Optimization of Mechanical Properties of Polymer Concrete and Mix Design Recommendation Based on Design of Experiments

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ABSTRACT: A series of polymer concretes using furan resin, silica aggregates, and microfiller were prepared for statistically designed combinations. The combinations were designed based on the mixture-design concept of design of experiments. The fillers chosen for the present investigation were high-purity naturally occurring silica of different particle sizes, their mix proportion optimized to have minimum void. For each polymer concrete combination, the mechanical properties were studied. Each response (mechanical property) was individually optimized for maximum values and compared with the experimental data. To obtain a single-input combination, having maximum values in all the responses, a combined optimization was done and a mix design was recommended. The coefficient of correlation between the experimental values and predicted values was found to be high, proving the fitness of the selected model. The effect of individual variables on the response was discussed. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 94: 1107–1116, 2004

Key words: resins; fillers; polymer concrete; mechanical properties; optimization

INTRODUCTION

Polymer concrete is a composite material in which the aggregates are bound together in a matrix of polymer binder.[1] The composites do not contain a hydrated cement paste, although portland cement has been used as a filler.[2] Setting time and time for development of maximum strength can be readily varied from a few minutes to several hours by adjusting the temperature and catalyst system.

Even though concrete is the most widely used construction material in the world, because of its low cost, ease of application, and good compressive strength, there are a few shortcomings, most of which are attributable to the portland cement binder. Shortcomings include poor flexural strength, poor tensile strength, high porosity, freeze–thaw deterioration, and destruction by corrosive chemicals,[3] to cite but a few. Most of the concretes in industries are subject to attack by corrosive gases and chemicals being handled there, with the result that they require suitable protection against such corrosive environments. The conventional methods of corrosion protection, including polymeric coatings and linings, lack long-term durability.[4] Developments in civil engineering have shown an interest in overcoming these limitations by incorporating polymers that are known for their good mechanical properties and chemical resistance. Thus polymer concrete composites possessing a unique combination of properties dependent on the formulation were developed.[5–8] These include rapid curing at ambient temperature, high tensile, flexural and compressive strengths, good adhesion to most surfaces, good long-term freeze–thaw durability, low permeability to water and aggressive media, and good chemical resistance.

Different types, properties, and applications of polymer concrete have been widely reported.[9–11] Application and performance of polymer concrete are dependent on the specific polymer binder as well as the type of aggregate and its gradation.[12] A review of the literature shows that the aggregates used in polymer concretes are either fine particles[13] or the particle size distribution is chosen on a theoretical basis,[14] suitable mainly for portland cement concretes. For polymer concretes, it is recommended that the aggregate mix proportion should have minimum void for optimum properties.[15,16]

Because polymer concrete is a heterogeneous material, the properties of polymer concrete may be highly variable. Contributions to the variability of the composite material include, but are not limited to, heterogeneity of the aggregate particles and polydispersion of the polymer binder. To optimize the process vari-

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ables suitable for polymer concrete mixtures, a reliable predictive mathematical model for polymer concretes, based on polyester and epoxy resins, has been proposed.[17] In the present study, polymer concretes based on furan resin were optimized and reported.

Design of experiments

Design of experiments constitutes a group of statistical techniques that can be used for model building, model exploitation, and optimizing multivariable systems.[18–20] Two such techniques are the mixture design and response surface methodology, in which the primary approach to the general problem is to optimize a mixture whose properties depend on the proportions of the component materials. In these techniques, rather than selecting one starting point, a set of trial batches covering a chosen range of proportions for each component is set up according to established statistical procedure.[21] Trial batches are performed and results are analyzed using standard statistical methods that yield reliable estimates of parameters from empirical models for each performance criterion. Each response is expressed as an algebraic function of factors. Once a response is characterized by an equation, any number of analyses is possible. It allows calculations to be made of the response at intermediate levels, which were not experimentally studied, and shows the direction in which to move if we wish to change the input levels so as to decrease or increase the response. For instance, the user could determine which mixture proportions would yield a desired response. Similarly the user could optimize any response function subject to constraints on the others, such as determining the lowest cost mixture with strength greater than the specified strength.[22]

Thus the objective of the present investigation is to recommend mix design for furan polymer concrete, by optimizing the input combination for different properties and to predict properties such as compressive strength, flexural strength, tensile strength and splitting tensile strength with high degree of accuracy, based on statistical analysis of experimental data.

EXPERIMENTAL

A method of optimizing the aggregate mix proportion, suitable for use in polymer concretes, was previously described.[15] Synthesis of furan polymer, selection of suitable catalyst for crosslinking the resin, and effect of silane coupling agent are described elsewhere.[23] Among the different furan resins, the furfuryl alcohol formaldehyde resin of mole ratio 1 : 0.5 was taken as the binder for the present investigation. Using this furan resin as one variable, optimized aggregate mix proportion as a second variable, and silica powder (microfiller) as the third variable, 15 combinations were designed using the mixture-design concept of design of experiments. The input variables, range chosen for the study, their coded values, and mixture design combinations are given in Tables I and II. Based on these combinations, polymer concretes for each composition were prepared, cured, and tested at different intervals of time, under identical conditions. The aggregate % shown in Table II is a mixture of three grades: I, II, and VI in the ratio of 39.6 : 33.5 : 26.9 by weight of grades I : II : VI, grade I having particle size from 4.76 to 9.52 mm, grade II having particle size from 2.38 to 4.76 mm, and grade VI having particle size from 0.15 to 0.3 mm. This ratio corresponds to the optimized aggregate mix proportion having the least void. Suffix "a" refers to repeat combinations to account for "lack-of-fit" statistics.

TABLE I
Range of Variables and Their Coded Form

Sample	Variable	Lower limit %	Lower limit Coded value	Upper limit %	Upper limit Coded value
1	Resin	7.5	0	15	0.6
2	Aggregate	75	0	87.5	1
3	Microfiller	5	0	15	0.8

TABLE II
Mixture Design Combinations for Polymer Concretes

Combination reference	Resin (%)	Aggregate (%)	Microfiller (%)
1	7.5	87.5	5
1a	7.5	87.5	5
2	7.5	77.5	15
2a	7.5	77.5	15
3	9.375	83.125	7.5
4	11.25	83.75	5
5	11.25	78.75	10
6	11.25	76.25	12.5
7	11.25	73.75	15
8	13.125	79.375	7.5
9	15	80	5
9a	15	80	5
10	15	75	10
11	15	70	15
11a	15	70	15

Polymer concrete mortars, of different composition as detailed in Table II, were prepared by mixing required quantities of resin with additive, aggregate, microfiller, and catalyst in a slow-speed mechanical mixer fitted with a paddle stirrer. A dry mix of aggregate, microfiller, and catalysts was first made in a powder mixer. Separately, an additive was added to the resin and homogenized. To this, the dry mixed filler was added slowly while stirring at slow speed to ensure that no air bubbles were trapped in the prepared polymer concrete mortar. After complete mix-

TABLE III
Experimental and Predicted Values for Mixture Design Combinations of Polymer Concrete

Combination reference	Compressive strength (kg/cm^2)		Flexural strength (kg/cm^2)		Tensile strength (kg/cm^2)		Splitting tensile strength (kg/cm^2)	
	Experimental	Predicted	Experimental	Predicted	Experimental	Predicted	Experimental	Predicted
1	779	792.15	237	234.63	130	128.53	124.4	122.84
1a	797	792.15	240	234.63	127	128.53	120.2	122.84
2	843	835.47	262	264.10	143	141.54	127	126.40
2a	831	835.47	266	264.10	139	141.54	125.1	126.40
3	830	806.74	207	224.30	126	122.60	136	131.32
4	646	653.98	213	215.25	111	113.77	120.9	120.44
5	666	680.22	225	220.11	120	121.42	128.8	135.11
6	675	694.20	230	227.28	122	124.07	135.8	136.87
7	712	708.77	239	237.62	129	125.93	138.6	134.92
8	710	682.33	221	212.65	120	119.12	123.9	124.23
9	726	737.59	220	214.89	124	122.00	103.7	103.21
9a	744	737.59	208	214.89	121	122.00	101.6	103.21
10	760	769.56	216	216.19	129	129.23	127.2	123.33
11	812	803.85	225	230.15	134	133.32	129.4	128.60
11a	803	803.85	232	230.15	132	133.32	125.8	128.6

ing of the resin and filler, the prepared polymer concrete mortar was used for casting specimens for studying compressive, flexural, tensile, and splitting tensile strengths, adopting standard techniques[24–27] for all the combinations given in Table II. Specimen casting were done using a vibrating table, operating under high-frequency electrical vibrators.

In the mixture-design approach the total amount of the input variables was fixed and constrained to sum 100. For each statistical combination, all properties of interest were measured and empirical models for each property as a function of the input variables were determined from regression analysis. The advantage of the mixture approach is that the experimental region of interest is more naturally defined. To simplify calculations and analysis, the actual variable ranges were transformed to dimensionless coded variables with a range of ± 1. Intermediate values were also translated similarly. The variables X_1, X_2, and X_3 were codified using the formula

$$\text{Pseudo} = (R_i - L_i)/(1 - L) \quad (1)$$

where $R_i = A_i/\sum A_i$, L_i is the lower constraint in real value, L is the sum of lower constraints in real value, A is the actual value, and A_i is the total of actual values.

The mathematical relationship between three independent variables and the response can be approximated by the second-order polynomial

$$Y = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \quad (2)$$

where β_1, β_2, and β_3 are linear coefficients; β_{12}, β_{13}, and β_{23} are cross-product coefficients; β_{11}, β_{22}, and β_{33} are quadratic coefficients; X_1 refers to the coded value of resin; X_2 refers to the coded value of aggregate; and X_3 refers to the coded value of microfiller.

A resin content of 9.375% in polymer concrete mortar was found to be sufficient to confer good workability suitable for many applications. Because polymer concrete mix with a lesser resin content is preferred, a lower limit of 8.5% was selected, based on the workability of the polymer concrete mortar. An upper limit of 12% was fixed on the basis of segregation observed in the polymer concrete mortar, even in the case of composition having 13.125% binder content and extensive segregation in the case of 15% binder

TABLE IV
Model Summary Statistics

Property	SD	R^2	Adjusted R^2	Predicted R^2
Compressive strength	16.86276	0.951299	0.924243	0.894916
Flexural strength	7.840583	0.870658	0.798802	0.727356
Tensile strength	2.495449	0.938797	0.904796	0.827659
Splitting tensile strength	3.565718	0.924001	0.881779	0.794089

TABLE V
ANOVA for Compressive Strength

Source	Sum of squares	DF	Mean square	F value	Prob > F
Model	49989.76	5	9997.952	35.1604	<0.0001
Linear mixture	11320.68	2	5660.338	19.90605	0.0005
AB	28888.6	1	28888.6	101.5943	<0.0001
AC	28624.18	1	28624.18	100.6644	<0.0001
BC	2.476754	1	2.476754	0.00871	0.9277
Residual	2559.173	9	284.3526		
Lack of fit	2122.673	5	424.5347	3.890352	0.1063
Pure error	436.5	4	109.125		
Cor total	52548.93	14			

trials, which is not acceptable for castable applications. For aggregates and microfiller, the ranges practically studied were retained for optimization studies. Thus, for combined optimization of all properties, the following ranges for the three variables were selected:

1. Resin = 8.5 to 12 wt %
2. Aggregate = 70 to 87.5 wt %
3. Microfiller = 5 to 15 wt %

RESULTS AND DISCUSSION

For each polymer concrete combination, designed on the basis of mixture-design concept of design of experiments, the properties studied are compressive, flexural, tensile, and splitting tensile strengths and are detailed in Table III. The experimentally studied responses at different intervals of time were previously reported[23] and, based on the results observed, the 7-day value was analyzed statistically using design-expert software (Statease Inc., Minneapolis, MN).

The individual response, such as compressive strength, can be predicted by the regression equations given below, which express the relationship between the input variables and the response:

$$Y_{CS} = 2044.794X_1 + 792.1593X_2 + 874.0463X_3 - 2414.91X_1X_2 - 335.42X_1X_3 - 14.2103X_2X_3 \quad (3)$$

$$Y_{FS} = 306.872X_1 + 234.632X_2 + 319.4057X_3 - 207.03X_1X_2 - 342.505X_1X_3 - 77.4687X_2X_3 \quad (4)$$

$$Y_{TS} = 256.3802X_1 + 128.5375X_2 + 143.0912X_3 - 250.394X_1X_2 - 238.116X_1X_3 + 19.15619X_2X_3 \quad (5)$$

$$Y_{STS} = -15.2457X_1 + 122.8415X_2 + 90.09536X_3 + 161.517X_1X_2 + 341.6094X_1X_3 + 90.96555X_2X_3 \quad (6)$$

where the suffixes CS, FS, TS, STS refer to compressive, flexural, tensile, and splitting tensile strengths, respectively.

The coefficients of the individual variables in each equation give a measure of the effect of variables on the predicted response. For variables having coefficients of large magnitudes, even a marginal increment will give a significant change in the response. However, for variables having coefficients of lower magnitudes, even a large increase will result in only a small change in the response. Thus significant and less significant variables can be identified from the equation. By solving the equation, an individual property can be maximized or minimized, leading to an optimum combination of inputs for polymer concrete.

The above equations are based on the quadratic model, suggested among other models such as linear,

TABLE VI
ANOVA for Flexural Strength

Source	Sum of squares	DF	Mean square	F value	Prob > F
Model	3724.327	5	744.8655	12.11661	0.0009
Linear mixture	3083.73	2	1541.865	25.08127	0.0002
AB	212.3189	1	212.3189	3.453758	0.0961
AC	615.6565	1	615.6565	10.01479	0.0115
BC	73.60856	1	73.60856	1.197379	0.3023
Residual	553.2727	9	61.47474		
Lack of fit	444.2727	5	88.85454	3.260717	0.1376
Pure error	109	4	27.25		
Cor total	4277.6	14			

TABLE VII
ANOVA for Tensile Strength

Source	Sum of squares	DF	Mean square	*F* value	Prob > *F*
Model	859.6879	5	171.9376	27.61044	<0.0001
Linear mixture	491.7635	2	245.8818	39.4847	<0.0001
AB	310.5775	1	310.5775	49.8738	<0.0001
AC	297.5641	1	297.5641	47.78405	<0.0001
BC	4.500846	1	4.500846	0.722764	0.4173
Residual	56.04541	9	6.227267		
Lack of fit	37.04541	5	7.409081	1.559807	0.3437
Pure error	19	4	4.75		
Cor total	915.7333	14			

cubic, and two-factor interaction, because this quadratic model fitted well with the experimental data. The fitness of the above model for an individual property can be explained from the model summary statistics, lack-of-fit statistics, and analysis of variance.[20]

Model summary statistics

The model provides several comparative measures for model selection. R^2 statistics, which give a correlation between the experimental response and the predicted response, should be high for a particular model to be significant. Adjusted R^2, which gives similar correlation after ignoring the insignificant model terms, should have good agreement with predicted R^2 for the model to be fit.[20] Table IV details the model summary statistics for the selected models for all four responses. It can be observed that the coefficient of correlation (R^2) is high, which indicates a high degree of correlation between the experimental and predicted response. Only $(1 - R^2) \times 100\%$ of total variation in the model has not been accounted for by the model. It can also be seen from the model that the predicted R^2 value is in good agreement with the adjusted R^2 value.

Analysis of variance (anova)

ANOVA for the model gives the sum of squares and degrees of freedom for the model terms from which mean square values of the model terms are calculated. ANOVAs for each response for the selected quadratic model are given in Tables V–VIII. The lack-of-fit test compares the residual error to the pure error from replication and gives *F*-values for all the models. The *F*-value must be lower if a particular model is to be significant. From the *F*-test, it was found that only the quadratic model passed the *F*-test. It can be seen that the model *F*-values are significantly higher than the tabulated *F*-value[18] of 2.96 for the given degrees of freedom at the 95% confidence level. Furthermore, the values of Prob > *F*, which is the probability that the model will not explain the variations in the response, are considerably less than 0.05, indicating that the quadratic model is highly significant.

The predicted responses obtained from the regression equations given in eqs. (3) to (6), are compared with the experimental values and are given in Table III. The close correlation between the experimental values and predicted values proves the fitness of the selected model. The above factors confirm the fitness of the selected model in analysis of the experimental data.[20]

Effect of variables

Contour plots are response surface plots that help to identify the type of interactions between the test variables on the response and are given in Figures 1–4. Three-dimensional (3D) contour plots help us to visu-

TABLE VIII
ANOVA for Splitting Tensile Strength

Source	Sum of squares	DF	Mean square	*F* value	Prob > *F*
Model	1391.227	5	278.2454	21.88437	<0.0001
Linear mixture	696.9826	2	348.4913	27.4093	0.0001
AB	129.229	1	129.229	10.16404	0.0110
AC	612.4399	1	612.4399	48.16922	<0.0001
BC	101.4915	1	101.4915	7.982444	0.0199
Residual	114.4291	9	12.71434		
Lack of fit	95.11908	5	19.02382	3.940718	0.1042
Pure error	19.31	4	4.8275		
Cor total	1505.656	14			

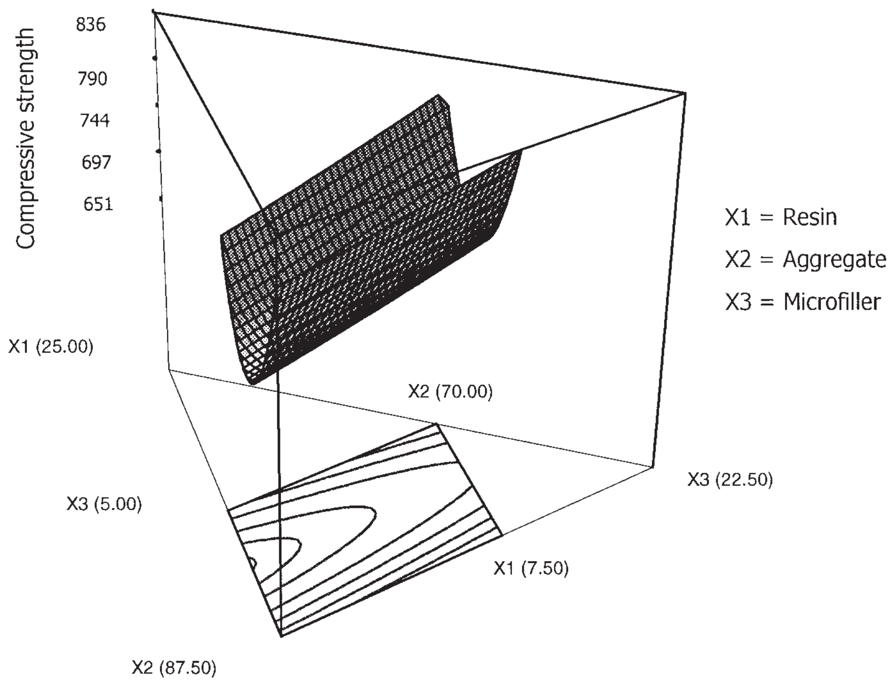

Figure 1 3D contour diagram showing the effect of individual variables on compressive strength.

alize the effect of variables in 3D space. The elliptical nature of the contour plots indicates that the interaction between the corresponding variables is significant. The optimum values are obtained at the point of intersection of the lines formed by joining the locus of the tip of the curve.[20]

Trace plots indicate the effect of changing each mixture component while holding all other components in a constant ratio, and are shown in Figures 5–8. The response is plotted while moving along an imaginary line from a reference blend to the vertex of the component being incremented. The default

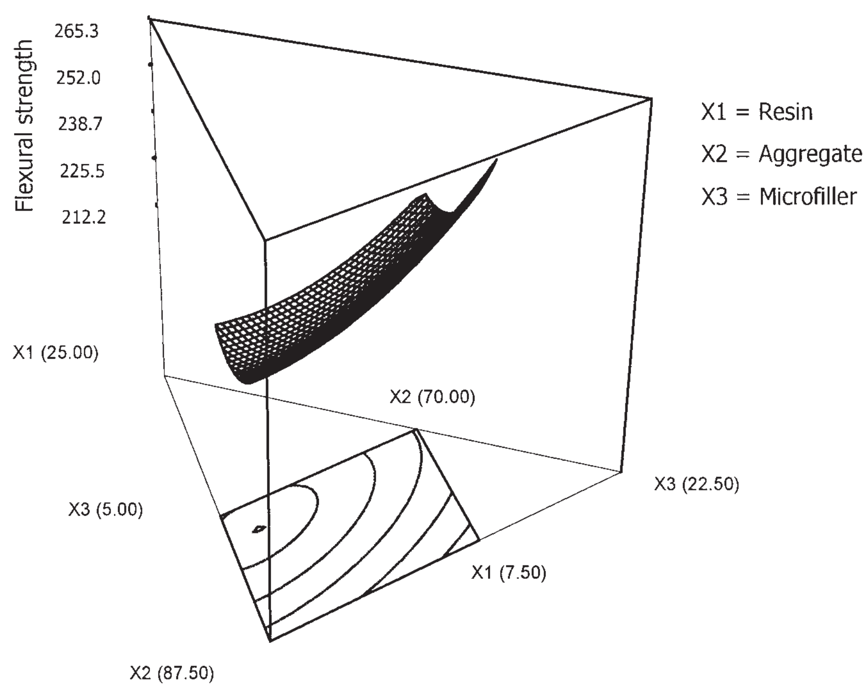

Figure 2 3D contour diagram showing the effect of individual variables on flexural strength.

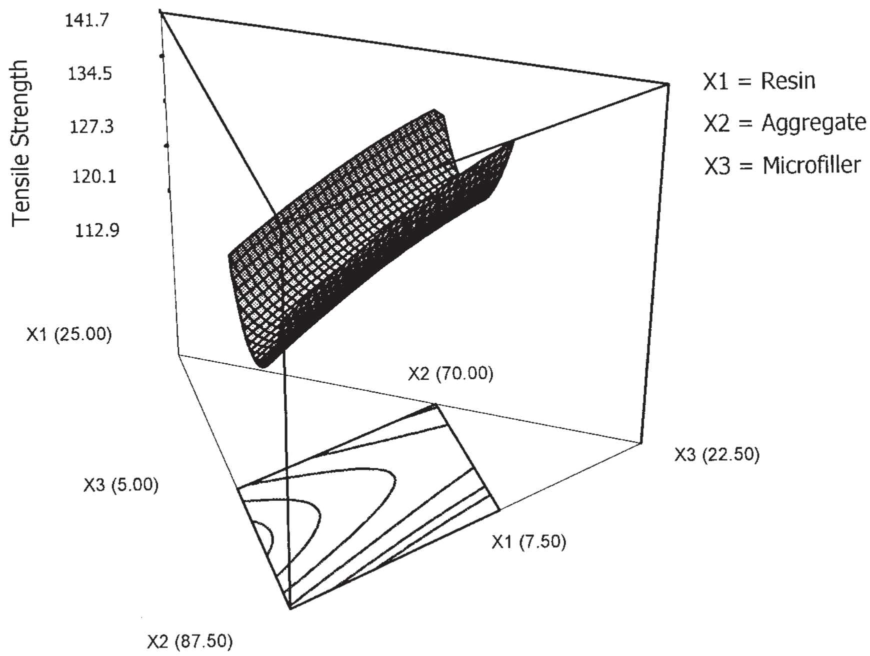

Figure 3 3D contour diagram showing the effect of individual variables on tensile strength.

reference is the centroid of the design. A steep slope or curvature in an input variable indicates a relatively high sensitivity of response. These influential variables are good ones to select for the axes on the 2D and 3D contour plots.

Optimization studies

Polymer concrete inputs, optimized for maximum in individual property as well as for all the properties (combined), are given in Table IX. It may be observed

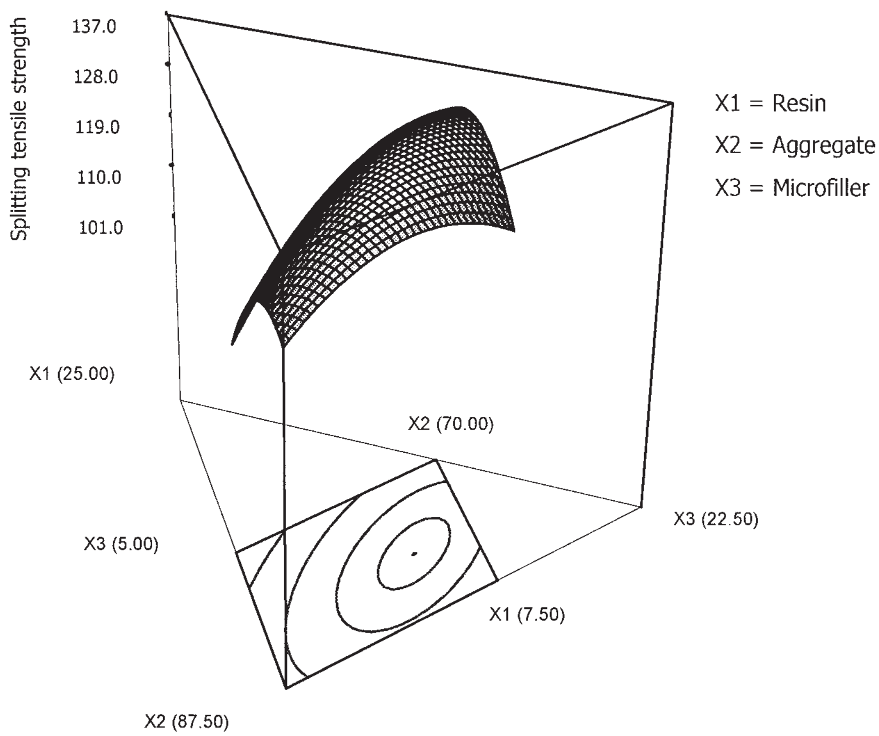

Figure 4 3D contour diagram showing the effect of individual variables on splitting tensile strength.

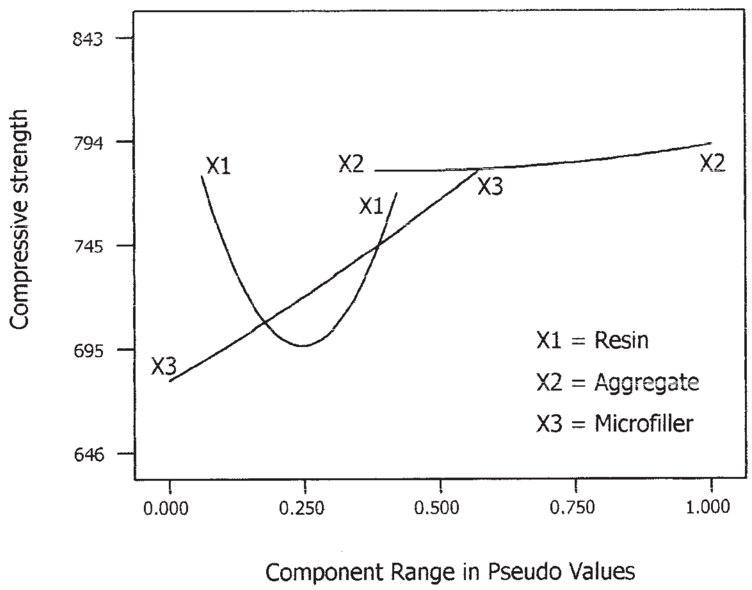

Figure 5 Trace plot showing the effect of variables on the compressive strength.

that for compressive, flexural, and tensile strengths, the resin content corresponding to the optimum was different from that of the practically studied maximum. Splitting tensile strength, when individually optimized as the maximum strength, was obtained corresponding to a resin input of 8.5%, which is in the lower limit of the range chosen for optimization. To have single-input combination for maximum values in all the properties, a combined optimization was carried out. The resin content for combined optimization was found to be 8.5% and the predicted optimum was equal to that obtained when individually optimized except in the case of splitting tensile strength, where it is of slightly less value, but within practical limits for such heterogeneous systems.[28]

CONCLUSIONS

The mechanical properties of polymer concretes based on furan resin, whose raw material is abundantly available from renewable resources, were studied. The fillers used for the study are unique in

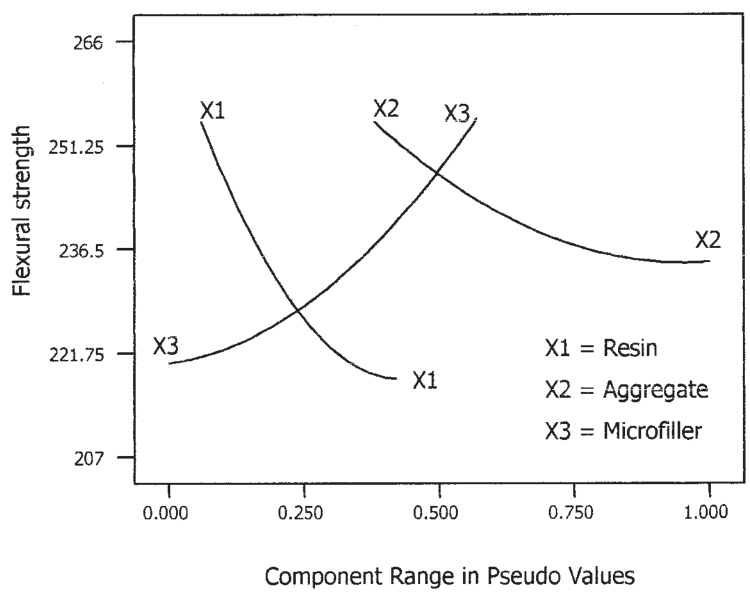

Figure 6 Trace plot showing the effect of variables on the flexural strength.

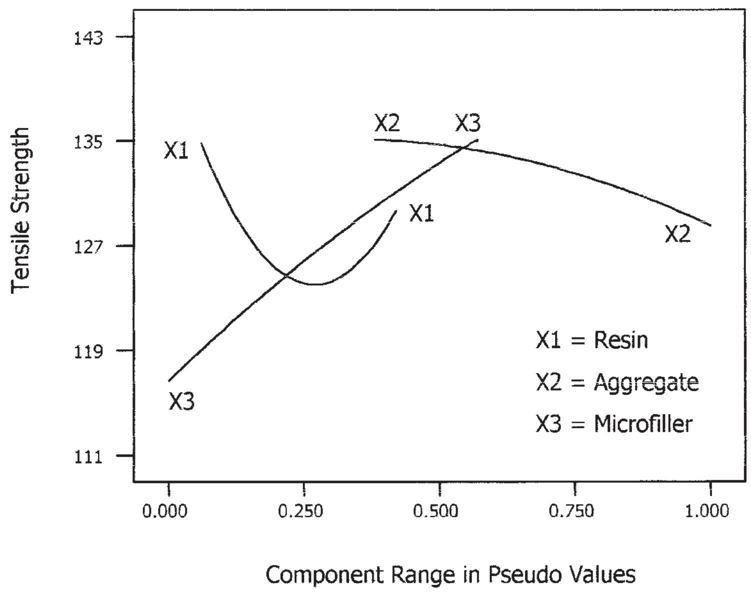

Figure 7 Trace plot showing the effect of variables on the tensile strength.

terms of minimum void content. By the use of statistical techniques, the experimental data were analyzed and an optimum mix design was recommended for each property individually. From the combined optimization results, a single combination is recommended that has an optimum value in all properties. Further, compressive, flexural, tensile, or splitting tensile strengths can be determined for any input combination within the experimental range of variables, by substituting the coded values in the corresponding regression equations. Based on this, depending on the application requirement and the significance of the property, the input combination can be chosen with a high degree of accuracy without the need for conducting actual experiments. The high correlation coefficient confirms the significance of statistical techniques in modeling experimental data.

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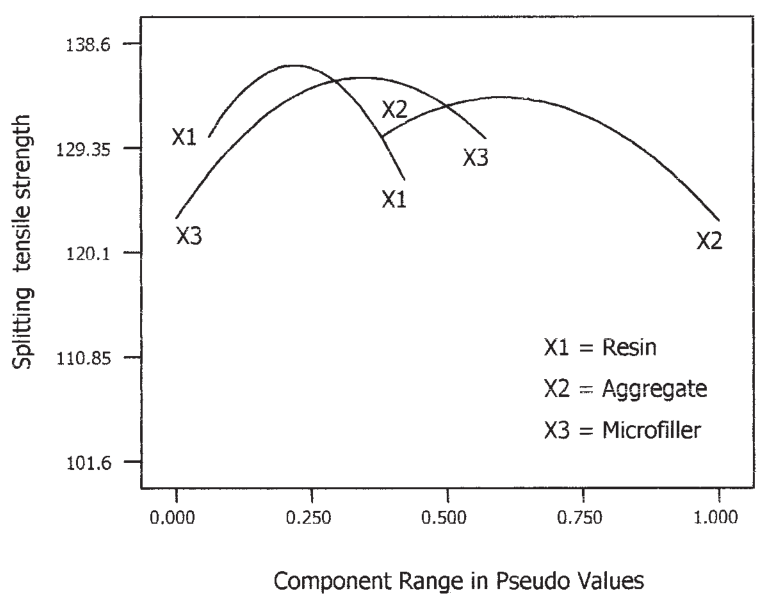

Figure 8 Trace plot showing the effect of variables on the splitting tensile strength.

TABLE IX
Experimental Maximum, Predicted Optimum for Individual Properties, and Optimum Combination for All Properties

Sample	Property	Description	Inputs (%) Resin	Aggregate	Microfiller	Value
1	Compressive strength, kg/cm^2	Experimental maximum	7.5	77.5	15	843
		Predicted optimum	8.5	76.5	15	780
2	Flexural strength, kg/cm^2	Experimental maximum	7.5	77.5	15	266
		Predicted optimum	8.5	76.5	15	255
3	Tensile strength, kg/cm^2	Experimental maximum	7.5	77.5	15	143
		Predicted optimum	8.5	76.5	15	135
4	Splitting tensile strength, kg/cm^2	Experimental maximum	11.25	73.75	15	139
		Predicted optimum	10.75	77.06	12.19	137
5	Compressive strength	Predicted optimum	8.5	76.5	15	780
	Flexural strength					255
	Tensile strength					135
	Splitting tensile strength					130

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