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Optimization of irradiated waste polyethylene terephthalate modified asphalt pavement using response surface methodology

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Abstract. The study focuses on the characterization of asphalt pavement mechanical properties containing two different sizes of irradiated plastic waste polyethylene terephthalate (IP) using gamma irradiation technique as fine aggregate substitutes in a 14mm asphaltic concrete wearing (ACW14). Response Surface Methodology (RSM) was employed in this study to determine the relationships between three independent factors (IP6, IP16, and bitumen content) on mix volumetric and Marshall Characteristics. To fabricate the samples, 0 to 1%, 1 to 2.5%, and 4 to 6% all by weight of aggregate particles were used as percentages for IP6, IP16, and bitumen contents, respectively. RSM statistical analysis demonstrates a high coefficient of correlation (R^2) of 0.9700, 0.9896, 0.9869, and 0.8946 for the responses bulk density (BSD), void in the mix (VIM), stability, and flow, respectively. The high correlation coefficient shows that the models developed are in reasonable agreement with the analyzed experimental outcomes. Investigation of the individual effect of the independent factors elucidates that interactions between the three factors influenced all the responses. In view of the outcomes accomplished 0.55%, 1.77%, and 4.63% were observed to be the optimized contents for IP6, IP16, and bitumen contents, respectively.

Keywords: asphalt concrete; gamma irradiation; mechanical properties; pavement; plastic waste; response surface methodology (RSM)

1. Introduction

Economic competitiveness and growth are among the main problems affecting almost all developing and developed nations over the previous two centuries. Though, financial competitiveness has resulted not just in an enormous rise in the manufacturing of products but also in the development of the most effective and safe transport equipment used to supply the products. As a consequence, companies are producing bigger cars and vessels with additional axle load as well as increased tire pressure to preserve financial competitiveness in the distribution of products across the worldwide market. Therefore, on the current roads, the amount of these large cars has risen significantly and substantially (Moghaddam *et al.* 2011).

The immense rise in the amount and frequency in traffic volume and axle loads has led considerably to both rapid deterioration and deformation of asphalt pavements earlier than expected (Moghaddam *et al.* 2011, Tortum *et al.* 2005, Tayfur *et al.* 2007). A prominent way of increasing asphalt mixture performance is to use additives like several kinds of fibers and polymers (Abtahi *et al.* 2010, Kalantar *et al.* 2012, Toghroli *et al.* 2018). Similarly, the utilization of plastic waste such as polyethylene terephthalate (PET) has been carried out in the cement and concrete industry to solve the issues relating to greenhouse carbon dioxide emission, waste disposal and in saving the natural ingredients of concrete such as aggregates and cement (Karanth *et al.* 2017, Paliwal and Maru 2017). In addition to that, the use of waste products as secondary materials to enhance the characteristics of asphalt mixture that will increase the service life of the pavement becomes the primary objective of engineers and experts.

Research conducted by (Kamada and Yamada 2002) explored the potential of using polyethylene (PE) and polypropylene (PP) as a portion of aggregate in asphalt blends. Their finding showed that using PE or PP contributes to a major enhancement in the porous asphalt mixes stiffness due to the reduction in the asphalt binder viscosity. They also demonstrate that adding polyethylene amounts to a substantial rise in anti-stripping characteristics and loss in fatigue cracking resistance of dense graded asphalt blends. Also, mixtures containing PE showed an improvement in the modified porous asphalt mixture oil-resistance characteristics.

The characteristics of asphalt concrete with varying amount of PET was investigated by (Modarres and Hamedi 2014a, b) and they discovered that adding 2% of PET by binder weight increases the blends rigidity and indirect

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tensile strength. Furthermore, the findings revealed that modification with PET could boost the fatigue characteristic of the mixture. In a study carried out by Moghaddam *et al.* (Moghaddam *et al.* 2012) it was discovered that the highest stiffness is attained at 1% PET amount in the asphalt blend by total aggregate weight, and then it recesses with an increase in PET amount. Another study found that PET-modified asphalt mixes had significantly higher permanent deformation properties than reference asphalt mixes as the permanent strain was considerably lower in PET-modified mixtures relative to the reference mixtures for all stress levels and temperatures (Moghaddam *et al.* 2014a). Recently, due to the large volume of waste PET generation globally being one of the main wrapping products, its usage in the pavement industry as sustainable alternative materials for green and cleaner pavements has been gaining interest. Various researchers have investigated the use of waste PET in asphalt blends for better performance and saving natural resources from depletion (Gürü *et al.* 2014, Modarres and Hamedi, 2014a, Moghaddam *et al.* 2015a). In these other, using waste PET as an alternative constituent material in road construction projects could reduce the use of natural resources and environmental pollution.

There is the need to analyze waste polyethylene terephthalate (WPET) modified asphalt mixes performance depending on different PET sizes to understand their effect in dense-graded asphalt blends. Mostly, WPET acquired from shredding waste bottles is typically flaky in shape; its utilization to substitute coarse aggregates in asphaltic concrete mixes is not encouraged. However, its application as a replacement for fine aggregates has been investigated with different size ranges. There is no agreement on the size of PET used so far, so several studies have reported the use of different PET sizes in asphalt mixtures, e.g., (Ahmadinia *et al.* 2012); (Modarres and Hamedi, 2014b); (Choudhary *et al.* 2018); (Arshadi, 2018, Taherkhani and Arshadi, 2018, Taherkhani and Arshadi, 2019); (Hassani *et al.* 2005); (Moghaddam *et al.* 2012, Moghaddam *et al.* 2014a). One critical parameter affecting PET modified asphalt mixtures properties is the size of PET utilized.

The literature review conducted reveals that mixed results were reported from research studies conducted on the effect of utilizing PET in conventional asphalt mixtures. Studies were carried out in concrete with the utilization of gamma irradiation on the WPET and the outcomes were promising as the strength lost while using regular, non-irradiated WPET were recovered (Schaefer *et al.* 2018). Additionally, in the study, they used two doses of gamma irradiation on the PET which were low dose and high dose of 50kGy (kilo gray) and 100kGy (kilo gray) respectively, and revealed that the high dose is optimum with better recovery of the lost strengths. However, the effect of exposing WPET to gamma irradiation doses to be used as aggregate replacement in asphalt mixtures has not yet been carried out. Hence, it becomes paramount to study the effect of gamma irradiation on the WPET to be used as a substitute for fine aggregates in AC mixes.

Response surface methodology (RSM) is a statistical technique strategy where the yield result is associated with autonomous factors in order to investigate the effects, association, and association between dependent factors (responses) and autonomous elements (Montgomery, 2017, Zhu and Gu, 2016). To fully describe the interactions among two or more independent variables that influence a particular response, the use of RSM statistical analysis was found reasonable. The utilization of statistical techniques in the investigation of pavement performance gives more understanding of the parameters that decide the performance of pavement during service life (Moghaddam *et al.* 2015a). RSM is regularly acknowledged and is a significant statistical apparatus utilized for the design of experiments, modelling, and optimization through assessment of both individual impacts and interaction impacts of various factors (independent variables) under lesser trial runs (Moghaddam *et al.* 2015a, Yadav *et al.* 2014).

To have a clear understanding of all the possible interactions among various design factors, a factorial design of experiments (DOE) gives a reasonable model that relates different variables and responses. DOE permits the evaluation of the concurrent impact of individual components at various levels (Khuri and Cornell, 1996, Myers *et al.* 2016). (Moghaddam *et al.* 2015b) utilize RSM to optimize SMA mixture bitumen and PET amounts. The finding shows that all the models developed were fitted successfully to the experimental data. They also discovered the optimum amounts of 5.88% bitumen and 0.18% PET (by weight of aggregate particles) that meets the design criteria of Marshall Mix. In another study, RSM was used to optimized Nano silica and bitumen contents for HMA mixtures (Bala *et al.* 2018)

In this research, the main aim is to optimize the amounts of irradiated WPET based on size and bitumen, and to also evaluate the effect of WPET size, WPET content and gamma irradiation on the properties of irradiated WPET modified asphalt blends. Mix volumetric and Marshall Parameters of irradiated WPET modified mixes are evaluated and results were statistically analyzed using RSM to determine interactive effects of the three independent variables.

2. Materials and methods

2.1 Materials

The materials utilized in this investigation are crushed granite aggregate with ACW14 gradation as per Jabatan Kerja Raya *(*JKR) (Malaysia, 2008), bitumen, Portland cement, and waste PET. The aggregate gradation selected is shown in Table 1 and the bitumen utilized for this investigation was 60/70 penetration grade. Table 2 depicts the physicochemical properties of the utilized materials. Portland cement and waste PET are used as filler and fine aggregates, respectively.

Waste PET flakes were obtained from enhanced Plastic Industry, Ipoh-Perak. Malaysia. Previous studies indicate that single-size PET particles in the range of 0.425–1.18mm result in the desired performance of stone matrix asphalt (SMA) (Ahmadinia *et al.* 2012). However, in this research,

Table 1 Aggregate gradation

Sieve size (mm)	Gradation limit (%)	Used gradation (%)
20	100	100
14	90-100	95
10	76-86	82
5	50-62	58
3.35	40-54	44
1.18	18-34	28
0.425	12-24	18
0.15	6-14	10
0.075	4-8	5

Table 2 Used materials properties

Property	Unit	Method	Result	Limits
Coarse aggregate				
L. A. Abrasion	%	ASTM C131	22.5	<30
Flakiness index	%	BS 812 Part 105.1	7.4	<20
Elongation index	%	BS 812 Part 105.2	13.23	<20
Aggregate crushing value	%	BS 812 Part 3	19.81	<30
Water absorption	%	ASTM: C127	0.5	<2
Specific gravity	g/cm^3	ASTM: C127	2.66	-
Fine aggregate				
Water absorption	%	ASTM C128	1.23	<2
Specific gravity	g/cm^3	ASTM C128	2.63	-
Bitumen				
Penetration at 25ºC	0.1mm	ASTM D5	64	60-70
Softening point	ºC	ASTM D36	51	49-56
Ductility (25ºC)	Cm	ASTM D113	>100	Min. 100
Waste polyethylene terephthalate				
Density	g/cm3	ASTM D792	1.35	-
Absorption	%	ASTM D570	0.11	-
Melting point	ºC	-	250	-
Glass transition temperature	ºC	-	75	-

in order to check the effects of WPET size and content, WPET particles were sieved. Those passing sieve No. 6 and remaining on sieve No. 16 were used as 1.18mmWPET particles and referred to as RP16 and IP16 for regular and irradiated respectively, as well as those passing sieve No. 4 and remaining on sieve No. 6, were used as 3.35mmWPET particles and referred to as RP6 and IP6 for this study, respectively, for regular and irradiated WPET. Table 2 shows the WPET properties.

2.2 Methods

2.2.1 Gamma Irradiation of WPET

Gamma Irradiation: Gamma rays are generated when radioactive atomic nuclei are disintegrated and when certain subatomic particles are decayed. In the electromagnetic spectrum, the commonly accepted definition of the region of gamma rays includes some wavelength overlap. Gamma radiation has a wavelength that is considerably shorter than a few tenths of an angstrom and gamma-ray photons with more than tens of thousands of electron volts (Bagher, 2014).

The adjustments in the surface's chemical structure are one of the benefits of using gamma radiation for PET recycling and improving the mechanical properties of concrete. Among the advantages of gamma radiation is that it’s accelerated initiation of a monomer’s polymerization

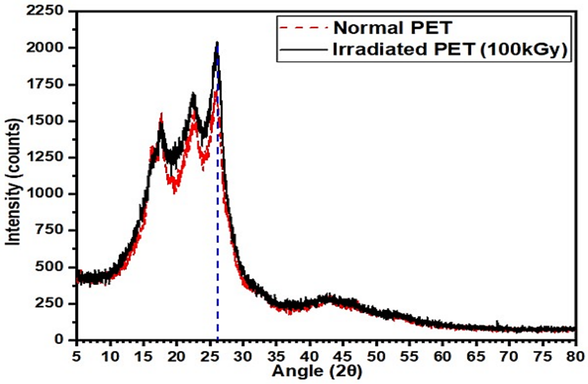
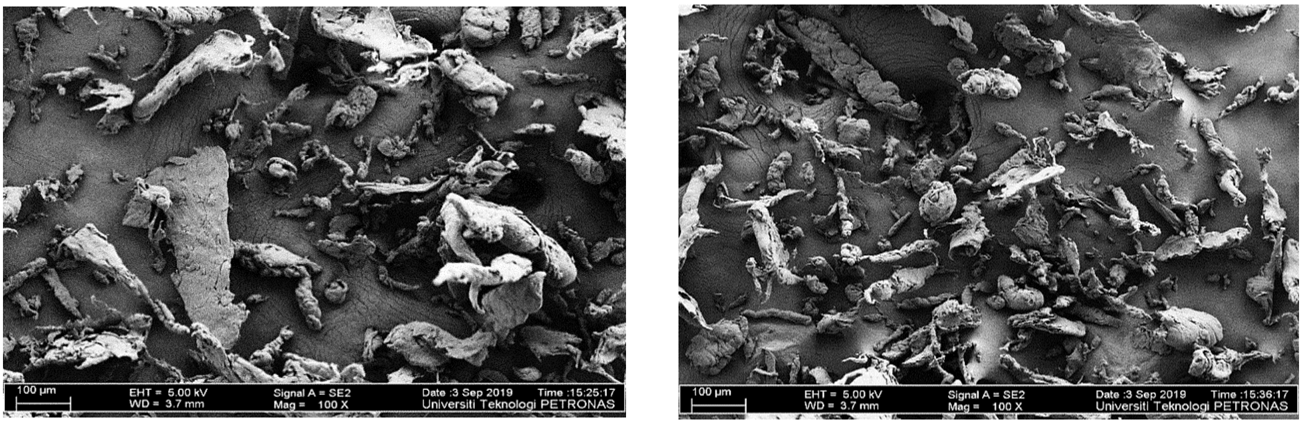


Fig. 1 X-ray diffraction pattern of non-irradiated (regular) and gamma irradiated WPET



(a) Regular WPET (b) Irradiated WPET

Fig. 2 FESEM micrographs of non-irradiated (regular) and irradiated WPET

into the ceramic network and can provide significant advantages, the most imperative advantage of gamma radiation is better adhesion between the fibers and the matrix (Patel *et al.* 2006). PET when irradiated has a higher modulus, toughness, stiffness, strength, and hardness with higher crystallinity. Then again, cross-linking can be defined as the chemical bonding between polymer chains. Radiation can induce cross-linking and has the effect of reinforcing the compound's chemical structure. Both chains splitting and cross-linking in PET can lead to greater strength along these lines (Plester, 1973).

PET flakes acquired from Enhanced Plastic Industry, Ipoh, Perak. Malaysia was utilized as the plastic aggregate in this study. Because of imperfections in recycling facilities, a manual sorting process was employed to remove metals and non-plastic impurities. The sorted PET flakes were then irradiated in a cobalt-60 irradiator that works at 58Gy/min at the Malaysian Nuclear Research Agency office. A radiation dose of 100kGy was utilized in this study.

2.2.2 Characterization of non-irradiated and irradiated WPET

Powdered X-ray diffraction (XRD) (Bruker D8 Advance X-ray diffractometer) and field emission scanning electron microscopy (FESEM) (Zeiss Supra 55 VP instrument) were utilized to characterize the grounded regular and irradiated WPET for the determination of the degree of crystallinity with graphite monochrome Kα radiation source and surface morphology, respectively.

Fig. 1 displays patterns of X-ray diffraction of both regular, non-irradiated WPET particles. Such specimens of WPET contain both amorphous and crystalline regions. The characteristics of the WPET particles are a prominent intensity peak for regular, non-irradiated, and irradiated WPET at 2θ=26.50. It is interesting to note that the rise in diffraction peaks intensity for irradiated WPET expose to 100kGy radiation dosage shows an improvement in the plastic crystallinity, but for regular, non-irradiated WPET, the decrease in diffraction peak intensity when compared with the irradiated plastic implies the disorderness of the former as opposed the later (Kumar *et al.* 2012, Mishra, 2016). This improvement in the intensity of diffraction peaks may be due to the breakdown of regular, non-irradiated WPET particles' original structure. The overall increase in the diffraction peak intensity shows a convergence of plastic particles after exposure to gamma radiation dose towards a more ordered arrangement. The degree of crystallinity of the regular, non-irradiated, and irradiated WPET were evaluated and found to be 41.15% and 46.07% respectively; likewise, amorphous contents were found to be 58.85% and 53.93% for regular, non-irradiated, and irradiated WPET. However, for the surface morphology FESEM was used, the results as depicted in the micrograph in Figs. 2(a)-2(b) reveals that WPET possessing finer particles when irradiated than the regular, non-

Table 3 Strength and Volumetric properties of regular and irradiated waste PET

Mixture type	BSD (g/cm^3)	VIM (%)	VMA (%)	VFB (%)	Stability (kN)	Flow (mm)
Control	2.3926	3.53	14.81	76.14	14.76	3.08
RP16(1.0)	2.3811	3.55	14.80	76.01	16.71	3.74
RP16(2.5)	2.3357	4.72	15.81	70.15	16.34	4.05
RP16(5.0)	2.2885	5.54	16.48	66.38	15.66	4.18
RP16(7.5)	2.2439	6.27	17.08	63.29	15.13	4.37
IP16(1.0)	2.3804	3.58	14.83	75.86	17.71	3.88
IP16(2.5)	2.3352	4.74	15.83	70.06	17.97	3.93
IP16(5.0)	2.2791	5.93	18.83	64.77	16.83	4.20
IP16(7.5)	2.2372	6.55	17.33	62.20	16.61	4.39
RP6(1.0)	2.3820	3.51	14.77	76.24	16.53	3.58
RP6(2.5)	2.3365	4.69	15.78	70.28	15.14	4.01
RP6(5.0)	2.2951	5.27	16.24	67.55	15.02	4.23
RP6(7.5)	2.2575	5.70	16.58	65.62	14.60	4.33
IP6(1.0)	2.3814	3.54	14.79	76.06	17.42	3.74
IP6(2.5)	2.3361	4.70	15.80	70.25	17.07	3.98
IP6(5.0)	2.2947	5.28	16.26	67.53	16.77	4.27
IP6(7.5)	2.2545	5.83	16.69	65.07	16.33	4.36

RP-regular PET; IP-irradiated WPET; RP16(1.0)-1.18mm size regular plastic retained on sieve number 16; the numbers in the parenthesis means weight of PET by total weight of aggregate particles

irradiated WPET. Such finer particles improve the accumulated surface area, which in turn enhances the crystalline structure in the irradiated WPET (Mishra, 2016). Furthermore, both chain scission and cross-linking occur simultaneously in the WPET after exposure to gamma irradiation, which resulted in increased strength.

2.2.3 Sample preparation

The mix design layout utilized to prepare asphalt blend specimens was based on the conventional Marshall Mix design procedure with 75blows giving to both sides of the cylindrical Marshall specimen. For the fabrication of each sample, about 1200g of aggregates and filler were weighed and heated in an oven to a temperature of 160ºC. Likewise, the bitumen was heated in an oven to a temperature of 160ºC which is the mixing temperature. The dry process was used to add the WPET into the mix but in this study, the WPET was introduced at the last step of the mixing. Firstly, the heated bitumen of various amounts (4%, 5%, and 6%) and aggregates were mixed for about 5minutes, and then the WPET was added and mixed for almost 2minutes. The primary reason behind this technique was just to preserve the WPET in its natural form with negligible modifications in its shape and features. The loose blends were then put in the preheated mold and compacted at 140ºC. In accordance with ASTM D1559, Stability and volumetric parameters were achieved. To determine the optimum bitumen content (OBC) in hot mix asphalt blends, it requires the average value of bitumen amount that offers maximum stability, maximum bulk specific density and particular air voids amount in this case 4% (Sengoz and Topal 2005). In this research, the optimal contents of irradiated WPET based on size and bitumen were measured via the RSM method of analysis.

2.2.4 Mix strength and volumetric

Using the conventional Marshall Mix design approach, the stability and volumetric properties are assessed. Marshall stability is the primary strength property based on the standard requirement while volumetric properties of compacted bituminous asphalt mixes comprise voids in the mix (VIM), voids in mineral aggregates (VMA) and the voids are calculated based on the bulk specific gravity of the compacted mix in accordance with ASTM D2726. Optimum bitumen content (OBC) of the control mix of 4.8 % was used to prepare samples containing WPET having various concentrations (1%, 2.5%, 5.0%, and 7.5%) by total mix in order to determine the optimum contents of the regular and irradiated WPET particles that will offer maximum stability and satisfies the Marshall mix design requirements. Based on the obtained stability values of 17.97kN and 17.42kN respectively for 2.5% IP16 and 1% IP6 contents, the optimum contents of the IP16 and IP6 were chosen and used for the RSM design of the experiment. The replacement levels of the irradiated WPET were also selected based on the outcomes reported by some studies of 1% by total aggregate weight (Moghaddam *et al.* 2012, Moghaddam *et al.* 2014a, Moghaddam *et al.* 2015b). Table 3 summarizes the strength and volumetric properties of four different blend types depending on WPET size, regular and gamma-irradiated WPET.

2.2.5 Method of analysis

One factor at a time (OFAT) methodology is a conventional approach to optimize multifactor experiments.

OFAT includes a changeable single factor for a specific experimental design while other factors are kept constant. OFAT is unable to provide appropriate output because the effect of interactions amongst all involved factors in the designs is not examined truly, and it is not capable of reaching the true optimum value (Frigon and Mathews, 1997, Montgomery, 2005, Mohammed *et al.* 2019). Hence, RSM methodology was introduced for parameter optimization in such a way that the number of experiments and interactions among the parameters are reduced to a minimum (Myers and Montgomery, 2002, Mojiri *et al.* 2017). In this study, to investigate the combined effect of IP6, IP16, and bitumen contents on mix volumetric and Marshall Characteristics, RSM was utilized. Numerical optimization was performed to explore the most appropriate amounts of the three independent factors by magnifying the stability and diminishing the bulk-specific density. Design expert software has been used for experimental design. Based on the Box-Behnken Design (BBD) method for three independent variables, mix design formulations for the variables were randomly selected (Khosroyar and Arastehnodeh, 2018). IP6 ranges from 0% to 1%, IP16 ranges from 1% to 2.5%, and bitumen content ranging from 4% to 6% all by weight of aggregates particles were the parameters considered in the design. These ranges were selected based on preliminary studies and related works of literature (Moghaddam *et al.* 2015b, Bala *et al.* 2018, Ahmadinia *et al.* 2011, Moghaddam *et al.* 2012, Moghaddam *et al.* 2014a, Moghaddam *et al.* 2014b). The responses of this study were: bulk specific density (BSD), void in the mix (VIM), stability, and flow. The RSM software version 11.1.0.1 developed seventeen mixes for the responses with five randomized replications at the center point per block. The five replications are the central points used by the software to improve the experiment's accuracy against any likely errors. Table 4 depicts the three independent variables with their ranges and corresponding responses. Using Eq. (1) numerical variables for the experiments are converted to coded form.

$$x_i = \frac{(X_i - X_0)}{\Delta X} \tag{1}$$

2.2.6 Response surface methodology (RSM)

Box and Wilson (1951) introduced RSM in 1951 and they proposed using a second-degree polynomial model. RSM has recently been used for process parameter optimization. RSM can be considered as a systematic calculation technique for the optimization problem. This method provides an appropriate experimental method that incorporates all the independent variables and utilizes the experiment's input data to subsequently create a set of equations that can offer an output's theoretical value (Said and Amin, 2015). The findings are achieved from a well-designed regression analysis that examines the relationship between independent variables'-controlled values. Based on the new values of independent variables, the dependent variable can then be forecast (Said and Amin, 2015). RSM is an effective statistical tool for both the modeling and optimization of multiple variables with a minimum number of experiments to forecast the optimum performance parameters (Baş and Boyacı, 2007, Bezerra, *et al.* 2008). By employing the RSM technique in the optimization process, the testing of all the variables relating to the product assessment requires just a short time, making the laboratory test stage more efficient (Said and Amin 2015).

Furthermore, the estimation of parameters that profoundly influence the model can determine which allows researchers to concentrate on those specific variables to improve the performance of the process (Said and Amin, 2015). In a set of experimental designs, one factor or process variable can depend strongly on or be dependent on another variable. In an attempt to discover the output-input relationship, understanding the interaction between the variables is critical, that is why taking a single factor at a time approach is seldom used to evaluate interrelationships between parameters. RSM can determine the relationship as well as interactions between the multiple parameters using quantitative data by creating a model equation. In RSM implementation, there are three steps; (i) experiment design, i.e., Box Behnken (BBD) and Central Composite Design (CCD); (ii) statistical and regression analysis to build model equations that describe the modeling of the response surface; and (iii) optimization of parameters/variables carried out via model Equation (Steppan, 1998).

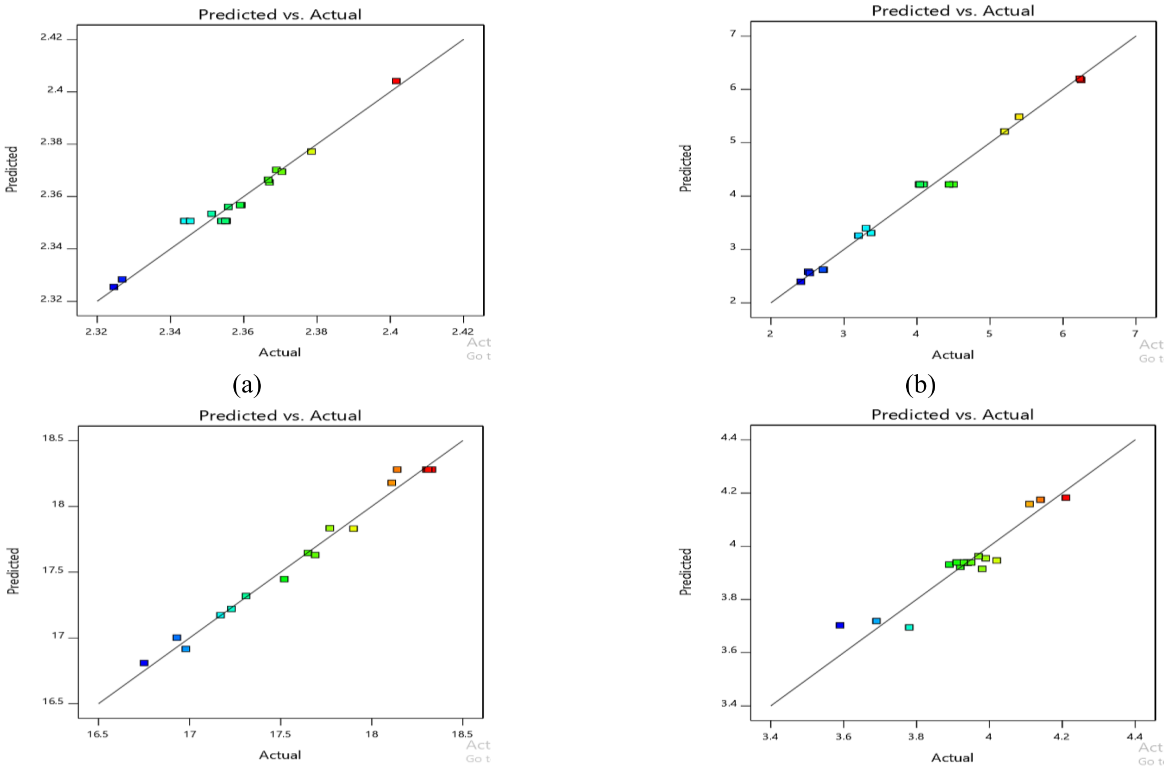
3. Results and discussions

In this research, response surface methodology (RSM) was used to establish models for irradiated WPET modified asphalt blends and to determine the relationships among independent components and dependent variables. Table 4 presents the range of actual values of the experimental design and the output for each response. Regression analysis was then employed to the responses and fitted quadratic equations were established for all the outputs with the exception of flow for which a linear equation was produced. The models produced for the responses BSD, VIM, stability, and flow were chosen by the RSM design software based on the highest order polynomial where the additional terms were significant and not aliased.

Table 5 provides an overview of the analysis of variance (ANOVA) for all the developed models. The determination coefficient (R^2) is being utilized to verify the model's degree of correlation. As shown in the table, bulk specific density (BSD), voids in the mix (VIM), stability and flow have R^2 values of 0.97, 0.9896, 0.9869, and 0.8946 respectively. Similarly, the higher R^2 values are demonstrating that the models have only 3%, 1.04%, 1.31%, and 10.54% correlation errors. Furthermore, a good measure of interactions between the predicted and actual results is indicated by the high R^2 values for all the models developed. Also, it was observed from the ANOVA analysis that predicted R^2 values are in strong agreement with the adjusted R^2 having a difference of less than 0.2. The signal-to-noise ratio is evaluated by adequate precision (Bala *et al.*) to contrast the variation of estimated quantities at the design levels with the median prediction error. In this research, reasonable discrimination of models was

Table 4 Experimental design matrix for Box-Behnken Design (BBD) method and responses

Run	IP6 (A) (%)	IP16 (B) (%)	Bitumen content (C) (%)	BSD (g/cm^3)	VIM (%)	Stability (kN)	Flow (mm)
1	0	1.75	4	2.3590	5.40	17.65	3.59
2	0	1.75	6	2.3670	2.51	17.31	3.98
3	0	2.5	5	2.3689	3.37	17.90	3.92
4	0.5	1.75	5	2.3553	4.03	18.32	3.94
5	0	1	5	2.4016	2.72	17.77	3.78
6	0.5	1.75	5	2.3550	4.04	18.30	3.91
7	0.5	1.75	5	2.3454	4.44	18.31	3.95
8	1	1.75	4	2.3268	6.25	17.23	3.97
9	0.5	1	6	2.3704	2.53	17.52	3.89
10	1	1	5	2.3785	3.20	18.11	3.99
11	1	1.75	6	2.3512	2.71	17.17	4.14
12	0.5	2.5	4	2.3245	6.23	16.93	4.02
13	0.5	1.75	5	2.3539	4.09	18.14	3.94
14	0.5	1.75	5	2.3438	4.50	18.33	3.93
15	1	2.5	5	2.3593	3.30	16.98	4.21
16	0.5	2.5	6	2.3558	2.41	16.75	4.11
17	0.5	1	4	2.3667	5.20	17.69	3.69



(a) (b)

(c) (d)

Fig. 3 Predicted against experimental plots for responses (a) BSD, (b) VIM, (c) Stability and (d) Flow

discovered when the adequate precision ratios of the models were determined for all the responses which are much greater than 4 (Ölmez, 2009, Mosabepranah and Eren, 2016, Mosaberpanah and Eren, 2017). The F-test lack of fit (LOF) has been employed to assess the model's adequacy. LOF shows the data variation around the fitted model and if the model does not fit the data excellently, the LOF p-value will be less than 0.05 ($p<0.05$)

which shows it is significant. For all responses with the exception of flow, the LOF p-values were greater than 0.05 ($p>0.05$) which invariably indicates that is not significant and the models fit the data excellently. For the flow, despite having a significant LOF a reasonable agreement was found between the predicted and the adjusted R^2 values which show that the selected model can be used to navigate the design space in order to determine the optimal conditions (Shafeeyan *et al.* 2012, Sánchez-Romeu *et al.* 2008).

The final response regression models are shown in Eqs. (2)-(5), where only the important impacting variables are provided. The synergistic and antagonistic impacts of the individual factors on all the responses are indicated by the positive and negative signs before the terms in the equation.

$$\begin{aligned} BSD = {} & +2.35 - 0.0101A - 0.0136B + 0.0084C \\ & + 0.0034AB + 0.0041AC + 0.0069BC \\ & + 0.0115A^2 + 0.0149B^2 - 0.0112C^2 \end{aligned} \tag{2}$$

$$\begin{aligned} VIM = {} & +4.22 + 0.1825A + 0.2075B - 1.62C - 0.1375AB \\ & - 0.1625AC - 0.2875BC - 0.4738A^2 \\ & - 0.5988B^2 + 0.4713C^2 \end{aligned} \tag{3}$$

$$\begin{aligned} Stability = {} & +18.28 - 0.1425A - 0.3162B - 0.0938C \\ & - 0.315AB + 0.07AC - 0.0025BC \\ & - 0.2363A^2 - 0.3538B^2 - 0.7037C^2 \end{aligned} \tag{4}$$

$$Flow = +3.94 + 0.13A + 0.1138B + 0.1063C \tag{5}$$

where A is irradiated WPET retained on sieve No. 6; B is irradiated WPET retained on sieve No. 16 and C is the bitumen content.

3.1 Statistical analysis

Plots of predicted against actual values are useful to have a better knowledge of models' satisfactoriness. Figs. 3(a)-3(d) demonstrates the predicted against the actual data plots for the removal of parameters for all the responses. It can be observed clearly that all the points in the plots are relatively near the equality line which further reveals that there is strong agreement between the predicted and experimental results of the models developed. Furthermore, the AP values of 21.55, 25.40, 20.62, and 18.41 for responses BSD, VIM, stability, and flow respectively are all greater than 4 (AP>4) as shown in Table 5 which is an indication that the predicted models can be used to navigate the design space defined by BBD.

Figs. 4(a)-(F) shows the effect of irradiated WPET sizes and bitumen content on the BSD of compacted bituminous samples, as can be seen from the 2D and 3D plots there is an insignificant difference in BSD values for mixes having bitumen amount between 5percent and 6percent, the BSD values were lowest for mixtures fabricated at lower bitumen content of 4percent. However, for blends fabricated between 1.3-2.1% and 0.3-0.7% for IP16 and IP6 respectively, the BSD values were the least. In addition, it was observed that mixes constructed at the highest bitumen and lowest irradiated WPET contents have the maximum BSD values. This trend can be ascribed to the higher melting point of PET and more crystalline part as the gamma irradiation increases the degree of crystallinity in PET as shown in the diffractogram in Fig. 1. Similarly, this decreases in BSD with an increase in irradiated WPET amount in the mixes relates to the reduced unit weight of PET relative to natural aggregates as well as more voids in the irradiated WPET modified mixes. A similar finding of a reduction in BSD was reported by (Huda *et al.* 2017) on the use of the palm oil industry's bi-products as coarse aggregates in structural lightweight concrete. Additionally, the effect of exposing the WPET to doses of gamma radiation decreases the molecular weight of the WPET when compared with regular, non-irradiated WPET which in turn increases its molecular mobility.

Void in the mix (VIM) is one of the essential variables of the bituminous mixes used for asphalt construction and optimum bitumen content evaluation. Figs. 5(a)-(f) depicts the effect of irradiated WPET depending on size and bitumen quantity in the asphalt blend. As can be seen from the 2D and 3D contour plots, the bitumen quantity has more pronounce impact on the VIM than the irradiated WPET in the blends because when the bitumen amount increases from 4% to 6%, the VIM value experiences a significant decline of 3.84% from 6.25% at 4% bitumen amount to 2.41% at 6% bitumen amount. Furthermore, it could be observed that for all the bitumen contents the VIM value increases with the addition of irradiated WPET content. For IP16, the VIM experiences an advancement trend from 1.25% to 2.275% after which it starts to recess. A similar trend was observed for IP6 but in this case from 0.2% to 0.8%. This increased in VIM value with irradiated WPET contents can be ascribed to the flaky shape of the WPET used in the blends that remained in the form of crystal which increases the surface area. The higher surface area, however, needs to be covered with the bitumen that would ultimately result in lower workability when blending and therefore lower bulk densities, resulting in increased VIM. Another reason that can cause VIM to increase with the addition of irradiated WPET into the blend might be due to the elastic deformation of the WPET particles under compaction that may lead to increased VIM values. Similar results were reported by (Moghaddam *et al.* 2015b).

In asphalt concrete wearing course design, among the most significant parameter considered for the asphalt blend is stability. Sufficient stability is necessary in wearing courses in order to withstand varying levels of traffic loads. Figs. 6(a)-6(f) demonstrates the relationship between dependent variable stability as well as independent variables IP16, IP6, and bitumen. From the 2D contour plots of figure 6, it can be deduced that the obtained oval shape shows there is excellent interaction between all the independent factors (Nassar *et al.* 2016, Bala *et al.* 2018). Additionally, an increase in both sizes of the WPET contents and bitumen quantity in the blends affect the Marshall stability significantly. The stability values improve significantly for mixtures cast at the intermediate binder amount of 5percent with an increase in the amount of IP16, from 1.225% to 2.275%, and IP6, from 0.25% to 1%.

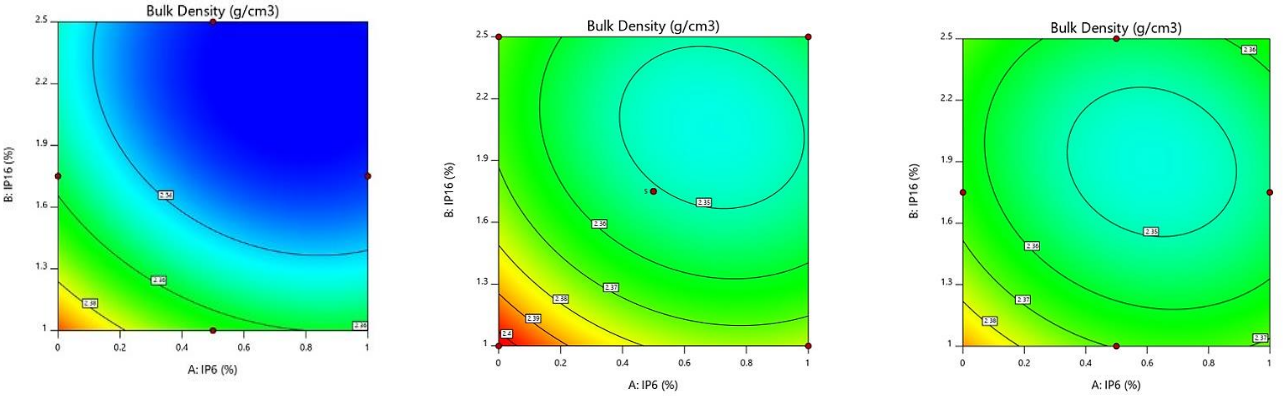
Table 5 ANOVA for stability and volumetric properties

Source	SS	Df	MS	F value	p value	remarks
Bulk density						
Model	0.0051	9	0.0006	25.16	0.0002	Significant
A-IP6	0.0008	1	0.0008	35.91	0.0005	
B-IP16	0.0015	1	0.0015	65.15	< 0.0001	
C-Bitumen	0.0006	1	0.0006	25.05	0.0016	
AB	0.0000	1	0.0000	2.01	0.1992	
AC	0.0001	1	0.0001	2.97	0.1287	
BC	0.0002	1	0.0002	8.40	0.0230	
A^2	0.0006	1	0.0006	24.66	0.0016	
B^2	0.0009	1	0.0009	41.08	0.0004	
C^2	0.0005	1	0.0005	23.30	0.0019	
LOF	0.0000	3	0.0000	0.3515	0.7919	not significant
AP	21.55					
R^2	0.9700					
Adj. R^2	0.9315					
VIM						
Model	25.29	9	2.81	73.67	< 0.0001	Significant
A-IP6	0.2664	1	0.2664	6.98	0.0333	
B-IP16	0.3444	1	0.3444	9.03	0.0198	
C-Bitumen	20.87	1	20.87	546.94	< 0.0001	
AB	0.0756	1	0.0756	1.98	0.2020	
AC	0.1056	1	0.1056	2.77	0.1401	
BC	0.3306	1	0.3306	8.67	0.0216	
A^2	0.9450	1	0.9450	24.77	0.0016	
B^2	1.51	1	1.51	39.57	0.0004	
C^2	0.9351	1	0.9351	24.51	0.0017	
LOF	0.0548	3	0.0183	0.3446	0.7960	not significant
AP	25.40					
R^2	0.9896					
Adj. R^2	0.9761					
Stability						
Model	4.55	9	0.5053	58.49	< 0.0001	Significant
A-IP6	0.1624	1	0.1624	18.80	0.0034	
B-IP16	0.8001	1	0.8001	92.61	< 0.0001	
C-Bitumen	0.0703	1	0.0703	8.14	0.0246	
AB	0.3969	1	0.3969	45.94	0.0003	
AC	0.0196	1	0.0196	2.27	0.1757	
BC	0.0000	1	0.0000	0.0029	0.9586	
A^2	0.2350	1	0.2350	27.20	0.0012	
B^2	0.5269	1	0.5269	60.99	0.0001	
C^2	2.09	1	2.09	241.38	< 0.0001	
LOF	0.0355	3	0.0118	1.89	0.2721	not Significant
AP	20.62					
R^2	0.9869					
Adj. R^2	0.9700					
Flow						

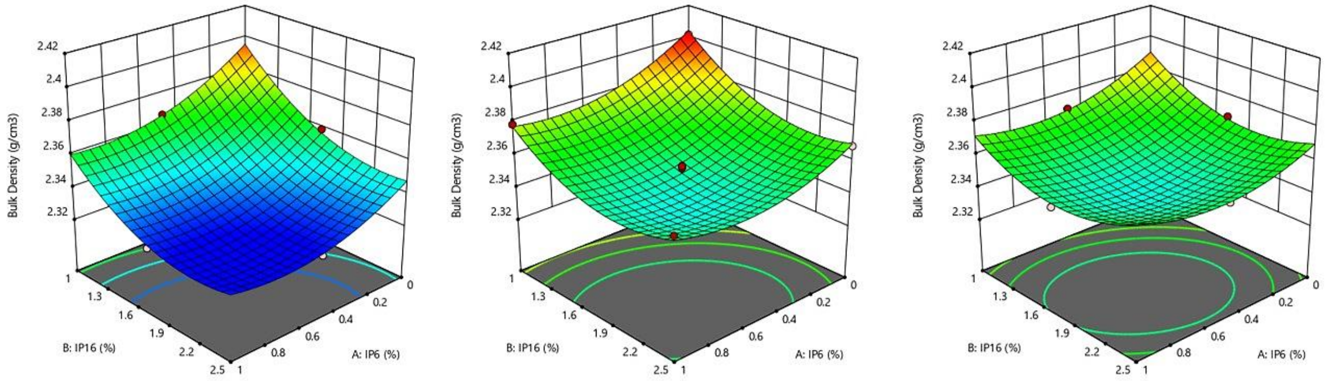
Table 5 Continued

Source	SS	Df	MS	F value	p value	remarks
Model	0.3290	3	0.1097	36.79	< 0.0001	significant
A-IP6	0.1352	1	0.1352	45.36	< 0.0001	
B-IP16	0.1035	1	0.1035	34.73	< 0.0001	
C-Bitumen	0.0903	1	0.0903	30.30	0.0001	
LOF	0.0378	9	0.0042	18.28	0.0066	significant
AP	18.41					
R^2	0.8946					
Adj. R^2	0.8703					

SS means Sum of Squares; Df means Degree of Freedom; MS means Mean Square; LOF means Lack of Fit



(a) 4% bitumen content (b) 5% bitumen content (c) 6% bitumen content



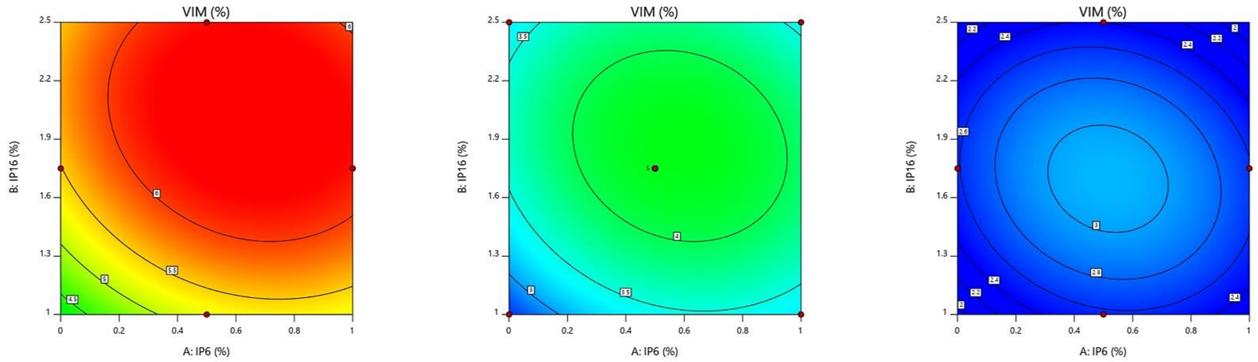
(d) 4% bitumen content (e) 5% bitumen content (f) 6% bitumen content

Fig. 4 2D contour and 3D plots for bulk specific density

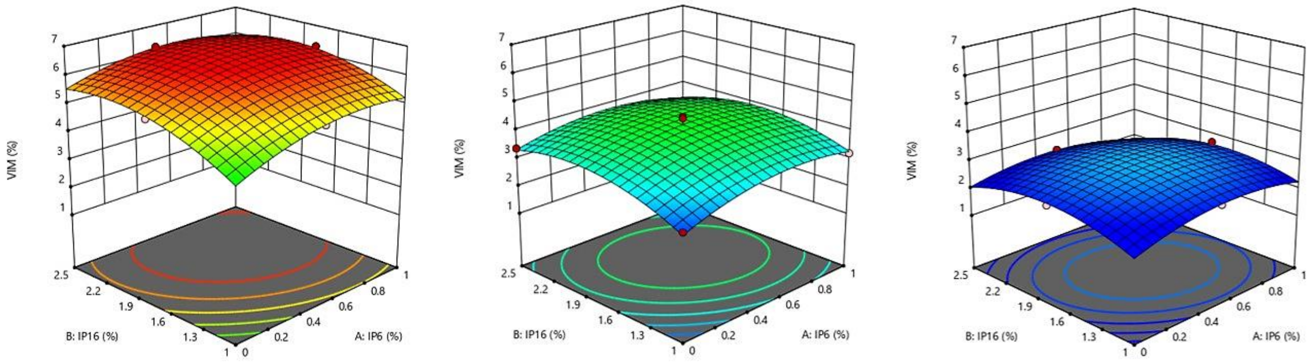
Though, stability reduces significantly as the bitumen amount rises from 5-6percent, whereas both sizes of irradiated WPET contents were between the ranges mentioned above.

The findings also showed that the increase in the amount of bitumen beyond 5percent negatively affects the stability of the blend and this is further emphasized for mixes made with the maximum binder quantity of 6percent. All the independent factors showed a significant relationship with the dependent factor. However, the IP16 has a greater influence on the stability, then followed by the IP6 with the bitumen content having the least influence among the three independent variables.

Flow is an asphalt mixture's capacity to adjust without cracking to gradual settlements and movements. For flow, linear interactions are found as demonstrated in Figs. 7(a)-(f) between all the three independent factors. From the figure, it can be observed that all the three independent variables have almost the same effect on the flow as the flow values are both increased by increasing all independent factors amount in the blend. This might be ascribed to reduced WPET-reinforced mixture internal friction owing to reduced irradiated WPET particle friction surface. Similar outcomes were revealed by (Moghaddam *et al.* 2015b) that adding regular, non–irradiated PET had almost the same impact on the flow rates of the blend as shown for irradiated WPET modified blends in this study.

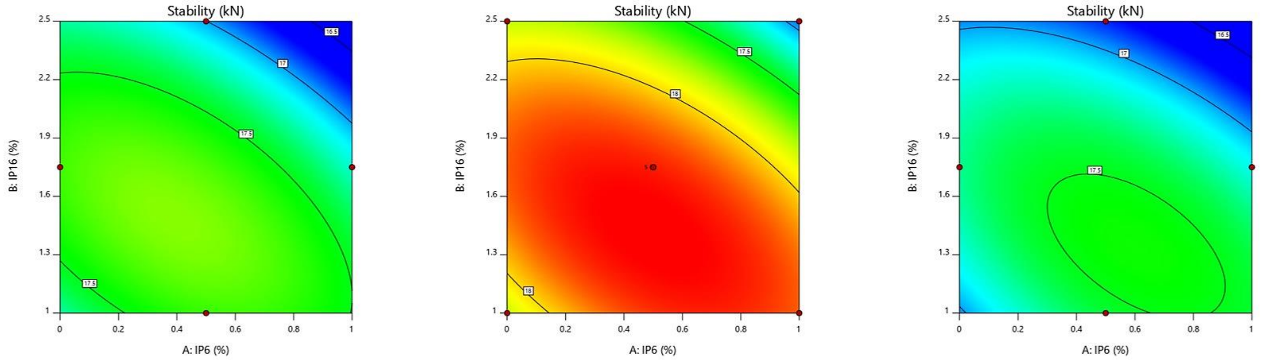


(a) 4% bitumen content (b) 5% bitumen content (c) 6% bitumen content

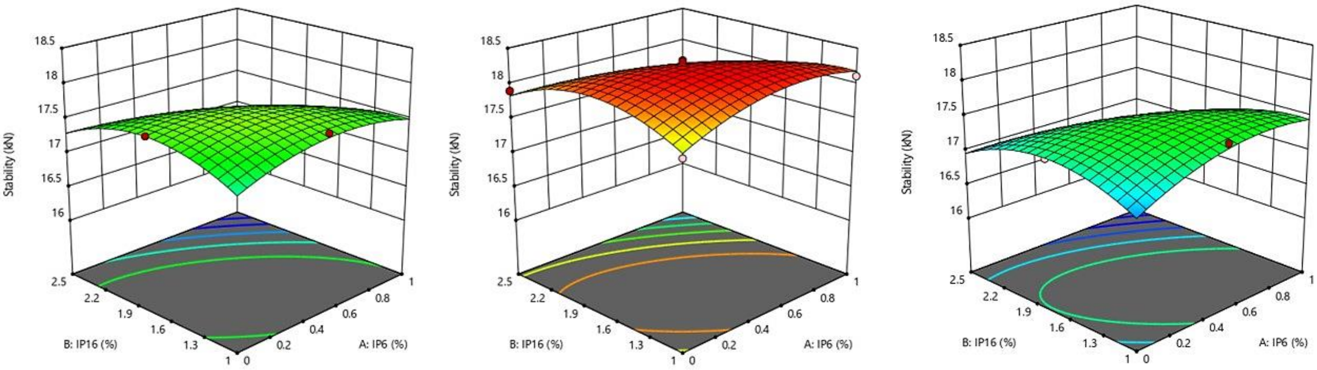


(d) 4% bitumen content (e) 5% bitumen content (f) 6% bitumen content

Fig. 5 2D contour and 3D plots for the void in the mix



(a) 4% bitumen content (b) 5% bitumen content (c) 6% bitumen content

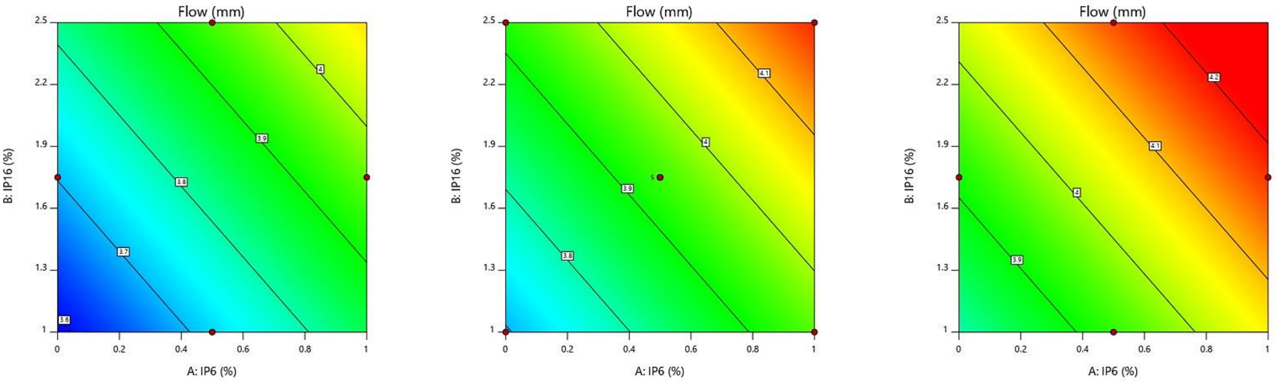


(d) 4% bitumen content (e) 5% bitumen content (f) 6% bitumen content

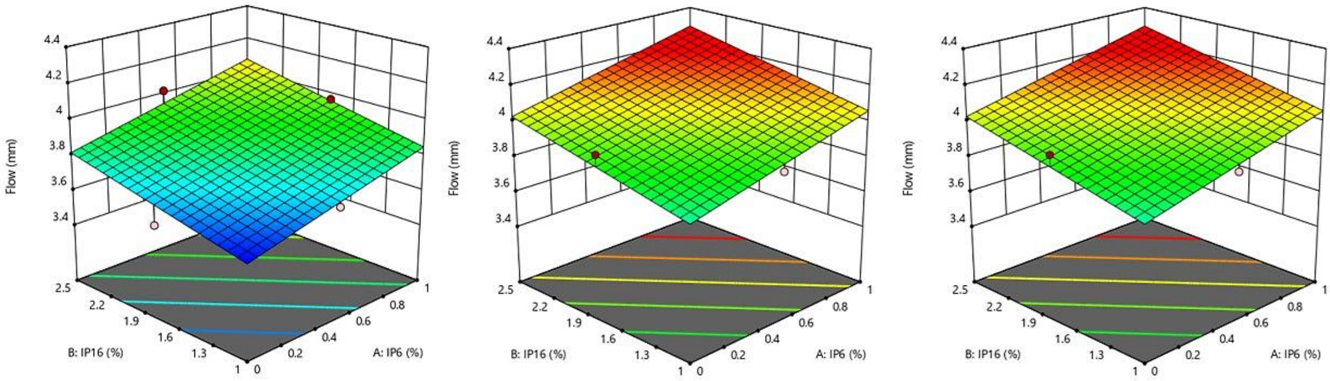
Fig. 6 2D contour and 3D plots for Marshall stability

3.2 Mix design parameters optimization

Due to its complication and too many amounts of data points for irradiated WPET modified blends, the utilization of the old methods for evaluating optimum bitumen content may not be very precise. Among the objective of this



(a) 4% bitumen content (b) 5% bitumen content (c) 6% bitumen content

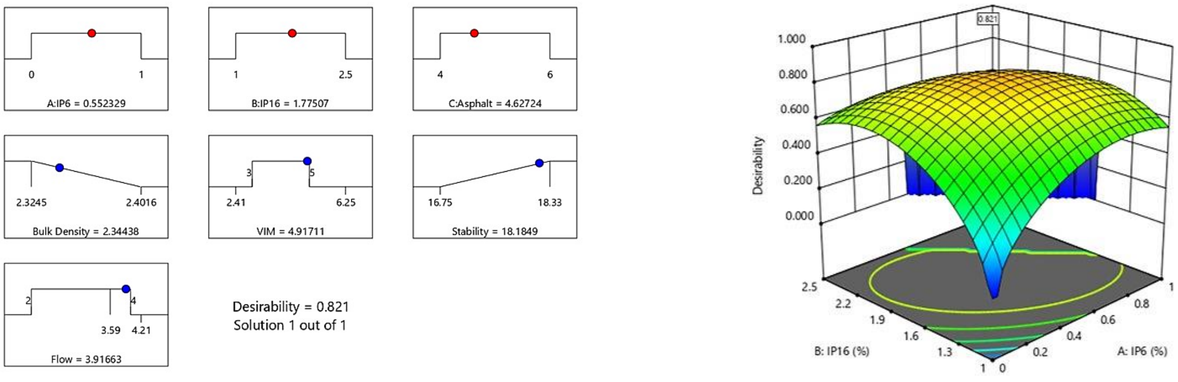


(d) 4% bitumen content (e) 5% bitumen content (f) 6% bitumen content

Fig. 7 2D contour and 3D plots for flow

Table 6 Experimental outcomes verification at optimal conditions

Responses	IP6 (%)	IP16 (%)	Bitumen (%)	Experimental	Predicted	Error (%)	Desirability (%)
BSD (g/cm^3)				2.3962	2.3444	2.16	
VIM (%)	0.55	1.77	4.63	4.76	4.92	3.36	82.1
Stability (kN)				18.83	18.18	3.45	
Flow (mm)				4.04	3.91	3.22	



(a) (b)

Fig. 8 DOE Numerical Optimization plots (a) ramps for all the responses (b) Desirability

research is performing a statistical analysis to determine the maximum contents of IP6, IP16, and bitumen that will satisfy the design specifications of the Marshall blend. A numerical multi-objective optimization technique was employed (Rahman *et al.* 2017) to evaluate the optimum quantities of IP6, IP16, and bitumen that will boost the Marshall stability of the asphalt mix.

The intended responses, stability, and BSD were set at maximum and minimum levels respectively to obtain a durable blend, whereas, VIM and flow values were set to the ranges specified for ACW14 as per JKR (Malaysia, 2008).

RSM design expert software suggested an optimal solution for the mix design based on the maximum desirability of 82.1%. Figs. 8(a) and 8(b) shows the DOE optimization ramps and desirability plot for the independent factors and responses. An additional experiment was performed to validate the predicted optimum based on the outcomes of the optimal predicted requirements and a comparison was made with predicted model values suggested by the software. Validation test results are shown in Table 6; the percentage error between the experimental and optimally predicted values is calculated utilizing Eq. (6).

$$Percentage\ Error = \frac{Experimetal - Predicted}{Experimental} \times 100 \quad (6)$$

4. Conclusions

This study targeted optimizing the contents of bitumen and two different sizes of irradiated WPET particles on the mix volumetric and strength of dense graded AC mixtures. In this investigation, RSM statistical analysis was used to determine the relationship between chosen factors.

The following conclusions were drawn based on the results analyzed in this paper.

- On the BSD: the highest values were obtained for blends made with bitumen contents between 5-6percent and lower contents of irradiated WPET. However, the lowest BSD values were gotten for mixes constructed at lower amounts of bitumen and intermediate-range amounts of the two sizes of irradiated WPET.
- On the VIM: the bitumen amount has a more pronounced impact than the other two independent factors in the blends. Also, the maximum value of VIM irrespective of bitumen quantity was obtained for blends made within the ranges of 1.6-1.98% and 0.4-0.65% for IP16 and IP6 contents, respectively.
- On Marshall Stability: the maximum values were gotten for mixes cast with bitumen quantity between 4-5percent after which it recesses drastically. These highest stability values were achieved for blends cast with intermediate ranges amount of the two sizes of irradiated WPET.
- On the Flow: All the three independent factors have the almost same effect on the flow values as the flow rates were increased with increasing amounts of all the known factors.
- Validation: the optimal amounts of all the three independent factors IP6, IP16, and bitumen contents as suggested by RSM that will satisfy the design specifications of the Marshall blend were 0.55%, 1.77%, and 4.63% all by weight of aggregates particles, respectively.
- Depending upon the outcome from optimization, errors between experimental outcomes and optimally predicted were less than 5percent for all the responses, showing that the results predicted were in good correlation with the experimental outcomes.

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