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Effects of marine microplastics on the mechanical performance of bituminous binder for road asphalt pavements

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Abstract

Microplastics, MPs, represent a apprehension as their accumulation causes irreversible damage to the marine environment and wildlife and consequently in the food chain. At the same time, it is well recognized that MPs can outstandingly improve the road performance of bitumen. In this regard, encapsulating marine MPs in bitumen can indeed both enhance the performance of bitumen and encourage their recycling. Therefore, the goal of this study is to investigate the feasibility of marine MPs as fillers or modifier in bitumen. Three different MPs compositions and two different dosage (3 and 6% wt. of bitumen) were analyzed, for a total of six solutions and compared with 1) asphalt mastics containing traditional limestone powder and 2) modified bitumen (PMB). The obtained asphalt mastics or modified bitumen were characterized in terms of empirical and rheological analyses. It was found that in the presence of 6% MPs, the softening points of neat B50/70 bitumen (B50/70) can be increased by more than 6.1°C, and the penetration can be reduced by more than 8.0 dmm. B50/70 and showed a significant improvement in the elasticity and stiffness since the phase angle is reduced while the complex shear modulus is increased. Marine MPs are innovatively employed to achieve modification of flexible road binders corresponding to significative reductions of heavy metals concentration up to 85%. Therefore, a recycling strategy of marine MPs is developed, offering a new solution for sustainability in the field of road engineering.

Keywords: Asphalt; rheology; leaching test; sustainability

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1. Introduction

An green issue that has increasing over the time is due to the huge amount of plastics in the aquatic environments (Agbekpornu and Kevudo 2023). Microplastics (MPs) are producing negative effects on the marine ecosystem (Kershaw 2016).

MPs are conventionally defined as particles with size ranging between 1 and 5000 μm, composed of synthetic polymers (either thermosets or thermoplastic materials), such as polypropylene (PP), polyethylene (PE), polyvinyl chloride (PVC), polyether sulfone resin (PES), polyethylene terephthalate (PET) and polystyrene (PS), as well as elastomers (e.g., styrene-butadiene rubber, SBR) (Jarlskog et al. 2020).

The MPs recycling and reuse is of great importance to the marine environment. About the 60% of the world's thermoplastic output is used in the building construction sectors. One of the usable disposals of plastic waste in the road construction sector is that it can be found in the production of bituminous pavements.

The current recycling routes include mainly physical and chemical methods. In the first case, sorting, washing and grinding, followed by density separation, sieving, filtration, and precipitation (Stang et al. 2022) are typically selected. In contrast, chemical methods usually involve the conversion of polymers into monomers or oligomers, capable of being reused in industrial production chains as fuels or as raw materials through decomposition or pyrolysis (dos Santos Ferreira et al. 2022).

Many research have been carried out studies focused on the introduction of MPs in bitumen as modifier, by using different MPs types as PVC, PET, LDPE, HDPE and PP; all the results revealed positive effects on base bitumen by appropriate techniques, such as reducing penetration (Ghabchi et al. 2021), increasing softening point (Machsus 2021), improving elastic recovery (Dalhat and Adesina 2020) and reducing permanent deformation (Ghabchi et al. 2021). The best rutting resistance (higher G* and lower δ values) can be achieved at 2% and 8% PVC concentration for 250 μm PVC microplastics mixed in bitumen at 200°C with a speed of 3000 rpm (Maharaj et al. 2014). The PET microplastics are usually added into bitumen at 4-20% wt. of bitumen (Silva et al. 2017) to reduce the penetration and to improve the softening point and resistance to permanent deformation.

It is worth noting that recent works focused on MPs found in road dust, while distinguishing between MPs released from the road itself and those released from nearby construction sites, tyres or other sources remains complex. Therefore, it is essential to further assess the environmental impact of the application of marine MPs to asphalt pavements. Furthermore, the modification of bitumen by existing MPs is often achieved by single MPs. However, MPs collected from marine a lot contain a variety of waste plastic particles, such as PP, PE, and PS, are the most common polymers detected in MPs, accounting for 27%, 26%, and 9% of the total MPs concentration, respectively (Shruti et al. 2021). The effect of MPs on bitumen properties is therefore different from that of single MPs on bitumen.

Therefore, the aim of the present study is to evaluate a sustainable strategy of combining MPs collected from the ocean with bitumen to obtain eco-friendly asphalt mastics and to reveal their effect on bitumen properties and the modification mechanism. The physical and rheological properties of the MPs-modified asphalt mastics were then characterized to verify the improvement of the MPs on the bitumen. Finally, the goal of the research was to find an innovative employment of the marine MPs to achieve modification of flexible road binders, in the light of a recycling strategy with zero leakage of marine MPs offering a new solution for sustainable development in the field of road engineering.

2. Materials

2.1. Microplastics

In this study, the microplastics (MPs) collected from the sea of Southern Italy were analyzed to identify their possible reemployment as replacement of the typical limestone powder for making asphalt mastics and/or probable modifier for bitumen. The MPs recovered were subject to a cut up process through a mechanical shredder to obtain particle less than 5 mm in size. Since the obtained MPs were a mixture of several types of plastic particles and different types of plastics stand for different effects on the performance of asphalt mastics, the study was focused on three different categories of MPs according to their melting points and crystallinity as follows: (1) MPs with high melting point (CH) above 110 °C; (2) MPs with low melting point (CS), belove 110 °C and (3) a mixture of 50% CH and 50%

CS (CHS). The composition and the presence of harmful, polluting and dangerous compounds were analysed in previous studies (Veropalumbo et al. 2023). The three types of microplastics are shown in Fig. 1. Limestone Powder (LP) was used as comparison purpose.

a) b) c)

Fig. 1 MPs: (a) CH; (b) CS and (c) CHS.

2.2. Bitumen

A neat bitumen 50/70 penetration grade (B5070), typical bitumen used in European countries, supplied by a refinery in Southern Italy was adopted in the research for the production of the asphalt mastics. At the same time a polymer modified bitumen PMB 10/40-70 (PMB) was adopted for the comparison (see Table 1).

Table 1. Properties of bitumen.

Properties	Standard method	B50/70	PMB
Penetration at 25 °C (dmm)	EN 1426	62	54
Softening point (R&B) (°C)	EN 1427	46	69
Dynamic viscosity at 150 °C (Pa·s)		0.36	0.42
Dynamic viscosity at 100 °C (Pa·s)	EN 13702	4.40	9.74
Dynamic viscosity at 60 °C (Pa·s)		276.30	5406.59

2.3. Asphalt mastic preparation

B50/70 was preheated in an oven at 160°C for 30 min, then LP or MPs filler was added to the preheated bitumen in stages, and finally the filler was dispersed at 160°C for 0.5h at 600rpm to complete the preparation of the mastic. The conventional limestone filler and three MPs fillers were dispersed into B50/70 at 3% and 6% bitumen weight ratio by high-speed shear dispersion method, respectively. A total of eight mastics were prepared, two limestone filler mastics (3% LP and 6% LP) and six MPs mastics (3% CH, 3% CHS, 3% CS, 6% CH, 6% CHS and 6% CS). The B50/70 and PMB binders were also used as references and controls for the performance evaluation of all the mastics.

3. Methods

3.1. Empirical and rheological tests

The physical properties tested in this study included viscosity, softening point and penetration. The viscosity test was conducted by the method specified in EN 13702, which tests the dynamic viscosity of bitumen mastics at 60°C, 100°C and 150°C, respectively. The softening point test was in accordance with the ring & ball method in EN 1427 standard. The penetration test of bitumen mastic at 25°C was carried out according to the method specified in EN 1426 standard.

The frequency sweep test was carried out on a Dynamic Shear Rheometer (EN 14770). Each bitumen mastic was tested in the frequency range of 0.1-10 Hz at intervals of 10°C in the range of -10°C to 60°C, respectively. 8 mm diameter parallel plate geometry with 2 mm gap was configured in the -10°C-30°C scanning; 25 mm diameter parallel plate geometry with 1 mm gap was configured in the 30°C-80°C scanning. The shear modulus G*, the phase angle δ, the storage modulus G' and the loss modulus G" of the bitumen and mastics were measured.

The multiple stress creep and recovery (MSCR) test was performed (EN 16659 standard) by using a DSR configuration with a 25 mm parallel plate geometry and a 1 mm gap. The sample was loaded at constant stress for 1 s, then allowed to recover for 9 s. Ten creep and recovery cycles are run at 0,100 kPa creep stress followed by 10 more cycles at 3,200 kPa creep stress. The non-recoverable creep compliance (Jnr) and the percent recovery (%R) are used to measure the rutting resistance under repeated loading.

4. Results and discussion

4.1. Physical properties

The softening points of neat B50/70 and mastics made up with MPs filler are shown in Fig. 2. Compared with the B50/70 (softening point of 46.0°C), the softening points of CH, CHS, and CS MPs mastics were increased by 6.5%, 10.2%, and 12.0%, respectively, at 3% content, and by 13.7%, 17.4%, and 21.3%, respectively, at 6% content. All three MPs can significantly increase the softening point of B50/70, and the softening point of mastic with high MPs dosing is more significantly increased. Comparing the softening points of the three MPs, it can be seen that CS mastic has the highest softening point and CH mastic has the lowest softening point. The softening point of CHS mastic is between that of CS mastic and CH mastic. Furthermore, it can be found from Fig. 2 that 3% and 6% LP only raise the softening point of the B50/70 by 2.8% and 7.0%, respectively, which is less than the increase in softening point of mastic by either of the MPs at the same content. The increase in softening point implies an increase in the ability of mastic to resist permanent deformation, and the above data indicate that MPs can improve the ability of mastic to resist permanent deformation more effectively than conventional limestone powder.

Penetration as a common consistency parameter can also reflect the ability of bitumen mastic to resist permanent deformation. Fig. 2b depicts the penetration of B50/70, limestone powder bitumen mastic and MPs bitumen mastic. The penetration of B50/70 is 62 (0.1 mm), which is reduced by 3% and 6% LP by 0.5% and 1.9%, respectively. However, the penetration of B50/70 is reduced by 4.8%, 10.6% and 23.4% by 3% CH, CHS and CS, respectively; and by 12.9%, 17.7% and 28.1% by 6% CH, CHS and CS, respectively. This indicates that (1) CS MPs have the most significant reduction in penetration, followed by CHS MPs and the worst by CH MPs; (2) MPs can more significantly improve the consistency and enhance the ability of asphalt mastic to resist permanent deformation compared with LP.

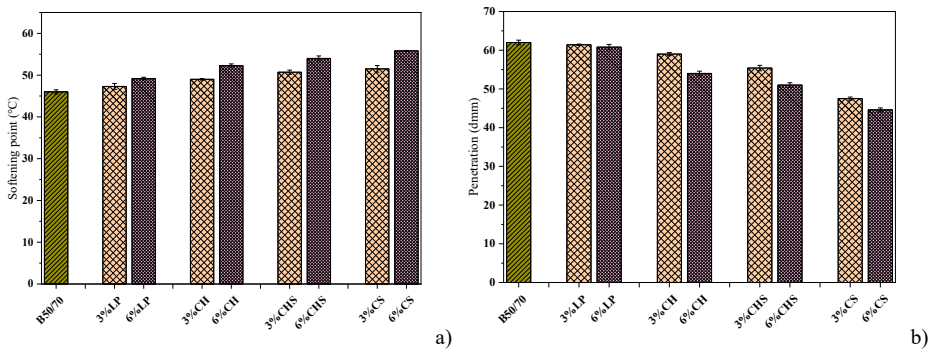

Fig. 2 a)Softening point and b) penetration of bitumen and asphalt mastics.

Table 2 Dynamic viscosity of bitumen and bitumen mastics.

Mastic type	Unit	B50/70	3%LP	6%LP	3%CH	6%CH	3%CHS	6%CHS	3%CS	6%CS
60 ℃	Pa·s	389.61	624.77	793.96	2277.68	2843.61	2928.82	3243.90	7913.27	12162.02
100 ℃		4.86	7.64	7.70	8.63	10.09	10.53	16.70	15.79	30.91
150 ℃		0.26	0.37	0.39	0.42	0.52	0.47	0.59	0.71	0.88

4.2. DSR results

It is first observed from Fig. 3a that the main curve of complex modulus of the 3% LP mastic is almost coincident with that of B50/70 except in the reduced frequency below 1.0E-02Hz (high temperature), where the main curve of complex modulus is slightly higher than that of B50/70. This indicates that the 3% LP only slightly improves the stiffness of B50/70 at high temperature, but has little effect on the medium and low temperature performance.

Secondly, in the range of 1.0E-05 Hz to 1.0E-01 Hz, the G* of 3% CH mastic is slightly higher than that of 3% LP mastic; while the G* of 3% CHS mastic is gradually and significantly higher than that of 3% CH mastic with decreasing reduced frequency (increasing temperature); The obtained results shows that (1) 3% CH and 3% CHS are more effective in improving the high temperature properties of the mastic compared to 3% LP; (2) 3% CH and 3% CHS are consistent with 3% LP in enhancing the rheological properties of mastic in the medium temperature range; (3) 3% CH and 3% CHS were detrimental to the low temperature performance of the mastic. Finally, the master curves of 3% CS mastic are significantly higher than those of B50/70, 3% LP mastic and other MPs mastic, and gradually coincide with those of B50/70 and 3% LP mastic after the reduced frequency is greater than 1.0E+03Hz (low temperature). This indicates that 3% CS MPs improves the rheological properties of B50/70 better than other fillers and has little effect on the low-temperature properties.

The most significant trend in the master curves of the complex shear modulus as shown in Fig.3b is that both 6% LPs and 6% MPs have positive effect on the G* in low frequency range (high temperature performance) of B50/70. The enhancement of G* in the low frequency range of B50/70 is different among the three MPs. In particular, in the low frequency range of 1.0E+00Hz-1.0E-05Hz, the G* of CH mastic starts to be gradually higher than that of LP mastic after the reduced frequency is less than 1.0E-03Hz, and the G* is improved by 1.0% on average compared to that of LP mastic; The G* of CHS mastic becomes gradually higher than that of LP mastic after the reduced frequency is less than 1.0E-02Hz, and the G* is improved by 2.0% on average; The G* of CS paste begins to be gradually higher than that of LP paste after the reduced frequency is less than 1.0E+00Hz, and the G* is increased by 3.7% on average. In contrast, in the range of 1.0E+00Hz-1.0E+06Hz, all the master curves of MPs mastics gradually overlap with that of LP mastic and then gradually fall slightly below that of 6% LP mastic as the reduced frequency increases.

As shown in Fig. 3c, the phase angles of both B50/70 and LP mastics demonstrate a tendency to increase as G* decreases. In particular, the phase angle master curves of B50/70, 3% LP mastic and 6% LP mastic almost overlap without significant differences, which indicates that the enhancement of B50/70 elasticity by LP is not obvious, and the addition of LP has not caused variations in the elasticity and viscosity parameters of B50/70. In contrast, the phase angles of all the MPs mastics are smaller than those of the LP mastics.

In contrast, the phase angles of all the MPs mastics are smaller than those of the LP mastics. Among all the MPs mastics, 6% CS returns the smallest phase angle, which is on average 41% lower than that of 6% LP; 3% CH returns the largest phase angle, which is on average 13% lower than that of 6% LP.

Unlike the phase angle of LP mastics, which tends to increase with decreasing G*, the phase angles of all MPs mastics show a tendency of increasing and then decreasing with decreasing G*. According to the point where the transformation of the material properties may occur, Fig. 3c can be divided into two regions, upper (A) and lower (B), bounded by G* (1.0E+06 Pa) at 30°C.

The tested temperature range in region A is -10°C to 30°C. The phase angles of B50/70 and all the mastics in this region increase with decreasing G*. In particular, no significant differences or patterns are found between B50/70 and all the mastics except that the phase angle of 6% CS is progressively smaller than that of the other mastics during G* from 1.0E+07 Pa to 1.0E+06 Pa. This indicates that all fillers have little effect on the low-temperature performance of B50/70, which is consistent with the results reflected by the trend in the high-frequency range of the complex shear modulus curves for each of the mastics in Fig. 3a.

Observing the region B in Fig. 3c, i.e., the region with G*<1.0E+06 Pa, it can be found that the phase angles of all MPs mastics show a trend of increasing and then decreasing with decreasing G*. In particular, (1) the 6% CS mastic exhibits the minimum G* of 3.5E+04 Pa which is much greater than the minimum G* of B50/70, 3% LP and 6% LP; the overall average of the phase angle of the 6% CS mastic is reduced by 42% compared to the overall average of the phase angle of B50/70, 3% LP and 6% LP. (2) The 6% CHS, 3% CS, 3% CHS and 6% CH reach the maximum phase angle (between 71° and 74°) at G* of 1.0E+05 Pa almost simultaneously. Compared to the phase angle of 6% CS mastic, the phase angle of 6% CHS mastic, 3% CS , 3% CHS and 6% CH is increased by 223%-236% and the

maximum phase angle corresponding to G* is reduced by a factor of 10 (from 1.0E+06 Pa to 1.0E+05 Pa). (3) The 3% CH has the largest phase angle of 83° among all MPs filler pastes, corresponding to a G* of 1.0E+04 Pa. However, compared with 3% LP and 6% LP mastics with maximum phase angle (89°) corresponding to a G* of 262 Pa, 3% CH is still a more effective elastic reinforcement solution than LP fillers. Overall, the addition of MPs not only enhances the G* of the bitumen mastic, but also reduces the phase angle. This effectively improves the elasticity of the bitumen mastic, especially under high temperature conditions.

From the above results it is evident that 6%CS shows the best performance among all the mastics. It is decided to compare the rheological performance of 6%CS with that of PMB to further evaluate the performance of 6%CS mastic. Fig. 3d depicts the master curve and phase angle of 6%CS and PMB.

It is first observed that G* of 6%CS is globally larger than G* of PMB by 12% on average over the entire reduced frequency range. Further, especially at high temperatures of 50°C and 60°C, the difference between 6%CS and PMB gradually increases as the reduced frequency (warming) decreases. This indicates that the 6% CS mastic exhibits higher stiffness than PMB in the range of -10°C to 60°C, and this advantage is especially significant at high temperature.

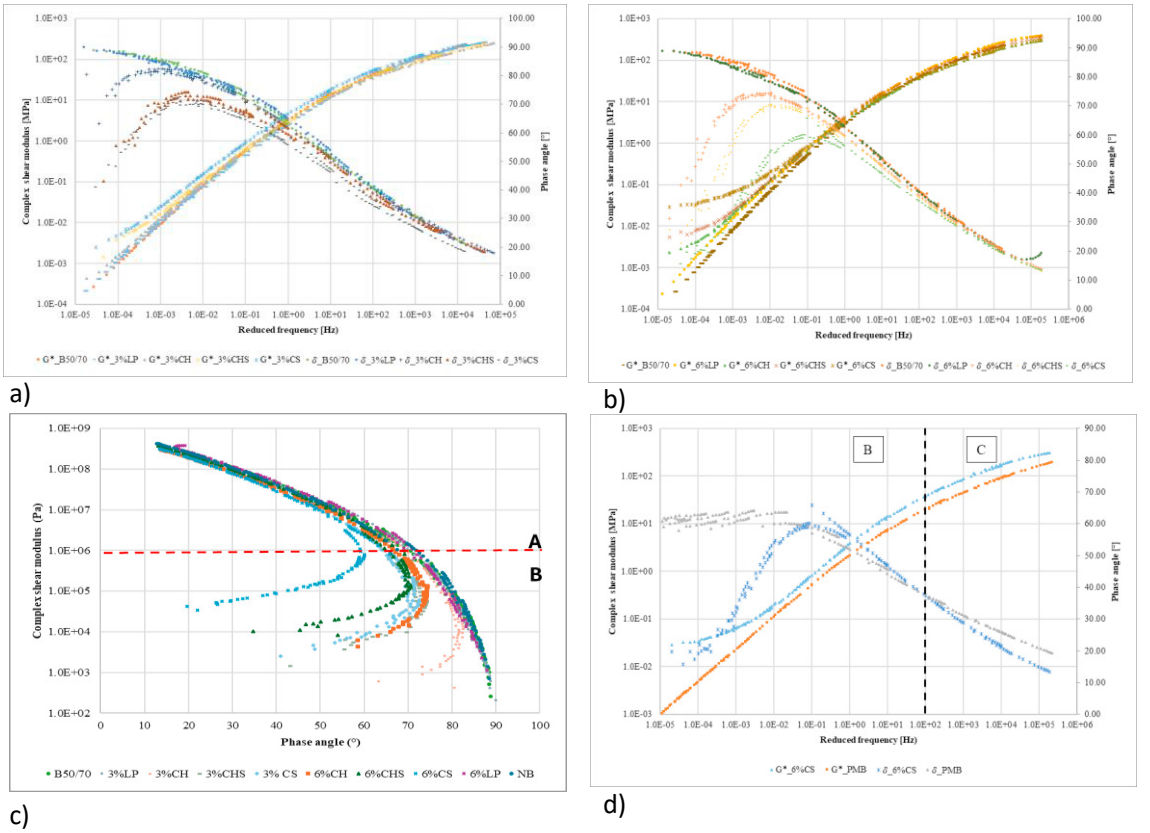

Fig. 3 FS results: master curves comparing a) 3% MPs and b) 6% MPs, c) black diagram comparing 3% and 6%MPs and d) master curve comparing 6%CS and PMB.

4.3. MSCR results

To better determine the rutting resistance of bitumen and mastics, Jnr and Rτ were calculated for all bitumen and mastics under two stresses of 0.1 kPa and 3.2 kPa at 40°C and 50°C, respectively, as shown in Fig. 4. It is reported that the lower the Jnr value, the better the ability of the mastic to resist permanent deformation, and the larger the Rτ, the more elastic the mastic is.

MPs mastics return less Jnr than that of LP mastics under the same content of filler. In particular, (1) when the filler is 3% by weight of B50/70, the Jnr0.1 and Jnr3.2 for CH mastic are 9.3% and 9.4% at 40 °C (31.4% and 24.7% at 50 °C) lower than those of LP mastic; The Jnr0.1 and Jnr3.2 of CHS mastic are 17.4% and 12.4% at 40 °C (47.6% and 30.8% at 50 °C) lower than those of LP mastic; The Jnr0.1 and Jnr3.2 of CS are 21.7% and 19.1% at 40 °C (49.4% and 48.7% at 50 °C) lower than those of LPs. (2) When the filler is 6% by weight of B50/70, The Jnr0.1 and Jnr3.2 of CH mastic are 65.8% and 34.7% at 40 °C (44.1% and 42.5% at 50 °C) lower than those of LP mastic; The Jnr0.1 and Jnr3.2 of CHS mastic are 75.2% and 41.3% at 40 °C (50.2% and 47.9% at 50 °C) lower than those of LP mastic. The above results show that MPs reduce the Jnr of B50/70 more effectively than LP and improve the creep recovery of B50/70, since even a mastic containing only 3% CS already returns a lower Jnr0.1 and Jnr3.2 than 6% LP mastic at 40°C (Fig. 4 (a)). Moreover, the Jnr0.1 and Jnr3.2 of any one of the MPs mastics at 50°C is less than those of 6% LP mastic (Fig. 4 (b)), which further supports the conclusion that MPs are more effective than LP in reducing the permanent deformation of B50/70 under repeated loading.

In addition, increasing the MPs content allows the mastics to obtain smaller Jnr0.1 and Jnr3.2. For example, Jnr0.1 and Jnr3.2 for 6% CH mastic are 68.6% and 40.4% at 40°C (30.3% and 35.1% at 50°C) lower than those for 3% CH mastics, respectively. By comparing the Jnr0.1 and Jnr3.2 values of different types of MPs mastics at the same content, it can be observed that, for example, Jnr0.1 and Jnr3.2 of 6% CHS mastics are 27.5% and 10.1% at 40°C(10.9% and 9.5% at 50°C) less than those of 6% CH mastics; And Jnr0.1 and Jnr3.2 of 6% CS mastic are 61.7% and 21.3% at 40°C (36.4% and 28.3% at 50°C) lower than those of 6% CH mastic. The relationship of Jnr0.1 and Jnr3.2 for each 3% MPs mastic is in the same trend as those of 6% MPs mastic, that is, the CS MPs have the strongest reduction effect on the Jnr0.1 and Jnr3.2 of B50/70. In summary, the values of Jnr0.1 and Jnr3.2 for the 6% CS mastic are the lowest among all bitumen and mastic, which means that this mastic has the best resistance to permanent deformation under repeated loading. This result is also the same as that found in the softening point, needle penetration, dynamic viscosity, and frequency sweep tests.

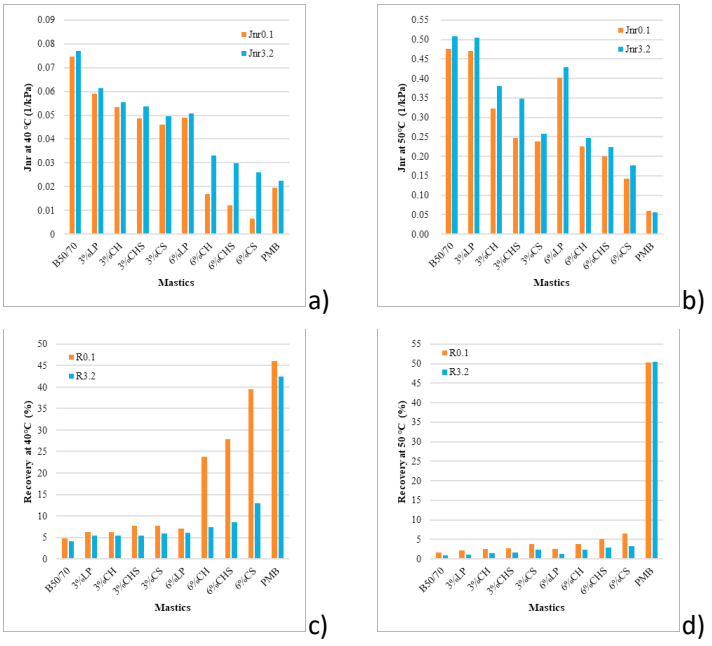

Fig. 4 MSCR results: a) Jnr at 40°C, b) Jnr at 50°C, c)R at 40 °C and d) R at 50°C.

From Fig. 4 (c) it can be observed that R0.1 and R3.2 of B50/70 returns at 40°C are 4.8% and 4.1%, respectively; 3% CH mastic and 3% LP mastic show almost the same recovery ratio (6.3% under 0.1 kPa and 5.4% under 3.2 kPa).

It is remarkable that the recovery ratio of 3% CS mastic is almost equal to that of 6% LP mastic at 3.2 kPa stress level (6%), while at 0.1 kPa stress level, the recovery ratio of 3% CS mastic is already about 10% higher than that of

6% LP mastic. Therefore, the MPs filler has a better effect on the B50/70 recovery ratio than LP filler, since the MPs filler requires only 50% of the weight of the LP filler to obtain the same recovery ratio of the mastic as that of the LP filler. Another remarkable feature in Fig. 4 (c) is that the recovery ratio of the mastic with 6% MPs content is much higher than that of the other bitumen and mastic.

Fig. 4 (d) depicts the recovery ratio of B50/70, mastics made up by LP and mastics made up by MPs at 50 ℃. Similar to the case of 40° C, both R0.1 and R3.2 of B50/70 are the lowest, at 1.6% and 0.96%. The addition of 3% LP filler only improves the recovery ratio of B50/70 at 0.1 kPa stress to 2.2%, while it hardly contributes to the recovery ratio of B50/70 at 3.2 kPa stress. LP filler is increased from 3% to 6%, and R0.1 and R3.2 are increased from 2.2% and 1.0% to 2.5% and 1.3%, respectively. Although the R0.1 and R3.2 of 3% CH is the lowest among all MPs mastics, it is still 0.05% and 1.5% higher than 6% LP, which once again indicates that MPs are more efficient fillers than LPs to improve the B50/70 recovery ratio. 6%CS is the best recovered mastic among B50/70, mastics made up by LP and mastics made up by MPs with R0.1 and R3.2 of 6.4% and 3.3%, which is 1.45% and 0.34% (2.55% and 1.05%) higher than R0.1 and R3.2 for 6% CHS.

5. Conclusions

In this study, three types of MPs collected from marine environment and classified according to their melting melting point (HMPs, MMPs and LMPs) are employed as filler or modifier of the bitumen for road pavement applications. The effect of marine MPs on the characteristics of bitumen is evaluated in terms of basic and rheological characterization.

The main conclusions are as follows:

1) MPs (6%wt of bitumen) raises the softening points of B50/70 by at least 6.1°C, and reduces the penetration by at least 8.0 dmm and 5.8 dmm. The addition of MPs reduces the temperature sensitivity of B50/70 and improves the resistance to high-temperature deformation.

2) MPs is a more desirable filler for B50/70 than LP since the G* of MPs mastics is overall higher than the G* of LP mastic, especially at high temperature. MPs reduces the phase angle of the bitumen while enhancing G*, improving the stiffness and elasticity of the bitumen.

Further studies will be carried out on the influence of the obtained mastics on the entire asphalt mixture, focus the attention on the mechanical response and environmental compatibility to provide if they produce positive or negative effect.

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