Engineering properties of irradiated waste polyethylene terephthalate (WPET) modified asphaltic concrete mixtures using the modified dry method

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Abstract. The large quantity of WPET production is causing adverse effects on the environment. A common approach to reusing these wastes is through recycling as aggregates in asphalt concrete (AC) mixes. However, because of the use of regular WPET in AC blends, some discrepancies in the mechanical properties were recorded. One way of dealing with this is to utilize gamma radiation on the WPET. This study mainly concentrates on the irradiation, volumetric, and Marshall parameters. The regular and irradiated WPET utilized in this research were in granules shape retained on BS sieve size 3.35mm, which would substitute natural aggregates of equal size by volume at four different WPET percentages, 0, 1, 3, and 5% by total aggregate weight. This study's findings showed that aggregate substitution up to 5% enhanced most of the characteristics of the asphalt mixture considered when compared with the control mix. Based on the effect of irradiation on the WPET, it was noticed that mixes containing irradiated WPET showed a better output in terms of the engineering properties than those incorporating regular WPET.

1. Introduction
Because of the increasing world population that needs additional raw materials in the agricultural and manufacturing sectors, natural resources are exhausted, leading to the annual dumping of most waste materials into landfills. As such, research on construction materials has attempted to devise successful ways to reduce the detrimental effects on the environment of such waste. One alternative to using waste materials, such as waste plastics, is to substitute the conventional materials for road building purposes. This, aside from showing the proper management of solid waste, contributes to green and sustainable development by not further plundering natural resources [1].

Polyethylene Terephthalate (PET) is a thermoplastic polymer of the polyester family which is a strong, transparent, and lightweight plastic used in the production of water bottles, food packaging containers, and fibers in clothes [2, 3]. Since PET takes almost 300 years to degrade as well as the roads have a similar life expectancy, the usage of PET as an improvisation material in the asphaltic mixture can be a solution to be studied [4]. In Malaysia, reports show that only 16% of PET waste bottles are recycled whereas the remaining 84% go to landfills and leaked into the environment. This can be a good breakthrough as the introduction of PET waste bottles into asphalt mixture can enhance the mechanical properties of asphalt pavements besides, being a key to reduce the dumping of PET bottle waste [5]. To
address these problems, the recycling of PET materials has received serious attention. Nowadays, waste PET materials are used in various industries such as plastic, textile, fiber, construction, etc. [6-8].

The propensity to use recycled PET in construction products such as concrete and asphalt mixtures have also been increasing recently [9-12]. The motives for the use of waste materials in asphalt mixtures are to minimize the use of natural resources and fossil fuels (such as aggregates, binder) in addition to minimizing the amount of accumulated waste, to stabilize contaminants in hazardous waste, and also to increase the efficiency and durability of asphalt pavements as the most common pavement types used globally.[13-15]. There have been numerous researches on the use of PET materials in asphalt mixtures. As PET materials’ melting point is very high (about 250 °C), they might not be evenly distributed in the mixture if used as bitumen modifiers. PET materials are, therefore, also used as modifiers for blends or replacements for aggregates [8, 16].

The findings of numerous studies have shown that the use of PET in asphalt mixtures has the potential to improve durability [17] and performance characteristics concerning resistance to plastic deformation [1, 16], tensile strength [18], rigidity [19] so far the PET amount is optimally chosen. However, the shape and size of PET materials may affect these outcomes [16].

In addition, both positive and negative impacts on Marshall stability was found when PET particles were used as modifiers. This indicates that when a certain amount of PET was used, an improvement was observed in Marshall stability. There was, however, a decrease in Marshall stability following the use of PET particles [10, 17]. The semi-crystalline nature of PET, which is retained in the mixture, can explain the increase in Marshall stability. Meanwhile, the drop in the stability as the PET content increases simultaneously is due to lower PET particle stiffness and friction relative to natural aggregates [10].

The prospect of utilizing PET waste in asphalt mixes as an aggregate substitute, which is also termed plastiphalt, was evaluated by Hassani et al. [20]. The study concentrated on variables such as stability, flow, Marshall quotient, and density, and the outcome revealed that the stability and quotient of Marshall were almost the same as those of the control samples. This study may mean that PET can be used in mixes as an aggregate. The shredded waste plastic in stone matric asphalt (SMA) was used by Sarang et al. [21] as a stabilizing additive to regulate drain down by stiffening the SMA mixture. It is possible to further process rigid plastics such as PET into the finer dimension and substitute fine aggregates that move through a 4.75-mm sieve. Dalhat et al. [22] found that the dense-grade asphalt mixture incorporating finer sizes ranging from 2.38 mm (No. 8) to 2 mm (No. 10) exhibited improved output than that containing plastic sizes ranging from 0.42 mm (No. 40) to 2.38 mm (No. 8) based on the moisture sensitivity test to replace fine aggregate with waste plastic.

The use of ionizing radiation, such as gamma rays, is one alternative method for polymer recycling. It is well-known that ionizing radiation, such as gamma rays, contributes to polymer chains being cross-linked and broken. Often, it modifies the crystalline structure. As the degree of crystallinity increases, for irradiated ones, the polymer becomes stronger, stiffer, and harder than the regular one. Besides, the polymer’s chemical composition is used to decide whether cross-link and split form in the same fractions or whether one or more controllers [23].

In the case of polyethylene terephthalate (PET), in addition to low-energy photons, gamma rays emit electrons that control the modification of their structure. Molecular shifts are due to the mechanism of chain splitting, caused by free radicals at around 10kGy. Split chains can then be started again to create cross-links with contiguous molecules, which increase to 200kGy if no chemical degradation occurs [23]. Several studies were conducted utilizing irradiated waste PET as either additives or aggregates substitutes in asphalt mixes, and the outcome has been promising as the engineering properties of the resulting mixtures when compared with mixtures containing regular waste PET were enhanced [8, 12].

Therefore, in the current work, the effects of gamma-irradiation and WPET granules on the volumetric and strength characteristics of dense-graded AC blends were evaluated owing to limited information on the use of gamma-irradiation on WPET for the production of high-performance AC mixtures. To do that, various WPET particles of size 3.35mm were evaluated at different contents (0, 1, 3, and 5)% as well as exposing the regular WPET to gamma rays to improve its crystallinity degree, mechanical, and thermal properties, an advanced and effective approach of recycling in the pavement construction.
2. Materials and Methods

2.1. Materials
Waste PET (WPET) obtained from Enhanced Plastic Industry, Portland cement produced by YTL, asphalt binder supplied by PETRONAS refinery, and crushed granite aggregate collected from Sunway Quarry with AC14 gradation are the materials used in this research. For this analysis, the asphalt binder used was 60/70 penetration grade. The Portland cement and WPET used were used, respectively, as the filler and plastic aggregates.

| Properties             | Value | Standard  |
|------------------------|-------|-----------|
| Asphalt binder         |       |           |
| Penetration @ 25°C (dmm) | 62    | ASTM D5-13|
| Softening point (°C)   | 50    | ASTM D36-12|
| Specific gravity @ 25°C| 1.03  | ASTM D70  |
| Aggregates             |       |           |
| Specific gravity (FA\textsuperscript{a}) | 2.63  | ASTM C128 |
| Absorption (%) (FA)    | 1.23  | ASTM C128 |
| Specific gravity (CA\textsuperscript{b}) | 2.66  | ASTM C128 |
| Absorption (%) (CA)    | 0.50  | ASTM C128 |
| WPET                   |       |           |
| Density                | 1.35  | ASTM D792 |
| Melting point (°C)     | 250   | -         |

\textsuperscript{a} Fine aggregate  
\textsuperscript{b} Coarse aggregates

2.2. Methods

2.2.1. Gamma Irradiation. Obtained WPET granules from the Enhanced Plastic Industry, Ipoh, Perak, was used as the plastic aggregate in this investigation. The manual sorting process has been used to exclude metals and non-plastic impurities due to imperfections in recycling facilities. In a cobalt-60 irradiator that operates at 58Gy / min at the Malaysian Nuclear Research Agency office, the sorted WPET was then irradiated. In this study, 100kGy gamma radiation was selected based on some selected literature [8, 12, 24].

2.2.2. Sample Preparation. The wet and dry procedures are the two methods that are typically used to incorporate the chosen material into the asphalt mixture. In the wet method, the added material is combined with the asphalt binder prior to adding the asphalt binder to the mixture. The material to be added is mixed with the aggregate during the dry method before adding the asphalt binder [25]. In this study, the dry approach was used with an innovation that, the asphalt binder is thoroughly blended with the heated aggregate before incorporating the WPET and further blended until all aggregate particles and added WPET granules are fully coated with the asphalt binder. The motive behind this method was to ensure that the properties of the added WPET are maintained with minimum changes in its shapes and properties due to mixing temperature. Three different types of mixes were fabricated in this investigation, these are mixtures made with conventional materials (control), mixtures incorporating regular WPET (RWPET), and mixtures containing irradiated WPET (IWPET).

2.2.3. Marshall Mix Design. To obtain the optimum asphalt binder content (OAC) of the AC14 aggregate gradation, Marshall specimen were made and tested according to ASTM D1559. In the Marshall stability test, the specimens are conditioned in a water bath to a temperature of 60°C for 30 minutes after which the conditioned specimens are then placed in the Multiplex Marshall stability and
flow testing machine with a constant loading rate of strain 50.8mm/min until failure. The total maximum load (kN) that the specimen could withstand before failure is recorded as the specimen Marshall stability. The total amount of deformation that occurs at the maximum load is recorded as the Marshall flow value. The 3.35mm size waste PET granules of various amounts (1%, 3%, and 5% by weight of total aggregates mix) were incorporated into the asphalt mixture using the principle of volumetric substitution as a consequence of the significant difference in the density between the waste PET and the conventional aggregates. Before the mixing, the aggregates particles and the asphalt binder were first heated to a temperature of 110 °C and mixed manually at a temperature of 160 °C until all aggregates particles are well coated by the asphalt binder. Then, the required content of the waste PET (regular or irradiated) was added to the already blended aggregate and asphalt binder and further mixed for a few minutes to ensure all added particles are fully coated. The mixes were at a temperature of 135±5 °C compacted employing the Superpave gyratory compactor with 100 gyrations.

3. Results and Discussion

3.1. Optimum Asphalt Binder Content (OAC) evaluation

The optimum asphalt binder content (OAC) was determined by preparing three specimens for every asphalt binder content within the range of 4.0 - 6.0% as prescribed in JKR [26] for AC14 gradation with an increment of 0.5%. The value of OAC was determined through the determination of Marshall stability, Marshall flow, bulk specific density as well as void analysis comprising of the determination of the percentage of air void in the compacted mix (VIM), percentage of air voids in mineral aggregates (VMA) and percentage of air voids filled with asphalt (VFA). In this investigation, the control mixture OAC was utilized for the mixes containing waste PET granules, and the OAC was estimated as the average of the asphalt binder content corresponding to maximum density, maximum stability, and the median of the ranges specified in JKR for flow, VIM, and VFA. The OAC was determined for the control asphalt mixture to be 5.25%.

![Graphs](image.png)

**Figure 1.** Volumetric and Marshall properties for OAC estimation (a) Density (b) AV (c) Stability (d) Flow (e) VFA.
3.2. Characterization of the waste PET (regular and gamma-irradiated)

3.2.1. Fourier Transform Infrared Spectroscopy (FT-IR). The chemical structural analysis of the regular and IWPET sample was performed through the FT-IR spectroscopy. The spectrum of the regular and IWPET samples as shown in Figures 2 (a-b) exhibit the same transmittance percentages proving that irradiation does not affect the chemical bonds of WPET. A few intense bands representing significant functional groups are interpreted. The intense spectrum bands at wavenumber 1715.36 cm\(^{-1}\) mark the presence of stretching of C=O from the carboxylic acid group, at 1239.93 cm\(^{-1}\) represents the terephthalate group, 1089.68 cm\(^{-1}\) is due to the methylene group which forms the polyethylene and wavenumber 724.18 cm\(^{-1}\) corresponds to a benzene ring and interactions among the polar ester groups. Other minorly obvious spectrums are 3431.73 cm\(^{-1}\) (hydroxyl group), 2965.52 cm\(^{-1}\) (CH symmetrical stretch).

![Figure 2. FT-IR spectra (a) regular waste PET (b) irradiated waste PET](image)

3.2.2. Thermogravimetric Analysis (TGA). The TGA thermogram can be seen in Figure 3 (a-b), and as shown in the figure, the weight loss of regular and IWPET was approximately 15% at 348.4°C and 350°C, respectively. This behavior indicates that, for IWPET, the initial decomposition reactions commence at a temperature marginally greater than that of RWPET. Therefore, IWPET is more stable than RWPET in terms of thermal decomposition. This pattern can validate the production of crosslinking after exposure to gamma irradiation. The increase in starting temperature (\(T_{\text{onset}}\)) is attributable to the effect of crosslinking on the material that occurs after irradiation; thus, materials with higher starting temperature values have higher disintegration heat, whereas materials with lower starting temperatures appear to degrade more rapidly under the impact of heat [27, 28].

![Figure 3. TGA thermogram (a) regular waste PET (b) irradiated waste PET](image)
3.3. Density and Voids analysis

3.3.1. Density. As observed from Figure 4a, the bulk specific gravity of both the RWPET and IWPET modified asphalt mixtures decreases with increasing WPET content. The density of WPET is much lesser than that of aggregates, thus attributing to lower specific gravity for the modified mixtures. Therefore, the incorporation of WPET as aggregate replacement material reduces the mixture's overall specific gravity compared to the control mixture. Nevertheless, the comparison of bulk specific gravity between regular and IWPET modified asphalt mixes indicates that mixtures modified with IWPET possess higher bulk specific gravity than the mixtures containing RWPET. This is because the crystalline structure in IWPET is higher due to gamma rays, which increases the degree of crystallinity of the IWPET compared with RWPET, thus contributing to a slightly higher specific gravity value.

![Figure 4. (a) Variation of density against WPET (b) Variation of air void against WPET](image)

3.3.2. Air Void (AV). Figure 4b shows that the air voids of RWPET mixtures decreased for mixes containing 1% and 3% PET content before increasing at 5% content. The mixes containing IWPET air voids content shows a sharp decrease at 1% IWPET content before posing an increase in air voids for 3% and 5% content. Comparatively, the RWPET modified asphalt mixture has higher air voids than the IWPET asphalt mixtures. The two WPET types possess higher air voids at 5% PET content compared to the control mix. The higher air voids content at 5% WPET content for both regular and IWPET modified mixtures can be confirmed to its elastic deformation property, whereby under compaction effort, it can result in higher air voids value. It must be noted that the excessive air voids in the mixture will cause cracking due to inadequate asphalt binder to coat the aggregate, whereas a very low air void may cause more rutting problems and asphalt binder bleeding. Nevertheless, the air voids of all WPET modified asphaltic mixture are well within the specification range of 3% to 5%, which confirms the PET usage's suitability as aggregate replacement.

3.3.3. Voids in Mineral Aggregate (VMA). Figure 5a shows the plot of VMA values against waste PET content for both types of PET. VMA is the void space between the aggregates, which includes effective asphalt binder content. It can be inferred that the VMA values of RWPET modified asphalt mixtures show an initial decrease at 1% and 3% PET content before reaching the maximum VMA value at 5% PET content. Whereas for IWPET modified asphalt mixture, the VMA value drops to a minimum value of 14.211% at 1% IWPET content before showing a steady increase up to 5% content. An increase in PET content affects the mixture's air voids due to the elastic deformation property of PET, hence causing the asphalt binder to decrease, filling the air voids of the mix. Therefore, as the WPET content increases regardless of whether it is regular or irradiated causes the VMA of the WPET modified asphalt mixture increase.
Figure 5. (a) Variation of VMA against WPET (b) Variation of VFB against WPET

3.3.4. The Voids Filled with Bitumen (VFB). It can be observed from Figure 5b that the VFB values for both RWPET and IWPET modified asphalt concrete mixture poses an initial increase in VFB values at 1% WPET content before decreasing at 3% and 5% WPET content, respectively. VFB represents the percentage of voids filled with bitumen hence inversely related to the air voids content. Generally, the air voids show an increment when WPET is added due to its elastic property under Marshall compaction. Hence, when the air voids increase, the VFB decreases. The values of VFB nevertheless conform to the specification range of 70% to 80%, thus enabling the addition of WPET as aggregate replacement material.

3.4. Marshall Properties

3.4.1. Marshall Stability. Figure 6 (a-b) shows both regular and IWPET's influence as an aggregate replacement on the mixture's Marshall stability and Marshall flow values. From Figure 6a, it was observed that the RWPET modified asphalt mixtures indicate that with the increasing WPET content, the stability value shows an initial decreasing pattern reaching a minimum value. Then it showed constant increment until it reached the highest stability value. The highest stability value happens when the addition of RWPET content is 5%, raising the mix's stability by 7.8% compared with the control mix. For the IWPET modified asphalt mixtures, a similar trend was observed as that of RWPET mixes was obtained for mixtures containing 1% and 3% content of the IWPET. Further increase in the amount of the IWPET to 5% resulted in an enhanced Marshall stability value. The stability value of the IWPET modified asphalt mixture increased by 23.1% in comparison to the control mix. This behavior of increasing stability is attributed to the amorphous region of WPET melts to a certain extent, coating the aggregate, thus increasing the interlocking ability between aggregates. Therefore, this improves the stability of the mixture. At the same time, the crystalline region of PET serves as an elastic aggregate in the mix. Regarding the effect of exposing the waste PET to 100kGy radiation dosage, the degree of crystallinity increases, which makes the WPET more rigid and harder. The IWPET after irradiation is more stable thermally than the RWPET, as seen in the TGA thermograms, which probably increases the Marshall stability values.
3.4.2. Marshall Flow. As shown in Figure 6b, the flow values for RWPET modified asphalt mixtures decreases at lower content of the added WPET before experiencing an increasing trend with the increasing quantity of the WPET. Whereas the IWPET modified asphalt mixes, flow values showed the same behavior as that achieved for the RWPET modified asphalt mixtures but with lower flow values than the latter. The flow value is generally seen to increase in both regular and IWPET modified mixtures compared to the control mix. This behavior may be a consequence of WPET particles' low internal friction in the modified asphalt mixes [29]. The increase in flow values indicated that the mixes become more flexible under repetitive loading. Nevertheless, the flow values are well within the required specification range of 2mm to 4mm, as recommended in the JKR standard [26].

3.4.3. Marshall Quotient (MQ). The Marshall quotient is defined as the rigidity ratio, which is the ratio of stability to the flow values. From Figure 7, it can be observed that the Marshall quotient values differ with each type of WPET of different contents. It can be inferred that the Marshall quotient of the control mix is higher than that of the RWPET mixes. However, for the IWPET modified asphalt mixtures, the Marshall quotient shows a better rigidity value at 5% of IWPET content. Therefore, this 5% IWPET content mix proves adequate resistance against permanent deformations due to the higher stability and Marshall quotient compared to the control mix.
4. Conclusions
Based on the results analyzed in this investigation, the following conclusions were summarised:

- On the volumetric properties, the density and VFB of both WPET modified asphalt mixtures reduce with an increase in WPET contents, whereas for AV and VMA, an increase was noted with an increase in the amount of WPET for both modified mixes.
- Based on the strength characteristics, the WPET modified asphalt mixes possess higher Marshall stability values when compared with the control mix. However, the mixture incorporating 5% IWPET showed a 23.1% increase in Marshall stability, while for the mix containing RWPET, only a 7.8% increment was observed at a 3% WPET amount. However, flow values were increased for both modified mixes with an increase in WPET content. The resistance against permanent deformation (MQ) decreases for all the modified mixes at all WPET content, except for 5% IWPET content modified asphalt mix.
- On the effect of gamma radiation on WPET, this investigation showed that irradiation causes an enhancement in the WPET thermal stability; thus, it can be considered an alternative for plastic re-use in asphalt concrete mixtures with improved engineering properties. This will help in the conservation of natural resources, waste disposal, and reduce environmental contamination.

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