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RESEARCH ARTICLE

# Properties of Waste Polyethylene Terephthalate (PET) Modified Asphalt Mixes: Dependence on PET Size, PET Content, and Mixing Process

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***Abstract***

*Management and disposal of waste polyethylene terephthalate (PET) bottles is an ever-growing challenge. The present study investigated the effect of incorporation of shredded waste PET bottles on properties of asphalt mixes in terms of: (i) process of PET addition, (ii) PET content, and (iii) PET size. Experimental design included three variables: two processes (dry process, and modified dry process), three PET contents (2.5%, 5.0%, and 7.5% by weight of binder), and two PET sizes (2.36–1.18 mm, and 0.30–0.15 mm). Volumetric properties, Marshall parameters, and moisture susceptibility characteristics of PET modified mixes were evaluated and compared with control mix (without PET). Analysis of variance (ANOVA) was performed to evaluate main and interaction effects of the variables. Results indicated that all the three variables had significant influence on the measured properties. Further, mixes prepared using modified dry process outperformed other mixes and showed highest resistance towards moisture induced damage.*

***Keywords***

*polyethylene terephthalate, asphalt mix, plastic waste, moisture damage*

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## 1 Introduction

Population growth coupled with urbanization and rise in the standard of living have led to an explosion in the quantity of solid wastes generated globally. Plastics comprise 9% of a total of ~120,000 tonnes per day of municipal solid waste generated in India [1]. Being non-biodegradable, they persist in the environment for longer durations of time and cause issues related to their disposal. Out of various forms of plastics, polyethylene terephthalate (PET) is a widely used packaging material for soft drinks, bottled water, food items and other products. PET is a semi-crystalline thermoplastic polymer formed by polycondensation of terephthalic acid with ethylene glycol [2]. Most of the global demand for PET is for production of synthetic fibers and PET bottles. PET bottles have preceded traditional packaging and storage materials like glass and tin, due to numerous advantages such as chemical resistance, lightweight, easy production and storage.

Rapid expansion of PET bottle industry has led to a fast growth in global PET consumption [3]. During 2009–2013, PET bottle industry grew at an average annual rate of 4.3% [4]. Global PET bottle consumption is nearly 20 million tonnes and is rising at a staggering rate of 15% annually [5]. At the same time, recycling rate of PET bottles is low at just 29.3% [6, 7]. To overcome the pollution menace of plastics in general, and waste PET bottles in particular, channels for reutilization of waste PET are being explored where it could be possibly used in bulk quantities. One such route of PET waste reuse has been in highway industry where it can be used as an additive to bituminous mixes or as substitute for fine aggregates [8–10].

## 2 Research background

Two approaches can be followed to introduce a plastic into an asphalt mix: (i) wet process, and (ii) dry process. The wet process consists of blending the plastic into bitumen with a mixer followed by addition of 'plastic modified bitumen' to aggregates. On the other hand, the dry process involves adding the plastic to heated aggregates before blending with bitumen. The wet process can be conveniently used for plastics such as low density polyethylene (LDPE), high density polyethylene

(HDPE), and polypropylene (PP) having melting points generally below 160°C, which is close to asphalt mix production temperatures. However, wet process is not feasible for plastics like PET due to its high melting point of about 250°C making it extremely difficult to achieve a uniform blend and its tendency to segregate from bitumen [11, 12]. Consequently, use of dry process has been reported in studies on PET modified asphalt mixes. However, adhesion between aggregates and binder may be compromised in case of dry process as some portion of PET may melt when added to the heated aggregates and coat them [13].

Recently, incorporation of PET in asphalt mixes has also been tried through the use of a procedure similar to the dry process but with a slight recourse that addition of PET is done after aggregates are mixed with bitumen [8, 13, 14]. In absence of a formal terminology, this procedure of fabrication of PET modified mixes is termed 'modified dry process' in the present work. It is hypothesized that modified dry process results in minimum changes in shape and properties of PET during mixing [8].

There is a need to compare performance of asphalt mixes prepared with PET using dry and modified dry processes, in order to understand effectiveness of one process over the other. Another significant parameter influencing properties of PET modified asphalt mixes is the size of PET used. As PET obtained from shredding waste bottles is usually flaky in shape, its use to replace coarse aggregates in the asphalt mix is not encouraged. Considering its application as a fine aggregate, there has been no consensus on the size to be adopted. Researchers have used PET in various size ranges, e.g., 4.75–2.36 mm [9], passing 2.36 mm sieve [10, 13, 15, 16], 2.36–1.18 mm [17], and 1.18–0.425 mm [8, 12, 14]. Therefore, there is need to investigate properties of PET modified mixes with different PET sizes.

## 3 Research objectives

The main aim of this study is to evaluate the effect of process of PET addition, PET size, and PET content on properties of PET modified asphalt mixes. Volumetric properties, Marshall parameters, and moisture susceptibility characteristics of PET modified mixes are evaluated and also compared with control mix (without PET). Results are statistically analyzed through analysis of variance (ANOVA) to evaluate main and interaction effects of the three variables: process type, PET size, and PET content.

## 4 Materials description and methodology

Granite-rich aggregates were used in the study, and were obtained from a local stone crusher plant. Aggregate gradation selected for the study was bituminous concrete (BC), which is a dense-graded asphalt mix of 19.0 mm nominal maximum aggregate size (NMAS). BC is recommended for use in surface courses of flexible pavements in India. Fig. 1 shows aggregate particle size distribution of BC gradation along with the upper and lower specification limits as per Ministry of Road Transport and Highways (MoRTH [18]). Bitumen of viscosity grade VG-30 was used as binder for both control and PET modified mixes. The VG-30 bitumen was provided by Tiki Tar Industries (Gujarat, India). Tables 1 and 2 respectively present the characteristics of aggregates and bitumen used in the study.

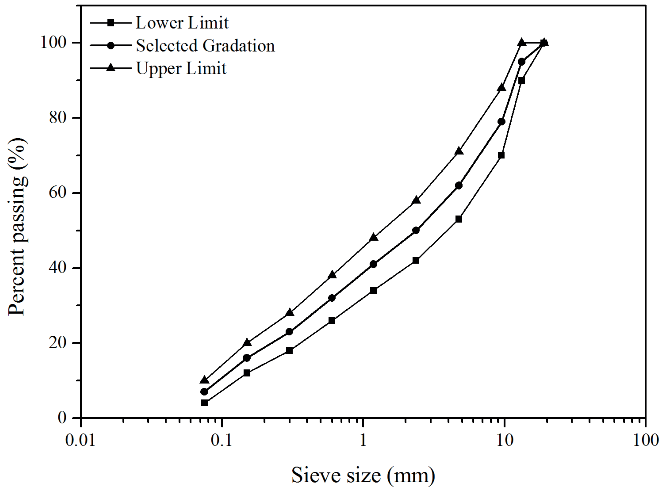


**Fig. 1** Aggregate gradation curves for BC mix with specification limits

PET was obtained from shredding waste plastic bottles. Shredded PET was in flaky form and was cleaned before use. Specific gravity of PET was found to be 1.297. Two main sizes of PET (Fig. 2) obtained from the Plastic Waste Management Centre (Guwahati, India) included: passing 2.36 mm sieve and retained on 1.18 mm sieve (designated as the 'coarse' PET size), and passing 300 µm sieve and retained on 150 µm sieve (designated as the 'fine' PET size). The percentages of added PET varied as 2.5%, 5.0% and 7.5% by weight of binder.


**Fig. 2** PET sizes used: (a) Size 1 (2.36–1.18 mm); (b) Size 2 (300–150 µm)

Marshall method of mix design, currently recommended by MoRTH [18] specifications of India, was used for design of control BC mixes. Mixing and compaction temperatures for VG-30 bituminous binder were determined in accordance to equiviscous method stated in Asphalt Institute Manual Series–2 (MS-2) [19]. Viscosity ranges of 170±20 cSt and 280±30 cSt have been established for determination of mixing and compaction temperatures, respectively. These temperatures were obtained as 155°C (for mixing) and 145°C (for compaction).

**Table 1** Properties of aggregates used in study

| Test | Requirement | Result |
|---|---|---|
| Cleanliness test, % | Max. 5 | 1.9 |
| Combined elongation & flakiness index, % | Max. 35 | 29.6 |
| Impact test,% | Max. 24 | 20.7 |
| Water absorption, % | Max. 2 | 0.47 |
| Stripping test,% | Min. retained coating 95 | 97 |

BC mix without PET was used as control mix in the study. To determine optimum binder content (OBC) of the control mix, three samples were fabricated at each binder content of 4.5%, 5.0%, 5.5%, and 6.0% by weight of mix. Compactive effort of 75 blows of Marshall impact compactor was used during the compaction. Bulk density and maximum specific gravity of the samples were determined in accordance to ASTM D2726 [20] and ASTM D2041 [21] respectively. Thereafter, the samples were tested for volumetrics and Marshall stability using digital Marshall testing machine. OBC was determined at 4% air voids and also checked for mix design criteria specified by MoRTH [18], and was found to be 5.2% by weight of mix. Mix design properties of control mix obtained at the OBC are presented in Table 3. In the present research, the same binder content (corresponding to OBC of control mix) was used for fabrication of PET modified mixes so as to facilitate comparison of properties of PET modified mixes with control mix without considering binder content as a separate variable.

**Table 2** Properties of VG-30 bitumen used in study

| Test | Requirement | Result |
|---|---|---|
| Absolute viscosity at 60°C, poise | Min. 2400 | 2889 |
| Kinematic viscosity at 135°C, cSt | Min. 350 | 490 |
| Penetration at 25°C, 0.1mm | 50–70 | 63 |
| Softening point (R&B), °C | Min. 47 | 50 |
| Solubility in trichloroethylene, % | Min. 99 | 99.8 |
| Flash point (Cleveland open cup), °C | Min. 220 | 280 |
| Tests on RTFO residue | | |
| Viscosity ratio at 60°C | Max. 4 | 2.8 |
| Ductility at 25°C, cm | Max. 40 | 67 |

In order to prepare PET modified mixes, two processes were used: (1) dry process, and (2) modified dry process. Under the dry process, PET was first mixed with heated aggregates before introducing the binder, whereas in the modified dry process, heated aggregates were first mixed and coated with asphalt binder and thereafter PET was added and mixed. PET particles with two sizes (2.36–1.18 mm, and 300–150 μm) and three contents (2.5%, 5.0%, 7.5% by weight of binder corresponding to OBC of control mix) were used for preparation of PET modified mixes. Three replicate samples were prepared for each combination of PET size and PET content and average results were reported.

**Table 3** Marshall mix design results of control BC mix at the OBC

| Property | Requirement | Result |
|---|---|---|
| Marshall stability at 60°C, kN | Min. 9 | 13 |
| Marshall flow, mm | 2–4 | 3.1 |
| Marshall quotient, kN/mm | 2–5 | 3.7 |
| Air voids, % | 3–5 | 4.0 |
| Voids in mineral aggregates, % | Min. 13 | 15.0 |
| Voids filled with bitumen, % | 65–75 | 73.5 |
| Bulk density, g/cc | - | 2.384 |

Moisture susceptibility of bituminous mixes indicates the vulnerability to damages caused by the effect of moisture. Moisture-induced distress is the most common distress observed on highways in India. Accumulation of moisture inside bituminous mixes may lead to several distresses such as stripping, cracking, raveling, and formation of potholes [8]. Tensile strength ratio (TSR) test, also called Modified Lottman test, is a commonly used test method for determining the moisture susceptibility tendency of bituminous mixes, and the same was used in this study. The test was conducted in accordance to the guidelines laid down in AASHTO T283 [22] using universal testing machine (UTM: 14 kN). To perform the test, six compacted specimens were first prepared at an average air void content 7±0.5%. One subset with three specimens (referred as conditioned subset) underwent moisture conditioning that included vacuum saturation followed by a freeze-thaw cycle. Indirect tensile strength (ITS) of both conditioned and unconditioned subset was then evaluated, and TSR was reported as the ratio of average ITS of conditioned specimens to that of unconditioned specimens (Eq. 1):

$$TSR = ITS_C / ITS_{UC} \quad (1)$$

where, $ITS_C$ = average $ITS$ for conditioned samples; $ITS_{UC}$ = average $ITS$ for unconditioned samples. Higher $TSR$ values evidently correspond to better resistance against moisture induced damages. Minimum acceptable limit of 80% $TSR$ is used in this study as per the MoRTH [18] specifications.

## 5 Statistical analysis

Results of the study were statistically analyzed by performing analysis of variance (ANOVA) to evaluate significance of main and interaction effects of the factors for each response variable. The study included three factors: process type, PET content, and PET size; and three response variables: Marshall stability, bulk density, and TSR. ANOVA was conducted using SPSS software at 5% level of significance. In cases where a significant interaction is observed, one may deduce that the effect of one factor on the response variable depends on the level of other factor [23].

## 6 Results and discussion

### 6.1 Results of mix volumetrics

Fig. 3 shows the results of bulk density of PET modified mixes at different PET contents and PET sizes. As can be seen from Fig. 3, there is reduction in bulk density of all PET modified mixes in comparison to the control (shown as a solid horizontal line); and with increase in PET content, the bulk density reduces further. As PET has much lower specific gravity than the aggregates, it will lower the bulk density on being added to the mix. Coarse PET particles generally yielded higher bulk density in comparison to finer ones at all PET percentages. This can be explained in terms of additional surface area to be coated with the binder. As the binder has to coat a larger surface area when finer PET size is used, it is likely to result in lower workability during mixing, and hence lower bulk density. The difference between bulk density of mixes produced with the two processes is less pronounced at PET contents of 2.5% and 5.0% while the difference is larger at higher (7.5%) PET content. In most cases, it is observed that modified dry process yields higher bulk density than dry process. Since the same total binder quantity is used for preparation of both control and PET modified mixes, PET will consume its own share of binder in order to get coated during mixing. This will render the mastic comparatively stiffer as compared to control mix, and hence addition of PET will lead to lesser bulk density with the same compactive effort and binder content. This aspect is likely to be less pronounced in case of modified dry process wherein asphalt coating over aggregates is already achieved before addition of PET. This explains comparatively higher bulk density obtained, when modified dry process is used.

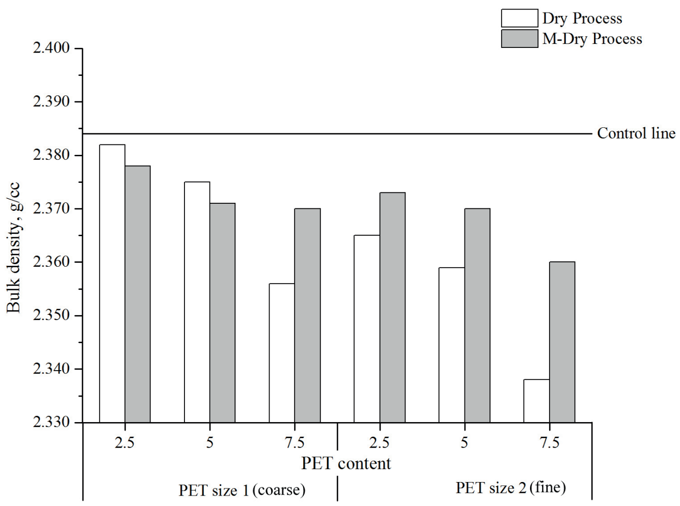


**Fig. 3** Results of bulk density

Table 4 shows the results of ANOVA performed on bulk density. Based on ANOVA results, main effects of all the three factors (process type, PET size, and PET content) are found to be statistically significant. From Table 4, all two-way interactions between the factors are also significant. In order to understand the results considering the effect of two factors at a time, plots with least-square means obtained during statistical analysis are plotted and shown in Fig. 4. From Fig. 4a, it is clear that bulk density values of mixes produced from modified dry process are higher than dry process at all PET percentages. Further, Figs. 4b and 4c indicate that use of coarser PET size yields higher bulk densities for both processes at all PET percentages.

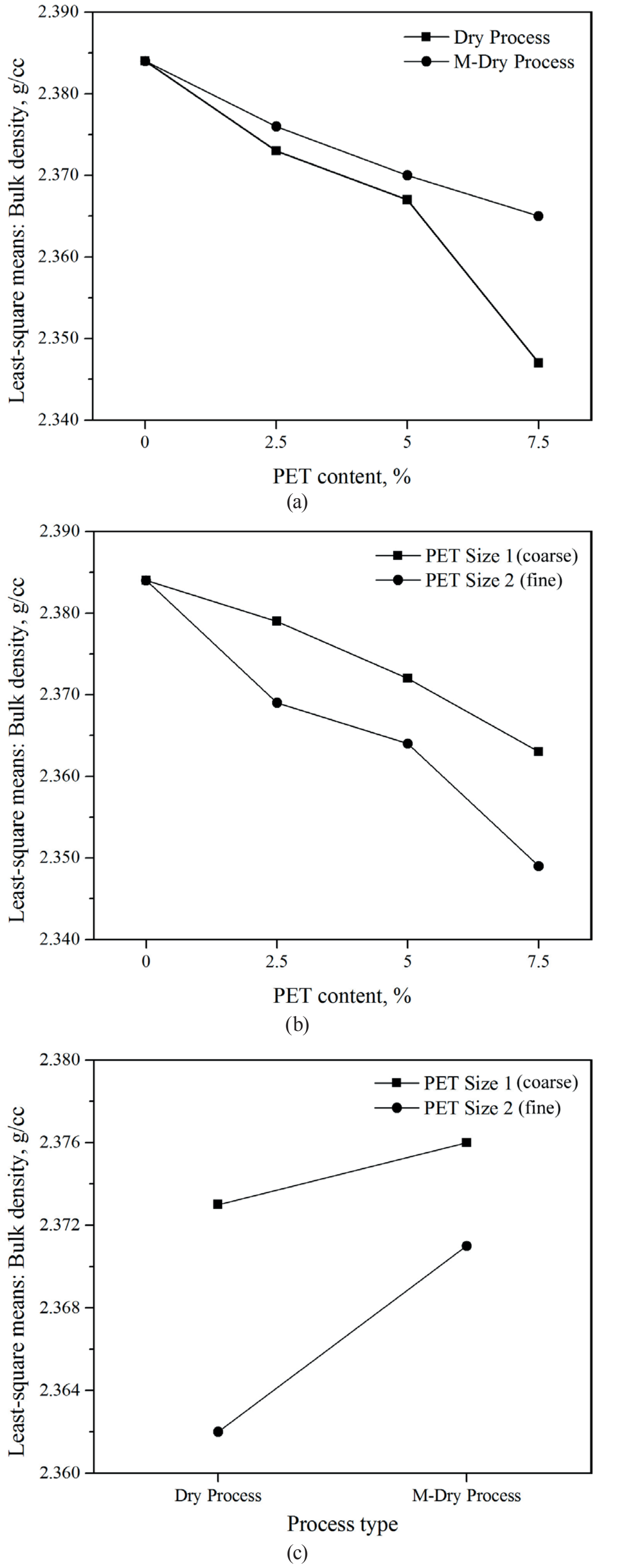


**Fig. 4** Least-square mean plots for bulk density: (a) PET content vs. process type, (b) PET content vs. PET size, and (c) Process type vs. PET size

**Table 4** Results of ANOVA

| Factor | Bulk density p-value, S/NS | Marshall stability p-value, S/NS | TSR p-value, S/NS |
|---|---|---|---|
| Process type | <0.001, S | <0.001, S | <0.001, S |
| PET content | <0.001, S | <0.001, S | <0.001, S |
| PET size | <0.001, S | <0.001, S | 0.038, S |
| Process type * PET content | <0.001, S | <0.001, S | <0.001, S |
| Process type * PET size | 0.011, S | 0.897, NS | 0.001, S |
| PET content * PET size | 0.008, S | 0.168, NS | 0.545, NS |
| Process type * PET content * PET size | 0.362, NS | 0.019, S | 0.042, S |

Note: 'S'—Significant difference; 'NS'—Non-significant difference

Air void content is an important volumetric parameter as it controls susceptibility of the mix to rutting, bleeding, cracking and ageing; apart from being a controlling parameter for selection of OBC for the mix. Results of air voids are presented in Fig. 5. Air void increases with the increase in PET content. In most cases, incorporation of PET using dry process results in higher air void content compared to modified dry process. This shows that addition of PET particles before addition of binder reduced the workability thereby causing an increase in air void content. Finer PET size is generally found to yield higher air void values, which corresponds to comparatively lower bulk density achieved with this size. However, all PET modified mixes meet the specified range of 3–5% air voids [18].

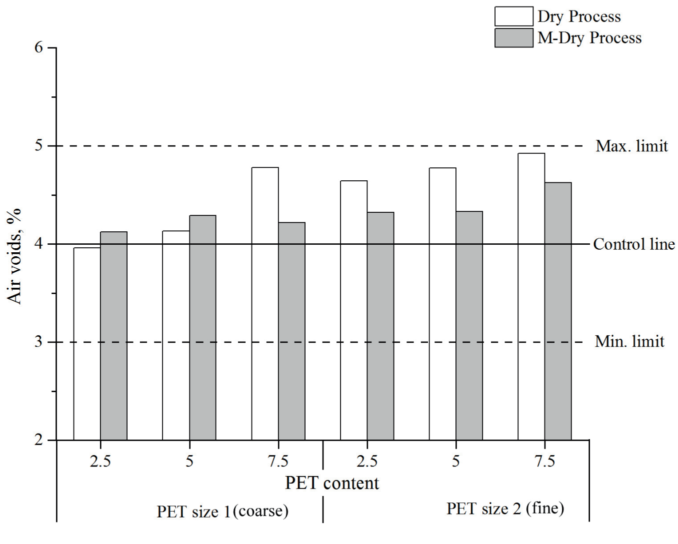


**Fig. 5** Results of air voids

Voids in mineral aggregate (VMA) is a volumetric parameter that ensures durability of asphalt mix by provision of adequate asphalt film thickness over aggregates. Minimum acceptable limits for VMA are generally specified by transportation agencies. Results of VMA are shown in Fig. 6. As can be seen from the figure, VMA increases with increase in PET content. VMA values for all PET modified mixes are higher than for the control mix, and meet the specified minimum limit of 13%. With the use of finer PET particles, a higher VMA content is observed at each PET content. VMA values are higher for mixes produced with dry process for finer PET size; however, a clear trend between the two processes is not discernible in the case of coarse PET. Results of voids filled with binder (VFB) are also presented in Fig. 7. As VFB is inversely related to the air voids, the trends are opposite to that observed for air voids in Fig. 5. Finer PET size is found to produce lower VFB values as compared to coarse size. All PET modified mixes are able to satisfy the VFB specifications of 65–75% as per MoRTH [18].

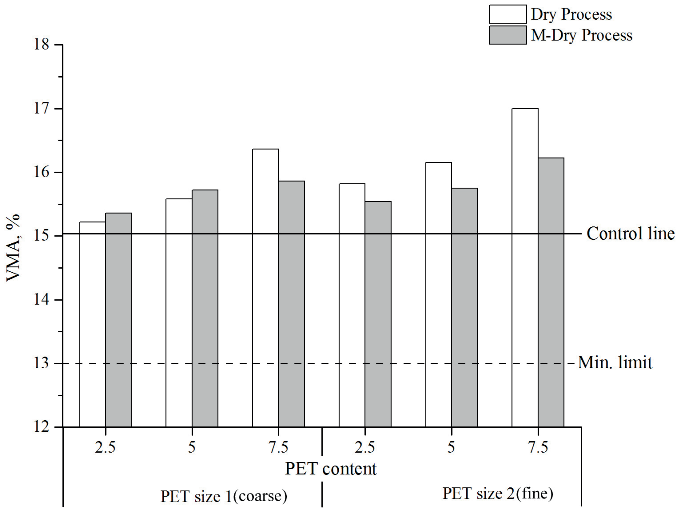


**Fig. 6** Results of VMA

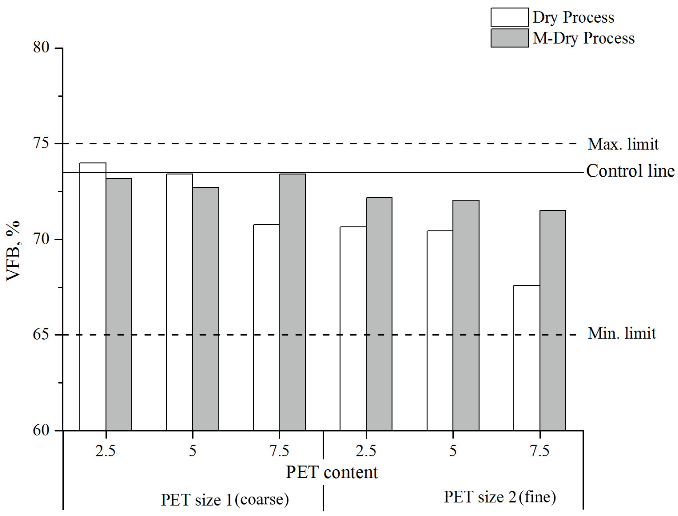


**Fig. 7** Results of VFB

## 6.2 Results of Marshall parameters

Fig. 8 presents the results of Marshall stability for control and PET modified mixes. For the mixes produced through modified dry process, it is seen that stability attains its peak value at 5% PET, and this trend is seen for both PET sizes. This shows that addition of PET through modified dry process increases the stability of mix up to a certain PET content. In case of modified dry process, addition of 5% PET caused 38%

and 30% increase in stability compared with the control mix, respectively, for coarse and fine PET sizes. However, peaked trend is not observed for mixes produced with dry process, and a steady decrease in stability is noted. Modified dry process produces stability higher/comparable to the control mix up to 7.5% PET contents for both PET sizes.

Results of ANOVA performed on stability values are presented in Table 4. Main effects of all three factors are found to be statistically significant. Least-square mean plots (Fig. 9a) show that stability values of mixes produced from modified dry process are higher than dry process at all PET percentages. Further, Fig. 9b and 9c indicate that use of a coarser PET size results in higher stability values for both processes at all PET contents. It is also seen that the effect of size remains consistent at all PET contents (Fig. 9b) and both process types (Fig. 9c), which corroborates the findings of non-significant two-way interactions between process type and PET size, and between PET content and PET size (Table 4).

Fig. 10 presents the results of Marshall flow of PET modified mixes. A clear trend with respect to PET percentage or PET size could not be delineated. For finer PET size, modified dry process produces marginally higher flow values than dry process. Nevertheless, all flow values meet the specified range of 2–4 mm.

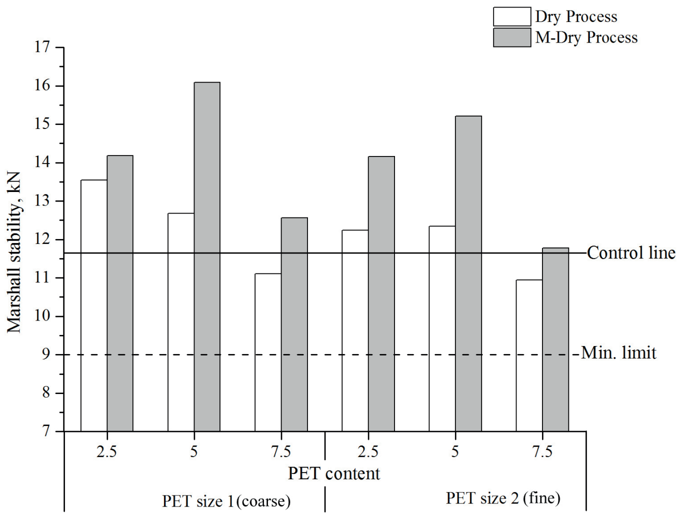


**Fig. 8** Results of Marshall stability

Marshall quotient (MQ) is the ratio of Marshall stability to flow. MQ is measured in the units of kN/mm. MQ is generally used as a simple measure to evaluate resistance against permanent deformation of a bituminous mix in-service, with higher values indicating a stiffer and more rut resistant mixture [24]. As shown in Fig. 11, there is increase in MQ in comparison to control mix up to 5% addition of PET for both processes and sizes. A peak is observed at 5% PET content as in case of stability values. At 7.5% PET content, the MQ values are marginally lower than that of control mix. It is also observed that MQ values of mixes produced through dry and modified dry processes are generally similar and no appreciable difference in terms of the production process is observed. Significant differences are also not observed in terms of the PET sizes. All PET modified mixes meet the acceptable MQ range of 2–5 kN/mm, except for the mix with 5% PET produced through modified dry process with coarse PET, in which case the value is slightly higher than the prescribed upper limit.

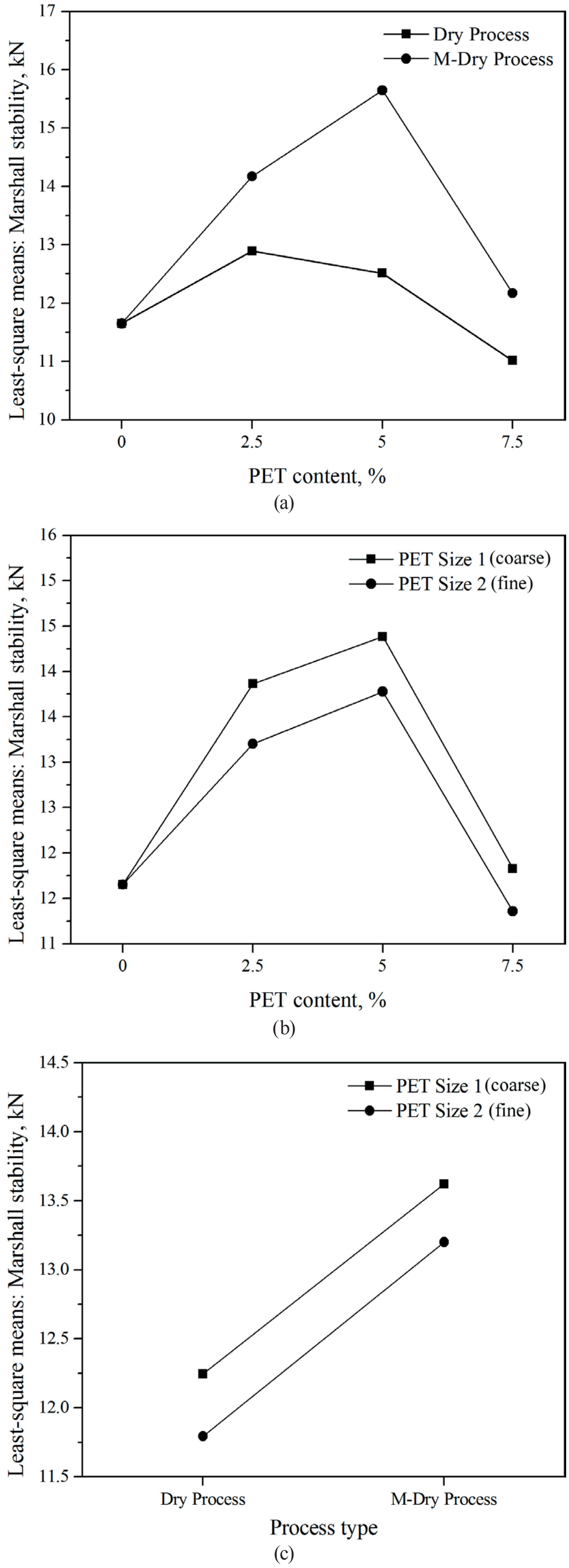


**Fig. 9** Least-square mean plots for Marshall stability: (a) PET content vs. process type, (b) PET content vs. PET size, and (c) Process type vs. PET size

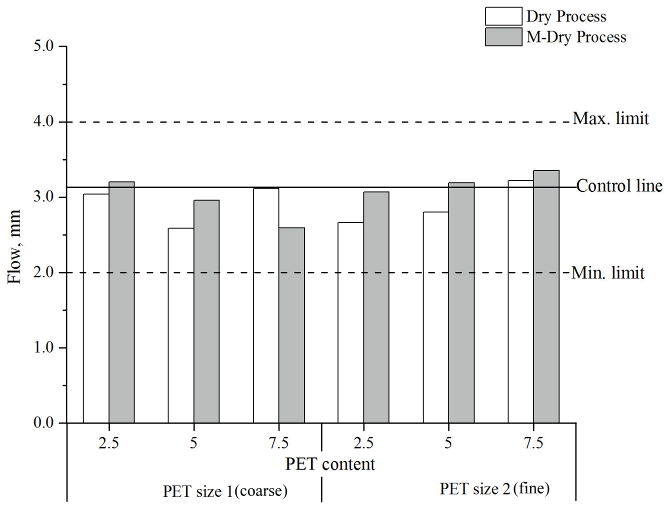


**Fig. 10** Results of Marshall flow

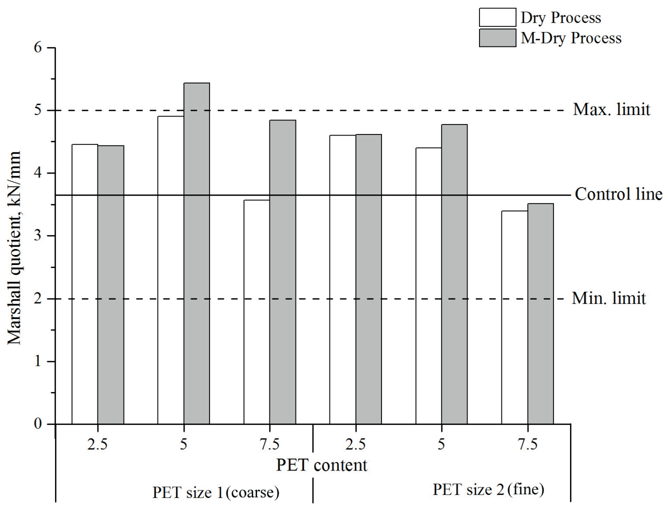


**Fig. 11** Results of Marshall quotient

Marshall characteristics (stability, flow and quotient), in most cases, indicate that PET modified mixes are stiffer than control mix as they exhibit comparatively higher MQ, higher stability, and lower flow. Increased stiffness of PET modified mixes can be explained through semi-crystalline property of PET [14,16]. Semi-crystalline nature implies that a portion of PET is amorphous while other portion is crystalline. Above its glass transition temperature of about 70°C, amorphous portion of PET exists in liquid form; however, crystalline portion of PET still exists as solid and rigid form as melting point of PET (about 250°C) is much higher than the mixing temperature (155°C) used in the study. The softened/molten portion likely improves aggregate-binder bond, and the rigid crystalline portion imparts stiffness to the mix.

### 6.3 Results of moisture susceptibility evaluation

Moisture susceptibility of the PET modified mixes was evaluated through TSR test which is the ratio of indirect tensile strength (ITS) of moisture conditioned samples to ITS of unconditioned samples. The average moisture conditioned ITS values of PET modified mixes are presented in Fig. 12. The results indicate that PET modified mixes show higher ITS values than control up to 5% PET for both PET sizes. At 5% PET content, mixes produced with modified dry process show about 20% higher ITS than those produced with dry process and control. This suggests that modified dry mixes are capable of resisting larger tensile stresses prior to cracking. Further, the results show that use of two different PET sizes does not lead to significant differences in the conditioned ITS values.

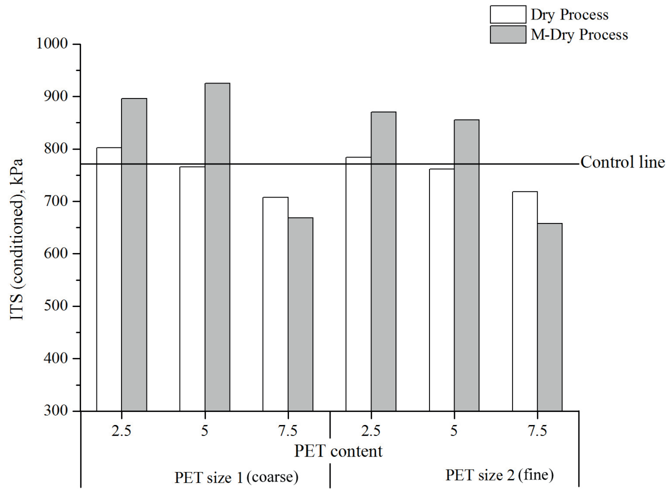


**Fig. 12** Results of conditioned ITS test

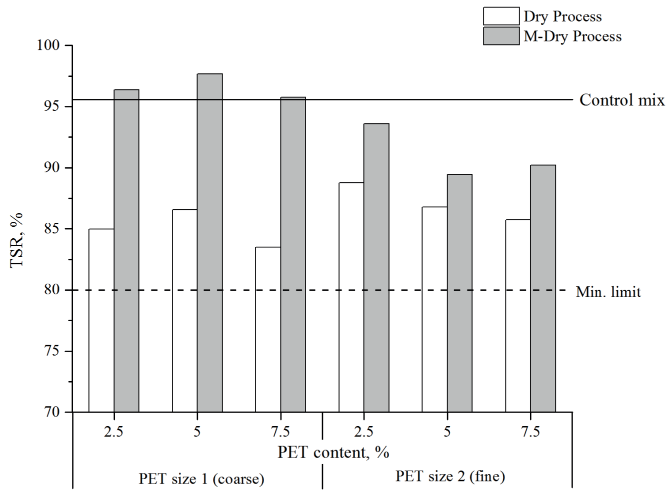


**Fig. 13** Results of TSR test

Fig. 13 shows TSR values of PET modified mixes with different PET contents, sizes and production processes. It is seen that mixes produced by modified dry process clearly outperform those with dry process in terms of resistance against moisture damage. When coarser PET size is used, the results are even better than the control mix. Nevertheless, all PET modified mixes satisfy the minimum requirement of 80% TSR.

ANOVA results for TSR (Table 4) indicate that main effects of all three factors (process type, PET content and PET size) are significant. This points out that modified dry process produces mixes with higher resistance against moisture induced distresses. In case of dry process, it is likely that binder coating around the aggregates would reduce as binder is poured over aggregates as well as PET during mix fabrication. However, such effect is not seen during production of mixes through modified dry process as PET is introduced in the mix after aggregates have already been coated with the binder. It is also interesting to see that dry process shows higher TSR values when finer PET particles are used, whereas higher TSR values are obtained with the use of coarse PET particles with modified dry process. This is corroborated from statistically significant two-way interaction between process type and PET size from ANOVA results for TSR shown in Table 4.

As shown in Fig. 14, least-square plots for TSR values indicate that TSR values of mixes produced from modified dry process are significantly higher than dry process at all PET percentages. Further, the significant two-way interaction between process type and PET size, noted from ANOVA results, can also be observed from Fig. 14c indicating how the effect of process type on TSR varies with different PET sizes. Fig. 14b indicates that use of coarse PET size yields comparatively higher TSR values than fine size.

## 7 Conclusions

The present study investigated the effect of mixing process, PET content, and PET size on performance of PET modified dense-graded asphalt mixes. Mix volumetrics, Marshall parameters, and moisture susceptibility characteristics of PET modified mixes were evaluated and compared with control mix. Based on results and analyses, the significant findings of the study are summarized as follows:

Modified dry process produced higher Marshall stability than dry process for all PET contents and sizes. Up to 5% PET content, the stability of PET modified mixes fabricated from both processes was significantly higher than the control mix.

In general, PET modified mixes showed higher resistance against deformation with higher stability, lower flow and higher Marshall quotient than the control mix.

Use of coarse PET size resulted in higher bulk density than fine size. This was also reflected from lower air voids, VMA and VFB for mixes with coarse PET.

Mixes produced with modified dry process showed better resistance to moisture damage with significantly higher TSR values than mixes fabricated using dry process. TSR values were higher for coarse PET size. Modified dry process also produced conditioned ITS values higher than dry process and control up to PET percentage of 5% for both PET sizes.

In general, all PET modified mixes complied with the requirements specified for volumetrics, Marshall criteria, and TSR irrespective of production process, PET content, and PET size.

Overall, PET modified mixes produced through modified dry process with coarser PET size (2.36–1.18 mm) showed comparatively superior performance considering volumetrics, Marshall parameters, and resistance against moisture induced damage.

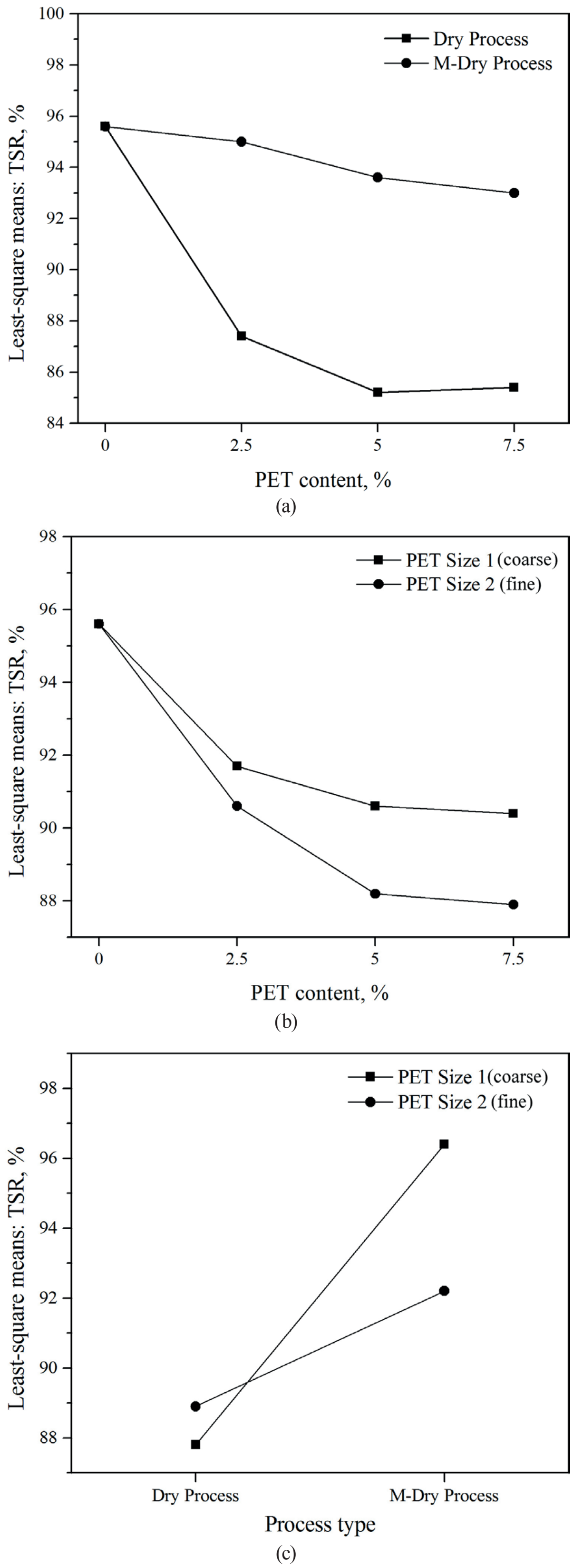


**Fig. 14** Least-square mean plots for TSR: (a) PET content vs. process type, (b) PET content vs. PET size, and (c) Process type vs. PET size

Based on the study, utilization of PET from waste bottles as additive in asphalt mixes is encouraged not only as a promising way to ease the environmental burden of its disposal but also as a technique to enhance performance of asphalt mixes.

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