any particular reactor size requires additional selection of modification conditions.

The continuous operation mode scheme has significant drawbacks in terms of loading of the raw materials (more expensive equipment is needed). In the batch scheme, the loading is performed by manual labor or using equipment with loading rate that can be regulated in a considerably wide range. In the case of the continuous mode, special dosage units are required that provide high accuracy and stability of operation. The cost of such equipment is at least 10 times greater than the cost of equipment for the batch process. However, in this case, automated control systems can be used, which would decrease labor costs and allow to automate the production.

The main difference of the continuous operation mode from the batch one is the material flow management. While in the batch scheme the weight or volume of the loaded components are preliminary measured, in the continuous process type the component ratios are determined by the dosage units.

Several dependencies of the binder properties were found out in the study of bitumen modification with PR:

- From the reaction mass stirring rate;
- From the modification time in combination with the operation mode (batch and continuous);
- From the bitumen to PR ratio.

Study of the reaction mass stirring rate on the binding component properties

The modification of BND 60/90 grade bitumen was performed at 100, 200 500 and 1000 rpm of the propeller stirrer.

- Flow reactor temperature: 180 °C;
- Stirring with a propeller stirrer;
- Production capacity 1100 g/h;

Table 4: Analysis of schemes modification bitumen

Unit	Scheme of periodic modification		Scheme of continuous modification	
	10 kg	200 kg	10 kg	200 kg
Bitumen loading	Manually	Pump without adjusting feed	Pump with adjustable flow	
PR loading	Manually	Manually or dispenser	Dispenser or a pump with adjustable flow	
Industrial oil loading		Manually	Pump with adjustable flow	
Solvent loading	Manually / Pump	Pump without adjusting feed	Pump with adjustable flow	
Efficient mixing		Possibly		
Fast heating	Possible	Possible with the increased power heaters	Possible	
The thermal inertia of the masses to be modified	Low	High	Low	High

– Sampling was carried out from a reactor valve placed on the 0.5 L height which corresponds to 30 min process time.

The main controlled binder parameter: penetration at 25°C.

The data on the dependency of penetration from the stirring rate are given in Table 5.

Analysis of the results shows that the value for penetration does not change for stirring rates above 500 rpm. So we selected this stirring rate for further experiments.

Table 5: The dependence of the penetration of the binder mixing speed

Bitumen BND 60/90	Penetration at 25 °C 0,1 mm and stirrer speed			
	100 rpm	200 rpm	500 rpm	1000 rpm
MNPZ	45	38	37	37
UNPZ	37	32	29	29
YANOS	42	35	31	31

Table 6: The dependence binder penetration at 25°C by modification time in a periodic and continuous modes

Bitumen BND 60/90	Penetration at 25 °C 0,1 mm	Penetration at 25 °C 0,1 mm during the modification, min				
		0	30	45	60	90
Periodic modification						
YANOS	75	63	55	53	52	50
UNPZ	60	48	44	42	40	39
MNPZ	60	48	41	37	34	32
Continuous modification						
YANOS	75	63	31	30	30	29
UNPZ	60	48	29	29	28	28
MNPZ	60	48	37	37	36	36

Table 7: The dependence binder softening temperature at 25 ° C by periodic and continuous modes

Bitumen BND 60/90	Softening temperature, °C	Softening temperature, °C during the modification, min			
		30	45	60	90
Periodic modification					
YANOS	53	53	54	54	54
UNPZ	51	52	52	53	53
MNPZ	51	53	53	53	54
Continuous modification					
YANOS	53	54	54	54	55
UNPZ	51	52	52	53	53
MNPZ	51	52	52	53	53

Study of the dependency of the binder properties from the modification time and comparison of results for batch and continuous operation modes

– Sampling was carried out from reactor valves placed on the 0.5 L and 1 L height which corresponds to 30 min and 60 min process time for the 1000 g/h production capacity, or 45 and 90 min for 670 g/h capacity.

Process conditions

- Flow reactor temperature: 180°C;
- Stirring with a propeller stirrer at 500 rpm;
- Production capacity 1000 g/h and 670 g/h;

The following properties of the binder were compared: penetration at 25 °C, ball-and-ring softening temperature, Fraas brittle point.

Table 8: The dependence Fraass breaking point temperature by modification time in a periodic and continuous modes

Bitumen BND 60/90	Fraass breaking point, °C	Fraass breaking point, °C during the modification, min			
		30	45	60	90
Periodic modification					
YANOS	-14,5	-9	-10	-10	-9
UNPZ	-14	-5,5	-6	-6,5	-6
MNPZ	-14	-10	-10	-10	-10
Continuous modification					
YANOS	-14,5	-1	-1	0	0
UNPZ	-14	-1,5	-1	0	1
MNPZ	-14	-9	-9	-9	-8

Table 9: Penetration at 25°C, 0,1 mm with a ratio of bitumen: PR in a continuous mode

Bitumen BND 60/90	Penetration at 25 °C 0,1 mm	Penetration at 25°C, 0.1 mm with a ratio of bitumen: PR		
		90:10	85:15	80:20
YANOS	75	43	31	25
UNPZ	60	32	28	23
MNPZ	60	42	38	27

Table 10: Fraass breaking point °C with a ratio of bitumen: PR in a continuous mode

Bitumen BND 60/90	Fraass breaking point, °C	Fraass breaking point °C with a ratio of bitumen: PR		
		90:10	85:15	80:20
YANOS	-14,5	-8	-1,5	1
UNPZ	-14	-7	-1	2
MNPZ	-14	-11	-10	-2

Experimental results are given in Tables 6-8.

Analysis of the data shows that for both the batch and the continuous operation modes the maximal decline of penetration values occurs during the first 30 minutes of modification. The most important difference is in the rate at which penetration values continue to decline after this time. While for the batch operation mode it is considerable – 5-10 units in the time range of 30-90 min, for the continuous mode it is only 1-2 units.

The presented data demonstrate that the properties of the product are almost identical for both modification modes.

However, Fraas brittle point values are significantly higher for the continuous mode compared to the batch mode. An exception are the results for MNPZ bitumen that has a difference of 1-2 degrees in this parameter.

Study of the bitumen to PR rate on the properties of the binder product

The bitumen – PR ratio is one of the most important parameters for the development of a continuous operation mode bitumen modification process.

While in the batch mode this ratio is easily controlled, in the continuous operation it is much more difficult to set the proper material flows. Of particular importance is to set acceptable limits for the ratio, as the dosage pumps have their own tolerable deviations. It is necessary for the bitumen to PR ratio to be within the set interval of deviation.

Process conditions

- Flow reactor temperature: 180 °C;
- Stirring with a propeller stirrer;
- Production capacity 1000 g/h;
- Sampling was carried out from a reactor valve placed on the 0.5 L height which corresponds to 30 min process time at 1000 g/L production capacity.

Table 11: Penetration and Fraass breaking point temperature by adding industrial oil to binder obtained continuously modified bitumen

Bitumen BND 60/90	Industrial oil, %	Penetration at 25°C, 0.1 mm with a ratio of bitumen: PR		Fraass breaking point °C with a ratio of bitumen: PR	
		90:10	85:15	90:10	85:15
YANOS	2 % I-20A	50	40	-13	-8
YANOS	3 % I- 20A	54	46	-16	-11
YANOS	5 % I-20A	62	61	-19	-15
YANOS	2 % I-30A	48	39	-9	-4
YANOS	3 % I- 30A	52	43	-13	-7
YANOS	5 % I-30A	57	49	-15	-10
UNPZ	2 % I-20A	41	39	-13	-6
UNPZ	3 % I-20A	47	44	-16	-11
UNPZ	5 % I-20A	59	56	-18	-14
UNPZ	2 % I-30A	37	35	-9	-3
UNPZ	3 % I- 30A	48	45	-13	-7
UNPZ	5 % I-30A	55	52	-15	-10
MNPZ	2 % I-20A	51	49	-16	-15
MNPZ	3 % I- 20A	56	55	-18	-18
MNPZ	5 % I-20A	64	62	-19	-19
MNPZ	2 % I-30A	48	46	-14	-12
MNPZ	3 % I- 30A	53	49	-16	-15
MNPZ	5 % I-30A	61	59	-18	-17

The bitumen to PR rate was: 90 : 10, 85 : 15, 80 : 20.

The controlled parameters were: penetration at 25°C and Fraas brittle point.

The experimental data are given in Tables 9 and 10.

It is clear that the bitumen to PR ratios of 90 : 10 for the continuous process and 85 : 15 for the batch process give very close results.

Preparation of the binder in continuous mode using mineral oil as an additive

Process conditions

- Flow reaction temperature 180°C;
- Stirring with a propeller stirrer;
- Production capacity 1000 g/h;
- Sampling was carried out from a reactor valve placed on the 0.5 L height which corresponds to 30 min process at 1000 g/h capacity.

The bitumen to PR ratio was: 90 : 10 and 85 : 15.

The controlled parameters were penetration at 25 °C and Fraas brittle point.

The oil was fed by a peristaltic pump at the 0.5 L valve level through an entrance point for the oil.

The results are presented in Table 11.

CONCLUSIONS

An analysis of the batch and continuous technological schemes of a bitumen binder modification was performed.

The minimal reaction mass stirring rate to provide the perfect-mixing reactor under continuous operation was found out – 500 rpm.

The maximal decline of penetration values occurs during the first 30 min for both operation modes.

The most important difference of the continuous mode from the batch one is the very slow rate of penetration decline after 30 min process time.

The Fraas brittle point in continuous mode is significantly higher compared to the batch mode.

Modification of bitumen with bitumen to PR ratios 90 : 10 and 85 : 15 (for the continuous and batch modes, correspondingly), give very close results.

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REFERENCES

1. Chigorina E. A., Razinov A. L., Retivov V. M. *Biosci., Biotech. Res. Asia*. **2014**, 11(3), 1679–1683.
2. Merentcova G.S. *Polzunovskiy vestnik*. **2014**, 1, 100–103.
3. Ubas'kina Y. A., Chigorina E.A., Razinov A. L., Ryabenko V. S., Kovtun I. D. *Orient. J. Chem.* **2016**, 32(1), 305–311.
4. Usmanov T.K., Abdullin A.I., Emelyanicheva E.A., Markov C.Yu. *Vestnik Kazanskogo tekhnologicheskogo universiteta*. **2013**, 2 (16), 117–121.
5. Dumskiy Yu.V., Cherednikova G.F., Dumskiy S.Yu. *Izvestia VSTU* **2010**, 7 (2), 127–130.
6. Fiterer E.P. *Russian Coatings Journal*. **2011**, 1–2, 84–89.
7. Kobayashi S., Müllen K. *Encyclopedia of Polymeric Nanomaterials*. Springer (**2015**),

- 1546–1550.
8. Lesnyak V.P., Gaponik L.V., Shiman D. I., Kostuk S.V., Kaputckiy F.N. Sintez, modifikaciya i primenenie neftepolimernikh smol na osnove monomersoderjashikh piroliznikh frakciy Minsk **2008**, 204–245.
9. Dumskiy Yu.V., Cherednikova G.F., Dumskiy S.Yu., Kostorubina E.V., Kuznetcova N.A. *Izvestia VSTU*. **2013**, 1-104 (6), 72–74.
10. Egorushkin A.V., Egorushkin V.O., Endjievskaya I.G., Vasilovskaya N.G. *Modern problems of science and education* **2014**, 6, 265–269.