

UDC 691.168
IRSTI 67.15.49
RESEARCH ARTICLE

POLYPROPYLENE-MODIFIED BITUMEN: RHEOLOGICAL AND PHYSICAL-MECHANICAL CHARACTERISTICS OF BINDERS AND ASPHALT CONCRETE MIXTURES

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Abstract. *The aim of this study was to evaluate the effects of isotactic polypropylene H-350 and sunflower foots (SF) on the empirical physical properties and short-term aging behavior of BND 100/130 road bitumen. The scientific novelty consists in the systematic experimental assessment of a locally sourced BND 100/130-PP H-350-SF system with simultaneous variation of PP and SF contents and blending technique, including evaluation of apparent phase heterogeneity and RTFOT behavior. Eleven formulations were prepared with 3-5 wt.% PP H-350 and 0-4 wt.% SF. Penetration at 25 °C, ring-and-ball softening point, and ductility were determined, while short-term aging of three formulations was evaluated using RTFOT. Penetration ranged from 31.4 to 76.4 × 0.1 mm, and softening point from 53.0 to 67.2 °C; phase-separated formulations showed paired values. RTFOT mass loss was 0.04-0.10%. For phase-matched samples, penetration retention was approximately 97.9% and 94.6% for the two reported phases of Mix 7 and 104.2% for Mix 9. An asphalt concrete test mixture prepared with a reported PP-modified binder showed water absorption of 2.95 vol.%, cohesion of 0.27 MPa, tensile crack resistance of 3.4 MPa at -10 °C, and compressive strengths of 3.1 MPa at 20 °C and 1.1 MPa at 50 °C. The results support empirical comparison of the formulations but do not establish EN 14023 compliance or Superpave Performance Grade. Further rheological, storage-stability, mixture-design, and statistical studies are required for definitive performance classification.*

Keywords: *polymer-modified bitumen; polypropylene; Superpave; Performance Grade; sunflower foots; asphalt concrete; RTFOT; pavement engineering; Kazakhstan.*

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ӘОЖ 691.168
ҒТАМР 67.15.49
ҒЫЛЫМИ МАҚАЛА

ПОЛИПРОПИЛЕНМЕН МОДИФИКАЦИЯЛАНҒАН БИТУМ: БАЙЛАНЫСТЫРҒЫШТАР МЕН АСФАЛЬТБЕТОН ҚОСПАЛАРЫНЫҢ РЕОЛОГИЯЛЫҚ ЖӘНЕ ФИЗИКА- МЕХАНИКАЛЫҚ СИПАТТАМАЛАРЫ

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Андатпа. Бұл зерттеудің мақсаты изотактикалық полипропилен Н-350 және күнбағыс соапстоктарының (SF) БНД 100/130 жол битумының эмпирикалық физикалық қасиеттері мен қысқа мерзімді қартаюына әсерін бағалау болды. Ғылыми жаңалығы ПП және SF мөлшерін және араластыру технологиясын бір мезгілде өзгерту арқылы жергілікті өндірілген БНД 100/130-ПП Н-350-SF жүйесін жүйелі эксперименттік бағалаудан, сондай-ақ көзбен байқалатын фазалық біртектілікті және RTFOT жағдайындағы мінез-құлқын зерттеуден тұрады. Құрамында 3-5 масс.% ПП Н-350 және 0-4 масс.% SF бар он бір құрам дайындалды. 25 °C температурада пенетрация, «сақина және шар» әдісі бойынша жұмсару температурасы және созылғыштық анықталды, ал үш құрамның қысқа мерзімді қартаюы RTFOT әдісімен бағаланды. Пенетрация 31,4-тен 76,4 × 0,1 мм-ге дейін, ал жұмсару температурасы 53,0-тен 67,2 °C-қа дейін өзгерді; фазаларға бөлінген құрамдар үшін жұп мәндер көрсетілді. RTFOT нәтижесінде масса жоғалту 0,04-0,10% құрады. Фазалары біркәнді сәйкестендірілген үлгілер үшін пенетрацияның сақталуы Мх 7 қоспасының екі көрсетілген фазасы үшін шамамен 97,9% және 94,6%, ал Мх 9 үшін 104,2% болды. Мәлімделген ПП-модификацияланған байланыстырғыш негізінде дайындалған асфальтбетон қоспасының су сіңіруі 2,95 көлем.%, ілінісуі 0,27 МПа, -10 °C температурадағы созылу кезіндегі жарықшаққа төзімділігі 3,4 МПа, ал 20 °C және 50 °C температуралардағы сығылу кезіндегі беріктігі тиісінше 3,1 МПа және 1,1 МПа болды. Алынған нәтижелер құрамдарды эмпирикалық тұрғыдан салыстыруға мүмкіндік береді, алайда EN 14023 талаптарына сәйкестікті немесе Superpave PG пайдалану сипаттамалары сыныбын растамайды. Пайдалану сипаттамаларын түпкілікті жіктеу үшін қосымша реологиялық зерттеулер, сақтау кезіндегі тұрақтылықты бағалау, қоспаның құрамын толық жобалау және статистикалық деректерді өңдеу қажет.

Түйінді сөздер: полимермен модификацияланған битум; полипропилен; Superpave; пайдалану класы (Performance Grade); күнбағыс фазасы; асфальтбетон; RTFOT; жол төсемдерін жобалау; Қазақстан.

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УДК 691.168
МРНТИ 67.15.49
НАУЧНАЯ СТАТЬЯ

БИТУМ, МОДИФИЦИРОВАННЫЙ ПОЛИПРОПИЛЕНОМ: РЕОЛОГИЧЕСКИЕ И ФИЗИКО-МЕХАНИЧЕСКИЕ ХАРАКТЕРИСТИКИ ВЯЖУЩИХ И АСФАЛЬТОБЕТОННЫХ СМЕСЕЙ

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Аннотация. Целью данного исследования являлась оценка влияния изотактического полипропилена Н-350 и подсолнечных соапстоков (SF) на эмпирические физические свойства и кратковременное старение дорожного битума БНД 100/130. Научная новизна заключается в систематической экспериментальной оценке системы БНД 100/130-ПП Н-350-SF местного производства с одновременным варьированием содержания ПП и SF и технологии смешивания, включая оценку визуально наблюдаемой фазовой неоднородности и поведения при испытании RTFOT. Были приготовлены одиннадцать составов с содержанием ПП Н-350 3-5 мас.% и SF 0-4 мас.%. Определяли пенетрацию при 25 °С, температуру размягчения по методу «кольцо и шар» и растяжимость, а кратковременное старение трех составов оценивали с использованием RTFOT. Пенетрация находилась в диапазоне от 31,4 до 76,4 × 0,1 мм, а температура размягчения - от 53,0 до 67,2 °С; для составов с разделением фаз приведены парные значения. Потеря массы после RTFOT составила 0,04–0,10%. Для образцов с однозначно сопоставленными фазами сохранение пенетрации составило приблизительно 97,9% и 94,6% для двух представленных фаз смеси Mix 7 и 104,2% для Mix 9. Испытанная асфальтобетонная смесь, приготовленная с заявленным ПП-модифицированным вяжущим, характеризовалась водопоглощением 2,95 об.%, сцеплением 0,27 МПа, трещиностойкостью при растяжении 3,4 МПа при -10 °С и пределом прочности при сжатии 3,1 МПа при 20 °С и 1,1 МПа при 50 °С. Полученные результаты позволяют проводить эмпирическое сравнение составов, однако не подтверждают соответствие EN 14023 или классу эксплуатационных характеристик Supergrade PG. Для окончательной классификации эксплуатационных характеристик необходимы дополнительные реологические исследования, оценка стабильности при хранении, полный подбор состава смеси и статистическая обработка данных.

Ключевые слова: полимерно-модифицированный битум; полипропилен; Supergrade; марка эксплуатационных характеристик (Performance Grade); подсолнечный фуз; асфальтобетон; RTFOT; дорожное строительство; Казахстан.

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1 INTRODUCTION

In recent years, the highway construction sector in Kazakhstan has increasingly incorporated test methods and specifications influenced by international pavement-engineering practice. Superpave is one such framework; however, the present study does not determine Performance Grade (PG), because dynamic shear rheometer (DSR) and bending beam rheometer (BBR) measurements after the required aging procedures were not performed. Therefore, the present work is limited to empirical physical properties of modified binders and selected physical-mechanical properties of an asphalt concrete mixture.

Polymer-modified bitumens (PMB) can improve the temperature sensitivity and deformation resistance of conventional binders through incorporation of polymeric modifiers. Styrene-butadiene-styrene (SBS) is widely used, while thermoplastic polyolefins such as isotactic polypropylene (PP) are of interest as alternative modifiers. In this study, PP H-350 supplied by the Atyrau Petrochemical Complex (APCP) was investigated as a modifier of BND 100/130 bitumen.

Polypropylene is incorporated into bitumen primarily through physical dispersion. Semi-crystalline PP domains can increase binder stiffness and reduce penetration, while the effect on low-temperature and viscoelastic performance cannot be established from penetration, softening point, and ductility alone. The homogeneity of a polymer-modified binder is also dependent on the blending procedure and cooling history. Accordingly, the present study interprets these effects only in terms of the measured empirical properties and observed phase heterogeneity.

The study focuses on the BND 100/130 (PPCP) - PP H-350 (APCP) system and examines sunflower foots (SF) as an additional component. The experimental data include formulations with SF contents calculated from the reported mass balances; these range from 0 to 4 wt.%. Because the available experimental record does not contain complete storage-stability, microscopic, or rheological characterization, statements concerning compatibility, solvation, and re-agglomeration are treated as interpretations rather than demonstrated mechanisms.

Kazakhstan experiences extreme continental climate conditions, with annual temperature amplitudes exceeding 80 °C in some regions, ranging from approximately -40 °C in winter to +45 °C in summer [1]. Such conditions impose severe demands on road pavement binders: conventional straight-run bitumens are susceptible to low-temperature cracking during freeze-thaw cycles and to permanent deformation (rutting) at high summer pavement temperatures. Polymer modification is widely recognized as one of the most effective strategies for broadening the functional temperature interval of road binders and improving resistance to both rutting and fatigue cracking [2].

Among thermoplastic polymers evaluated as bitumen modifiers, isotactic polypropylene (PP) is of practical interest due to its semi-crystalline structure, relatively high melting temperature (approximately 160-165 °C), and commercial availability from domestic sources [9]. Unlike elastomeric modifiers such as styrene-butadiene-styrene (SBS), isotactic PP acts primarily as a stiffening agent by introducing rigid crystalline domains into the bituminous matrix. When dispersed above its melting temperature and subsequently cooled, PP may form partially crystallized inclusions that restrict bitumen flow at elevated temperatures, potentially raising the softening point and reducing penetration [2, 10]. The melt flow index of PP H-350 (~35 g/10 min at 230 °C/2.16 kg) indicates moderate molecular weight, which may facilitate dispersion under high-shear or mechanical-stirring conditions at 165-175 °C [3]. Field experience in several countries has shown that PP can improve rutting resistance of asphalt mixtures at elevated service temperatures, although the low-temperature performance is a recognized concern for cold climates [4].

The principal challenge in PP-bitumen systems is the thermodynamic incompatibility between non-polar polyolefin chains and the complex polar and aromatic matrix of bitumen [2]. Without appropriate compatibilization, PP may not disperse homogeneously and can segregate upon cooling, producing multi-phase binders with position-dependent empirical properties. Sunflower foots (SF), a lipid-rich by-product of vegetable oil refining, contains phospholipids and free fatty acids whose amphiphilic character - polar head groups combined with non-polar aliphatic tails - may facilitate wetting of PP particle surfaces by the bitumen continuous phase and promote a more homogeneous

macroscopic distribution [3]. This potential interfacial activity of SF is consistent with the empirical observation in the present study that selected SF-containing formulations yielded single-valued (non-phase-separated) penetration and softening-point results, while some formulations with minimal SF exhibited pronounced within-sample heterogeneity. However, the present experimental record does not include direct microstructural evidence (e.g., optical microscopy, fluorescence imaging) or thermodynamic measurements to confirm a specific compatibilization mechanism; the role of SF is therefore discussed as a hypothesis consistent with the measured empirical data.

The present study addresses a combination of locally available materials - BND 100/130 bitumen from Pavlodar Petrochemical Plant (PPCP) and PP H-350 from the Atyrau Petrochemical Complex (APCP) - that has not been extensively characterized in the published literature for Central Asian climatic conditions [3, 9]. By systematically varying PP content (3-5 wt.%), SF content (0-4 wt.%), and blending method (mechanical stirring vs. high-shear dispersing), the study generates a dataset of empirical binder properties and short-term aging indicators that can inform formulation screening for pavements exposed to Kazakhstan's extreme climate [1].

The scientific novelty of this study lies in the systematic experimental evaluation of a locally sourced BND 100/130-PP H-350-sunflower foots (SF) system, with simultaneous consideration of PP and SF contents and blending technique. Unlike studies focusing primarily on polymer-modified binders using conventional polymer modifiers, the present work examines isotactic PP H-350 supplied by the Atyrau Petrochemical Complex in combination with SF as a secondary component and identifies the changes in empirical binder properties and visible phase heterogeneity across eleven formulations. The study additionally provides short-term RTFOT aging data for selected formulations, including phase-specific penetration retention where phase matching is possible. The new experimental result is the identification of formulation-dependent changes in penetration, softening point, ductility, and apparent phase uniformity, particularly the single-valued empirical response observed for Mix 3 containing 3 wt.% PP and 0.9 wt.% SF. These findings provide an experimentally grounded basis for further development of locally sourced PP-modified binders, while the study does not claim definitive compatibility, Superpave PG classification, or normative compliance.

The main objective of this study was to experimentally evaluate the effect of PP H-350 and sunflower foots (SF) on the empirical physical properties and short-term aging behavior of BND 100/130 bitumen and to determine how the composition and blending technique influence the apparent phase uniformity of the resulting binders. To address these questions, eleven experimental PMB formulations were prepared by varying PP H-350 concentration (3-5 wt.%), SF content (0-4 wt.%, calculated from the reported mass fractions), and blending method. The objectives were: (1) to evaluate the effect of PP H-350 on penetration, softening point, and ductility of BND 100/130 bitumen; (2) to examine the effect of SF content on the measured binder properties and visible phase heterogeneity; (3) to compare the reported results for mechanical stirring and high-shear dispersing; and (4) to evaluate short-term aging of selected formulations by RTFOT. The asphalt concrete results are reported as measured values; normative compliance is not claimed where the source data do not provide sufficient mixture-design and standard-test details.

2 MATERIALS AND METHODS

2.1. Raw Materials

Base Bitumen: Straight-run petroleum road bitumen of grade BND 100/130 produced by Pavlodar Petrochemical Plant (PPCP, Kazakhstan), reported as conforming to GOST 33133-2014. The available manuscript data do not include the complete passport certificate or the triplicate test results for the unmodified reference binder (Mix 0); these data are therefore not reconstructed or inferred.

Table 1 - Standard requirements for BND 100/130 road bitumen per GOST 33133-2014 [14]

Indicator	Unit	Requirement for BND 100/130	Test Method
Needle penetration at 25 °C	×0.1 mm	100–130	GOST 33133-2014 / EN 1426
Softening point (R&B), min.	°C	43	GOST 33142-2014 / EN 1427
Ductility at 25 °C, min.	cm	65	GOST 33136-2014 / EN 13589
Brittleness temperature (Fraass), max.	°C	–18	EN 12593
Flash point (open cup), min.	°C	230	GOST 4333
Kinematic viscosity at 135 °C, min.	mm ² /s	150	ASTM D2170 / EN 12595
Mass loss after RTFOT, max.	%	0.8	EN 12607-1

Polymer Modifier: Isotactic polypropylene homopolymer grade H-350 (PP H-350) supplied by APCP (Kazakhstan). The polymer is characterized by a melt flow index (MFI) of ~35 g/10 min (at 230 °C / 2.16 kg, ISO 1133), density of 0.905 g/cm³, and a melting temperature of 160-165 °C. These supplier-reported characteristics are retained from the original manuscript.

Sunflower Foots (SF): a lipid-rich processing residue derived from industrial sunflower oil processing, reported to have a kinematic viscosity of 40-60 mm²/s at 50 °C and to contain predominantly fatty acid triglycerides and phospholipids. In the present article, the role of SF is discussed in terms of the measured empirical properties; direct evidence of molecular solvation or chemical compatibilization was not obtained.

2.2. Preparation of PMB Formulations

A total of eleven modified formulations (Mix 1 through Mix 11) and an unmodified reference sample (Mix 0 - neat BND 100/130) were prepared according to Table 1. The PP content was 3-5 wt.%, while SF content calculated from the reported bitumen and PP fractions was 0-4 wt.%. Mechanical stirring was performed in a temperature-controlled vessel equipped with a paddle stirrer at approximately 300 rpm and 165-175 °C for 2 h. Polypropylene pellets were introduced incrementally. Mix 2 and Mix 7 are identified in the RTFOT dataset as disperser-prepared formulations; however, the available experimental record does not specify the disperser model, rotor/stator geometry, power, shear rate, loading sequence, or accelerated-cooling conditions. These parameters should be added from laboratory records before the method is considered fully reproducible.

Table 2 - Experimental formulation matrix of polymer-modified bitumen binders

Mix ID	BND 100/130 Bitumen	PP H-350	SF	Mixing Temp.	Mixing Time	Blending Technique
Mix 0 (Control)	100%	0%	0%	-	-	Unmodified Base Bitumen
Mix 1	96%	4%	0%	165–175 °C	2 h	Mechanical Stirring
Mix 2	95.70%	4%	0.30%	165–175 °C	2 h	High-Shear Dispersing
Mix 3	96.10%	3%	0.90%	165–175 °C	2 h	Mechanical Stirring
Mix 4	95.10%	4%	0.90%	165–175 °C	2 h	Mechanical Stirring
Mix 5	94.5%	4%	1.50%	165–175 °C	2 h	Mechanical Stirring

Mix 6	93.9%	4%	2.10%	165–175 °C	2 h	Mechanical Stirring
Mix 7	93.5%	4%	2.50%	165–175 °C	2 h	High-Shear Dispersing
Mix 8	93%	4%	3.00%	-	-	Mechanical Stirring
Mix 9	92%	4%	4.00%	-	-	Mechanical Stirring
Mix 10	92%	5%	3.00%	-	-	Mechanical Stirring
Mix 11	91%	5%	4.00%	-	-	Mechanical Stirring

2.3. Laboratory Testing Methods

All reported standardized characterization tests were performed in triplicate, and the manuscript presents arithmetic mean values. The original dataset does not provide individual replicate values; therefore, standard deviations and confidence intervals cannot be calculated retrospectively from the information available here.

- Needle Penetration at 25 °C was measured according to GOST 33133-2014 / EN 1426 (expressed in units of 0.1 mm). For samples exhibiting macroscopic phase separation, measurements from the different observed layers are reported separately. The phase identity of the layers was not independently characterized.

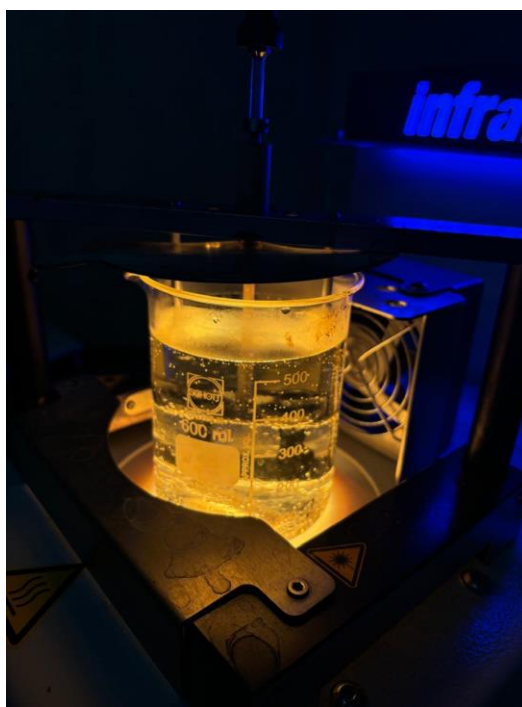


Figure 1- Penetration test of the bitumen sample at 25 °C using a laboratory penetrometer in accordance with GOST 33133-2014 / EN 1426.

- Softening Point (Ring-and-Ball method) was conducted according to GOST 33142-2014 / EN 1427.



Figure 2- Determination of the softening point of bitumen using the Ring-and-Ball method in accordance with GOST 33142-2014 / EN 1427.

- Ductility at 25 °C was evaluated according to GOST 33136-2014 / EN 13589 at an elongation rate of 5 cm/min.

- Short-Term Thermo-Oxidative Aging (RTFOT) was carried out according to ST RK EN 12607-1-2023 / EN 12607-1 at 163 °C for 85 min. Residual binder was analyzed for mass loss, penetration, and softening point. Because phase matching between pre- and post-RTFOT measurements is not documented for all phase-separated samples, retention ratios are calculated only where the corresponding phases can be identified unambiguously.

- Physical-Mechanical Asphalt Concrete Properties were determined on compacted cylindrical specimens according to ST RK 2373. The reported indicators include bulk density, water absorption, binder drainage, shear resistance (cohesion C and internal friction parameter $\tan \phi$), tensile crack resistance at -10 °C, and compressive strength at 20 °C and 50 °C). The source manuscript does not provide the aggregate source and properties, gradation curve, binder content, mixture-design procedure, mixing/compaction temperatures, specimen dimensions and number, or conditioning regime; consequently, the measured values are retained, but full normative compliance is not asserted. Data limitations. The available manuscript dataset does not contain the complete Mix 0 passport/test record, individual replicate values, full asphalt-mixture design details, exact ST RK 2373 clauses applicable to the tested mixture, or complete operating parameters for the high-shear disperser. These omissions are explicitly reflected in the revised interpretation, and unsupported compliance, PG, compatibility, and comparative-SBS claims have been removed.

3 RESULTS AND DISCUSSION

3.1. Influence of PP H-350 Concentration on Binder Physical Properties

The experimental penetration, softening point, and ductility data for the formulated binders are summarized in Table 3. The Mix 0 column is retained as the unmodified reference designation, but quantitative Mix 0 results were not reported in the available dataset and are therefore not used for numerical baseline comparisons.

Table 3 - Physical and empirical rheological properties of unaged PMB formulations.

Property	M0	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10	M11
Penetration at 25 °C, $\times 0.1$ mm	Not reported	46.8	69.2/35.1	57.3	53.7/42.2	31.4	76.4/45.8	42.4/47.5	46.0	43.0	38.1	66.5
Softening Point (R&B), °C	Not reported	61.2	54/66	56.6	59.8/66.6	62.2	53/63.8	63.4/61.2	61.4	66.2	67.2	56.0
Ductility at 25 °C, cm	Not reported	10.05	17.9	14.1	11.0	7.7	18.6	7.9	8.9	6.6	-	-

The addition of PP H-350 produced substantial changes in the measured empirical properties. For example, Mix 1 (4 wt.% PP) had a penetration of 46.8×0.1 mm and a softening point of 61.2 °C, while Mix 10 (5 wt.% PP) had a penetration of 38.1×0.1 mm and a softening point of 67.2 °C. These results indicate increased stiffness in the measured empirical tests. Because the unmodified Mix 0 results and complete replicate statistics are unavailable, the magnitude of improvement relative to the base binder cannot be quantified reliably from the present dataset. No PG or rheological grade is assigned.

Ductility values varied across the formulations. Mix 1 showed 10.05 cm, whereas Mix 9 showed 6.6 cm. The observed differences demonstrate that SF and PP content can affect ductility, but the present dataset does not support a unique quantitative relationship because the compositions and blending methods vary simultaneously.

From a physicochemical standpoint, the stiffening effect of PP H-350 may be attributed to the introduction of semi-crystalline polymer domains into the continuous bitumen phase. Isotactic PP crystallizes upon cooling from the melt into a monoclinic alpha-crystal form, and the resulting crystalline inclusions act as relatively rigid filler particles that can restrict the flow of the surrounding viscoelastic bitumen matrix [2, 10]. At 4 wt.% PP (Mix 1), the penetration of 46.8×0.1 mm and the softening point of 61.2 °C suggest enhanced resistance to deformation compared with the nominal BND 100/130 grade range (100-130 $\times 0.1$ mm penetration, ≥ 43 °C softening point per GOST 33133-2014). Increasing PP content to 5 wt.% (Mix 10) further reduced penetration to 38.1×0.1 mm and raised the softening point to 67.2 °C, consistent with a higher fraction of crystalline domains contributing to binder stiffness. These trends are broadly consistent with observations reported for PP-modified bitumens in the literature [2, 10]. Figure 3 illustrates the penetration values for single-phase (non-separated) formulations, and Figure 4 shows the corresponding softening point values.

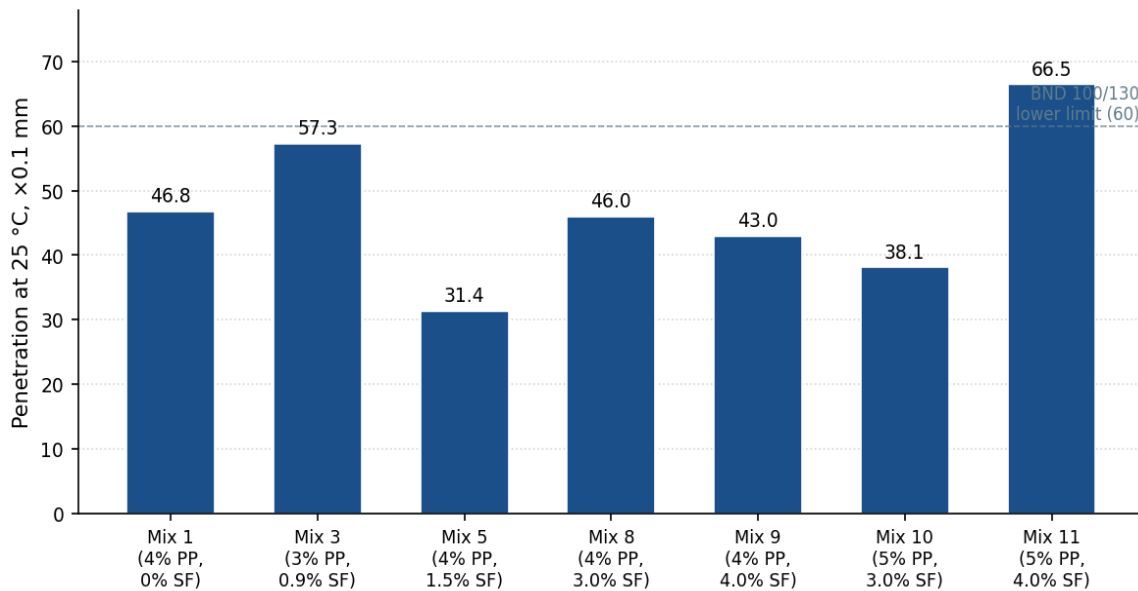


Figure 3 - Penetration at 25 °C (×0.1 mm) for single-phase PMB formulations (Mix 1, 3, 5, 8, 9, 10, 11). Phase-separated mixes (2, 4, 6, 7) are excluded.

The non-polar character of isotactic PP chains is thermodynamically unfavorable for dissolution in the polar aromatic matrix of bitumen [2]. Consequently, PP disperses as discrete particles or domains rather than forming a true molecular blend. The extent of dispersion- and therefore the uniformity of the stiffening effect - depends on blending temperature, shear intensity, and polymer particle size [13]. At 165-175 °C (above the melting point of PP H-350), the polymer is in the molten state and can be mechanically dispersed into the bitumen. However, upon cooling, recrystallization of PP can lead to domain coarsening and, in extreme cases, to macroscopic phase separation evidenced by the paired penetration/softening-point values observed for Mix 2, Mix 4, Mix 6, and Mix 7 in Table 2. This behavior is consistent with the thermodynamic instability of polyolefin-bitumen systems when no compatibilizer or sufficient shear is applied [11].

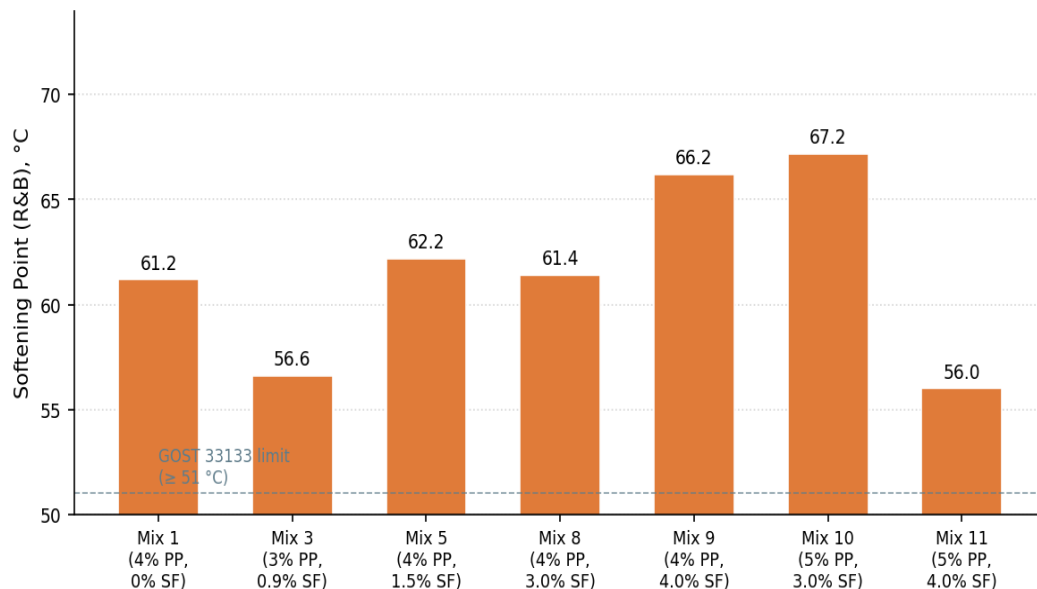


Figure 4 - Softening point (°C) for single-phase PMB formulations (Mix 1, 3, 5, 8, 9, 10, 11). Dashed line indicates reference BND 100/130 minimum requirement (43 °C per GOST 33133-2014 [14]).

The ductility results (Table 3) indicate that PP addition generally reduces binder elongation at break, which can be explained by the replacement of the viscoelastic bitumen network with a stiffer, less deformable PP-reinforced matrix. Mix 1 (4% PP, no SF) showed a ductility of 10.05 cm, while

higher SF-content formulations that remained single-phase (Mix 8: 8.9 cm; Mix 9: 6.6 cm) showed further reduction. The relatively low ductility of PP-modified binders compared to SBS-modified or unmodified base bitumen has been documented in the literature and is attributed to the absence of an elastic polymer network in the crystalline PP domains [2, 13]. Table 7 summarizes the proposed physicochemical mechanisms linking PP and SF additions to the observed empirical properties.

Table 4 - Proposed physicochemical mechanisms linking PP and SF modifications to observed empirical binder properties

Observation	Proposed Mechanism	Key Reference(s)
Reduced penetration with increasing PP content	Semi-crystalline PP domains act as rigid inclusions restricting bitumen flow	[2, 10]
Increased softening point with increasing PP content	Crystalline PP network restricts ring-and-ball deformation at elevated temperature	[2, 10]
Reduced ductility at higher PP content	Rigid crystalline inclusions replace viscoelastic bitumen; no elastic network	[2, 11]
Phase separation at low SF content	Thermodynamic incompatibility of non-polar PP and polar bitumen matrix; insufficient compatibilizer or shear to maintain dispersion on cooling	[2, 13]
Single-phase results at intermediate SF (0.9–3.0 wt.%)	Amphiphilic lipids/phospholipids in SF may reduce interfacial tension and stabilize PP dispersion	[3, 13]
Low RTFOT mass loss (0.04–0.10%)	PP does not contribute significantly to volatile light fractions; oxidation occurs mainly in bitumen maltene phase	[12]
Penetration retention $\geq 97\%$ after RTFOT	PP domains partially shield the bitumen matrix from severe oxidative hardening; overall stiffness increase upon aging is moderate	[12]

3.2. Role of Sunflower Fats as a Compatibility Agent

Mix 2 contained a calculated 0.3 wt.% SF and showed substantial differences between the reported layers: penetration $69.2/35.1 \times 0.1$ mm and softening point 54/66 °C. These differences are consistent with macroscopic phase heterogeneity. Mix 3 contained a calculated 0.9 wt.% SF and showed a single reported penetration value of 57.3×0.1 mm and a single softening point of 56.6 °C. These observations suggest improved apparent uniformity in Mix 3 relative to Mix 2, but they do not by themselves prove thermodynamic compatibility or storage stability.

The available results therefore identify Mix 3 (3 wt.% PP + 0.9 wt.% SF) as the lowest-SF formulation in the reported dataset with single-valued penetration and softening-point results. However, this is an empirical observation, not a validated optimum. A definitive optimum requires a pre-specified optimization criterion and comparison across all formulations using replicate statistics and, preferably, storage-stability and rheological measurements.

The potential role of sunflower fats as an interfacial agent may be linked to the chemical composition of this by-product. Sunflower fats from industrial oil extraction contains predominantly fatty acid triglycerides (principally linoleic and oleic acid chains), phospholipids (principally phosphatidylcholine and phosphatidylethanolamine), and minor amounts of sterols and waxes [3]. The phospholipid molecules, with their hydrophilic phosphate head groups and hydrophobic fatty acid chains, are amphiphilic and may adsorb at the interface between non-polar PP particles and the surrounding bitumen, effectively lowering interfacial tension and promoting a finer, more stable dispersion [3]. The fatty acid components may additionally contribute to plasticization of the bitumen matrix, which can be reflected in the somewhat higher ductility values observed at intermediate SF contents compared to the PP-only Mix 1 (Table 2). The kinematic viscosity of SF (40–60 mm²/s at 50 °C) is substantially lower than that of bitumen at mixing temperature, suggesting it also acts as a processing aid that reduces the apparent viscosity of the system during blending [3].

The influence of SF on ductility is illustrated in Figure 5. For single-phase formulations, ductility was highest at low SF levels (Mix 3: 14.1 cm) and decreased at higher SF contents (Mix 8: 8.9 cm; Mix

9: 6.6 cm). This non-monotonic trend may indicate that, while moderate SF addition improves binder homogeneity and thereby restores some ductility relative to the more heterogeneous low-SF formulations, excessive SF at higher concentrations may interact with the bitumen fraction components (particularly the asphaltenes) in a way that reduces the cohesion of the binder film. However, this interpretation is speculative in the absence of rheological or microstructural data, and the simultaneous variation of PP and SF concentrations across mixes prevents attributing the ductility changes to SF alone. A controlled systematic study varying SF content at constant PP content would be required to isolate this effect.

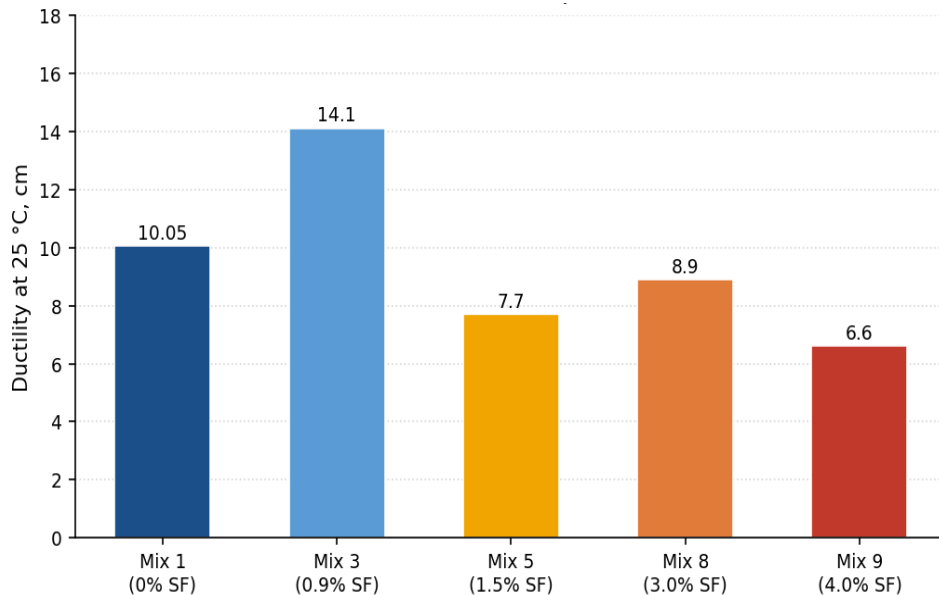


Figure 5 - Ductility at 25 °C (cm) for single-phase PMB formulations (Mix 1, 3, 5, 8, 9). Phase-separated mixes are excluded. Mix 10 and Mix 11 had no reported ductility values.

From a practical standpoint, the observation that Mix 3 (3 wt.% PP, 0.9 wt.% SF) and Mix 8 (4 wt.% PP, 3.0 wt.% SF) both yielded single-valued, non-phase-separated results with softening points of 56.6 °C and 61.4 °C respectively suggests that the SF addition can contribute to formulation stability under the mechanical-stirring conditions used. For Kazakhstan's high summer pavement temperatures, a softening point above 60 °C would be desirable for rutting resistance [1]. Mix 8 and Mix 9 (SP: 61.4 and 66.2 °C respectively) represent formulations that met this criterion among the single-phase samples. However, a complete assessment would require the unmodified BND 100/130 reference test results, replicate statistics, and ultimately DSR testing to verify rutting resistance at the target high-temperature grade [4].

3.3. Physical-Mechanical Properties of Asphalt Concrete Mixtures

A dense-graded Type A asphalt concrete mixture was reported as being prepared using a PMB containing 4 wt.% PP and SF in the range stated in the original manuscript. Because the source data do not identify one exact Mix ID or provide the complete mixture-design record, the asphalt concrete should be described as a test mixture based on the reported modified binder rather than as a uniquely optimized formulation.

Table 5 - Reported physical-mechanical properties of the asphalt concrete test mixture (normative compliance not asserted).

Parameter	Measured value	Normative status
Bulk Density, g/cm ³	2.519	Not verified from available documentation

Water Absorption, vol. %	2.95	Not verified from available documentation
Binder Draindown, %	0.12	Not verified from available documentation
Internal Friction Parameter ($\tan \varphi$)	0.27	Not verified; exact clause/category required
Internal Friction Angle ($\tan \varphi$)	0.94	Not verified from available documentation
Tensile Crack Resistance (R at -10°C), MPa	3.4	Not verified from available documentation
Compressive Strength at 20°C , MPa	3.1	Not verified from available documentation
Compressive Strength at 50°C , MPa	1.1	Not verified from available documentation

The measured asphalt concrete properties are presented in Table 3 without a claim of full ST RK 2373 compliance. The reported values were: bulk density 2.519 g/cm^3 , water absorption 2.95 vol.%, binder draindown 0.12%, cohesion $C = 0.27\text{ MPa}$, $\tan \varphi = 0.94$, tensile crack resistance 3.4 MPa at -10°C , compressive strength 3.1 MPa at 20°C , and 1.1 MPa at 50°C . Because the manuscript does not provide the exact standard clauses, mixture category, control mixture, aggregate characteristics, or complete specimen/conditioning details, these values should be interpreted as experimental results rather than proof of normative compliance.

3.4. Short-Term Thermo-Oxidative Aging Resistance (RTFOT)

The behavior of three representative formulations subjected to simulated plant aging in the RTFOT apparatus is documented in Table 4.

Table 6 - RTFOT short-term aging results for selected PMB formulations

Property	Mix 2 (Disperser)	Mix 7 (Disperser)	Mix 9	Normative Limit (EN 12607-1)
Mass Loss after RTFOT, %	0.04	0.10	0.10	$\leq 0.8\%$
Post-RTFOT Penetration, $\times 0.1\text{ mm}$	34.2	41.5 / 44.95	44.8	-
Softening Point after RTFOT, $^\circ\text{C}$	66.4	62.9 / 84.0	60.6	Not used for classification
Penetration retention, %	Not calculated (phase not matched)	97.9 / 94.6	104.2	-

RTFOT mass loss was 0.04% for Mix 2, 0.10% for Mix 7, and 0.10% for Mix 9. These values are below the EN 12607-1 mass-loss limit reported in the original manuscript. Penetration retention requires phase matching. For Mix 2, the original penetration was $69.2/35.1 \times 0.1\text{ mm}$ and the post-RTFOT value was $34.2 \times 0.1\text{ mm}$; because the corresponding phase is not identified, a unique retention ratio cannot be justified. For Mix 7, pairing the reported phases gives approximately 97.9% (41.5/42.4) and 94.6% (44.95/47.5). For Mix 9, the ratio is approximately 104.2% (44.8/43.0). These calculations are based only on the reported mean values. No comparison with SBS is made because no directly comparable SBS control experiment was performed in this study.

The Rolling Thin Film Oven Test (RTFOT, ST RK EN 12607-1-2023 / EN 12607-1) simulates short-term thermo-oxidative aging that a binder undergoes during hot-mix asphalt plant production and initial field service. In the RTFOT, a thin binder film is rotated in glass bottles inside an oven at 163°C for 85 min while an air jet promotes oxidation and volatilization of light fractions. The primary aging mechanisms during this stage include: (i) oxidative condensation of aromatic and naphthenic components, particularly in the maltene fraction, leading to an increase in asphaltene content; (ii)

evaporation of light volatile fractions (low-molecular-weight oils and resins), which increases the viscosity and stiffness of the residual binder; and (iii) cross-linking reactions that may contribute to further stiffening [12]. Because PP does not contain the unsaturated or heteroatom-rich functional groups that are most susceptible to thermal oxidation in bitumen, the polymer component may be expected to be relatively stable under RTFOT conditions; the observed mass loss and stiffness changes are therefore primarily attributable to changes in the bitumen fraction [12].

The RTFOT mass loss values of 0.04% (Mix 2), 0.10% (Mix 7), and 0.10% (Mix 9) are all well below the EN 12607-1 limit of 0.8%, indicating that the PP-modified binders do not exhibit excessive volatility during simulated plant production. Mass loss in bitumen-based materials is primarily associated with evaporation of low-boiling aromatic and naphthenic oils; the relatively low values observed here may suggest that the incorporated PP and SF components do not introduce additional volatility beyond that of the base BND 100/130 binder. The slightly higher mass loss for Mix 7 (0.10%) compared to Mix 2 (0.04%) may be associated with the higher SF content (2.5 wt.% vs. 0.3 wt.%), as fatty acid components in SF can volatilize at elevated temperatures; however, this interpretation requires confirmation from controlled testing at different SF levels.

Penetration retention after RTFOT provides a measure of aging susceptibility: a value close to 100% indicates that the binder stiffness did not change substantially during simulated plant aging, while values significantly below 100% indicate oxidative hardening. The penetration retention of approximately 97.9% and 94.6% for the two phases of Mix 7, and approximately 104.2% for Mix 9, are noteworthy. Values near or above 100% suggest that the RTFOT-aged binder has not become substantially stiffer than the unaged material in terms of needle penetration; values slightly above 100% may reflect the statistical uncertainty inherent in comparing mean values from limited replicates, or may indicate a minor softening effect associated with structural reorganization of PP domains during the aging process. It should be noted that PP crystallinity and domain morphology can be influenced by thermal history, and the RTFOT thermal cycle (163 °C for 85 min followed by cooling) may alter the degree of crystallinity of the PP phase, potentially affecting the measured penetration of the residual binder [12].

3.5. Comparison with Previous Studies

The penetration values of 38.1–57.3 $\times 0.1$ mm for single-phase PP-modified BND 100/130 formulations (3–4 wt.% PP) are broadly consistent with the range reported in the literature for PP-modified bitumens, where increasing PP content progressively reduces penetration relative to the base binder [10]. The softening point values of 56.6–66.2 °C observed in the present study likewise suggest that PP addition elevates the high-temperature stiffness of the binder, consistent with the findings of Yildirim [2], who reported that semi-crystalline polymers generally increase softening point due to the formation of a reinforcing network at service temperatures. The present results therefore support the general trend observed for polyolefin-modified bitumens, with SF acting as a secondary modifier that partially moderates the stiffening effect of PP and improves ductility.

In contrast to SBS-modified bitumens, which are commonly reported to improve both high-temperature stiffness and low-temperature flexibility [11,15], PP modification in the present study resulted in substantially reduced ductility at 25 °C for all formulations (10.05–14.1 cm, compared to the GOST 33133-2014 [14] minimum of 40 cm for BND 100/130). This is consistent with the semi-crystalline nature of PP, whose crystalline domains restrict deformation capacity of the binder, unlike the elastomeric phase structure of SBS that provides both stiffness and flexibility [11]. The incorporation of SF partially mitigated ductility reduction (from 10.05 cm in Mix 1 to 14.1 cm in Mix 3), presumably because the fatty acid and phospholipid components of SF contribute to a more deformable interfacial phase around PP domains [3,13].

The RTFOT mass loss values of 0.04–0.10% observed in this study are well within the acceptable range for PMBs, as the EN 12607-1 threshold of 0.8% was not approached by any formulation. This is broadly comparable to aging behavior reported for other PMBs [12], and suggests that PP-modified binders prepared using BND 100/130 as the base binder do not introduce unacceptable volatility risks during hot-mix plant production. Airey et al. [12] showed that polymer-

modified bitumens subjected to combined RTFOT and PAV aging retained identifiable polymer contributions to rheological properties, which suggests that PP network elements in the present formulations would likely survive short-term aging; however, this inference cannot be confirmed without dynamic rheological testing. Table 8 provides a comparative summary of key properties for selected formulations alongside qualitative reference data from the cited literature.

Table 7 - Comparative summary of key binder properties for selected formulations and qualitative comparison with literature data.

Property	This Study (range, single-phase)	PP-modified bitumens [2, 10]	SBS-modified bitumens [11, 15]	BND 100/130 standard [14]
Penetration ($\times 0.1$ mm)	31.4–76.4	Reduced vs. base binder	Variable (grade-dependent)	101–130
Softening Point ($^{\circ}\text{C}$)	51.7–67.2	Increased vs. base binder	Increased vs. base binder	≥ 43
Ductility at 25°C (cm)	10.05–14.1	Generally reduced	Generally improved	≥ 40
RTFOT mass loss (%)	0.04–0.10	≤ 0.8 (pass)	≤ 0.8 (pass)	≤ 0.8
Phase stability	SF-dependent; separation in Mix 7	Generally problematic at high PP	Generally stable (SBS)	N/A

3.6 Overall Classification and Practical Implementation

Table 5 consolidates the empirical binder properties. EN 14023 grade designations and Superpave PG grades are not assigned because the available dataset does not include the complete mandatory specification characteristics or the DSR/BBR testing required for PG determination.

Table 5 - Empirical binder properties; EN 14023 and Superpave PG grades are not assigned from the available tests.

Mix ID	Penetration, $\times 0.1$ mm	Softening Point, $^{\circ}\text{C}$	Ductility, cm	Post-RTFOT SP, $^{\circ}\text{C}$	EN 14023 / PG classification
Mix 0	—	—	—	—	Not assigned
Mix 1	46.8	61.2	10.05	—	Not assigned
Mix 2 *	69.2 / 35.1	54 / 66	17.9	66.4	Not assigned
Mix 3	57.3	56.6	14.1	—	Not assigned
Mix 4	53.7 / 42.2	59.8 / 66.6	11.0	—	Not assigned
Mix 5	31.4	62.2	7.7	—	Not assigned
Mix 6	76.4 / 45.8	53 / 63.8	18.6	—	Not assigned
Mix 7 *	42.4 / 47.5	63.4 / 61.2	7.9	62.9 / 84.0	Not assigned
Mix 8	46.0	61.4	8.9	—	Not assigned
Mix 9	43.0	66.2	6.6	60.6	Not assigned
Mix 10	38.1	67.2	—	—	Not assigned
Mix 11	66.5	56.0	—	—	Not assigned

The results show that 3-5 wt.% PP H-350 can substantially alter the measured penetration and softening point of the investigated BND 100/130 formulations. However, the available data are insufficient to designate an EN 14023 PMB grade, determine a Superpave PG, establish a single optimum composition, or claim full compliance of the asphalt concrete with ST RK 2373. For practical application, the missing mixture-design, binder-statistics, storage-stability, and rheological data should be added before performance claims are generalized.

4 CONCLUSIONS

This study investigated eleven PMB formulations based on BND 100/130 bitumen, PP H-350, and sunflower foots using empirical binder tests and RTFOT aging. An asphalt concrete test mixture was also characterized using selected physical-mechanical indicators. The conclusions below are restricted to the measurements documented in the available dataset:

1. The investigated formulations containing 3-5 wt.% PP H-350 showed penetration values of 31.4-76.4 $\times$ 0.1 mm and softening points of 53.0-67.2 °C, with some phase-separated samples showing paired values. These results demonstrate changes in empirical stiffness indicators but do not establish Superpave PG grades.

2. The reported mass balances correspond to SF contents of 0-4 wt.%. Mix 2 (0.3 wt.% SF) exhibited pronounced within-sample heterogeneity, whereas Mix 3 (0.9 wt.% SF) showed single-valued penetration and softening-point results. This supports an empirical association between SF content and apparent uniformity, but does not prove molecular compatibility or storage stability.

3. Mix 2 and Mix 7 were identified as disperser-prepared in the RTFOT dataset, while the original formulation table did not identify their blending technique. The revised Table 1 resolves this documentary inconsistency, but detailed disperser operating parameters remain to be added from laboratory records.

4. RTFOT mass loss for the three reported formulations was 0.04-0.10%. Penetration retention was not assigned to Mix 2 because the post-aging value cannot be matched unambiguously to one of its two initial phases; for Mix 7, phase-specific retention was approximately 97.9% and 94.6%, and for Mix 9 approximately 104.2%. No superiority over SBS is claimed.

5. The asphalt concrete test mixture showed water absorption of 2.95 vol.%, cohesion $C = 0.27$ MPa, tensile crack resistance of 3.4 MPa, and compressive strengths of 3.1 MPa at 20 °C and 1.1 MPa at 50 °C. These are reported experimental values; full ST RK 2373 compliance cannot be confirmed from the current documentation.

Further work should include: (a) DSR and BBR testing after the required aging procedures for actual PG determination; (b) PAV aging; (c) storage-stability testing and/or microscopy to substantiate compatibility claims; (d) complete replicate data with standard deviations or confidence intervals; (e) full asphalt-mixture design and aggregate characterization, including an unmodified-bitumen control mixture; and (f) complete documentation of disperser geometry, shear rate/power, loading sequence, temperature-time profile, and cooling conditions.

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FUNDING

This research received no external funding.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

ARTIFICIAL INTELLIGENCE STATEMENT

During the preparation of this manuscript, the authors used artificial intelligence (AI) tools solely for text-related purposes, including language editing, grammar checking, improving wording, and enhancing the clarity of the text. All scientific content, results, analyses, interpretations, and conclusions remain the sole responsibility of the authors.

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Received: 02 July 2026

Revised: 08 August 2026

Accepted: 29 September 2026
