Evaluation of bitumens consistence modified by polymers at equipenetrating temperature

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ABSTRACT

At present the penetration (P25) and the softening point (TṘB) remain the main quality characteristic of both bitumens and PMBs. The penetration has more informative valeu than TṘB. The reason for this is that P25 is actually a shear strength property of bituminous cements while dipping the indenter (penetration needle) into them under the effect of stable load.

However, the softening point is valuable only for the bitumens with a low penetration index but useless in respect of the bitumens which are modified by more than 2,5 – 3,0 % of the SBS-type polymer. A clear proof of this are the valeus of stability determed during storage, when a system is formed in the upper part of the tube in which the polymer is the medium and the phase is the bitumen. Such a system is characterized by high penetration and immensely high TṘB.

According to designers idea the softening point temperature is informative when the 800×0,1 mm penetration is correspondant. This rule which underlies the ring-and-ball test (ṘB) can not be applied to the the PMB. Moreover, long ago I. Ph. Pfeiffer and P. M. Doormal prefered to use T800 for specifying the penetration index of bitumens, which subsequently transformed into TṘB because of the proximity of the values of the softening point temperature and T800 for the "sol-type" bitumen. As for the PMB the substitution results in dangerous errors while forecasting on the basis of TṘB rut resistance of asphalt-concretes. In accordance with W. Heukelom viewing the softening point temperature in a historic retrospective as the equipenetrating temperature will allow to enhance the reliability of the PMB consistence evaluation at high summer temperatures, to simplify the testing process by means of the traditional and durable penetrometer will be by using a prolonged needle to 120 mm.

Keywords: Mechanical Properties, Modified Binders, Standardisation, Temperature susceptibility, Testing

1. INTRODUCTION

Resistance of asphalt to the development plastic (irreversible) deformations at high temperatures is mainly determined by consistency of bituminous binders. These binders are the only component of asphalt possessing rheological properties. They can flow to a greater or lesser extent depending on the temperature and affecting stress value.
According to the traditional requirements system, bituminous binders' consistency at high temperature is estimated by their softening point. Substantively, the conditions for determining the softening point imply bitumen flowing under constant load with increasing temperature for a given regime. As a matter of fact, the method for determining the softening point which had been used for over 100 years became a forerunner of methods for determining the temperatures of transition (vitrifying and melting point) of amorphous polymers from glassy to fluid state [1].

2. REVIEW

A feature of the softening point ($T_{R\&B}$) of road bitumen definition is the fact that at the beginning of the test it is already in the plastic state and that the temperature determined by this way has nothing to do with the physical transitions in the bitumen. It is identified as the temperature at which the bitumen layer that initially has turned into a hemisphere gains afterwards a cylindrical shape capable of stretching under the ball's mass influence to a fixed length of 25 mm. Unlike polymers which melting point can be calculated on the basis of their chemical composition [1], such calculations are not possible for bitumen due to the fact that they it is a mixture of a wide variety of hydrocarbons. Physical uncertainty of softening point is its first drawback.
The second drawback is uncertainty of stressed state pattern: at the beginning of testing it includes bending compression and then binder shear in the layer of (3,1 mm) thickness between the inner surface of the ring and the surface of the ball. It is followed by bitumen stretching to a level of a bottom plate (25 mm). It does not meet any of the basic patterns used to determine the rheological properties of viscoplastic systems. The third disadvantage is ungrounded selection of stress occurring in the layer of binder under the influence of the ball's mass. Taking into account the fact that bitumen depending on the type of its structure may have different structural strength limit, it can be concluded that the stress in the layer for some bitumen can be lower than this limit and for other bitumen it can be essentially higher [2]. This leads to different flow velocities and hence, different time values during which the ball coated with bitumen can reach the bottom reference plate. As a result, incompatible softening point values may be obtained for different bitumen. Speeds of reaching the bottom plate by bitumen having penetration of 50×0,1 mm, 75×0,1 mm, 105×0,1 mm respectively equal to: 0,14 mm/s; 0,26 mm/s; 0,42 mm/s.
If you change the weight of the ball, softening point (melting of amorphous polymers) should be shifted toward higher temperatures and for the ball with lower weight – vice versa. Thus, the softening point is no more than a special case of the flow corresponding to different parts of the bitumen flow curve that differ by consistency and type of structure. To resolve ambiguity in the interpretation of the determined values and prevent errors in predicting bitumen flow at its softening point, the developers postulated that the softening point can be objective only in case when it corresponds to the penetration of 800×0,1 mm. Taking into account disadvantages of the method of determining the softening point, I. Ph. Pfejffer and P. M. Doormal preferred to use the temperature (T_{800}) at which penetration is 800×0,1 mm for determining penetration index of bitumen instead of it.
W. Heukelom [3] by using the value T_{800} derived generalized temperature- penetration dependence for five different types of bitumen: oxidized and distillation, road bitumen, high and low paraffined. Despite convincing conclusions about the expediency of penetration index evaluation using T_{800}, virtually everywhere, except for France where the LCPC method is used, they still keep on using the traditional softening point for this purpose. The justification for this serves the fact that for the distillation of road bitumen with the structure of "sol" type and the penetration from 40×0,1 mm up to 220×0,1 mm, the temperatures are practically the same.
In case of bitumen such as "sol-gel", and especially "gel" type, the use of $T_{R\&B}$ to evaluate temperature sensitivity of bitumen is connected with significant errors. And it is even truer for polymer modified bitumen, where the content of the polymer is such that it results in the formation of PMB of transition type for which the phase is formed consisting of a mixture of bitumen and polymer, and especially when polymer acts as medium and bitumen acts as phase.
Objective assessment of temperature sensitivity of T_{800} is based on the fact that penetration, according to [4], is a measure of resistance to immersion of an indenter of a variable shape (first conical, then cylindrical) into bituminous binder, i.e. acting as a measure of shear strength of bitumen.
This shear strength with increasing penetration from 30×0,1 mm to 800×0,1 mm, according to the data of [4], is modified as follows:

Penetration, ×0,1 mm	30	50	70	90	110	130	150	170	190	210	230	800
Shear strength, τ×10 MPa	2,54	1,14	0,66	0,46	0,35	0,29	0,24	0,21	0,18	0,16	0,15	0,04

Penetration of 800×0,1 mm corresponds to shear strength close to 0,004 MPa. Thus, T_{800} indicates the temperature at which certain bitumen reaches this stress level. The higher the value T_{800} - the higher bitumen's capability to resist the development of plastic deformations and flow.

3. EXPERIMENTAL RESULTS AND DISCUSS

To determine the temperature corresponding to the penetration value of 800×0,1 mm, a standard modified penetrometer with a needle of 100×0,1 mm length was used. This method consists in determining penetration at temperatures of 15; 25; 35 and 45 °C. At each temperature, a new penetration glass was used previously conditioned at the test temperature for at least 1,5 hours. Typical temperature- penetration dependence is semi-logarithmic (Figure 1). This allows using extrapolation of penetration in case of very high T_{800} to a level of 800×0,1 mm.

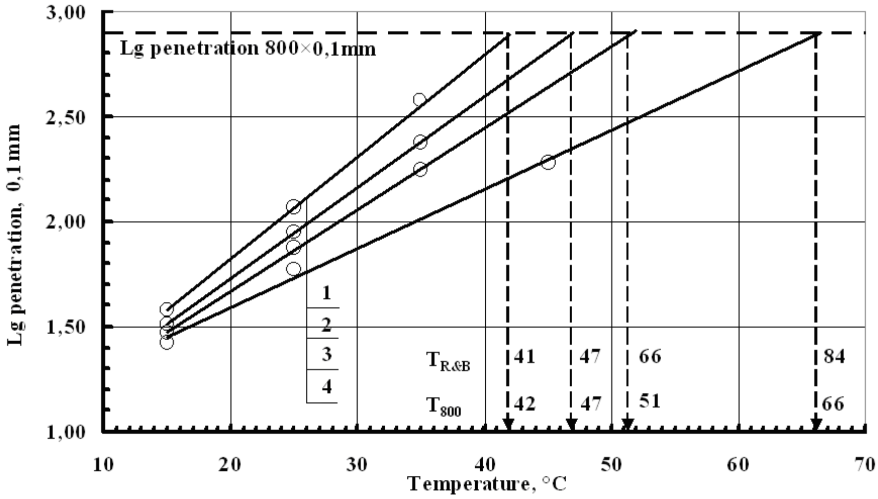

Figure 1: Temperature dependencies of PMB penetration with different content of:
1 – 0; 2 – 1,5 %; 3 – 3,0 %; 4 – 5,0 %

For the research purposes, bitumen of different origin was used: oxidized bitumen (LB) manufactured in Ukraine and distillation bitumen (AN) produced by Nynas (Sweden); bitumen created in the laboratory, PMB based on oxidized and residual bitumen with different content of thermoplastic elastomers polymer SBS - linear (SBSL-1) and radial (SBSR-2). The difference between oxidized and residual bitumen of equal penetration is that the first have a higher softening point (about 5 °C) and lower brittleness temperature (about 5 °C). This is due to difference in their structure and composition: in the first case, oil and asphaltenes prevail; in the second case resins prevail. The first can be attributed to the "sol-gel" type (penetration index ranges from minus 0,29 to + 0,36, respectively, with a penetration from 50×0,1 mm to 172×0,1 mm); the latter is a typical bitumen of "sol" type (penetration index ranges from minus 1,55 to minus 1,70, respectively, for the penetration from 52×0,1 mm to 182×0,1 mm). The data in Table 1 indicate that $T_{R\&B}$ and T_{800} for residual bitumen differ by no more than 1 °C, whereas for oxidized bitumen it reaches 2,5 °C.

Table 1: Temperature-penetration properties

Bitumen	Penetration at T °C, 0,1 mm					$T_{R\&B}$, °C	T_{800}, °C	$T_{R\&B}$-T_{800}, °C	T_{Fr}, °C
	5	15	25	35	45				
LB1	7	19	50	125	327	53,8	54,0	-0,2	-14
LB2	10	22	75	210	537	49,5	48,5	1,0	-16
LB3	14	32	105	313	827	47,0	44,5	2,5	-18
LB4	23	57	172	463	-	42,6	40,0	5,6	-20
AN1	4	15	52	200	580	48,3	47,5	0,8	-9
AN2	7	24	79	297	727	44,7	44,5	0,2	-10
AN3	12	40	118	341	-	40,8	42,0	-1,2	-13
AN4	18	63	182	678	-	37,3	36,5	0,8	-15

When mixed of oxidized bitumen having a penetration of 24×0,1 mm with short residue having $T_{R\&B}$ = 32 °C at the laboratory, bitumen of different consistencies were obtained (Table 2). At high content of short residue (69 %) temperature $T_{R\&B}$ and T_{800} are practically equal. Reduction of short residue content and transition to bitumen of increased consistency (123; 77; 48×0,1 mm) is accompanied by an increase in the difference $T_{R\&B}$-T_{800}, respectively, by 5,8 °C, 7,1 °C, 10,6 °C. In this case, taking $T_{R\&B}$ as a criterion of bitumen flow resistance at high temperatures will result in the distortion of objectivity of such predictions towards oversized estimation.

"Waxing" the bitumen with additives in the amount of 3 % that lower processing temperature leads to an increase in its consistency, rise in softening point by 31 °C – 46 °C and higher penetration index that actually means the transition to bitumen of "gel" structural type. At the same time, the difference $\Delta T = T_{R\&B}-T_{800}$ sharply grows to 53,9 °C and 36,9 °C.

Introduction of polymer in bitumen changes its structure. Particles of thermoelastic polymer in a small amount are swelling in bitumen oils, reduce oil content, penetration and increase softening point; preserve brittleness temperature of the original bitumen and give it elasticity. At that, bitumen is being medium. With high content the polymer becomes medium. This dramatically increases softening point of PMB and lowers brittleness temperature due to formation of elastic polymer grid. In case of moderate polymer content (transition zone), elasticity of PMB is stabilized, softening point increases but brittleness temperature is not much different from that of the original bitumen.

Table 2: Comparison of softening points and equipenetration temperatures of bitumen obtained by dilution of highly viscous oxidized bitumen

Short residue content in bitumen, %	Penetration, 0,1 mm	Temperatures, °C		
		$T_{R\&B}$	T_{800}	$\Delta T = T_{R\&B} - T_{800}$
30	48	57,4	68,0	- 10,6
48	77	49,4	56,5	- 7,1
64	123	43,2	49,0	- 5,8
69	160	42,8	43,5	- 0,7

At the same time, the difference between T_{800} и $T_{R\&B}$ starts growing rapidly already in the transition zone and reaches its maximum in the zone of high polymer content (Table 3). Consistency of bitumen affects the difference between $T_{R\&B}$ and T_{800}. Adding of 3 % of linear polymer SBSL-1 in short residue with penetration of 435×0,1 mm and in the bitumen with penetration of 174×0,1 mm and 89×0,1 mm leads to the difference of $T_{R\&B}$ and T_{800} respectively in 13,4 °C, 2,4 °C, 2,3 °C. Introduction of 6 % of polymer in the same bitumen increases the difference up to 41,4 °C, 14,2 °C, 7,6 °C respectively. In the first case, it is due to the fact that the conditions for distribution, wetting and swelling of polymer particles in the binders of low consistency are much better due to the content of oils. In the second case, the same trend is kept, but it is enhanced by increasing the polymer content to the level at which a polymer matrix is formed, i.e. phase inversion occurs.

Table 3: Effect of polymer type and consistency of bitumen on the temperatures $T_{R\&B}$ and T_{800}

Polymer	Bitumen (standard of Ukraine)	Polymer content	Properties of original bitumen and PMB			
			P_{25}, 0,1 mm	$T_{R\&B}$, °C	T_{800}, °C	$T_{R\&B}-T_{800}$, °C
SBSL-1	Short residue	0	435	34,0	29	5,0
		3	252	47,4	34	13,4
		6	164	89,4	48	41,4
	BND 130/200	0	174	41,3	39	2,3
		3	114	46,4	44	2,4
		6	72	79,2	65	14,2
	BND 60/90	0	89	46,4	47	-0,6
		3	67	54,3	52	2,3
		6	48	76,6	69	7,6
	BND 40/60	0	50	54,9	56	-1,1
		3	41	62,8	63	-0,2
		6	34	84,6	79	5,6
SBSR-2	BND 130/200	0	134	41,3	42	-0,7
		1,5	94	47,0	47	0
		3	76	66,0	51	15,0
		5	59	84,0	66	18,0

At the same time, this difference $T_{R\&B}-T_{800}$ depends on the quality of the polymer itself. Introduction of 3 % and 5 % of radial polymer SBSL-2 into the bitumen with a penetration of 134×0,1 mm leads to exceeding of $T_{R\&B}$ over T_{800} by 15 °C and 18 °C.

The results of testing stability at storage of PMB containing 5 % SBS of Russian production (SBSL-3), as well as of linear (SBSL-1) and radial (SBSR-2) for the compliance with EN 13399, evidence about the lack of objectivity of $T_{R\&B}$ (Table 4). After the test, highly elastic system with higher penetration and a very high softening point is formed in the upper part of the tube and a system with lower penetration, softening point and elasticity is formed in the lower part of the tube. This is contradicts to the well-known principles of bituminous binders, whereby the higher the penetration, the lower the softening point.

Table 4: Effect of a long-term heating at storage on the properties of PMB containing 6 % of polymer SBS in the upper and lower thirds of the tube

Polymer type	Indicators								
	Penetration at 25 °C, 0,1 mm			Softening point ($T_{R\&B}$), °C			Elasticity at 25 °C, %		
	Before the test	Top	Bottom	Before the test	Top	Bottom	Before the test	Top	Bottom
SBSL-1	130	74	45	94	69	58	99	96	57
SBSR-2	103	59	41	113	84	72	98	98	43
SBSL-3	126	60	76	99	76	60	99	97	52

In the paper [5], even more apparent than in [2], a necessity in replacing the softening point, at least in case of PMB, with equipenetration temperature is substantiated.

Table 5: Difference between $T_{R\&B}$ and T_{800} of bitumen modified by linear (SBSL-1) and radial (SBSR-2) [5]

Temperature, °C	SBS content, %							
	SBSL-1 (linear)				SBSR-2 (radial)			
	0	3	5	7	0	3	5	7
$T_{R\&B}$	44,4	58,5	69,2	76,7	44,4	63,7	74,2	80,9
T_{800}	44,1	49,7	52,4	53,2	44,1	47,4	50,9	52,7
$T_{R\&B}$-T_{800}, °C	0,3	8,8	16,8	23,5	0,3	16,3	23,3	28,2

4. CONCLUSION

Softening point which is widely applied as an indicator for the evaluation of stability of bitumen at high temperatures does not meet its intended purpose neither on its physical nature, nor stressed state pattern, nor the level of stresses affecting the tested layer of bitumen. Stress value from the ball's impact in one case can be higher than the limit of structural strength of bitumen and in the other case – just on the contrary. Accordingly, in the first case, $T_{R\&B}$ is lower and in the second case it is higher. At the same time, in hot weather, the stress value of asphalt is always higher than that limit [2].

Historically and substantively, the temperature corresponding to a penetration of 800 is more justified because the stressed state pattern at penetration reflects shear strength of bitumen at the immersion of an indenter in it. At penetration of 800×0,1 mm the stress occurring in bituminous binder is a constant close to 4×10^{-3} MPa. The higher the temperature at which the binder reaches this stress value, the more it is flow resistant, the higher is rut resistance of asphalt in which this binder is used.

The temperature corresponding to 800×0,1 mm is lower than the softening point. The value of this difference depends on the features of structure and composition of bitumen (bitumen of "sol", "sol-gel" or "gel" type), presence and content of additives in bitumen (energy saving and / or polymer).

Feasibility of replacing the indicator of bitumen consistency at high temperatures is conceptually supported by previous experience and stored scientific knowledge. The method itself is very simple; it does not require sophisticated equipment, highly skilled personnel, it is easily applicable in industrial laboratories.

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