Optimizing the mix design of cold bitumen emulsion mixtures using response surface methodology.

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Abstract

Cold mix asphalt (CMA) has been increasingly recognized as an important alternative worldwide. One of the common types of CMA is cold bitumen emulsion mixture (CBEM). In the present study, the optimization of CBEM has been investigated, to determine optimum proportions to gain suitable levels of both mechanical and volumetric properties. A central composite design (CCD) with response surface methodology (RSM) was applied to optimize the mix design parameters, namely bitumen emulsion content (BEC), pre-wetting water content (PWC) and curing temperature (CT). This work aimed to investigate the interaction effect between these parameters on the mechanical and volumetric properties of CBEMs. The indirect tensile stiffness modulus (ITSM) and indirect tensile strength (ITS) tests were performed to obtain the mechanical response while air voids and dry density were measured to obtain volumetric responses.

The results indicate that the interaction of BEC, PWC and CT influences the mechanical properties of CBEM. However, the PWC tended to influence the volumetric properties more significantly than BEC. The individual effects of BEC and PWC are important, rather than simply total fluid content which is used in conventional mix design method. Also, the results show only limited variation in optimum mix design proportions (BEC and PWC) over a range of CT from 10°C to 30°C. The variation range for optimum BEC was 0.42% and 0.20% for PWC. Furthermore, the experimental results for the optimum mix design were corresponded well with model predictions. It was concluded that optimization using RSM is an effective approach for mix design of CBEMs.

Keywords: Cold Bitumen Emulsion Mixtures; Response surface method; Analysis of variance; Optimum bitumen Emulsion content; Optimum Pre-wetting water content

1. Introduction

Several benefits are gained from using cold mix asphalt (CMA) instead of hot mix asphalt (HMA). The benefits include conservation of materials and energy, preservation of the environment and reduction in cost [1, 2]. One of the common types of CAM is cold bitumen emulsion mixture (CBEM). Although the advantages of CMAs are real, they attract relatively little attention and are considered inferior to HMA as structural layers due to their less satisfactory performance [3]. This may be at least partially due to the wide variation in available mix design procedures, tests and criteria. Some authorities and researchers have proposed mix design procedures, based on empirical formulae, laboratory tests or past experience [1, 4]. However, there is no global agreement on mixture design method or structural design methodology for CMAs [5]. Thus, it is clear that optimization of mixture parameters has to be made more consistent in order to promote the technology [4] whereas the variations in material proportions will generate differences in performance [6]. It is therefore essential to design and optimize mixture components in order to achieve appropriate properties [4, 7].

Most of the studies reported in the literature on CBEMs have focused on using the method adopted by the Asphalt Institute (Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design), with some modifications [1, 8]. There would therefore appear to be potential to explore the use of a statistical tool to optimize the mixture design of CBEMs.

In response to the above need, the present study has been undertaken in order to develop a performance based mix design incorporating a statistical approach using response surface methodology (RSM). RSM is used as the optimization technique to adjust the mixture parameters of CBEM to achieve acceptable mechanical strength and suitable volumetric properties. The study aimed to investigate the interaction effect of mixture parameters on the mechanical and volumetric properties of CBEM. RSM and a three-level factorial experimental design have been applied to satisfy these conditions. The central composite design (CCD) method has been used. CCD is a fractional factorial experimental design able to provide the relationship between responses and factors over a range of factor levels [9, 10].

RSM is regularly applied in disciplines such as concrete [11-13], material and mechanical engineering technologies [14-16]. Recently, there has been growing attention to the application of RSM in asphalt research [17-24]. Chávez-Valencia et. al. [17] also implemented RSM to evaluate the ageing phenomenon of bituminous binder in HMA. Haghshenas et. al. [18] studied the effects of frequency, temperature and their interaction, on rutting of HMA using RSM. Hamzah et. al. [19] used RSM to optimize the binder content of warm mix asphalt incorporating Rediset by evaluating the volumetric and strength properties of mixes. Kavussi et. al. [20] investigated the effect of aggregate gradation, hydrated lime content and Sasobit content on moisture damage of warm mix asphalt. An experimental study [21] used RSM to assess the effects of aggregate gradation and lime content on stripping of HMA in terms of the strength and stiffness. Also, Khodaii et. al. [22] evaluated the effects of aggregate gradation, lime content, Sasobit content and binder content on stripping potential of warm mix asphalt. RSM was used to investigate the effects of short term aging on asphalt binder rheological properties [23]. A laboratory study [24] assessed the properties of stone mastic asphalt mixtures incorporating waste polyethylene terephthalate using RSM.

There is therefore a potential benefit to apply RSM as an alternative approach for the optimization of mix design parameters in CBEMs.

2. Design of experiment using RSM

Montgomery [9] defined RSM as a mathematical and statistical technique used for designing experiments in order to establish relationships between multiple factors and to optimize the relevant conditions of parameters in order to predict the best responses.

A fractional factorial design such as CCD is usually used in RSM [10]. It has been reported as a potentially useful approach which is able to provide a suitable functional relationship between the responses and the factors (i.e. input parameters) [21]. Design Expert 9.0.6.2 software (Stat-Ease Inc., Minneapolis, USA) was used for the design, mathematical modelling, statistical analysis, and optimization of the process parameters. Analysis of variance (ANOVA) was conducted in order to obtain the interaction among the different parameters and the influence of each individual parameter.

The appropriate regression model, recommended by [9, 10], was applied, as shown in the following equation:

$$Y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X_j^2 + \sum_{i} \sum_{<j=2}^{k} \beta_{ij} X_i X_j + e_i \quad (1)$$

Where Y is the response, X_i and X_j are the parameters, β is the regression coefficient, k is the number of parameters included in the experiment, and e is the random error.

There are two kinds of fluid inside CBEM, which are water and bitumen. The water content includes two sources of water: (1) a proportion of the bitumen emulsion content (BEC) and (2) the additional water in the mix, termed the pre-wetting water content (PWC). PWC is defined as the amount of water added to the mixture prior to the addition of bitumen emulsion. This addition is to improve the ability of bitumen emulsion to coat the aggregate and to improve the workability of the mixture.

An experimental program was undertaken in order to consider the effects of certain important parameters on CBEM mix design. The parameters (independent variables) considered were BEC, PWC and curing temperature (CT). BEC and PWC are presented as a percentage of total mass of dry aggregate. These three parameters together with their respective ranges were selected based on a preliminary study and extant literature [4, 25, 26].

It is well known that the curing temperature significantly affects the properties of the CMAs [27-30]. Therefore, the CT was considered as a parameter in mix design, 10°C, 20°C and 30°C being taken to represent cold, moderate and warm climatic conditions, respectively. The ranges and the levels of all the parameters investigated are given in Table 1.

Table 1: Independent parameters and their coded levels for CCD.

Parameters	**Code**	**Unit**	**Coded parameter levels**		
			-1	**0**	**+1**
BEC	X_1	%	5.0	6.0	7.0
PWC	X_2	%	0.5	2.0	3.5
CT	X_3	°C	10	20	30

(-1) refers low level; (0) refers to mean level; (+1) refers to high level

The literature shows that CMA design methods are similar to those of HMA, but with no universally accepted method or procedure [31]. There are two sets of tests commonly conducted when assessing HMA, mechanical tests and volumetric tests respectively. In the current study the responses considered represented the mechanical and the volumetric properties of CBEMs. Indirect tensile stiffness modulus (ITSM) and indirect tensile strength

(ITS) were performed in order to evaluate the mechanical properties. Air voids and dry density were measured to assess the volumetric properties, calculated according to Asphalt Institute [5] recommendations.

The total number of experiments carried out was 20 (= 2^k +2k +6), where k is the number of parameters (k= 3). Fourteen different combinations were supplemented with six replicates of the mean case. The set of 14 mixes considered three levels of each studied parameter; all factors were varied in this way. The set of six replicates mixes considered the mid-level of each studied parameter; this point is often replicated in order to improve the precision of the experiment and minimize any possible sources of bias. The CCD matrix employed is presented in Table 2.

Table 2: Matrix of experimental design by CCD.

Run No.	**Mix design parameters**			**Total fluid content (%)**
	BEC (%)	PWC (%)	CT (°C)	
Mix 01	5.0	3.5	10	8.50
Mix 02	7.0	0.5	10	7.50
Mix 03	7.0	3.5	10	10.5
Mix 04	5.0	0.5	10	5.50
Mix 05	6.0	2.0	10	8.00
Mix 06	6.0	3.5	20	9.50
Mix 07	6.0	0.5	20	6.50
Mix 08	7.0	2.0	20	9.00
Mix 09	6.0	2.0	20	8.00
Mix 10	6.0	2.0	20	8.00
Mix 11	6.0	2.0	20	8.00
Mix 12	6.0	2.0	20	8.00
Mix 13	5.0	2.0	20	7.00
Mix 14	6.0	2.0	20	8.00
Mix 15	6.0	2.0	20	8.00
Mix 16	6.0	2.0	30	8.00
Mix 17	7.0	3.5	30	10.50
Mix 18	7.0	0.5	30	7.50
Mix 19	5.0	3.5	30	8.50
Mix 20	5.0	0.5	30	5.50

3. Material and experimental procedures

3.1. Aggregate

The aggregate used in this study was crushed limestone. The aggregate gradation used is shown in Fig. 1. In order to ensure appropriate interlocking of the dense graded surface course mix, a gradation was selected according to BS 4987-1 [32].

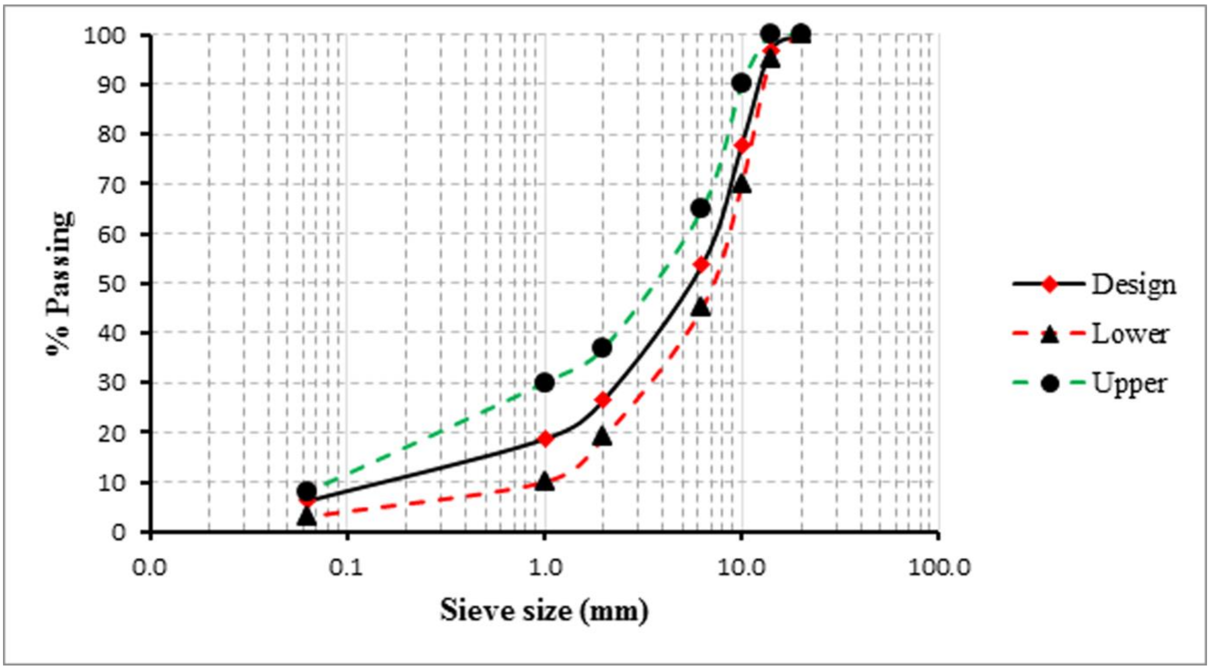

Fig. 1. Limestone aggregate gradation.

The physical properties of the limestone aggregate are shown in Table 3.

Table 3: Physical characteristics of limestone aggregate.

Properties	Value
Density- Oven Dried	2.68 Mg/m^3
Density- Saturated Surface Dried	2.69 Mg/m^3
Density- Apparent	2.70 Mg/m^3
Water Absorption	0.4 %
Aggregate Abrasion Value (AAV)	11.0
Polished Stone Value (PSV)	31
Los Angeles Coefficient (LA)	28

3.2. Bitumen Emulsion

The binder used was cationic slow setting bituminous emulsion (C60B5) to ensure high adhesion between aggregate particles [1]. The relevant properties of the selected bituminous emulsion are shown in Table 4.

Table 4: Bitumen emulsion properties.

Property	Value	Standard
Appearance	Black to dark brown liquid	
Breaking Behaviour	> 170	EN 13075-1
Softening Point (°C)	52	EN 1427
Viscosity - Efflux time 2mm - 40°C (sec)	15-70	EN 12846
Adhesiveness	>90	EN13614
Particle surface electric charge	Positive	EN 1430
Bitumen content (%)	60	EN 1428
Penetration (dmm)	47	EN 1426
Density (g/cm^3)	1.016	

3.3. Sample manufacturing

The mix proportions presented in Table 2 were used to prepare Marshall specimens.

The procedure followed for preparing the specimens was such that the PWC was first added to the dry batched mixture and mixed using a Sun and Planet mixer for 60s. This was followed by mixing using a spatula for 30s ensuring that the aggregate materials were thoroughly blended and wetted ready for the addition of the bitumen emulsion. The required emulsion was subsequently added and the mixture mixed for another 60s. To ensure homogeneity and consistency in the mix, the materials were then mixed by hand using a spatula for 30s. These timings were found suitable for such mixes by [31, 33]. Impact compaction (Marshall Hammer) was utilized to compact the specimens; 75 blows applied to each face. The selection of 75 blows was made based on a pilot study performed to investigate the effective compaction effort for CBEMs. After compaction, the curing protocol followed was such that the specimens were left in the moulds for 24hrs (in a sealed condition) at the same ambient temperature before they were carefully extruded. After that, specimens were conditioned for 28 days in a thermostatically controlled air chamber at temperatures of 10ºC, 20ºC, and 30ºC as stated in Table 2.

3.4. Laboratory testing program

The mix design considered here relies on the information obtained from fundamental tests, which are used to evaluate the mixes proposed using CCD, as shown in Table 2. Two response types have been investigated in order to identify mixture performance, namely: mechanical responses and volumetric responses.

3.4.1. Mechanical responses

The mechanical responses were evaluated by using ITSM and ITS tests.

Indirect tensile stiffness modulus

The ITSM is a non-destructive test used mainly to evaluate the stiffness modulus of bituminous mixes. Stiffness modulus is considered as an indicator of the structural behaviour of mixtures because it is related to the capacity of the material to distribute traffic loads. The test was carried out according to BS EN 12697-26 [34] and was performed under the conditions presented in Table 5. Four specimens per mix were tested under the same conditions.

Table 5: ITSM test conditions.

Item	Range
Specimen diameter	100±2 mm
Transient peak horizontal deformation	3 µm
Rise time	124±4 ms
Poisson's ratio	0.35
Test temperature	20 °C
Specimen thickness	45-60 mm
Compaction	75 blows/face
Specimen temperature conditioning	20^{o}C over the night before day of testing

Indirect tensile strength

The ITS test involved applying diametric compression with a constant deformation rate of (50 ± 2) mm/min to the samples between two loading strips, which creates tensile stresses along the vertical diametral plane causing a splitting failure. The test was conducted at 20°C using an INSTRON test equipment in accordance with BS EN 12697-23 [35].

3.5. Volumetric responses

It was demonstrated by Thanaya [1] that satisfactory volumetric properties are essential to the design of CBEMs. The volumetric properties of mixes were evaluated using the methodology proposed by the Asphalt Institute [5].

4. Results and discussion

A total of 80 Marshall specimens were produced for the 20 mixes proposed using CCD, four for each mix. The results are presented in Table 6 and discussed in the following paragraphs.

Table 6: Experimental factors and experimental responses

Parameters				**Responses**									
				Mechanical Responses						**Volumetric Responses**			
Run No.	**BEC (%)**	**PWC (%)**	**CT (^{o}C)**	**ITSM$_{10}$ days (MPa)**	**SD**	**ITS dry$_{28}$ days (kPa)**	**SD**	**ITS wet$_{28}$ days (kPa)**	**SD**	**Dry density (kg/ cm^{3})**	**SD**	**Air voids (%)**	**SD**
Mix 01	**5.0**	**3.5**	**10**	2141	(113.77)	639	(31.24)	444	(27.28)	2232	(0.063)	13.70	(0.072)
Mix 02	**7.0**	**0.5**	**10**	2142	(265.68)	688	(54.41)	543	(4.56)	2212	(14.45)	13.85	(0.787)
Mix 03	**7.0**	**3.5**	**10**	2551	(259.80)	662	(29.22)	477	(31.52)	2203	(3.532)	13.11	(0.156)
Mix 04	**5.0**	**0.5**	**10**	1149	(244.79)	401	(47.77)	307	(11.86)	2129	(11.08)	18.01	(0.433)
Mix 05	**6.0**	**2.0**	**10**	2859	(194.05)	858	(26.49)	502	(41.46)	2280	(0.645)	11.09	(0.031)
Mix 06	**6.0**	**3.5**	**20**	2928	(152.39)	697	(33.05)	597	(30.92)	2229	(15.07)	12.85	(0.569)
Mix 07	**6.0**	**0.5**	**20**	1735	(230.94)	614	(74.77)	524	(29.98)	2167	(7.11)	15.44	(0.341)
Mix 08	**7.0**	**2.0**	**20**	2999	(234.07)	1015	(40.87)	865	(41.29)	2257	(12.67)	11.27	(0.536)
Mix 09	**6.0**	**2.0**	**20**	2953	(161.68)	924	(42.38)	798	(36.21)	2243	(8.16)	10.82	(0.296)
Mix 10	**6.0**	**2.0**	**20**	2957	(203.94)	918	(15.09)	783	(20.82)	2253	(7.04)	10.85	(0.341)
Mix 11	**6.0**	**2.0**	**20**	3055	(245.03)	941	(31.25)	788	(40.25)	2286	(9.28)	11.36	(0.325)
Mix 12	**6.0**	**2.0**	**20**	2953	(161.68)	909	(28.15)	773	(35.01)	2273	(8.14)	10.92	(0.301)
Mix 13	**5.0**	**2.0**	**20**	2576	(181.15)	910	(33.95)	790	(30.33)	2294	(1.81)	11.075	(0.065)
Mix 14	**6.0**	**2.0**	**20**	2988	(161.68)	928	(42.38)	793	(33.58)	2279	(9.28)	11.36	(0.325)
Mix 15	**6.0**	**2.0**	**20**	2955	(203.94)	934	(15.09)	778	(24.28)	2248	(7.04)	10.85	(0.341)
Mix 16	**6.0**	**2.0**	**30**	4313	(260.56)	997	(51.74)	837	(35.07)	2276	(3.79)	11.24	(0.148)
Mix 17	**7.0**	**3.5**	**30**	3306	(213.82)	817	(22.97)	736	(3.74)	2218	(13.55)	12.65	(0.708)
Mix 18	**7.0**	**0.5**	**30**	3354	(299.96)	985	(65.47)	861	(24.98)	2215	(13.05)	13.14	(0.475)
Mix 19	**5.0**	**3.5**	**30**	3750	(230.55)	705	(69.08)	601	(38.91)	2235	(8.16)	13.59	(0.128)
Mix 20	**5.0**	**0.5**	**30**	2192	(109)	503	(74.77)	343	(30.81)	2118	(8.12)	18.34	(0.348)

* SD refers to Standard Deviation

4.1.Analysis of mechanical responses

CMA performance is influenced by the time and temperature of the curing process [36-38]. Doyle et al. [36] found that it was necessary to vary both time and temperature of curing in order to represent the material achieved in the field. Logically, measurement of evaporated water will enable a better understanding of the performance of these mixtures. Therefore, periodically, specimen weights were recorded over 28 days. The results of average loss of water for all 15 individual mixes are shown in Fig. 2, in which for example (10°C Mix 5, 3.5) refers to curing at 10°C with 5% BEC and 3.5% PWC. The percentage of water loss was calculated based on the weight of specimens after demoulding directly. It can be observed that around 85% to 95% of the total evaporation occurs during the first 10 days and 5% to 15% through the remainder of the period.

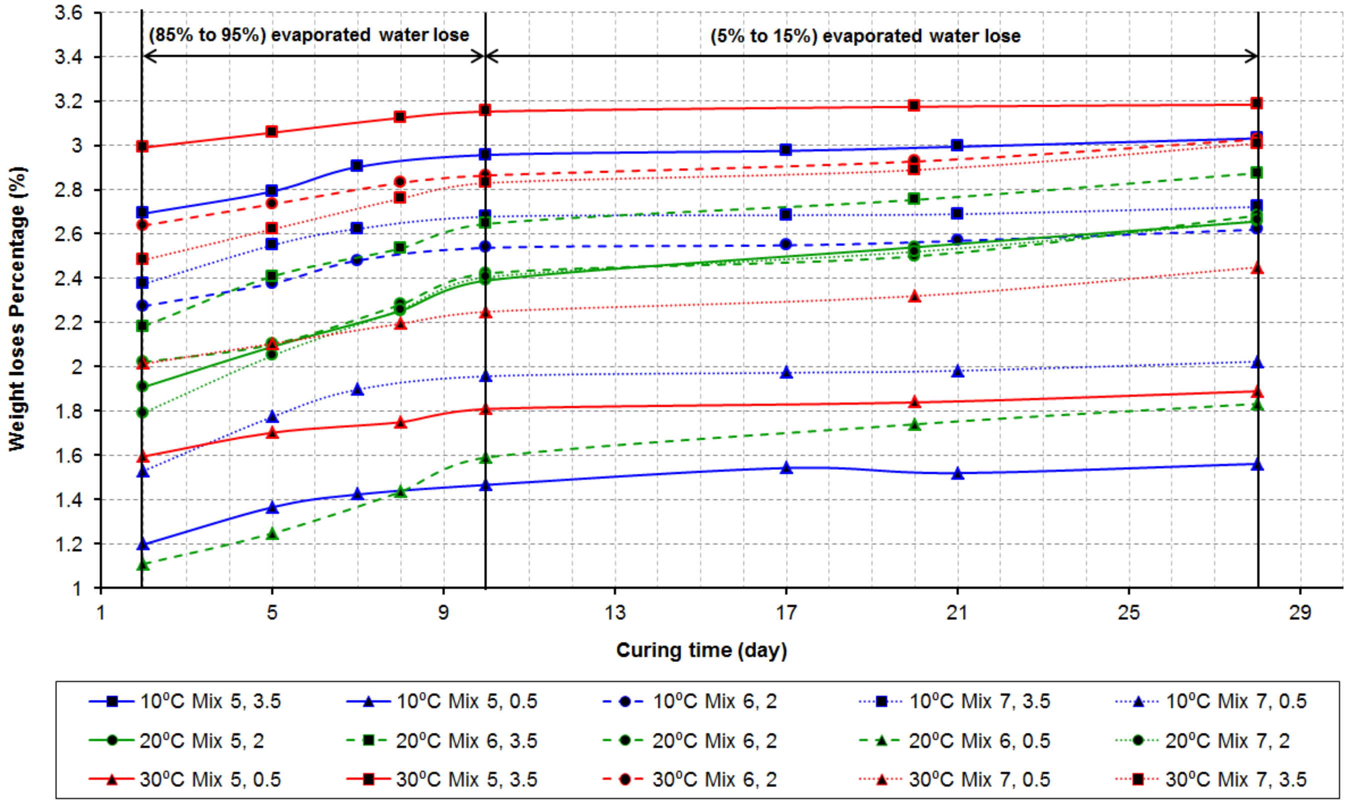

Fig. 2. Weight loss over curing period.

The evaluation of stiffness was performed at 10 days and 28 days. This is broadly consistent with South African Bitumen Association [39] recommendation to evaluate CBEMs at room temperature at 7 days and 28 days.

4.1.1. Indirect tensile stiffness modulus

This test has often been used as an indicative test for ranking CBEMs during mix design [1, 31]. Following a conventional mix design method [5], the performance of CBEM was initially evaluated based on the relationship between total fluid content, which is the sum of BEC and PWC, and mechanical properties. The $\mathrm{ITSM}_{10\ \mathrm{days}}$ and $\mathrm{ITSM}_{28\ \mathrm{days}}$ are plotted against total fluid content in Fig. 3 in order to give a better understanding of the performance of CBEM.

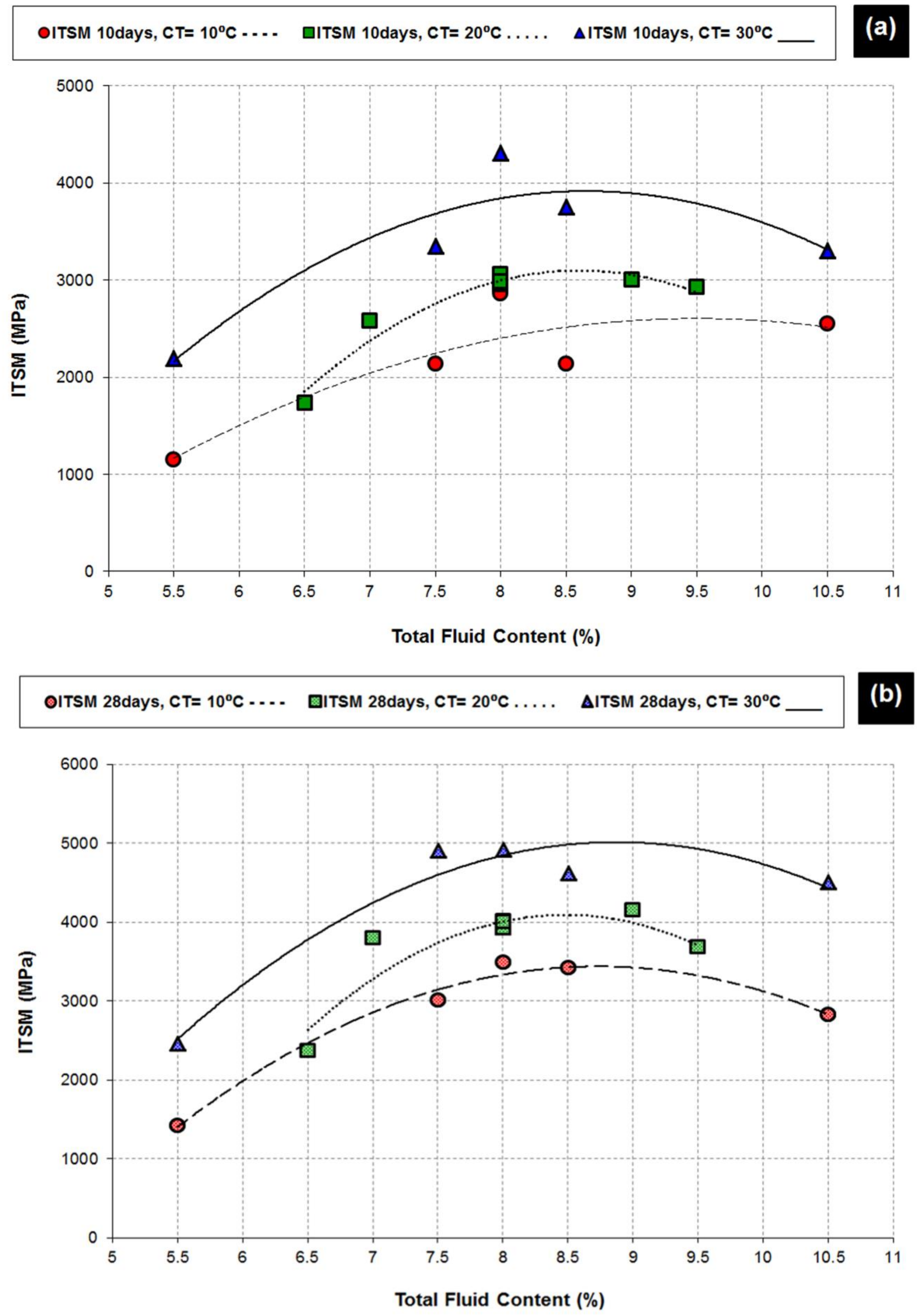

Fig. 3. The relation between ITSM and total fluid content of CBEMs under different CT after (a) 10 days and (b) 28 days

As can be seen from Fig. 3, the stiffness values show the same trend at 10 and 28 days. Peak ITSM values occurred at between 8.5% to 9.5% total fluid content at both 10 and 28 days. Therefore, it is reasonable to propose that evaluation at 10 days will probably give the designer appropriate information to optimize the mix design of CBEM, although, a wide range of curing times has been used by different researchers [4]. Also, as expected, ITSM increases with CT at both 10 and 28 days. This is consistent with results obtained in previous studies [1, 6, 40] and can at least partly be attributed to rapid water loss at higher temperature, which yields higher stiffness values with time. The results in Fig. 3 show that the lowest stiffness values were obtained from mixtures with the least total fluid content. This may be due to insufficient total fluid content in these mixes restricting the degree of compaction and increasing the air voids content in the mixture. It is worth noting the sensitivity of CBEMs' stiffness to the variation of individual mix components. Consequently, to assess the interactive relationship between the mix design parameters and the properties of CBEMs, RSM model has been used to generate a contour plot for $ITSM_{10\ days}$ shown in Fig.4.

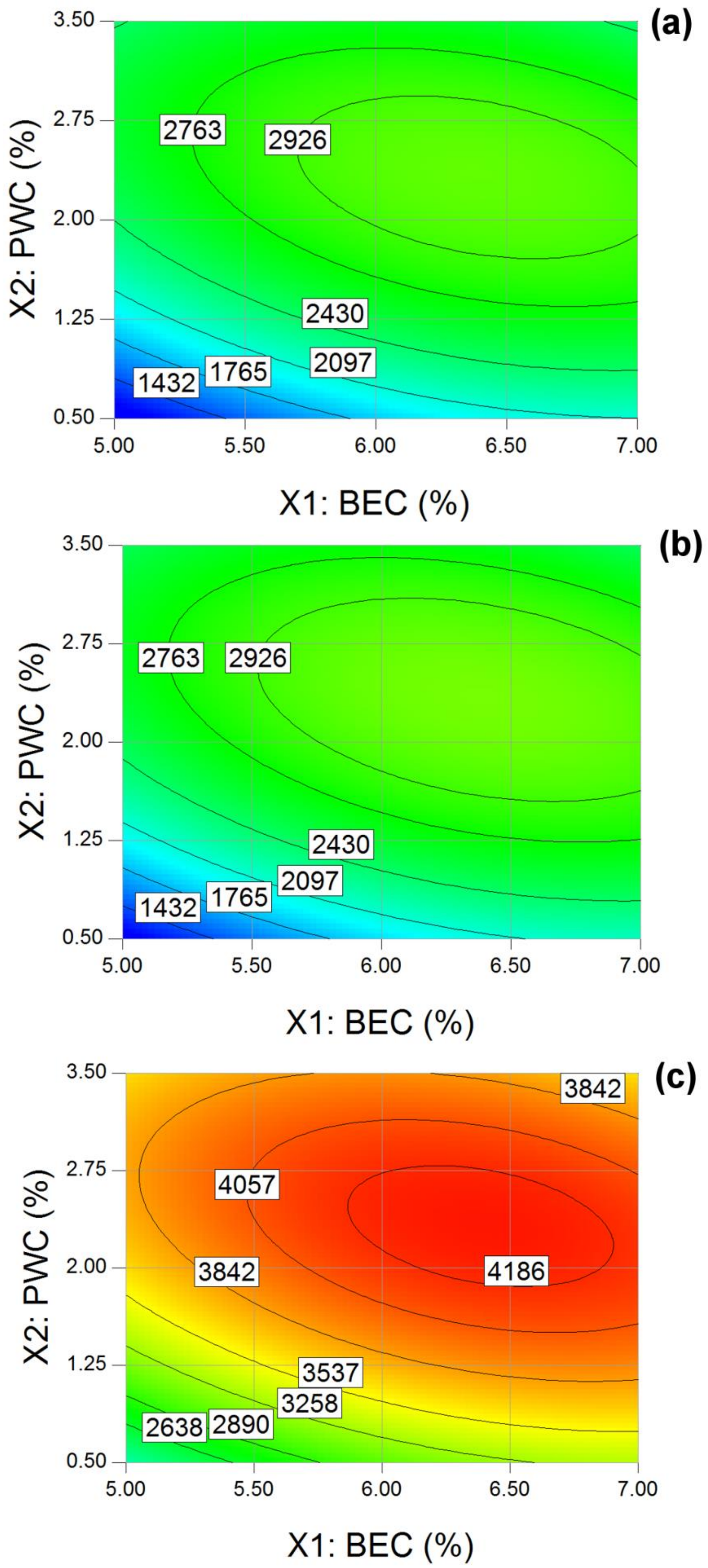

Fig. 4. Contour plots of ITSM$_{10\ \text{days}}$ versus BEC and PWC; (a) CT =10^{o}C, (b) CT =20^{o}C and (c) CT =30^{o}C.

The results in Fig. 4 show the effect of BEC and PWC versus $\mathrm{ITSM}_{10\ \mathrm{days}}$ at different CT. From these contour plots $\mathrm{ITSM}_{10\ \mathrm{days}}$ values tend to increase markedly with increasing PWC from 0.50% to 2.75% and with increasing BEC from 5.0% to 6.5%. However, $\mathrm{ITSM}_{10\ \mathrm{days}}$ markedly decreases when increasing PWC from 2.75% to 3.5% and increases slightly when increasing BEC from 6.5% to 7.0%. Moreover, the results in Fig. 4 show that in reality the individual effects of BEC and PWC are important, rather than simply total fluid content. The response surface presented in Fig. 4 shows elliptical contours which is the pattern obtained when there are perfect interactions between independent variables [41, 42]. Accordingly, there is a region of optimum performance at around 6.0- 6.5% BEC and 2.0- 2.5% PWC, whereas ITSM is lower with different BEC/ PWC proportions, even at the same total fluid content. The effect of increasing CT is to increase ITSM (by approximately 1.25-1.60 times as the CT increases from 10°C to 30°C) but optimum BEC and PWC are not significantly affected.

4.1.2. Indirect tensile strength

The ITS test was conducted on two sets of specimens: the first set (dry) was tested at 28 days immediately after curing; the second set (wet) was cured and then subjected to a vacuum (with 6.7 kPa pressure) for 30±5 minutes and immersed in a water bath for 3 days at 40°C before being tested. The reason for performing the ITS test on conditioned specimens is to take into consideration water damage as a criterion in mix design as recommended by several researchers [1, 40]. A similar picture to that in ITSM is presented in Fig. 5; the results show that relation between total fluid content and ITS for dry and wet specimens. The strength values for both sets display similar behaviour to that seen for ITSM. The peak ITS values for all mixtures were consistently found to lie between 8% and 9% total fluid content which is a similar range as for ITSM. The effect of increasing CT is generally to increase ITS, although the difference in peak ITS between CTs of 20°C and 30°C is slightly.

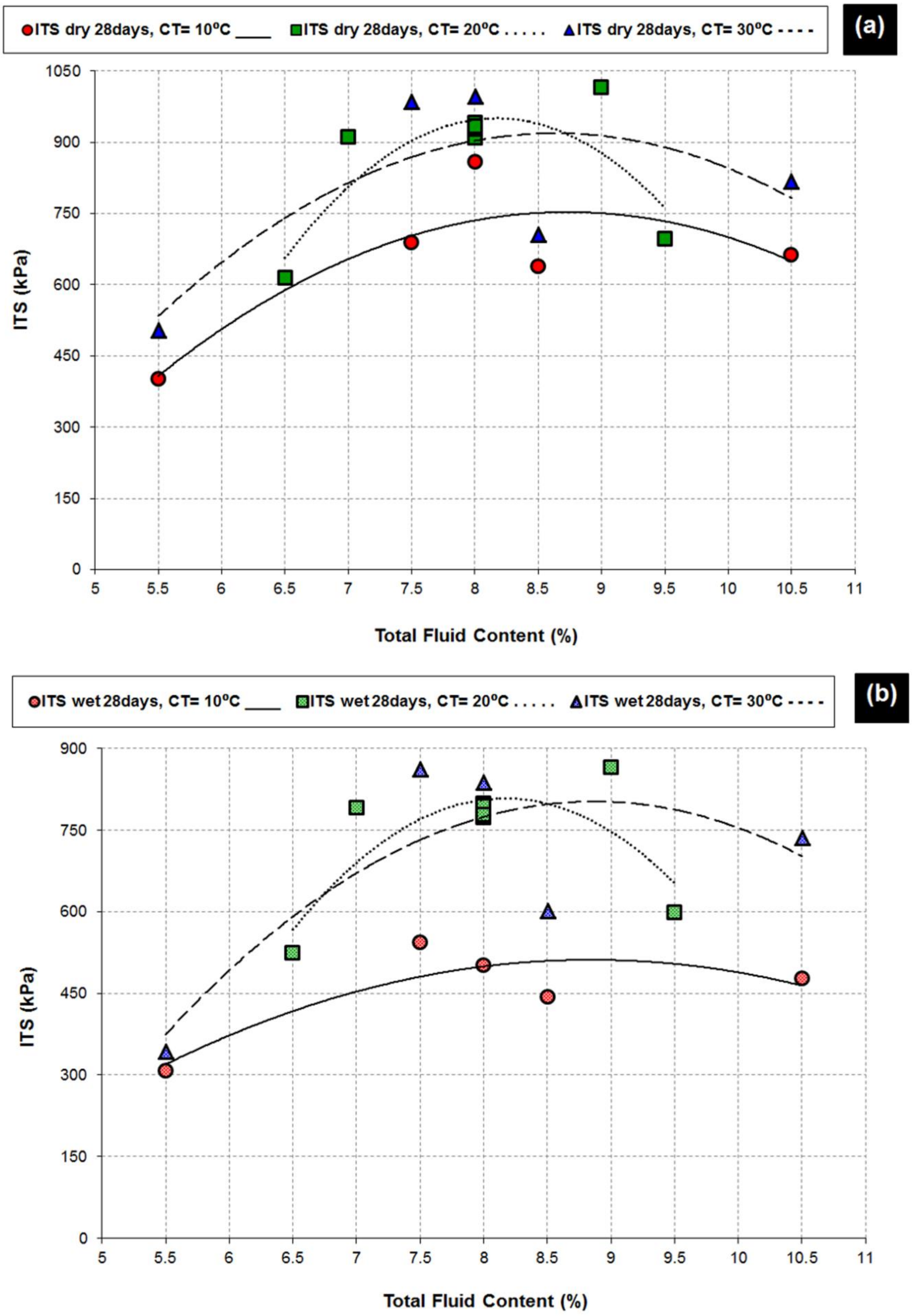

Fig. 5. The relation between ITS and total fluid content of CBEMs under different CT (a) dry condition and (b) wet condition.

Based on the developed model, Fig. 6 and 7 present the interaction effect of BEC and PWC on ITS values under different CT for dry and wet specimens, respectively.

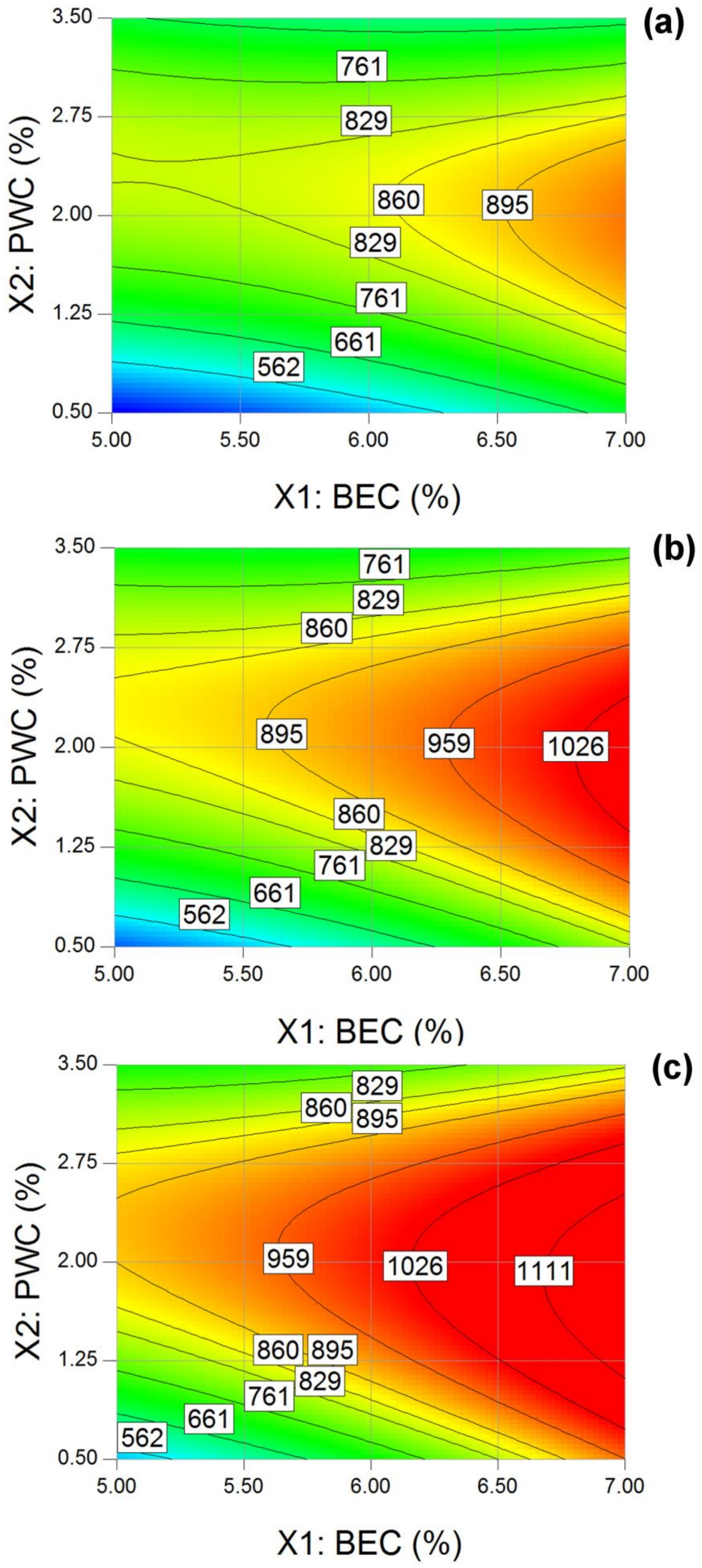

Fig. 6. Contour plots of ITS dry$_{28\ days}$ versus BEC and PWC; (a) CT =10^{o}C, (b) CT =20^{o}C and (c) CT =30^{o}C.

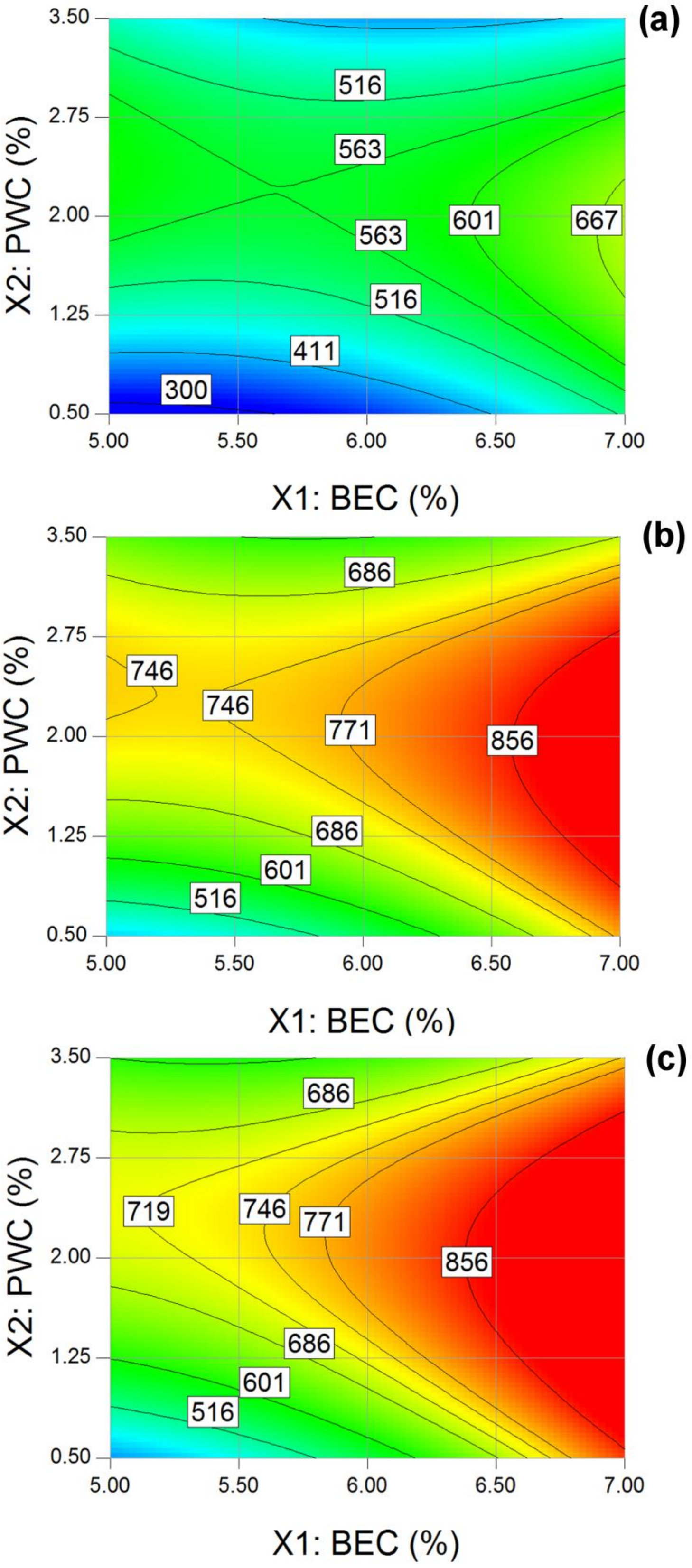

Fig. 7. Contour plots of ITS wet$_{28\ days}$ versus BEC and PWC; (a) CT =10^{o}C, (b) CT =20^{o}C and (c) CT =30^{o}C.

From Fig. 6 and 7, at a given CT, the results again indicate that ITS values significantly increase by increasing PWC from 0.50% to 2.375% and always increase with BEC while they markedly decrease by increasing the PWC from 2.375% to 3.5%. Over all, increasing ITS was observed by increasing BEC and CT. The response surface presented in Fig. 6 and 7 shows distorted parabolic contours which are obtained in cases with fewer interactions between independent variables [41, 42]. This means that the optimum region is less clear than was the case of ITSM. Optimum PWC is between 1.5- 2.5%, but optimum BEC may be around 6.5- 7.0% or higher. The effect of CT on these optimum is again slight and both dry and wet data sets present a similar picture. A more general conclusion from both ITSM and ITS, is that the interaction of BEC and PWC (and to a lesser extent CT) is complex and that CBEMs must be carefully and accurately designed. This conclusion is consistent with the findings of Gómez-Meijide and Pérez [2].

4.2. Analysis of volumetric responses

A volumetric analysis was carried out to determine the void content and dry density present in each mix.

4.2.1. Air voids:

The results are presented in Table 6. The air voids ranged from 10.82% to 18.34%, higher than 9 to 14% suggested by others [4, 5, 25]. Fig. 8 shows the relation between the total fluid content and resulting air voids content.

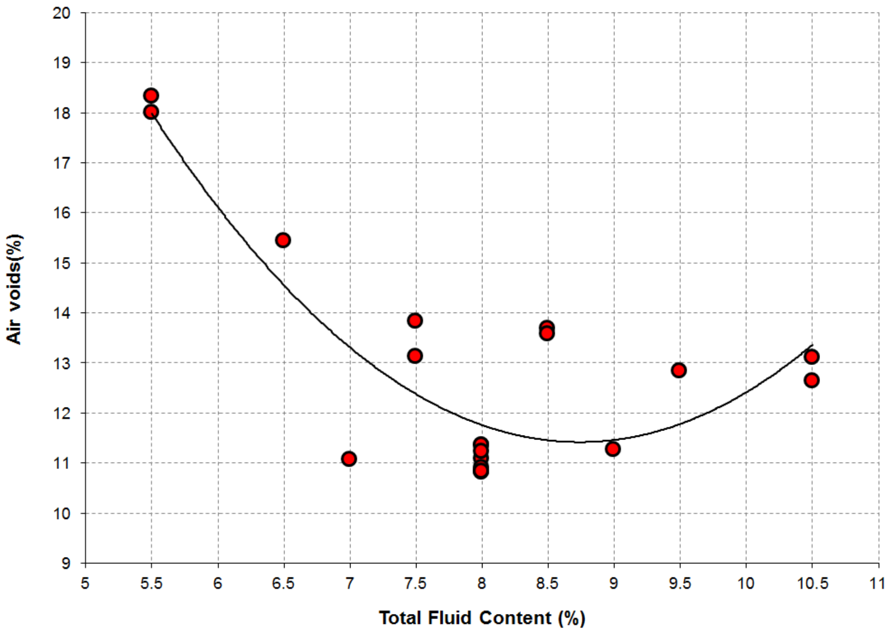

Fig. 8. The relation between air voids and total fluid content of CBEMs.

The highest values of air voids were observed at the lowest total fluid content, whereas the lowest air voids values were found at 8% to 9% total fluid content. The results imply the role of fluid inside the CBEM to determine the degree of compatibility of mixes.

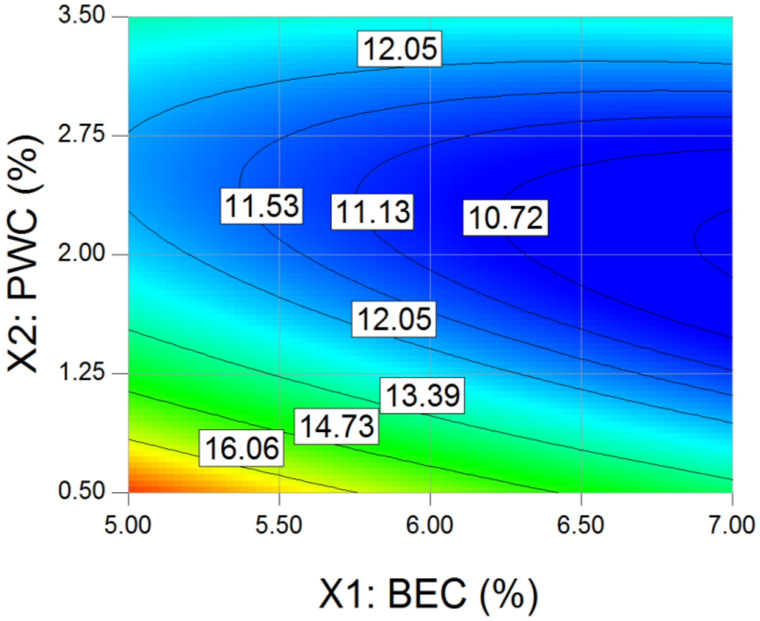

Fig. 9. Contour plot of air voids versus BEC and PWC.

Based on RSM modelling, Fig. 9 presents a contour plot for the air voids measurement in which it is clear that the individual effects of BEC and PWC are both important. Air voids values decreased when increasing the BEC from 5.0% to 7.0% and markedly decreased when increasing the PWC from 0.50% to 2.0% then significantly increased when increasing PWC from 2.0% to 3.5%. As for ITS there is a clear optimum region for PWC, about 1.5- 2.5%, but optimum BEC would appear to be at 7% or more and is therefore less clear on the plot. It is evident that PWC plays a key role in determining air voids in CBEM.

4.2.2. Dry density:

The dry density results are shown in Table 6. Fig. 10 presents the relation between dry density and total fluid content. Dry density values peak at around 8 to 9% total fluid content.

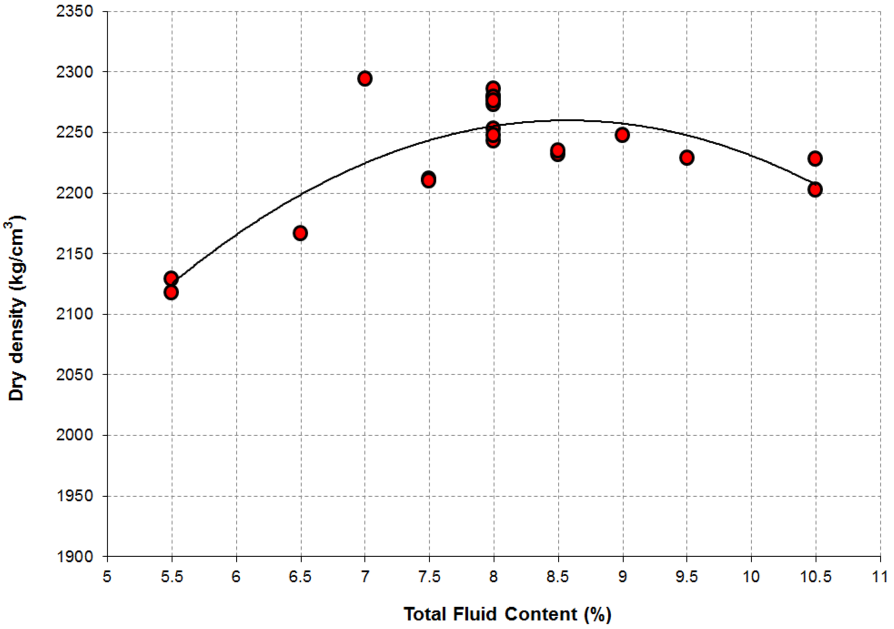

Fig. 10. The relation between dry density and total fluid content of CBEMs.

Based on RSM model, Fig. 11 presents a contour plot for dry density in terms of BEC and PWC.

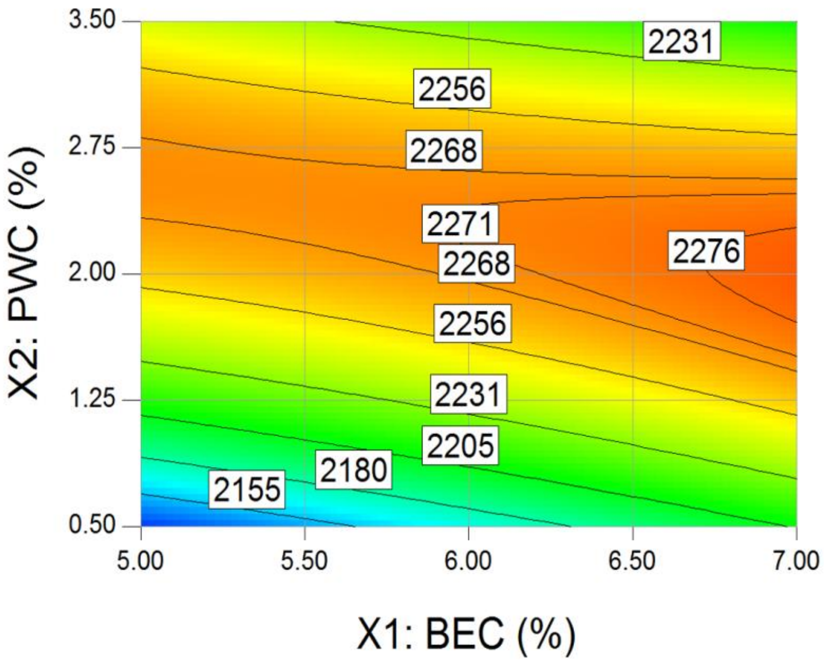

Fig. 11. Contour plot of dry density versus BEC and PWC.

Fig. 11 indicates that the dry density of CBEMs increased dramatically with an increase of PWC from 0.50% to 2.375% then decreased with an increase of PWC from 2.375% to 3.5%. The dry density slightly increased when BEC increased from 5.0% to 7.0%. The optimum PWC is in a narrow band either side of 2%, while optimum BEC is less definite but appears to be close to 7%. Low dry density corresponds to high air voids and poor mechanical performance, while the optimum for each measure approximately coincides.

4.3. Statistical analysis of responses

A statistical analysis was conducted to evaluate mix performance in terms of the above-mentioned tests. A quadratic model was developed for prediction purposes. The quality of the developed model was evaluated based on the coefficient of determination, R^2 and also the standard deviation values. Determination coefficients were obtained as 0.96, 0.98, 0.95, 0.95 and 0.90 for ITSM$_{10\ \text{days}}$, ITS dry$_{28\ \text{days}}$, ITS wet$_{28\ \text{days}}$, air voids and dry density, respectively.

For a good model fit, the coefficient of determination should be a minimum of 0.80. A high R^2 value close to 1.00 demonstrates a desirable and reasonable agreement between the calculated and observed results [43].

An additional tool used to evaluate the developed model was ‘‘adequate precision’’ (AP). *AP* compares the range of the predicted values at the design points to the average prediction error. In this particular case, the *AP* values of the models were 29.3, 35.3, 21.1, 28.5 and 17.6 for, respectively. They are greater than 4 and therefore confirm that the model can be used to navigate the space defined by the CCD [12].

The results of ANOVA analysis presented in Table 7 show that the models' *F-values* of 44.76, 94.77, 32.64, 77.77 and 36.82 and low *P-values*, which mean that the models are statistically significant for $\text{ITSM}_{10\ \text{days}}$, ITS $\text{dry}_{28\ \text{days}}$, ITS $\text{wet}_{28\ \text{days}}$, air voids and dry density, respectively. Only a 0.01% chance exists that a model *F-value* of this magnitude can occur because of noise.

ANOVA results confirm that all the main parameters in the mix design of CBEMs (BEC (X_1), PWC (X_2) and CT (X_3)) have significant effects on mechanical response according to the *t-test* at a 5% significance level ($P < 0.05$). Both BEC and PWC have a significant effect on volumetric response, as shown in Table 7. Insignificant terms, which have limited influence ($P > 0.1$), were excluded from the study to improve the models. The lack of fit (LOF) F-test was also used to evaluate the adequacy of the model. LOF depicts the variation of the data

Table 7: ANOVA for analysis of variance and adequacy of the quadratic model for responses.

Response	SoD	SoS	DoF	MS	*F-value*	*P-value*>F	Comment
ITSM $_{\textbf{10 days}}$							
	Model	8.88E+06	7	1.27E+06	44.76	< 0.0001	
	X_1	6.47E+05	1	6.47E+05	22.84	0.0004	
	X_2	1.68E+06	1	1.68E+06	59.44	< 0.0001	SD= 168.34
	X_3	3.69E+06	1	3.69E+06	130.15	< 0.0001	Mean= 2792.8
	X_1^2	1.78E+05	1	1.78E+05	6.27	0.0277	$R^2 = 0.96$
	X_2^2	1.39E+06	1	1.39E+06	48.94	< 0.0001	Adj. $R^2 = 0.94$
	X_3^2	8.15E+05	1	8.15E+05	28.76	0.0002	AP =29.2
	$X_1 X_2$	5.99E+05	1	5.99E+05	21.14	0.0006	
	Residual	3.40E+05	12	28338.13			
	Lack of Fit	3.318 E+05	7	47402.40	28.76	0.0010	
	Pure Error	8240.83	5	1648.17			
ITS dry $_{\textbf{28 days}}$							
	Model	5.81E+05	8	72628.92	94.77	< 0.0001	
	X_1	1.02E+05	1	1.02E+05	132.84	< 0.0001	
	X_2	10824.1	1	10824.1	14.12	0.0032	
	X_3	57608.1	1	57608.1	75.17	< 0.0001	
	X_1^2	2497.61	1	2497.61	3.26	0.0985	SD= 27.68
	X_2^2	2.49E+05	1	2.49E+05	325.16	< 0.0001	Mean= 802.25
	$X_1 X_2$	50244.5	1	50244.5	65.56	< 0.0001	$R^2 = 0.98$
	$X_1 X_3$	10082	1	10082	13.16	0.004	Adj. $R^2 = 0.97$
	$X_2 X_3$	3960.5	1	3960.5	5.17	0.0441	AP =35.3
	Residual	8430.39	11	766.4			
	Lack of Fit	7781.05	6	1296.84	9.99	0.0116	
	Pure Error	649.33	5	129.87			
ITS wet $_{\textbf{28 days}}$							
	Model	5.767E+005	8	72081.68	32.64	< 0.0001	SD= 46.99
	X_1	99496.64	1	99496.64	45.06	< 0.0001	Mean= 656.95
	X_2	7623.12	1	7623.12	3.45	0.0901	$R^2 = 0.95$

X_3	1.223E+005	1	1.223E+005	55.38	< 0.0001	Adj. $R^2 = 0.93$
X_1^2	10170.78	1	10170.78	4.61	0.0550	AP =21.1
X_2^2	1.163E+005	1	1.163E+005	52.66	< 0.0001	
X_3^2	25776.15	1	25776.15	11.67	0.0058	
X_1 X_2	43087.27	1	43087.27	19.51	0.0010	
X_1 X_3	18294.98	1	18294.98	8.29	0.0150	
Residual	24289.82	11	2208.17			
Lack of Fit	23837.05	6	3972.84	43.87	0.0004	
Pure Error	452.77	5	90.55			
Air voids						
Model	92.97	4	23.24	77.77	< 0.0001	
X_1	11.44	1	11.44	38.28	< 0.0001	SD= 0.55
X_2	16.59	1	16.59	55.51	< 0.0001	Mean= 12.78
X_2^2	57.27	1	57.27	191.66	< 0.0001	$R^2 = 0.95$
X_1 X_2	7.66	1	7.66	25.65	< 0.0001	Adj. $R^2 = 0.94$
Residual	4.48	15	0.30			AP = 28.5
Lack of Fit	4.14	10	0.41	6.12	0.0295	
Pure Error	0.31	5	0.062			
Dry Density						
Model	41661.05	4	10415.26	36.82	< 0.0001	
X_1	940.90	1	940.90	3.33	0.0882	SD= 16.82
X_2	7617.60	1	7617.60	26.93	0.0001	Mean= 2232.35
X_2^2	26718.05	1	26718.05	94.44	< 0.0001	$R^2 = 0.90$
X_1 X_2	6384.50	1	6384.50	22.57	0.0003	Adj. $R^2 = 0.88$
Residual	4243.50	15	282.90			AP = 17.6
Lack of Fit	2636.17	10	263.62	0.82	0.6321	
Pure Error	1607.33	5	321.47			

SoD: source of data; SoS: sum of squares; DoF: degree of freedom; MS: mean square.

X_1 = BEC, X_2 = PWC and X_3 =CT

around the fitted model. For this investigation, the small p values for LOF ($p < 0.05$), as presented in Table 7, show that except the dry density, which was insignificant, other responses including ITSM$_{10\ \text{days}}$, ITS dry$_{28\ \text{days}}$, ITS wet$_{28\ \text{days}}$ and air voids were significant. It is worth noting that while LOF values were significant, reasonable agreement between the predicted and adjusted R^2 were found for all responses such that it can be concluded that the suggested models for all responses can be used to navigate satisfactorily into design space to find optimum mix design parameters. Similar observations were reported by [23, 24, 44].

The final regression models, in terms of the significant influencing factors, are expressed by the following second-order polynomial equations:

$$ITSM_{10\ days} = -10708.36 + 3668.86X_1 + 2630.56X_2 - 157.01X_3 - 254.13X_1^2 - 315.61X_2^2 + 5.44X_3^2 - 182.41X_1X_2 \quad (2)$$

$$ITS\ dry_{28\ day} = 369.05 - 199.68X_1 + 864.71X_2 - 10.74X_3 + 27.93X_1^2 - 124.02X_2^2 - 52.83X_1X_2 + 3.55X_1X_3 - 1.48X_2X_3 \quad (3)$$

$$ITS\ wet_{28\ day} = 1344.55 - 627.82X_1 + 677.51X_2 + 21.09X_3 + 60.81X_1^2 + 60.81X_2^2 - 0.96X_3^2 - 48.92X_1X_2 + 4.78X_1X_3 \quad (4)$$

$$Air\ voids = 33.06 - 2.37X_1 - 10.79X_2 + 1.50X_2^2 + 0.65X_1X_2 \quad (5)$$

$$Dry\ Density = 2268.90 + 9.70X_1 + 27.60\ X_2 - 73.10X_2^2 - 28.25X_1X_2 \quad (6)$$

The significant interactions between variables are presented by 3D-surface plots, as shown in Fig. 12. These plots give more information on the interaction between mix design parameters affects the mechanical and volumetric responses. As clear in Table 7, where the interactive term is not statistically significant, their surface plots were not represented in Fig. 12.

The curvature of the surface plot in Fig. 12 (a) indicates that both BEC and PWC have interaction effect on $\text{ITSM}_{10\ \text{days}}$. Also, Fig. 12 (b-f) depicts the effects of mix design parameters, BEC, PWC and CT, on ITS values in both conditions (dry and wet). Based on the curvature of the surface plots, it is clear that BEC and PWC have the more powerful effect. However, the other interactions of parameters BEC and CT and PWC and CT are clearly observed on the ITS results, which means that ITS is influenced more strongly than ITSM in terms of mix design parameters. This is further confirmed by the result presented in section 4.1.2. Fig 12 (g and h) presents the interaction effects of BEC and PWC on the volumetric properties of CBEMs. The curvature of the surface plot in Fig. 12 (g and h) shows that PWC tended to influence the volumetric responses more markedly than BEC.

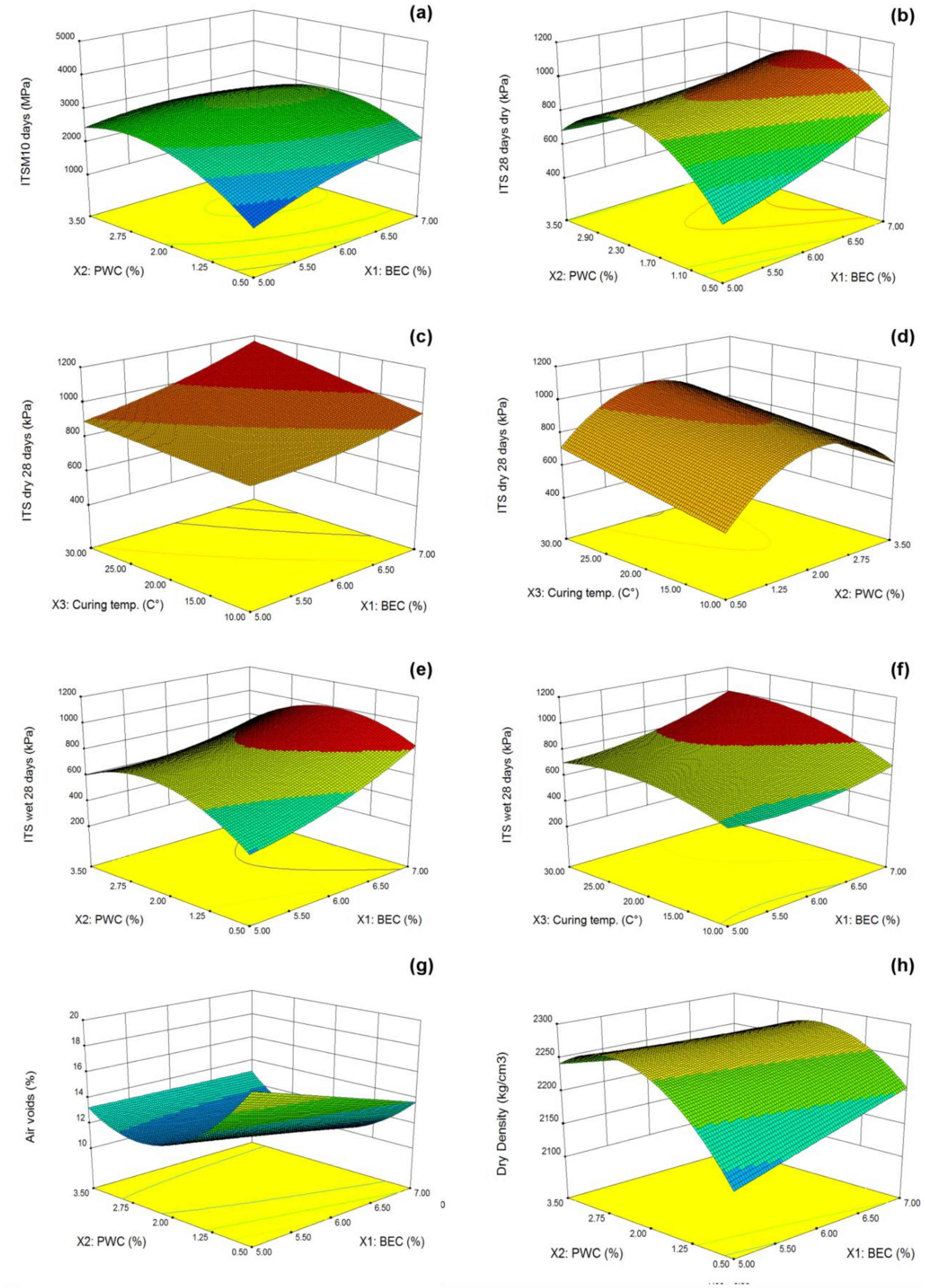

Fig. 12. 3D-surface plots of ITSM$_{10\ \text{days}}$, ITS dry$_{28\ \text{days}}$, ITS wet$_{28\ \text{days}}$, air voids and dry density. (a) ITSM$_{10\ \text{days}}$ versus BEC and PWC. (b) ITS dry$_{28\ \text{days}}$ versus BEC and PWC. (c) ITS dry$_{28\ \text{days}}$ versus BEC and CT. (d) ITS dry$_{28\ \text{days}}$ versus PWC and CT. (e) ITS wet$_{28\ \text{days}}$ versus BEC and PWC. (f) ITS wet$_{28\ \text{days}}$ versus BEC and CT. (g) Air voids versus BEC and PWC. (h) Dry density versus BEC and PWC.

4.4. Optimization the mix design components

An optimization process was carried out to determine the optimum value of BEC, and PWC under different CTs, using the Design Expert 9.0.6.2 software (Stat-Ease Inc., Minneapolis, USA). According to the software optimization step, the desired goal for each mix design parameter (BEC and PWC) was chosen within the range shown in Table 1. The desirable mechanical responses ($\text{ITSM}_{10\ \text{days}}$, ITS $\text{dry}_{28\ \text{days}}$ and ITS $\text{wet}_{28\ \text{days}}$) were defined as being a maximum to achieve the highest performance, the desirable air voids was defined as a minimum and desirable dry density was defined as a maximum in order to achieve a dense mixture with the lowest value of air voids. The derived second order polynomial models were used to interpolate the mix design parameters within the range and based on the desired responses. The results are presented in Table 8.

Table 8: The optimum BEC and PWC at different CT with their responses.

Items	Model prediction			Laboratory experiment
CT (^{o}C)	**T= 10 ^{o}C**	**T= 20 ^{o}C**	**T= 30 ^{o}C**	**T= 20 ^{o}C**
BEC (%)	7.00	6.75	6.58	6.75
PWC (%)	1.96	2.12	2.16	2.12
$\text{ITSM}_{10\ \text{days}}$ (MPa)	2936	3063	4235	3049
ITS dry $_{28\ \text{days}}$ (kPa)	946	1015	1086	1000
ITS wet $_{28\ \text{days}}$ (kPa)	684	883	894	889
Air voids (%)	10.02	10.26	10.42	10.34
Dry density (kg/cm^3)	2279	2276	2275	2278

An additional laboratory experiment was carried out to validate the optimum mix design proportions obtained by the RSM model. The experimental work was performed at CT equal to 20^{o}C. The results in Table 8 demonstrate that the experimental results are close to the predicted results by the developed model at CT of 20^{o}C.

The results in Table 8 show a limited variation of optimum mix design proportions (BEC and PWC) over the range of CT from 10°C to 30°C. The maximum variation of BEC is 0.42% and 0.20% for PWC. The total fluid content (BEC + PWC) for the optimized mixes lies within the range from 8.74% to 8.96%. Therefore, it can be concluded that the optimum proportions (BEC and PWC) tend to be only slightly influenced by CT. Overall, the results are comparable with those published by other authors about the mix design of CBEM [1, 26, 31].

5. Conclusions

The current research introduces a novel performance based mix design approach for CBEM involving mechanical and volumetric properties. A statistical approach was adopted in order to optimize the mix design parameters using RSM. Based on the laboratory experiments and analyses, the following conclusions can be drawn:

1. An alternative mix design approach for CBEMs was investigated using RSM. This approach involves the mechanical and volumetric properties. It was statistically demonstrated that the alternative approach output results were consistent with the laboratory tests.
2. The RSM approach offers a more comprehensive view of the effect of the variation of each mix design parameter on the mechanical and volumetric responses of CBEMs than would otherwise be the case. It has the advantage that all parameters are investigated at one time.
3. The individual effects of BEC and PWC are important, rather than simply total fluid content which is used in conventional mix design method. The results show a lower

strength/ stiffness of CBEM at the same total fluid content and with different BEC/ PWC proportions.

4. The evaluation of stiffness modulus of CBEMs after 10 days is likely to give the designer appropriate information to optimize the mix design of CBEMs in a relatively short time.
5. Based on the optimization by RSM, it can be concluded that the optimum mix design proportions (BEC and PWC) tend to be only slightly influenced by CT.

6. Acknowledgments

The authors would like to acknowledge the University of Nottingham which has funded this research through the Dean of Engineering Research Scholarship for International Excellence. The authors wish to express their sincere gratitude to the technicians at the Nottingham Transportation Engineering Centre for their support and assistance.

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