



Polymer-modified Bitumen from the Wastes of Petrochemical Plants

Ali Akbar Yousefi

Institute for Colorants, Paint and Coatings, P.O. Box: 16765/654, Tehran, Iran
Iran Polymer and Petrochemical Institute, P.O. Box: 14965/115, Tehran, Iran

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ABSTRACT

Waste management is a major concern in petrochemical plants. Almost all types of wastes are used to modify bitumen. Following this concept polymer and non-polymer wastes of petrochemical production lines were used to modify bitumen. It was found that incorporation of the wastes separately or as a combination of some high performing polymer-modified bitumens are obtained. Almost all modifiers did not improve the low temperature properties of the modified bitumen and in some cases deteriorated this property. The low molecular weight polyethylene (LMP) and *N*-methylpyrrolidone (NMP) wastes increase high temperature performance of bitumen, whereas styrene-butadiene rubber (SBR) waste improves low temperature performance at moderate concentrations. The best performance grades (PG) obtained by introducing these modifiers are PG = 106-10, PG = 76-22 and PG = 94-16 at a rate of 5% modification, respectively. The best performance grades obtained with mixtures of NMP and SBR were PG = 82-16 and PG = 76-22. The most useful performance grades resulted from incorporation of mixtures of LMP and SBR in bitumen were PG = 94-16 and PG = 100-10. In fact, mixtures of LMP and SBR wastes were found to be the most beneficial compositions for bitumen modification.

INTRODUCTION

Environmental aspects of polymers are enormous. Industrial and post-consumer wastes are widely studied [1,2]. However, the production plants of polymers also produce a large volume of off-grade polymeric materials. These polymeric materials by no means are useful in the downstream petrochemical industry. Meanwhile, some other materials used in the polymerization process are converted into residues which cannot be used in a petrochemical plant. Any finding to be able to use these wastes is welcome by industry. In a parallel sector of petroleum

industry that is petroleum refining sector, a large amount of vacuum bottom residue is produced. Depending on the crude oil from which this residue is taken, one is able to obtain bitumen directly or via air-blowing process [3-6]. Bitumen is a well-known construction material which is widely used in roads and pre-fabricated (built-up) roofing membranes worldwide [7-8]. Depending on the countries between 75% and 85% of bitumen production is consumed in road construction. The rest is used in roofing membrane or other applications.

Key Words:

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(*) To whom correspondence to be addressed.

E-mail: a.yousefi@ippi.ac.ir

Due to the increases in speed of passenger cars and tyre pressure for lorries the conventional non-modified bitumens are not able to resist against the dynamic mechanical loads of the past traffic. The thermal stresses resulting from the changes in temperature during the days and seasons result in transverse and alligator cracking and permanent deformation of an asphalt layer. These have to be added to the distresses on an asphaltic pavement. Also in some situations special functions such as shock absorbing and formation of porous asphalt is expected from bitumen. The bitumen is not able to meet all these requirements and characters alone.

Natural bitumen was pioneer material in road construction in 19th century [2]. In 20th century the petroleum refineries merged as main sources of bitumen. In the refinery at first crude oil is distilled under atmospheric pressure to separate light cuts. In the second step, the residue of the first tower is distilled under reduced pressure (vacuum) to isolate heavy medium to heavy cuts. The residue exited from the bottom of the second tower known as vacuum bottom is bitumen (straight run bitumen) or converted to bitumen via the well-known air-blowing process (blown bitumen) [3-6]. None of these bitumens are able to undergo the conditions mentioned in the previous paragraph. This is due to the inherent characteristics of the bitumen constituents. Bitumen is composed of wide varieties of homologues and interchangeable molecules which results in a thermoplastic colloidal structure [9]. This is a noncross-linked nanocomposite which its behaviour converts from a highly brittle elastic solid to a Newtonian viscous fluid in an 80-degree range of temperature. Actually, it is very good for mixing bitumen with aggregates in hot-mix asphalt production. But this very high temperature sensitivity of bitumen is not useful from the service-life point of view. Very high temperature sensitivity of bitumen properties stems from this fact that bitumen is composed of some solids (asphaltenes) and glass-forming materials (oils and resins, maltenes). Depending on the parent crude and process of production, bitumen represents some glass-transition temperatures (T_g) below 20°C. In the vicinity of T_g as temperature increases normally the modulus of the glass-forming materials reduces 1000 times. In the case of bitumen the changes in the properties can

also be controlled by the colloidal structure of bitumen. This structure can be sol (separated asphaltene particles) or gel (networked asphaltene particles). However, this complicated structure of bitumen does not provide the necessary consistency which is needed in roads and thin roofing membranes [2].

To meet new technical needs, bitumen has been subjected to different types of chemical and physical modifications [2,6]. Amongst different bitumen modifiers, the polymers are the most effective ones [6,10-12]. Different polymers are used in bitumen modifications. The styrene-butadiene rubber (SBR) is one of the bitumen modifiers. The bitumen modification has been reported in different research works [10-12]. However, the latex of SBR is successfully used in bitumen modification. The polymeric waste materials are also used in bitumen modification [13]. These materials are very different, e.g. industrial ashes, cellulose wastes, lignin, glass and scrap rubber of old tires. The first three of these materials were used as extenders, and recycled glass was used to reduce air voids in asphalt concrete. Scrap rubber is widely used to improve asphalt properties [2,13]. The wastes of polyethylene and polypropylene and some other polymers have also been used in asphalt [2,14-16]. One of the interesting aspects of bitumen modification is the possibility of using industrial wastes via simple physical blending.

In SBR production plant of Bandar Imam Petrochemical Complex (BIPC) (the BD-SR plant from Basparan sub-company of BIPC, Iran) there are two annoying wastes; popcorn [17] and NMP wastes [18]. The first one is an agglomerated fine powder of SBR which is an off-grade SBR. The second one is the bottom residue of the *N*-methylpyrrolidone (NMP) purification tower. This is composed of a complex mixture of chemicals and contains also a low percentage of SBR fine particles. The Iranian bitumen production plants are presently faced with a shortage of feedstock. The NMP waste (with a bituminous appearance) can act as a bitumen extender which makes it commercially very attractive. Meanwhile, another polymeric waste is available from high-density polyethylene (HDPE) plant of Basparan sub-company of BIPC. This waste called low molecular weight polyethylene (LMP). This is a waxy polymer which is isolated in hexane washing

unit of this plant. Similar works are already published [19-21]. A special technique named reactive mixing was also developed by Dabir et al. to improve compatibility of the polymeric wastes with bitumen [22].

In this paper we report on the positive effects of these three polymeric wastes from petrochemical plants on bitumen properties. To evaluate these effects the physical properties of the resulting modified bitumen are measured and compared with those of the base and control bitumens. The well-known softening point, penetration and Frass breaking point of bituminous blends were used to achieve this goal. The morphology of the resulting modified bitumen which represents the state of dispersion of the modifier in the bituminous matrix affecting the performance and engineering properties of the modified bitumen is also studied. According to our awareness this is for the first time that these types of wastes are used in bitumen modification and reported.

EXPERIMENTAL

Materials

Materials which were used are *N*-methylpyrrolidone (NMP), low molecular weight polyethylene (LMP) and SBR wastes all from Bandar Imam Petrochemical Complex (BIPC, Iran). Bitumen used in all modifications is 60/70 penetration grade from Tehran Refinery. The physical properties and composition of this bitumen are reported in Tables 1 and 2 [18].

Procedures

The base bitumen and modifiers were blended in a

duplex high-shear mixing system composed from a Polytron 6000 connected to a high-shear aggregate PT-DA 3020/2 along with a low-shear propeller mixer Polymix (Kinematika Co, Switzerland) [18,23]. To mix the waste components with bitumen, at first bitumen was melted in an oven at 160°C and transferred into the mixing chamber at 170°C. The mixing chamber was heated and controlled using a Haake oil-circulation heater. Here, while the high-shear mixer was operated at 14000 rpm the modifiers were introduced into the mixing chamber and mixing was continued for the next 30 min. At the end of mixing time, the mixture was taken out of the mixer into a water-bath cooled metallic can by opening the drain valve of the mixer. At this stage the necessary samples were taken for different tests.

The penetration, softening point, Frass breaking point tests were carried out according to National Iranian Standards ISIRI 2950 (ASTM D5), 2951 (ASTM D36), and 3867 (IP 80), respectively. The performance grade (PG) of the bitumens was estimated using the following semi-empirical relations [23]:

$$T_{DSR} \cong T_{R\&B} + 20, \quad T_{BBR} \cong 2(T_{Frass}),$$

$$PG = T_{DSR} + T_{BBR}$$

where T_{DSR} is the high temperature performance criterion, $T_{R\&B}$ is the bitumen softening point, T_{BBR} is the low temperature performance criterion and T_{Frass} is the Frass breaking point.

The morphology of the blends was viewed through a Zeiss FX optical microscope, Jenapol model. The FTIR spectrum of the NMP waste was obtained on a

Table 1. The physical properties of 60/70 penetration grade from Tehran Refinery.

Penetration at 25°C (0.1 mm)	Softening point (°C)	Frass breaking Temperature (°C)	Performance grade (PG)
58	50	-12	70-22

Table 2. Composition of 60/70 penetration grade from Tehran Refinery (crude from Asmari reservoir of Ahwaz in Khuzestan, Iran) [21].

Saturates (wt%)	Naphthene aromatics (wt%)	Polar aromatics (wt%)	Asphaltenes (wt%)
15.66	39.15	35.04	10.15

Brucker model IFS 48 Spectrophotometer. The Mooney viscosity of the popcorn waste was measured on a Mooney viscometer (Zwick 4809). The flow curve of the LMP was measured on a Paar-Physica MCR300 Rheometer in dynamic oscillatory mode using 25 mm parallel-plates geometry at 170°C.

RESULTS AND DISCUSSION

The raw materials are not well-characterized. At first the raw materials were characterized using the proper techniques. An optical microscopic view of the morphology of the NMP waste was reported [18]. It was found that this waste is not a simple single component material and that the microscopic rubber particles are observable in an oily matrix. The case of LMP waste is a little different. It is well understood that this waste is waxy and composed of low molecular polyethylene molecules. The rheology of this waste is very important. During mixing this waste with bitumen at 170°C, the only overwhelming parameter, is the viscosity of the materials to be mixed. Therefore, the dynamic oscillatory measurement was carried out on this waste. At 170°C this polymeric waste rheological behaviour is rubber-like and we observe a rubbery plateau in G' and G'' curves, whereas a shear-thinning behaviour is observed for complex viscosity (η^*) (Figure 1). Although a rubber plateau is observed for this waste, the values of the moduli are very small (around 20 and 200 Pa for G'' and G' , respectively). Due to the shear-thinning behaviour of the complex viscosity of

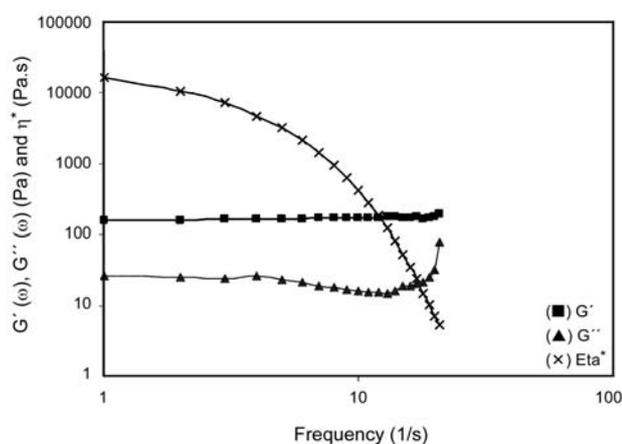


Figure 1. Rheological material functions of LMP at 170°C.

Table 3. Physical and rheological properties of the wastes.

Raw material	LMP	NMP	Popcorn
Penetration at 25°C	0	>400	--
Mooney viscosity (ML 1+4)	--	--	36
Complex viscosity at 170°C and 1 Hz (Pa.s)	~104	--	--

LMP a 1000-fold decrease in this material function can be attained in moderate shear rates (frequencies $\sim 20 \text{ s}^{-1}$). This observation makes it certain that at very high shear rates applied by the used duplex mixer, the viscosity of the LMP and bitumen matches each other. Normally standard bitumen is a Newtonian fluid with a viscosity of 3 Pa.s at 135°C [24]. The SBR waste (popcorn) is a rubbery material and like other rubbers is well-characterized with Mooney viscosity. The experimentally determined value 36ML1+4 was obtained for this waste (Table 3). This value is almost 10 units less than that of the standard SBR 1500, which is an indication of lower molecular weight of the waste polymer. A lower molecular weight simply translates to the ease of dispersion of these highly elastic chain molecules in bitumen under shear. A finer dispersion of rubber in bitumen helps to stabilize the emulsion and improves the engineering properties of the modified bitumen.

Blends and their Physical Properties

Binary Blends

Modified bitumens were prepared by blending with a single waste and also by a binary mixture of these waste modifiers. The physical properties of bituminous blends containing only one waste modifier are summarized in Tables 4-6. The rate of modification was limited to 1-10 wt% of modifier. In Table 4 the physical properties of the base and control (the bitumen undergone mixing conditions without addition of any modifier) bitumens are compared to those of 3 wt% to 10 wt% SBR waste-modified bitumens. A decrease followed by an increase in penetration as SBR waste content increases from 3 wt% to 10 wt% is observed. Meanwhile, the softening point of these blends reaches a maximum at 7% of modification and then it declines. The inverse is true for the Frass breaking point of these samples and a minimum is

Table 4. Effect of popcorn (SBR) waste on properties of Tehran 60/70 bitumen.

Sample	SBR (%)	Penetration (0.1 mm)	Softening point (°C)	Frass temperature (°C)	Performance grade (PG)
Base	0	58	50	-12	70-22
Control	0	50	55	-11	70-22
P1	3	43	56	-10	70-16
P2	5	42	55	-13	70-22
P3	7	41	59	-11	76-22
P4	10	45	57	-9	76-16

Table 5. Effect of low molecular weight polyethylene (LMP) waste on properties of Tehran 60/70 bitumen.

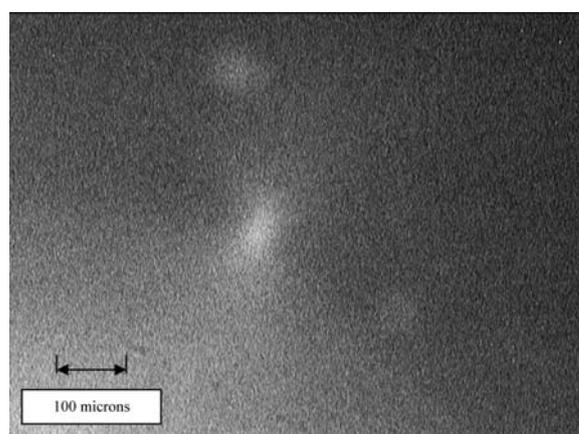
Sample	LMP (%)	Penetration (0.1 mm)	Softening point (°C)	Frass temperature (°C)	Performance grade (PG)
L1	1	41	61	-10	76-16
L2	3	33	68	-10	88-16
L3	5	34	76	-8	94-16
L4	7	32	81	-7	100-10
L5	10	24	86	-6	106-10

Table 6. Effect of *N*-methylpyrrolidone (NMP) residue on properties of Tehran 60/70 bitumen.

Sample	NMP (%)	Penetration (0.1 mm)	Softening point (°C)	Frass temperature (°C)	Performance grade (PG)
N1	3	44	59	-9	76-16
N2	5	39	60	-11	76-22
N3	7	45	61	-8	76-16
N4	10	37	61	-10	76-16

observed for this property. According to the calculated performance grades (PG) of the bitumen, the best performance is observed at 7% modification (PG = 76-22). From these results this can be concluded that SBR waste is of a positive effect on bitumen physical properties up to 7% modification and thereafter loses its effectiveness. As a matter of fact, the morphologies represented in Figures 2 and 3 divulge the reason behind this behaviour. It seems that up to 7 wt% the morphology goes towards a very fine morphology and this improves the properties. At higher SBR content, the polymer phase forms a continuous network and absorbs the lubricating constituents of bitumen, which directly results in a lower softening point and increases in penetration (the effect of soft network formation at medium temperatures) and Frass breaking point

(the effect of brittleness of bitumen at low temperatures). Similar results were found in vacuum bottom

**Figure 2.** The optical microscopic picture of P1 sample morphology.

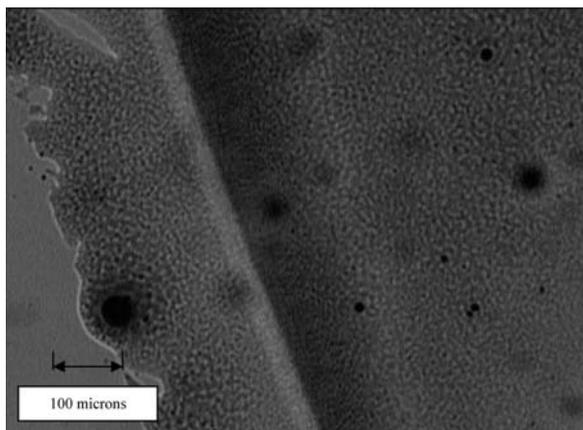


Figure 3. The optical microscopic picture of P4 sample morphology.

part for this waste [25-27].

In Table 5 the physical properties and estimated PG of the LMP-modified bitumens are reported. A sharp decrease in penetration and a moderate decrease in Frass breaking point along with a sharp increase in softening point are observed for these blends. These observations stem from low molecular weight and crystallinity of LMP. Due to low molecular weight and viscosity of LMP as compared with those of polyethylene this material can be well dispersed in bitumen. As shown in Figures 4 and 5 the morphology of the prepared formulations is finer than that of their polyethylene counterparts [11,18,19,23]. From a performance point of view it is seen that at high concentrations of LMP some high-performing bitumens at high temperatures are obtained (L4 and L5). These modified bitumens are best fitted with conditions of

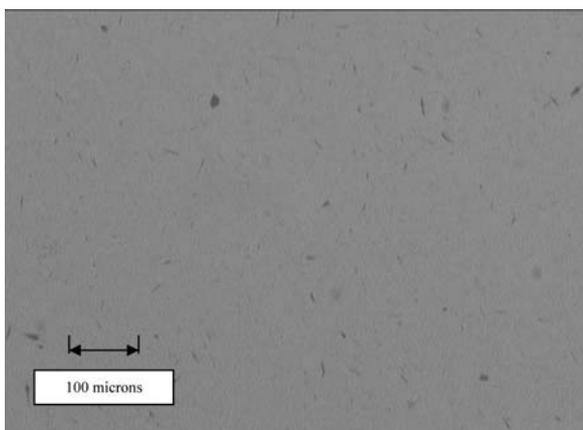


Figure 4. The optical microscopic picture of L1 blend morphology.

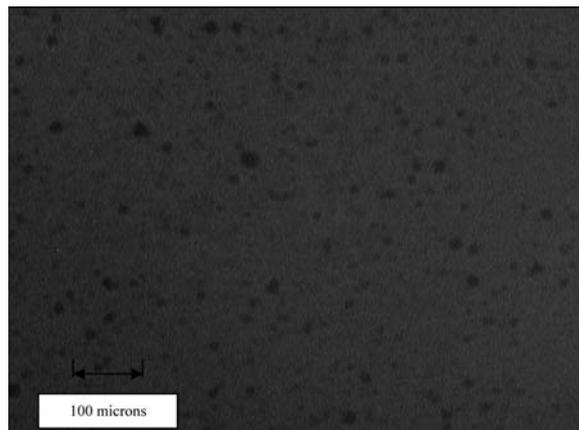


Figure 5. The optical microscopic picture of L5 blend morphology.

hot or tropical climate; these properties out-perform some commercial polymer-modified bitumens. The interesting point about these high concentrations of polymer is that these fine polymeric inclusions are less sensitive to phase separation and expected to be stable at high temperatures.

Table 6 summarizes the effect of addition of NMP waste on engineering properties of 60/70 penetration grade bitumen from Tehran Refinery. As it is noticed, in the range of experimental errors all the prepared blends are of engineering properties very close to those of the control bitumen. Lower penetrations and higher softening points as compared to those of the control bitumen are indications of the reactions taken place between bitumen and NMP waste components. In a view point of performance, we observe nearly a constant performance for these series of bituminous blends. All these contraries to what was expected for NMP waste/bitumen are indicating that this waste is of a hardening effect on bitumen. Therefore, NMP waste cannot be used to function as lubricating oil (at these concentrations). The morphology of these blends was not reported because its similarity to that of the base bitumen. These observations suggest NMP wastes act as an extender for bitumen which can increase the bitumen production volume.

Ternary Blends

In Table 7 the physical properties on the ternary blends of NMP waste/SBR waste/bitumen are summarized. Reduction in penetration and augmentation of softening point with a nearly un-touched Frass

Table 7. Effect of *N*-methylpyrrolidone (NMP) and styrene-butadiene rubber (SBR) wastes on properties of Tehran 60/70 bitumen.

Sample	NMP (%)	SBR (%)	Penetration (0.1 mm)	Softening point (°C)	Frass temperature (°C)	Performance grade (PG)
PN1	1	5	45	58	-10	76-16
PN2	2	5	46	60	-10	76-16
PN3	3	5	44	62	-9	82-16
PN4	1	7	43	60	-9	76-16
PN5	2	7	41	61	-8	76-16
PN6	3	7	42	61	-11	76-22

breaking point and performance grade is observed for these ternary blends. The morphology of the resulting blends is presented in Figure 6. The other blends' morphologies were not reported for the sake of brevity. As it is clearly observed, the rubber particles are much larger than those in P-series blends (Figures 2 and 3). This can be the result of polymer swelling by NMP waste components. It is obvious that these two wastes do not improve bitumen properties extensively; however, we simply observe an extender function for this couple.

In Table 8 the composition of ternary blends of LMP/SBR waste/bitumen along with corresponding engineering properties are compiled. These blends are very different from others. It is evident from this table that as LMP content increases (at constant SBR waste content) penetration intensively decreases, whereas the softening point and Frass breaking temperature dramatically increase. These changes result in a

drastic improvement in high temperature criterion of PG. Meanwhile, the low temperature criterion of PG is deteriorated. These observations suggest these bituminous blends suitable for very hot climates. The

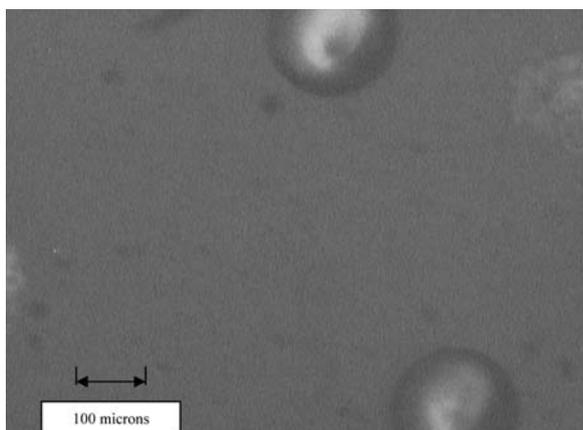


Figure 6. The optical microscopic picture of PN4 blend morphology.

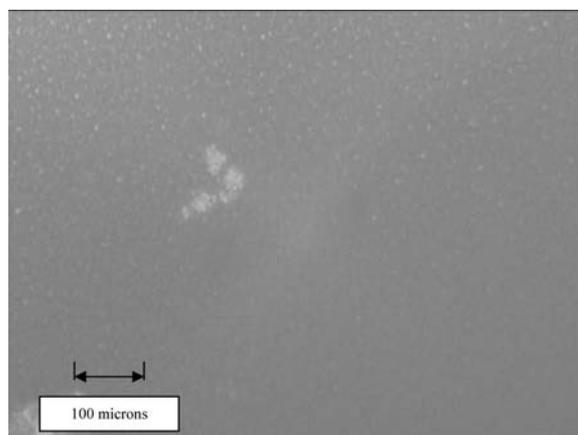


Figure 7. The optical microscopic picture of LP3 blend morphology.

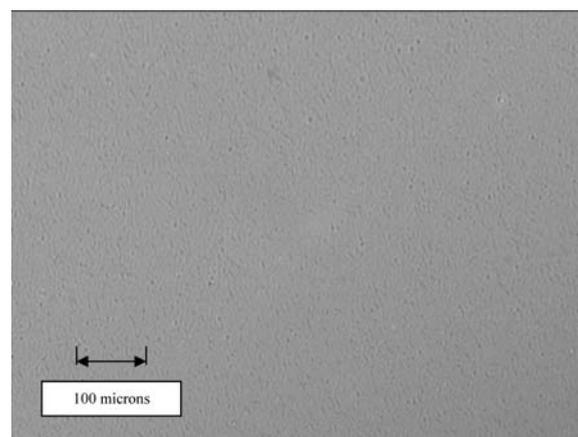


Figure 8. The optical microscopic picture of LP5 blend morphology.

Table 8. Effects of low molecular weight polyethylene (LMP) and styrene-butadiene rubber (SBR) wastes on properties of Tehran 60/70 bitumen.

Sample	LMP (%)	SBR (%)	Penetration (0.1 mm)	Softening point (°C)	Frass temperature (°C)	Performance grade (PG)
LP1	3	3	35	68	-10	84-16
LP2	5	3	33	77	-7	94-10
LP3	7	3	25	80	-5	100-10
LP4	10	3	25	85	-4	100-4
LP5	3	5	34	77	-9	94-16
LP6	5	5	30	78	-7	94-10
LP7	7	5	29	82	-7	100-10
LP8	10	5	25	88	-6	106-10

morphology of these blends is depicted in Figures 7 and 8. This is a very well developed morphology which is observed from these optical microscopic pictures. The existence of very fine particles witnesses the stability of polymers in bitumen medium. Some large rubber particles are also observed which can be avoided in an industrial scale by using very efficient colloid mills.

CONCLUSION

Polymeric wastes produced as by-products in polymer plants are annoying waste which should be recycled or reused safely. The polymeric and non-polymeric wastes (LMP, SBR and NMP) of HDPE and BD-SR plants of Bandar Imam Complex were used to modify bitumen. From the experimental results, it was concluded that NMP can act as an extender for bitumen, whereas LMP and SBR wastes are very good bitumen modifiers at moderate and high temperatures. The morphology of the binary and ternary blends of polymer modified blends is so that the polymer is finely dispersed in bitumen. In the case of LMP containing bituminous blends, the result lends out-perform many polymer-modified blends.

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