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Investigation of Rheological Characteristics and Bleeding Behavior of PPA-Modified Bitumen Emulsion for Microsurfacing

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ABSTRACT

The widespread application of bitumen emulsion, especially in surface treatment as the commonest approach to road maintenance in the world, indicates the necessity of finding ways to improve the bitumen emulsion performance. The present research is aimed at investigating the effect of polyphosphoric acid (PPA), as an admixture, on rheological characteristics of the bitumen emulsion. For this purpose, PPA was used at different dosages (i.e., 0.4, 0.8, 1.2, and 1.6 wt.% by weight of the residual bitumen emulsion) to modify the bitumen emulsion. Evaluation of the modified bitumen emulsion samples was performed based on a number of physical parameters (Saybolt Furol viscosity, softening point, penetration, and thermal sensitivity) and dynamic shear rheometer (DSR) and multiple stress creep recovery (MSCR) tests. Results of the bitumen tests showed that the use of PPA improved the bitumen emulsion characteristics in terms of reduced penetration, increased softening point, and decreased thermal sensitivity. With increasing the PPA dosage to up to 0.8 wt.% by weight of residual bitumen emulsion, the resultant changes in the penetration and softening point were significant and improved the bitumen emulsion performance. It was also observed that the value of $G^*/\sin(\delta)$, as a measure of resistance to bleeding in hot weather, increases with PPA dosage in all samples, with the best performance obtained with the samples containing PPA at 0.8 and 1.2 wt.%. An increase in the operating temperature of the PPA-modified samples was seen, so that the sample containing PPA at 0.8 wt.% exhibited 28.6% higher operating temperature than the control sample. The values of R% and %Rdiff parameters increased with the PPA dosage. The observed reduction of percent recovery due to increased temperature in the modified sample indicated the lower sensitivity of the modified bitumen emulsion to temperature rise. The value of Jnr parameter decreased with increasing the PPA dosage, indicating a boost in the resistance of the bitumen emulsion to bleeding.

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1. Introduction

Road maintenance is of paramount importance, especially for Iran with its severe financial limitations. Due to one reason or another, any pavement is susceptible to damage over time, and such damages differ in severity considering the pavement material, traffic load, meteorological conditions, local topography, etc. This highlights the necessity of road maintenance operation as the most common approach to maintain road performance and extend its useful life [1]. A major technique of road maintenance is what is usually referred to as preventive maintenance, and the most popular means of preventive maintenance is surface treatment. Offered in a variety of types (e.g., microsurfacing, chip seal, slurry seal, etc.), surface treatments are prepared into different material compositions. The common element in almost any surface treatment mixture is, however, the bitumen emulsion, which serves as asphalt binder [2].

Application of bitumen emulsion, which is currently a widespread precursor in the road construction industry, initiated in early 20st century. As of present, it is being used for preparing various types of plant hot and cold asphalt or mixed-on-site asphalt, surface treatment, bituminous coating, sealing, patching, soil and sand stabilization mixtures, among other applications. Because of many reasons, including reduced energy consumption, alleviated environmental impacts due to abandoning petroleum solvents, reduced production cost, etc., the bitumen emulsion has been acknowledged as the best and most cost-effective alternative to cut-back bitumen [3]. Composed of bitumen, water, and emulsifier, bitumen emulsions are usually produced to reduce viscosity and improve workability at ambient temperature. In some cases, other admixtures may also be used [4]. bitumen emulsion plays a key role in maintaining the performance of asphalt

mixtures and its high sensitivity to temperature, making the treatment susceptible to bleeding and cracking at high and low temperatures. respectively, numerous efforts have been made to modify the bitumen emulsion in such a way to achieve improved performance characteristics and durability, reduced thermal sensitivity to boost resistance to cracking and rutting, better integrity and cohesion, high early strength, and enhanced setting time [5].

Modification of rheological characteristics of the bitumen by introducing different admixtures can alter the asphalt mixture properties in terms of resistance to cracking, fatigue, or rutting [6]. Recently, modification of bitumen with acids has been recalled because of its economic efficiency and avoidance of disadvantages associated with air blown bitumen. In particular, polyphosphoric acid (PPA) has been highly regarded as it allows for increasing the bitumen consolidation in a controlled manner [7]. Using a series of linear amplitude sweep tests, researchers figured out that PPA-modified bitumen exhibits increased consolidation coupled with reduced thermal sensitivity, considering the rate of shear modulus changes of the mixture (G^*), and significantly improve rheological characteristics [8]. In another piece of research, was used the PPA at different dosages to modify bitumen. Results of their research showed that the PPA can improve the bitumen grade according to the performance grade (PG) system and increase high-temperature (HT) performance for all samples without compromising their low-temperature (LT) performance significantly; application of PPA further reduced cracking along the wheel tracks [9]. On the other hand, Some other researchers investigated the effect of PPA on the bitumen behavior and concluded that PPA-modified bitumen performance is directly linked to the base bitumen used in the bitumen emulsion [10]. Another study where LT rheological properties of PPA-modified

bitumen were investigated by means of a mechanical-dynamic analyzer, it was observed the effect of PPA at low temperature is highly associated with the bitumen composition (e.g., wax and asphaltene contents), and that, in general, PPA tends to improve LT performance of the bitumen by increasing the consolidation [11]. On the other hand, it is recommended to use modified emulsion bitumens to improve the performance of surface treatments, reduce damages, better integrity and cohesion, high early strength, and enhanced setting time. Also, changing the rheological and mechanical behavior of bitumen is one of the most important reasons for bitumen modification by additives[12, 13].

Numerous pieces of research have elaborated on the modification of bitumen with PPA, while the number of studies on the modification of bitumen emulsion with the same admixture is very limited. Accordingly, in the present research, physical properties of residual bitumen emulsion upon modification with PPA at different dosages were investigated and compared by conducting softening point test, penetration test, and thermal sensitivity experiments. Subsequently, in order to evaluate the effect of PPA on rheological characteristics of the bitumen, dynamic shear rheometer (DSR) and multiple stress creep recovery (MSCR) tests were conducted on the control and admixture-containing samples according to ASTM D7175 and ASTM D7405 standard test procedures.

2. Materials

In this research, cationic bitumen emulsion was prepared from a bitumen with a penetration grade in the range of 60 – 70, which was procured from Pasargad Oil Company, as well as a quick-setting cationic emulsifier commercially known as CECA (made in France). Then, modification of the bitumen emulsion was done with a PPA of 1.9 g/cm³ in density at 25°C, which was

manufactured by Sigma Aldrich Company (Fig.1). The manufacturer's provided specifications of the PPA are presented in Table 1.

Table 1. Specifications of the PPA used in this research.

Product name	Polyphosphoric acid
Abbreviation	PPA
Chemical formula	Hn+2PnO3n+1
Boiling point	300°C
Density at 25°C	1.9 g/cm ³



Fig. 1. The used PPA.

In this work, a colloid mill operating at 2840 rpm was used for preparing the bitumen emulsion. In order to modify the bitumen emulsion with PPA, a fixed formulation was employed with pre-batching method. Accordingly, appropriate amount of the modifying agent, as per the weight of solid bitumen, was added to a soap solution containing water, acid, and emulsifier and then mixed for 60 s in the emulsifying machine. Then, bitumen was added to the mixture at 62% at a temperature of 140°C followed by mixing and emulsification. The bitumen emulsion preparation steps were followed according to ASTM D2397 and studies by other researchers[4, 14]. The modified bitumen emulsion was subjected to low temperature evaporative test according to ASTM D7497. Next, softening point and penetration tests were conducted on the residual bitumen from the LT evaporative technique[15]. Indeed,

since the bitumen emulsion is usually used in cold asphalt, HT evaporation and/or distillation process for recovering the residual bitumen cannot simulate actual curing in the field and rather tends to cause, damage to modifier, and over-oxidation beyond actual in-field conditions [16, 17].

3. Experimental program and tests

In this study, first, the cationic bitumen emulsion was modified with PPA at 0.4, 0.8, 1.2, and 1.6 wt.% by weight of solid bitumen emulsion. Next, the modified bitumen samples were evaluated on the basis of physical criteria like penetration, softening point, Saybolt Furol viscosity, and thermal sensitivity (PI). Considering the obtained results, performance of the samples was further assessed by conducting DSR and MSCR tests according to ASTM D7175 and ASTM D7505 standard test procedures.

3.1. Basic tests for characterizing the modified bitumen emulsion

In this study, common physical characteristics of the base bitumen emulsion and modified residual bitumen emulsion were investigated by conducting penetration, softening point, and Saybolt Furol viscosity tests to measure hardness (*i.e.*, penetration), softening point, and viscosity, respectively. In addition, the thermal sensitivity-penetration index presented in Equation (1) was used to evaluate thermal sensitivity of the used samples [18].

$$PI = \frac{1952 - 500 \times \log(Pen_{25}) - 20 \times SP}{50 \times \log(Pen_{25}) - SP - 120} \quad (1)$$

3.2. DSR test

DSR test was performed to measure viscoelastic properties of the bitumen. The results help describe elastic and viscoelastic behaviors of the bitumen at moderate to high temperatures. Knowing that proper study of bitumen behavior is not possible unless the effects of temperature and loading time are

simultaneously considered, one should identify the parameters that include the effects of temperature and loading time adequately – these are complex shear modulus (G^*) and phase angle (δ), which are the main outputs of a DSR test. The complex shear modulus provides a measure of resistance to loading-induced deformation, while the phase angle indicates recoverable and non-recoverable relative deformations and ranges from 0° to 90° [19]. Rutting was evaluated according to ASTM D7175 at a frequency of 10 rad/s and different temperatures in the range of 4 - 88°C or dynamic shear modulus varies between 100 Pa and 10 MPa. A stronger resistance to rutting is expected at a higher G^* coupled with a lower δ . For $1 \text{ kPa} < G^* < 100 \text{ kPa}$, experiments were performed on bitumen samples with a thickness of 1 mm and a diameter of 25 mm at temperatures ranging from 40 to 80°C . However, when $100 \text{ kPa} < G^* < 30 \text{ MPa}$, tests were performed on bitumen samples with a thickness of 2 mm and a diameter of 8 mm at temperatures ranging from 0 to 40°C . This test was performed using smartpave 301 dynamic shear rheometer apparatus (Fig.2).



Fig. 2. A view of the DSR test apparatus.

In the PG system, the value of $G^*/\sin(\delta)$ serves as an indication of rutting performance for thick asphalts under repeated loading, referring to the temperature at which the value

of $G^*/\sin(\delta)$ exceeds 1000 Pa. In surface treatments, however, since the thickness is pretty small, the rutting is no longer a concern and one the value of $G^*/\sin(\delta)$ is rather used in the surface performance grade (SPG) system which is specially designed for surface treatments and indicates the resistance to bleeding in hot weather and It indicates the temperature at which the value of $G^*/\sin(\delta)$ exceeds 650 MPa. In other words, an increase in the value $G^*/\sin(\delta)$ suggests a reduction in bleeding probability [20]. In the PG system, the temperature is measured at 20 mm below the surface treatment, while the SPG system considered the temperature directly at the surface of the treatment [21].

3.3. MSCR test

MSCR test results are used to evaluate bitumen sensitivity to permanent deformation or rutting. This test was performed on a DSR apparatus with a plate of 25 mm in diameter and 1 mm in thickness at a high operating temperature for the bitumen according to ASTM D7405. In this test, at a high operating temperature, the samples were subjected to stress levels of 0.1 and 3.2 kPa and percent recovery (R%) and non-recoverable modulus (Jnr) were calculated after 10 loading cycles[22]. Each loading cycle included 1 s of loading followed by 9 s of relaxation. Beginning with 10 cycles of loading at 0.1 kPa, the procedure was followed by another 10 loading cycles at 3.2 kPa. The required time for performing the entire set of loading cycles was about 300 s. This test method was suitable for all unaged and aged bitumen samples by either rolling thin-film oven (RTFO) or pressure aging vessel (PAV). This test provides two key parameters, namely R% and non-recoverable creep compliance (Jnr) at two stress levels of 0.1 and 3.2 kPa. Normally, the higher the recoverability (R%) of the bitumen, the more desirable the bitumen elasticity and the larger the recovered strain during the relaxation time (*i.e.*, 9 s). Moreover, the closer

the non-recoverable creep compliance to zero, the higher the bitumen resistance to deformation. The non-recoverable creep compliance measures the ratio of residual strain after loading-unloading cycle to the applied stress, indicating the sample resistance to permanent deformation.

4. Results and discussion

4.1. Results of basic tests on modified bitumen emulsion

Table 2 reports the values obtained from the penetration, softening point, Syabolt Furol viscosity, and thermal sensitivity tests on the samples modified with PPA at 0.4, 0.8, 1.2, and 1.6 wt.% by weight of residual bitumen emulsion. As seen, modification of bitumen emulsion with PPA at 0.4 and 0.8 wt.% increased the softening point of the bitumen emulsion by 4.84 and 9.86%, respectively, compared to the control bitumen emulsion, while lowering the penetration by 5.08 and 15.25%, respectively. Adding the PPA at any higher dosage rather decreased the softening point while increasing the penetration. The asphalt mixture prepared from a bitumen of lower softening point exhibit lower thermal sensitivity. Moreover, a decrease in the penetration indicates the higher hardness of modified bitumen emulsion, improving the asphalt performance against applied loads.

PI provides a measure of thermal sensitivity, so that a higher PI indicates a bitumen sample of lower thermal sensitivity and smaller permanent deformations [3]. In the bitumen emulsion samples containing PPA at 0.4 and 0.8 wt.%, the PI increased by 22.64 and 37.36% compared to the control, while the modified bitumen emulsion samples containing PPA at 1.2 and 1.6 wt.%, PI decreased. Moreover, results of the Saybolt Furol viscosity tests shows that the introduction of PPA at up to 0.8 wt.% increases the bitumen emulsion viscosity by up

to 27%, although any higher dosage of PPA tends to reduce the viscosity.

Table 2. Physical characteristics of the modified bitumen emulsions.

Sample	SB	PPA0.4	PPA0.8	PPA1.2	PPA1.6	Test range	Standard procedure
Saybolt Furol viscosity (seconds)	22	26	28	25	24	100-20	ASTM D88
Penetration(0.1 mm)	59	56	50	60	68	90-40	ASTM D5
Softening point(°C)	62	65	68	68	64	> 57	ASTM D36
PI	2.65	3.25	3.64	2.72	2.48	-	-

The results presented in Table 2 are further visualized on a graph in Figure 3. In general, it is evident that, among other modified samples, the one containing PPA at 0.8 wt.% exhibited the best performance in terms of softening point, penetration, Saybolt Furol viscosity, and PI, and that any increase in the PPA dosage beyond 0.8 wt.% tends to reduce the bitumen emulsion performance.

The reason for improving the performance of emulsion bitumen modified with polyphosphoric acid due to the changes created in the microstructure of bitumen and its chemical compounds can be considered to be the increase of the effective amount of asphaltene and the reduction of resins and saturated oils of bitumen, which improves the performance properties of bitumen and it shows the thermal stability of modified bituminous compounds[10, 23].

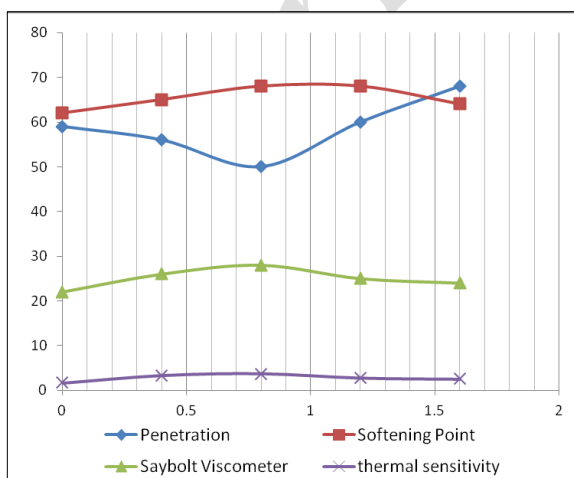


Fig. 3. Results of using PPA for modifying bitumen emulsion.

4.2. Results of rheology assessments on bitumen emulsion

4.2.1. DSR test

The results obtained from the DSR tests are presented in Figures 4 to 6, where the values of G^* and $G^*/\sin(\delta)$ are plotted in logarithmic scale. As is clear on Figures 4 and 5, introduction of PPA increased the complex shear modulus and phase angle for all PPA-containing samples, as compared to the control sample, although the level of reduction differs for the samples containing PPA at different dosages.

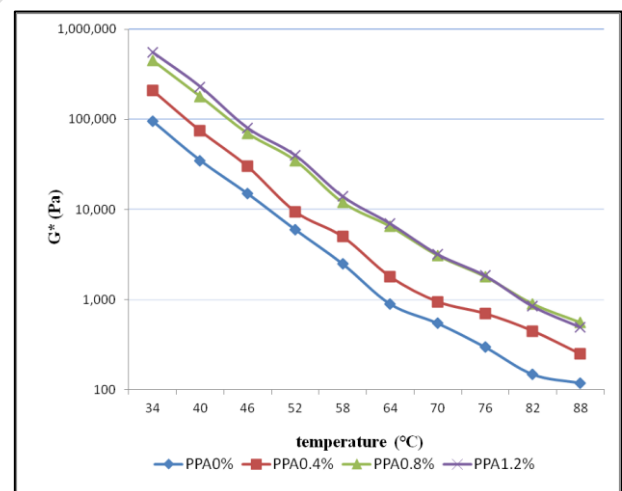


Fig. 4. Cross plot of complex shear modulus against temperature at different PPA dosages.

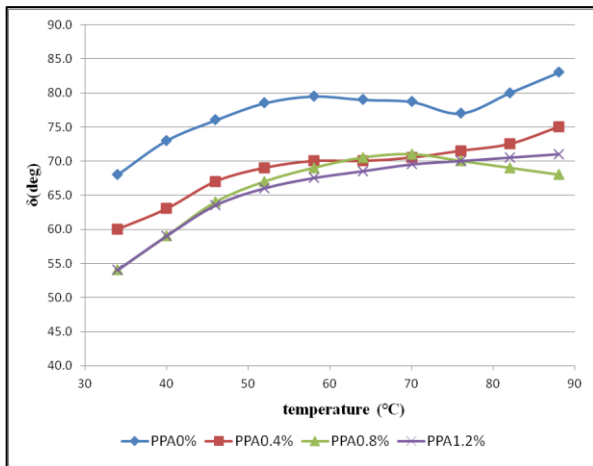


Fig. 5. Cross plot of phase angle against temperature at different PPA dosages.

In order to obtain a better insight into these two variables, Figure 6 presents the plot of $G^*/\sin(\delta)$, capturing the effects of G^* and δ simultaneously. As is evident from this figure, $G^*/\sin(\delta)$, which measures, in the SPG system, the resistance to rutting in hot weather, monotonically increases with PPA dosage in all modified samples, with the best performance shown by the samples containing PPA at 0.8 and 1.2 wt.% - showing closely similar performances at different temperatures. At moderate temperatures, the bitumen emulsion containing PPA at 1.2 wt.% outperformed the other samples while the bitumen emulsion containing PPA at 0.8 wt.% was marginally better performing than the one containing PPA at 1.2 wt.% at higher temperatures.

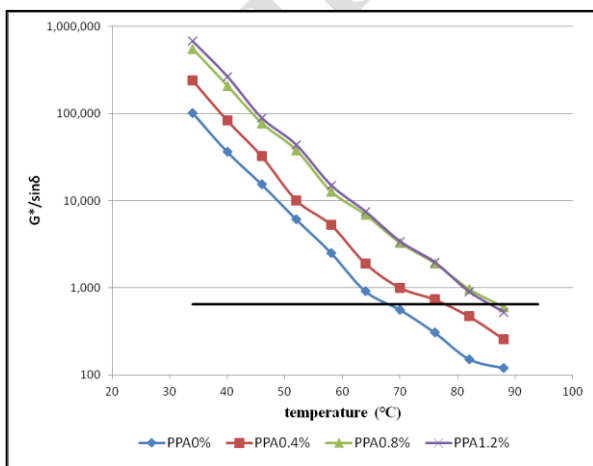


Fig. 6. Cross plot of $G^*/\sin(\delta)$ against temperature at different PPA dosages.

Table 3 reports the values of SPG temperature index. The results clearly indicate the effectiveness of PPA modification on increased operating temperature of residual bitumen emulsion. As is evident from the results, the sample containing PPA at 0.8 wt.% exhibits the best performance among all samples. Indeed, this sample showed an operating temperature that is 1.8 and 28.6% better than those of the sample containing PPA at 1.2 wt.% and the control sample, respectively.

Table 3. Operating temperatures of modified bitumen emulsion samples.

Sample	PPA%	Operating temperatures
Bitumen emulsion	0	67.8
Modified bitumen emulsion	0.4	78
	0.8	87.2
	1.2	85.7

4.2.2. MSCR test

Results of the MSCR tests at 58 and 64°C are shown in Figures 7 through 11. The results show that the highest R% is associated with the PPA-modified bitumen emulsion at 0.8 wt.% operating at 58°C under a stress level of 0.1 kPa. At the other end of the spectrum, the worst performance was that of unmodified bitumen emulsion operating at 64°C under a stress level of 3.2 kPa. On the other hand, it is evident that not only the stress level but also temperature rise (regardless of the stress level) impose adverse impacts on the samples, reducing the R% in all samples. At both stress levels, it was observed that an increase in the PPA dosage increases the R% although the increase in PPA dosage from 0.8 to 1.2 wt.% did not contribute to increased R%. Conversely, this effect is highly evident at PPA dosages of 0.4 and 0.8 wt.%.

Results of bitumen emulsion modification shows that R% increases with the PPA dosage. On the other hand, %Rdiff results demonstrate that the percent difference in recovery between the two stress levels decreases with increasing

PPA dosage, thereby reducing the bitumen emulsion sensitivity to stress buildup.

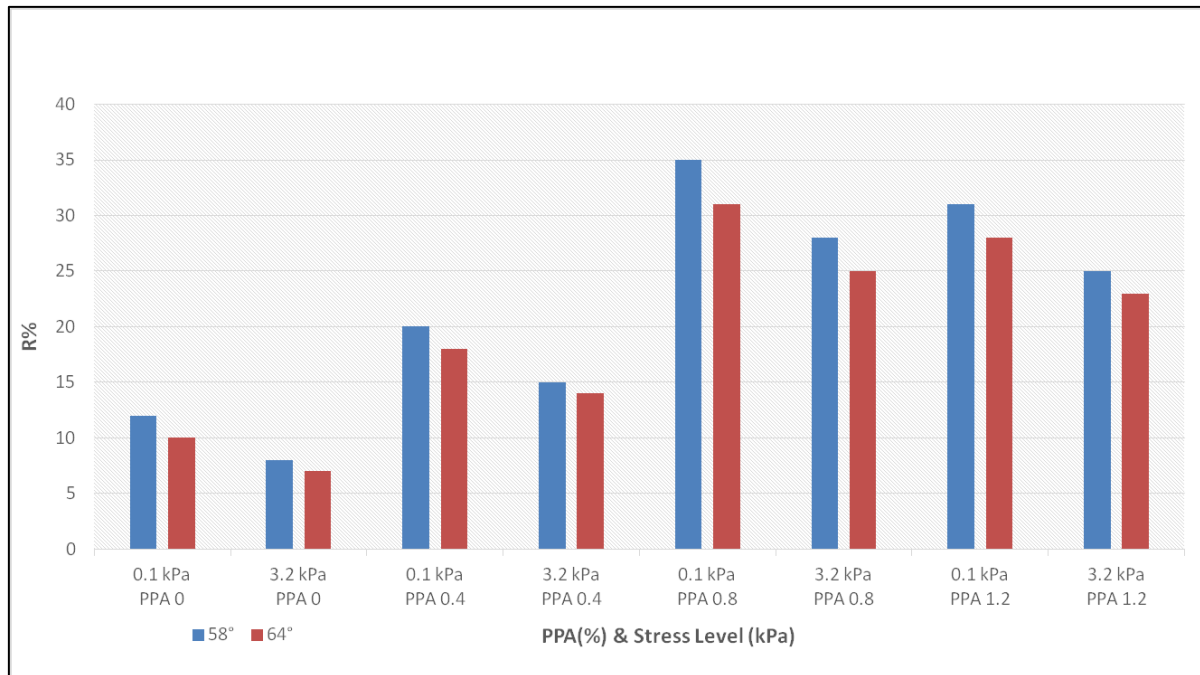


Fig. 7. Plot of R% for PPA-modified bitumen emulsion at different levels of stress and temperature.

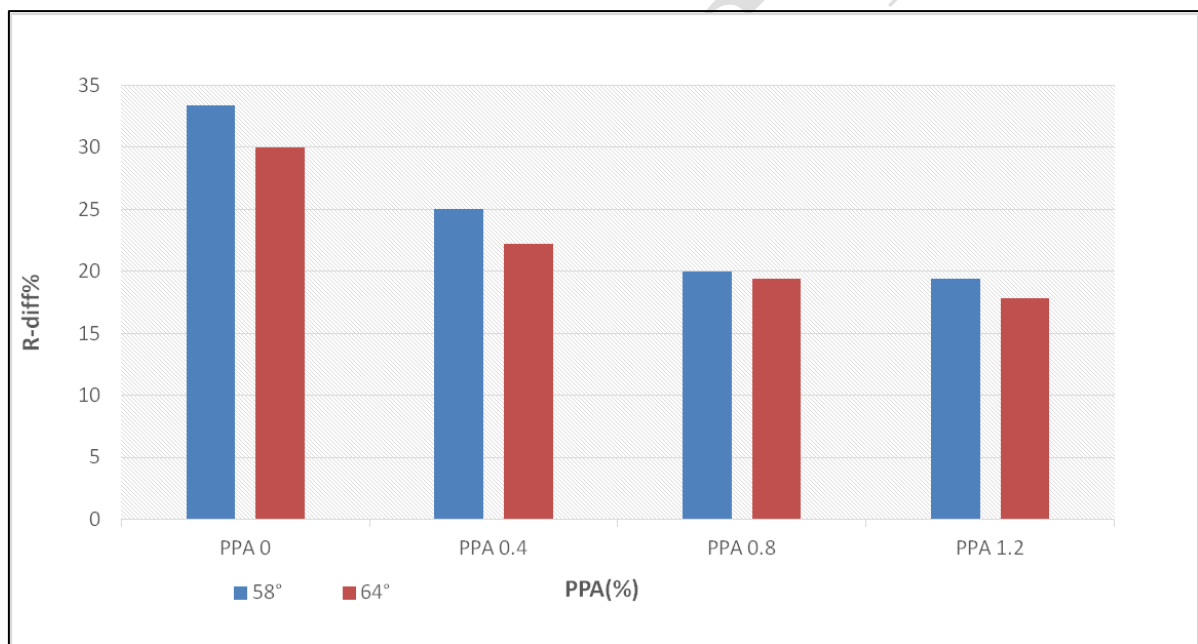


Fig. 8. Plot of %Rdiff for PPA-modified bitumen emulsion at different temperatures.

From the results, it is evident that, at all stress levels and PPA dosages, the reduction in %R due to temperature buildup is lower than that of non-modified bitumen emulsion, indicating reduced sensitivity of the modified bitumen emulsion to temperature buildup. Accordingly, the best stress level was exhibited by the bitumen emulsion modified with PPA at 0.8 wt.%.

Regarding the non-recoverable creep compliance, as shown in Figure 9, it was figured out that this parameter decreases significantly with increasing the PPA dosage to above 0.4 wt.%, with the sample containing PPA at 1.2 wt.% showing the lowest value of non-recoverable creep compliance, closely ahead of the sample containing PPA at 0.8 wt.%. Another finding was that an increase in

temperature at both low and high stress levels added to the non-recoverable creep compliance in all samples, highlighting the negative impact of increased temperature. The results indicated more than 50% reduction in non-recoverable creep compliance at the stress level of 3.2 kPa for the samples containing PPA at 0.8 and 1.2 wt.%, as compared to the base bitumen emulsion, marking very good performance of the modified bitumen emulsion. As is clear from Figure 10, for all

samples at all stress levels, the percent increase in J_{nr} at 64°C compared to that at 58°C followed an ascending trend with increasing the PPA dosage, reflecting the adverse effect of temperature on modified bitumen emulsions. All by all, based on the obtained results for the non-recoverable creep compliance, one may expect that the modified bitumen emulsion exhibits more resistance to rutting.

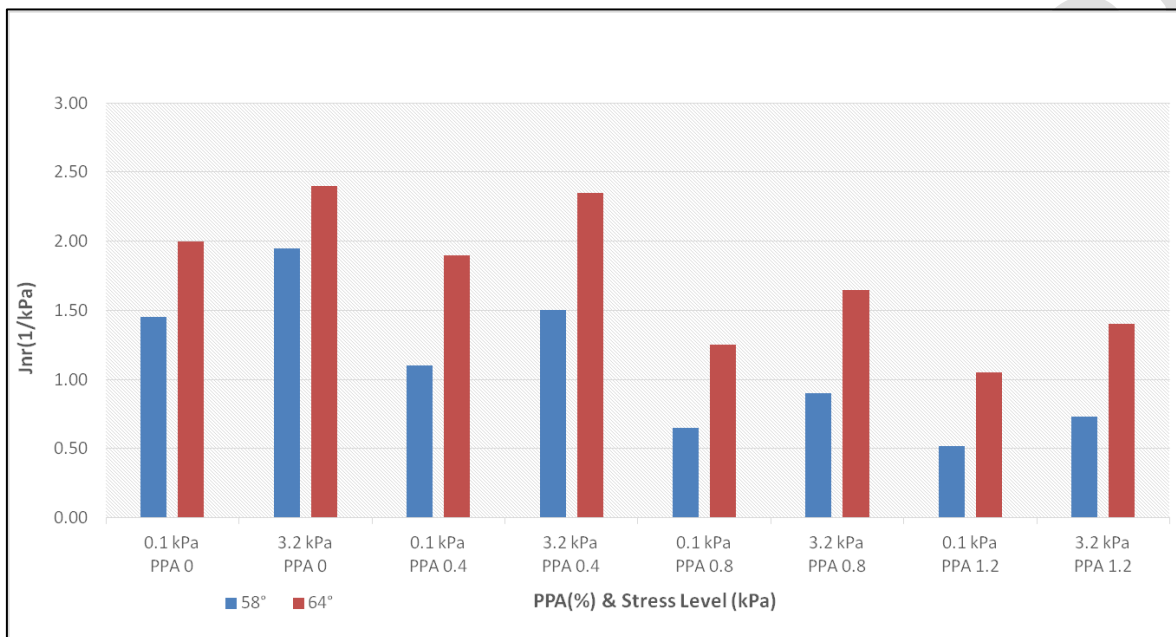


Fig. 9. Plot of non-recoverable creep compliance for PPA-modified bitumen emulsions at different levels of stress and temperature.

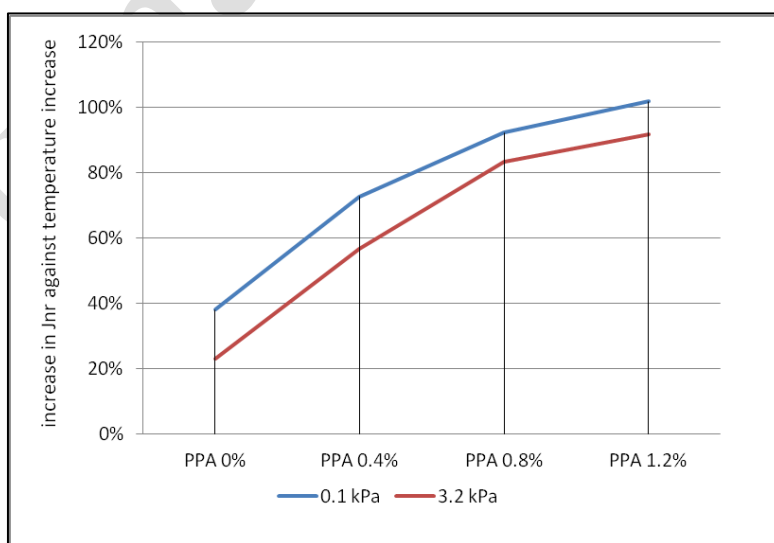


Fig. 10. Plot of percent increase in non-recoverable creep compliance against temperature increase for PPA-modified bitumen emulsions at different levels of stress.

As demonstrated in Figure 11, the percent difference in non-recoverable creep compliance ($J_{nr-diff}$) followed a decreasing trend with increasing the PPA dosage, indicating a drop of the samples' sensitivity to applied stress and enhanced performance of

bitumen emulsion. According to the results, the bitumen emulsion modified with PPA at 1.2 wt.% was found to be the best-performing sample, closely ahead of the one modified with PPA at 0.8 wt.%.

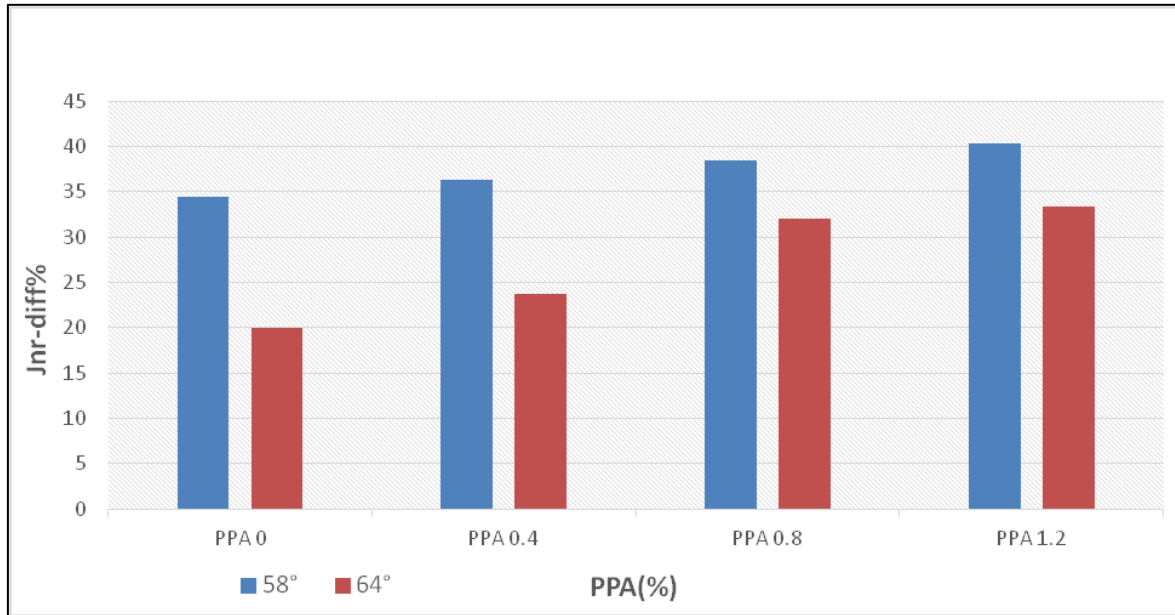


Fig. 11. Plot of percent difference in non-recoverable creep compliance of PPA-modified bitumen emulsions at different temperatures.

5. Conclusion

In this research, effect of modifying the bitumen emulsion with PPA was evaluated and analyzed. For this purpose, softening point, penetration, Saybolt Furol viscosity, and thermal sensitivity tests were performed and DSR and MSCR experiments were further conducted. Based on an analysis of the obtained test results, the following conclusions were drawn:

1. Results of bitumen tests showed that the use of PPA at up to 0.8 wt.% by weight of residual bitumen emulsion led to increased softening point and viscosity coupled with decreased penetration of the bitumen emulsion, thereby improving the bitumen emulsion performance. At higher PPA dosages, opposite results were obtained.
2. increase in the PPA dosage increased the value of $G^*/\sin(\delta)$ in all PPA-modified samples, which is a measure of resistance to rutting in hot weather under the SPG system, and the best performance at moderate and hot temperatures was shown by the bitumen emulsion containing PPA at 1.2 wt.% and 0.8 wt.%, respectively.
3. Temperature values of the SPG index were higher in the modified samples rather than the control and the sample containing PPA at 0.8 wt.% exhibited the best performance in terms of this index.
4. The percent recovery (%R) increased with the PPA dosage at both stress levels of 0.1 and 3.2 kPa. The increase in %R with PPA dosage was significant at PPA dosages up to 0.8 wt.%, although a further increase in PPA dosage from 0.8

to 1.2 wt.% imposed no significant effect on increasing the %R.

5. With increasing the PPA dosage, %R increases and percent difference in recovery decreases at both stress levels, indicating decreased sensitivity of the bitumen emulsion to increased stress, *i.e.*, increased resistance to bleeding at higher stress levels.
6. At all stress levels, the value of Jnr decreased with increasing the PPA dosage. At the stress level of 3.2 kPa, this reduction exceeded 50% for the samples containing PPA at 0.8 and 1.2 wt.%, as compared to the control sample. As a general conclusion while considering relevant technical and mechanical considerations, it is recommended to use PPA at 0.8 wt.% by the weight of residual bitumen emulsion. Future studies that can be valuable as a complement to this work include more detailed economic and environmental analyzes of different fillers in the life cycle of microsurfacing mixtures.

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Conflicts of interest

There are no conflicts of interest to declare.

Authors contributions

The main author, MJAB, conducted the experiments while the supervisor, MMK, provided guidance and the original idea for the study.

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