Silicon (2020) 12:1513–1528
https://doi.org/10.1007/s12633-019-00247-w

ORIGINAL PAPER

Response Surface Modeling and Optimization of Gelcast Fused Silica Micro Hybrid Ceramic Composites

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Received: 29 June 2019 / Accepted: 12 August 2019 / Published online: 17 August 2019

Abstract
In the current work, Response Surface Methodology (RSM) was effectively implemented to the technique of fabrication of fused silica (SiO_2) advanced hybrid ceramic composites using gelcating, a near net shaping process. The influence of process variables like solid loading (SL), monomer ratio (MR) and monomer content (MC) on flexural strength (FS), porosity (P) and dielectric constant (ε) was explored using central composite face centered design (CCFCD) with six centre points approach to an experimental work. The interaction between the process parameters on the responses was studied and modeled. Three mathematical models were created through RSM related independent process parameters to portray the flexural strength, porosity and dielectric constant as the responses. The acceptability of the derived model was examined with the help of Analysis of Variance (ANOVA) at 95% confidence level and through other parameters. The statistical analysis of the outcomes demonstrated that in extend considered the three input factors have critical impact on the response. The RSM models obtained have high R^2 values (0.999, 0.994, and 0.995) which demonstrate exceptional relation between the actual and predicted models. The optimum values obtained through RSM was experimentally confirmed and it was 87.36 MPa for flexural strength, 35.66% for porosity and 4.783 for dielectric constant (ε) obtained at 52 vol% solid loading, 15:1 monomer ratio and 5 wt% monomer content respectively.

Keywords RSM. Gelcasting. Fused silica. Flexural strength. Porosity. Dielectric constant. Additives

1 Introduction

Structural ceramic materials have been considered suitable materials for radomes and electromagnetic windows used as parts of space crafts. These materials require good mechanical, dielectric, thermal properties and excellent ablation resistance to withstand harsh reentry environment [1]. Among the structural ceramics, fused silica, silicon nitride, Boron nitride, Alumina and zirconia are candidate materials for such aerospace applications. Fused SiO_2 ceramics have high resistance to corrosion, good thermal shock resistance (withstands greater than twenty thermal cycles between room temperature and 1000 °C), very low coefficient of thermal expansion (0.54×10^{-6}/ °C, 25–800 °C), low ε (3–4 from 25 to 1000 °C), low dielectric loss (~0.0004), less weight loss rate (0.0025 mg/s at 2500 °C) and excellent insulating property (resistivity = 10^{15} Ωm at room temperature). These characteristics make fused SiO_2 potential material for antenna windows in aerospace, electronics, metal and polysilicon industries [2–4]. The strength of the SiO_2 ceramics is relatively low which is not enough to achieve the required characteristics of advanced hypersonic space craft applications. In order to improve these shortfalls, additives such as Al_2O_3 [1], Zr [3] Si_3N_4 [5] and BN [6] were included to give appreciable amount of improvement in mechanical properties of ceramic composites. The added reinforcements possess good dielectric, mechanical and chemical properties. A lot of porous ceramic manufacturing routes are available like forming, storing of pre sintered granules or fibers, aero gel or sol-gel and pyrolysis of several organic reinforcements and gelcasting [7, 8]. Oak Ridge National Laboratory introduced the latest ceramic forming technique called gelcasting in 1990 [9]. Gelcasting is a process borrowed from the traditional ceramic forming. The process is

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simple and no special equipment is required. The complex and complicated shapes with different sizes are prepared for various industrial applications using gelcasting which minimizes the huge machining costs [10–12].

In this paper, fused silica is combined with Si_3N_4 and Al_2O_3 as additive to improve the mechanical properties without disturbing other properties. The strength of these additives is excellent but dielectric properties are not on par compared to fused silica. These ceramics offer low ε (~5) for the applications in electromagnetic wave transparent windows.

Conventional optimization techniques are used to optimize one single factor at a time. These techniques are time consuming, complicated, require huge experimental data sets and invite no interactions among the parameters. Therefore some experimental runs and interaction among the parameters are greatly needed. So, some statistical techniques are being considered to build the interactions among the process parameters for generating response. In such techniques, RSM is one of the statistical modeling methods that can be applied to get the relation between the input variables and output. RSM is widely used in engineering problems and the industrial world to know the effect of process parameters on the outputs. RSM comprises observational methods committed to the advancement of connection existing between the procedure parameters of an investigation and the deliberate reactions, helpful for creating, enhancing and streamlining the procedure by directing low quantities of tests [13–15]. RSM is utilized to decide the factor levels that will at the same time fulfill an arrangement of wanted detail and to decide the ideal blend of components that yield a coveted reaction and depict that the reaction is close to the ideal. It is similar to determining how a particular output is influenced by the alterations in the level of the parts over the foreordained levels of interest and to attain a quantitative appreciation of the system [16]. Neural network modeling is applied to model gelcasting technique for the preparation of porous silicon nitride (Si_3N_4) ceramics [8]. RSM is used for the optimization of the sintering method for the fabrication of calcia partially stabilized zirconia (CaO-PSZ) [13]. Tougher ZTA ceramics were prepared through RSM optimization [14]. RSM is applied for the optimization of processing of Polydimethylsiloxane (PDMS) ceramic composite pervaporation membranes [17]. The sintering method is optimized for the fabrication of magnesia partially stabilized zirconia (Mg-PSZ) using RSM [18]. RSM is successfully applied for the optimization of Tetracycline photo degradation by $Bi_{3.84}W_{0.16}O_{6.24}$-graphene oxide (BWO-GO) [19].

Limited literature is available on the application of RSM for the optimization of gelcasting process. In the present experimental analysis, CCFCD with 6 centre points is adopted. Mathematical modeling and optimization of the gelcasting technique using RSM is successfully applied.

2 Experimentation

2.1 Basic Materials

The commercially available ceramic powders and other chemicals utilized in the present study are represented in Table 1.

Table 1 Base materials utilized for the preparation of hybrid ceramic composites

S. No.	Material	Role	Average particle size	Density (g/cm^3)	Provider
1	Fused silica powder (SiO_2)	Ceramic Powder	1–5 μm	2.2	M/S Ants Ceramics Pvt. Ltd., Thane- India
2	Silicon Nitride (Si_3N_4)	Ceramic Powder	1–5 μm	3.44	M/S Ube Industries, Japan
3	Alumina (Al_2O_3)	Ceramic Powder	50–200 μm	3.98	Alfa Aesar- USA
4	Methacrylamide CH_2-$C(CH_3)CONH_2$	Monomer		1.235	Sigma Aldrich Chemie- Germany
5	N N′-Methabisacrylamide (MBAM) ($C_7H_{10}N_2O_2$)	Cross linker		1.24	Sigma Aldrich Chemie, Germany
6	Darvan 821A	Dispersant		1.25	Vanderbilt Minerals LLC- USA
7	Polyethelene glycol 400 (PEG-400) $H(OCH_2CH_2)nOH$	Surfactant		1.126	Sigma Aldrich Chemie- Germany
8	Tetramethylethylenediamine- (TEMED) $C_6H_{16}N_2$	Catalyst		1.982	Sigma Aldrich Chemie- Germany
9	Ammonium persulfate (APS) $H_8N_2O_8S_2$	Initiator		0.775	Alfa Aesar- USA
10	Diluted Nitric acid (HNO_3) and Sodium hydroxide (NaOH)	pH adjustment			S. D. fine chemicals, India

2.2 Fabrication Process

The manufacturing technique adopted for the processing of ceramics is represented in Figure 1. The ceramic specimens were manufactured at various SL, MR and MC. First distilled water was mixed with dispersant Darvan 821A (1 wt% of MAM + MBAM), PEG, monomers (10 wt% of SiO_2) and ceramic powders [10]. The dispersing medium was stirred with the help of magnetic stirrer for seven hours. Deaeration was applied to remove the entrapped air molecules from slurry. APS and TEMED were mixed to the slurry which performs as initiator and catalyst respectively. After solution was poured into glass mold and after polymerization, the green samples were demolded. Green bodies were dried in a supervised temperature and humidity heater for 22 h. Later, binders were burnt out at 600 °C for 2 h with a heating rate of 4 °C/min and followed by pressure less sintering at 1250 °C for 1 h under nitrogen environment.

2.3 Characterization

A top high velocity diamond cut-off saw (MTI, Corporation, USA) was used to cut the sintered hybrid ceramic samples of bars of dimensions $3 \times 4 \times 40$ mm^3 from a volume of dimensions $5 \times 50 \times 50$ mm^3. 3-point bend experiment was utilized to examine the flexural strength with cross head speed of 0.5 mm/min and a length of 30 mm on universal testing machine (H10K-S, Tinus Olsen testing machine company, USA). Five specimens were examined to measure the mean value.

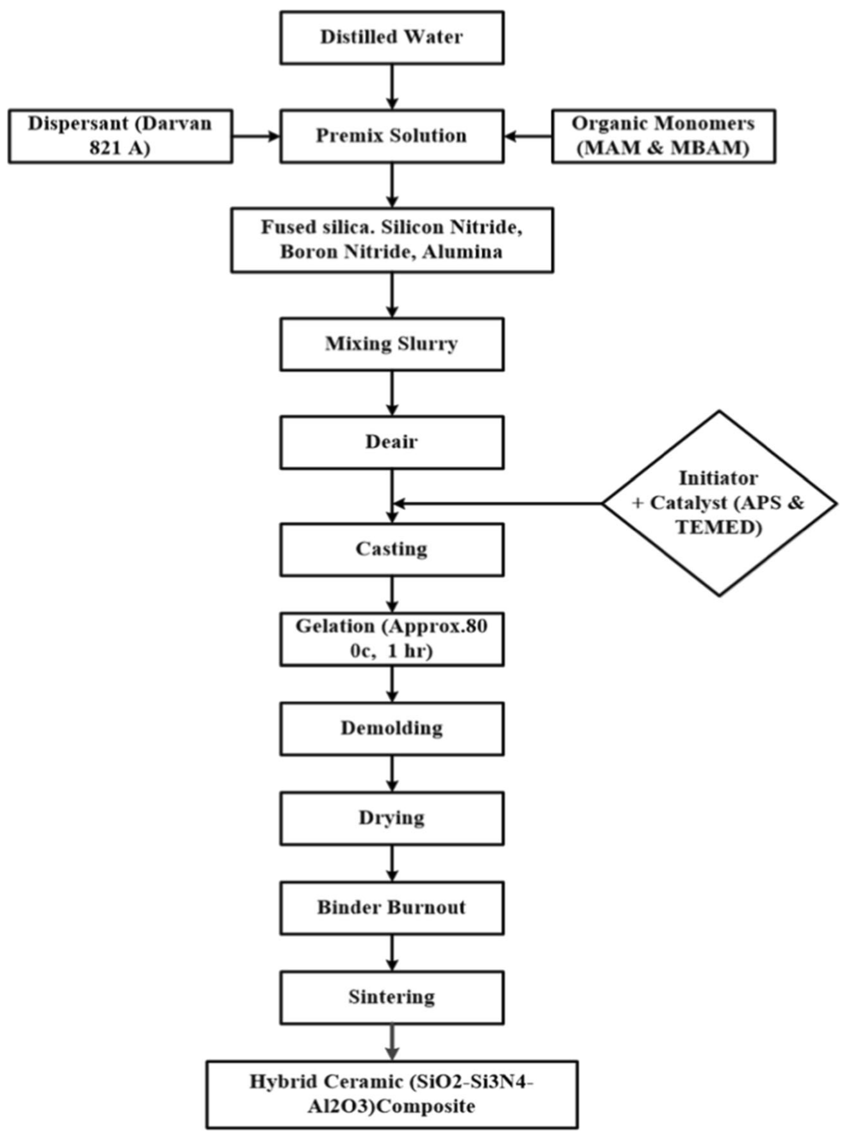

Fig. 1 Gelcasting process

Table 2 Experimental design of process variables and their levels

Symbol	Factors	Levels		
		1	0	−1
A	Solid loading (vol%)	48	50	52
B	Ratio of monomers (MAM:MBAM)	5:1	10:1	15:1
C	Monomers content (Wt%)	5	10	15

Archimedes principal was adopted to find porosity of the sintered specimen. Porosity was measured using ASTM standard C914–09 using Eq.1:

$$porosity = \frac{w_3 - w_1}{w_3 - w_2} \quad (1)$$

Where W_1 = Dry weight of the specimen. W_2 = Suspended weight of the specimen. W_3 = Saturated weight the specimen.

Room temperature dielectric constant was measured with the help of an Impedance Analyzer (MTZ-35, Biologic Sciences Instruments Pvt. Ltd., France) at 30 MHz frequency on speciemens of 1.5 mm thick and 10 mm diameter. The experiment was conducted 3 times on each sample to determine the mean dielectric constant [20–23].

2.4 Experimental Design

RSM is a statistical and numerical technique that is utilized for the design and optimization of engineering problems. It is mainly used to find an approximate mathematical model for estimating the future response and to find the process parameters that optimize the predicted model [17]. It usually consists of 3-steps (i) design of experimentation (ii) response modeling through regression (iii) optimization. CCD is a type of RSM. The basis for the development of CCD test design method was two levels full factorial. The quadratic surface generally suits well for the process optimization [13].

The aim of the current experimental analysis is to find the optimum settings of one to one factor influences and simultaneous interaction effects of the input process parameters (SL, MR and MC) that result in optimum responses (flexural strength, porosity and dielectric constant). Consequently RSM was decided for the present exploratory examination since it is perfect and a reasonable outline of multivariable measurable techniques. The second order model is exceptionally appropriate, organized, adaptable and enhanced with a specific end goal to locate the ideal qualities and where the collaboration impact between the parameters are required. Hence second order model is required for the present design. Design expert 7. 0 software was utilized to develop quadratic models with CCD. The aggregate number of trials with 3-

Table 3 Experimental design and test data

Run	Solid loading (vol%): A	Ratio of monomers: B	Monomer content (wt%): C	Flexural Strength (MPa)	Porosity (%)	Dielectric Constant (30 MHz)
1	50	10	15	75.38	34.95	5.782
2	52	15	5	87.2	35.5	4.788
3	52	5	15	85.2	34.124	5.15
4	50	15	10	74.12	35.92	5.366
5	48	15	5	48.12	37.544	4.47
6	52	10	10	95.12	28.5	6.982
7	50	10	10	80.63	31.72	6.57
8	50	5	10	73.15	32.08	5.957
9	50	10	5	77.13	33.4	6.01
10	50	10	10	80.92	32.01	6.45
11	50	10	10	80.63	31.72	6.57
12	48	5	15	60.81	36.5	4.64
13	48	5	5	50.9	35.12	5.2
14	52	15	15	80.13	36.5	4.792
15	48	15	15	63.41	38.1	4.02
16	48	10	10	67.23	34.544	6.485
17	50	10	10	81.03	31.93	6.32
18	50	10	10	80.75	30.02	6.86
19	50	10	10	79.36	31.09	6.06
20	52	5	5	76.22	33.433	6.02

Table 4 ANOVA analysis for response surface model of flexural strength

Source	Sum of Squares	Df	Mean Square	F Value	*p* value Prob > F	
Model	2582.45602	11	234.76873	1462.41642	< 0.0001	Significant
A-Solid loading	1779.556	1	1779.556	11,085.17273	< 0.0001	significant
B-Monomer ratio	4.489	1	4.489	27.9627842	0.0007	significant
C-Monomer content	1.53125	1	1.53125	9.538430229	0.0149	significant
AB	4.6360125	1	4.6360125	28.87855136	0.0007	significant
AC	67.8030125	1	67.803013	422.3570965	< 0.0001	significant
BC	14.2311125	1	14.231113	88.64814607	< 0.0001	significant
A^2	0.17438409	1	0.1743841	1.086269704	0.3278	
B^2	146.073384	1	146.07338	909.9172457	< 0.0001	significant
C^2	59.9277841	1	59.927784	373.3008897	< 0.0001	significant
ABC	57.4056125	1	57.405613	357.5898316	< 0.0001	significant
B^2C	29.0873025	1	29.087303	181.1900117	< 0.0001	significant
Residual	1.28427841	8	0.1605348			
Lack of Fit	1.28427841	3	0.4280928			
Pure Error	0	5	0			
Cor Total	2583.7403	19				

factors was 20 (2 k + 2 k + 6) where k is the quantity of autonomous factors, represented in Table 2.

14 tests were performed with 6 replications at the centre values (zero) to measure pure error. Analysis of Variance (ANOVA) is used for recommended and balanced models. The F-values were utilized to test the statistical immensity of recommended and changed models for ANOVA. It takes a shot at partitioning the variety in the trial information into parts. Every one of the terms in the condition was figured and organized, in an ANOVA table. There are parts of parameters which were proposed to assess the nature of the scientific models and how ably they suit the exploratory information like coefficient of determination (R^2), adjusted coefficient of determination (Adj. R^2), coefficient of variation (C.V.), adequate precision and lack of fit (LOF) [18]. The significance of any model in statistics is defined with a high F-value (>1) and low *p* value (less than

Table 5 ANOVA analysis for response surface model of porosity

Source	Sum of Squares	Df	Mean Square	F Value	*p* value Prob > F	
Model	115.25071	12	9.604225902	290.0140245	< 0.0001	Significant
A-Solid loading	18.264968	1	18.264968	551.5381386	< 0.0001	Significant
B-Monomer ratio	7.3728	1	7.3728	222.6327683	< 0.0001	significant
C-Monomer content	1.20125	1	1.20125	36.27354776	0.0005	significant
AB	0.0219451	1	0.021945125	0.662666006	0.4424	
AC	0.0075031	1	0.007503125	0.226568127	0.6486	
BC	0.327405	1	0.327405006	9.886485835	0.0163	significant
A^2	0.0331531	1	0.033153125	1.001108398	0.3504	
B^2	12.511112	1	12.51111151	377.7918008	< 0.0001	significant
C^2	14.648299	1	14.64829901	442.3273869	< 0.0001	significant
AC^2	6.780699	1	6.780699025	204.7533901	< 0.0001	significant
B^2C	0.1655082	1	0.165508225	4.99776941	0.0605	
BC^2	1.1878362	1	1.187836225	35.86849868	0.0005	significant
Residual	0.2318149	7	0.033116419			
Lack of Fit	0.2318149	2	0.115907466			
Pure Error	0	5	0			
Cor Total	115.48253	19				

0.05). Further coefficient of determination and adjusted coefficient of determination are also two important parameters for model significance. The coefficient of determination values which were appeared to be nearly to one were considered good acceptance between the calculated and measured values within the scope of experimentation. The signal to noise ratio is measured by adequate precision where a value of more than 4 is acceptable [19]. The co. effective of variety (CV) figures out the leftover fluctuation in the information. 3D mapping is utilized to discover the impact of every parameter exclusively and their connections to the reaction.

3 Results and Discussion

The monomer content (MAM and MBAM), ratio of monomers and other constituents were selected based on literature Adding of small amount of alumina has significant influence on flexural strength, thermal shock resistance without disturbing the dielectric properties of sintered fused silica ceramics. Flexural strength increased with increase in ratio of monomers, reached maximum value and then decreased, which shows that there is an optimum value in the ratio of monomers. Addition of silicon nitride to silica did not deteriorate the dielectric properties. While to some extent, the composite ceramic display better dielectric properties compared to silica ceramic, especially at high measuring temperature [1, 2, 5, 24–27]. The experimental designs of SiO_2 micro hybrid ceramic samples with measured responses are presented in Table 3.

Accordingly 20 tests were conducted using CCFCD with 6 centre points to optimize the three process variables and to find their influence on the three responses (Flexural strength, porosity and dielectric constant). The flexural strength and dielectric constant monotonically increase with solid loading, whereas porosity decreases.

The mathematical relationships of flexural strength, porosity and dielectric constant are obtained from the analysis in Table 2. Polynomial equations were fitted for flexural strength, porosity and dielectric constant, as shown in Eq. 2, Eq. 3 and Eq. 4 respectively.

$$\begin{aligned} \textit{Flexural strength}\ (MPa) = 80.75 + 13.34 \times A + 0.067 \\ \times B - 0.875 \times C + 0.761 \\ \times A \times B - 2.91 \times A \\ \times C - 1.33 \times B \times C + 0.252 \\ \times A^2 - 7.29 \times B^2 - 4.67 \\ \times C^2 - 2.68 \times A \times B \times C \\ + 4.26 \times B^2 \times C \end{aligned} \tag{2}$$

$$\begin{aligned} \textit{Porosity}\ (\%) = 31.78 - 3.02 \times A + 1.92 \times B + 0.775 \\ \times C + 0.05 \times A \times B - 0.031 \times A \\ \times C - 0.06 \times B \times C - 0.35 \times A^2 + 2.13 \\ \times B^2 2.31 \times C^2 + 2.06 \times A \times C^2 - 0.32 \\ \times B^2 \times C - 0.86 \times B \times C^2 \end{aligned} \tag{3}$$

Table 6 ANOVA analysis for response surface model for Dielectric constant

Source	Sum of Squares	df	Mean Square	F Value	*p* value Prob > F	
Model	14.72598	10	1.4726	197.6118887	< 0.0001	significant
A-Solid loading	0.850889	1	0.85089	114.1830555	< 0.0001	significant
B-Monomer ratio	1.246796	1	1.2468	167.3109007	< 0.0001	significant
C-Monomer content	0.442682	1	0.44268	59.40462696	< 0.0001	significant
AB	0.0072	1	0.0072	0.966187242	0.3513	
AC	0.002592	1	0.00259	0.347827407	0.5699	
BC	0.121032	1	0.12103	16.24160754	0.0030	significant
A^2	0.012261	1	0.01226	1.645355419	0.2316	
B^2	2.778825	1	2.77883	372.8979722	< 0.0001	significant
C^2	1.633556	1	1.63356	219.2113063	< 0.0001	significant
ABC	0.072962	1	0.07296	9.79096577	0.0121	significant
Residual	0.067068	9	0.00745			
Lack of Fit	0.067068	4	0.01677			
Pure Error	0	5	0			
Cor Total	14.79305	19				

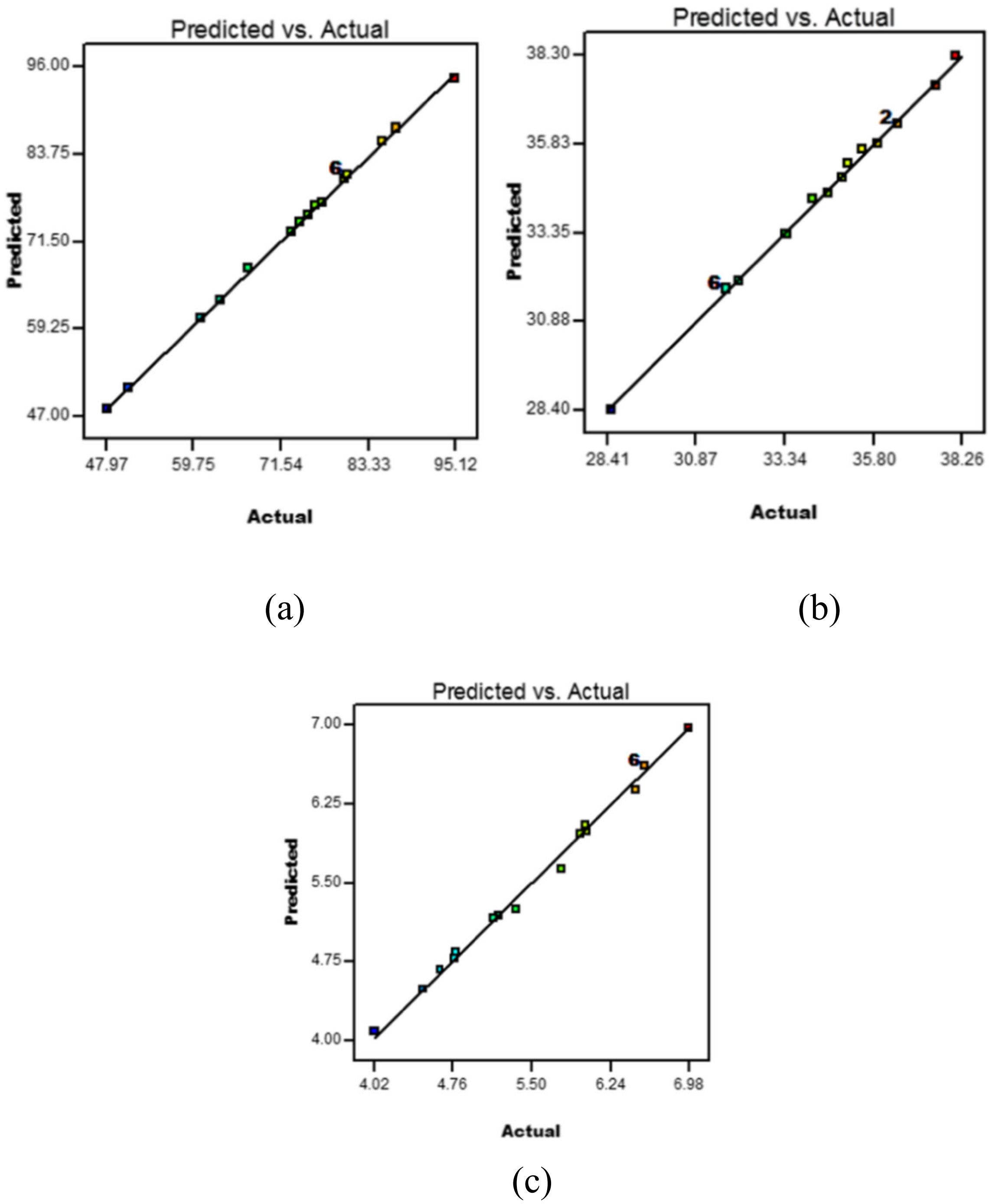

Fig. 2 Predicted versus actual (**a**) Flexural strength, (**b**) Porosity and (**c**) Dielectric constant

$$\begin{aligned} \textit{Dielectric constant} = {} & 6.61 + 0.29 \times A - 0.35 \times B - 0.21 \\ & \times C - 0.03 \times A \times B + 0.018 \times A \\ & \times C + 0.123 \times B \times C + 0.067 \\ & \times A^2 - 1.001 \times B^2 - 0.771 \times C^2 \\ & + 0.095 \times A \times B \times C \end{aligned} \tag{4}$$

The ANOVA statistics for three responses are presented in Tables 4, 5 and 6.

R-Squared: 0.999, Adj R-Squared = 0.998, Pred R-Squared = 0.97, Adequate Precision: 149.417, C.V.: 0.535.

R-Squared: 0.997, Adj R-Squared = 0.994, Pred R-Squared = 0.8838, Adequate Precision: 67.149, C.V.: 0.5349.

R-Squared: 0.995, Adj R-Squared = 0.99, Pred R-Squared = 0.8962, Adequate Precision: 45.038, C.V.: 1.50.

They reveal that the obtained polynomial equations might be utilized to explore the design space. The F-values for flexural strength, porosity and dielectric constant are 1462.41, 290.01 and 197.61 respectively. The *P*-values of less than 0.05 for the three models are considered to be significant. The high R^2 values (0.999, 0.997 and 0.995) indicate that the amount ofdifference in the output might be described by the models of flexural strength, porosity and dielectric constant. The highly Adj. R^2 values are 0.998, 0.994 and 0.990 indicating the high significance of these mathematical models. The adequate precisions are found to be 149.41, 67.14 and 45.03 respectively. The low CV values (0.53, 0.54 and 1.50) estimate that these tests

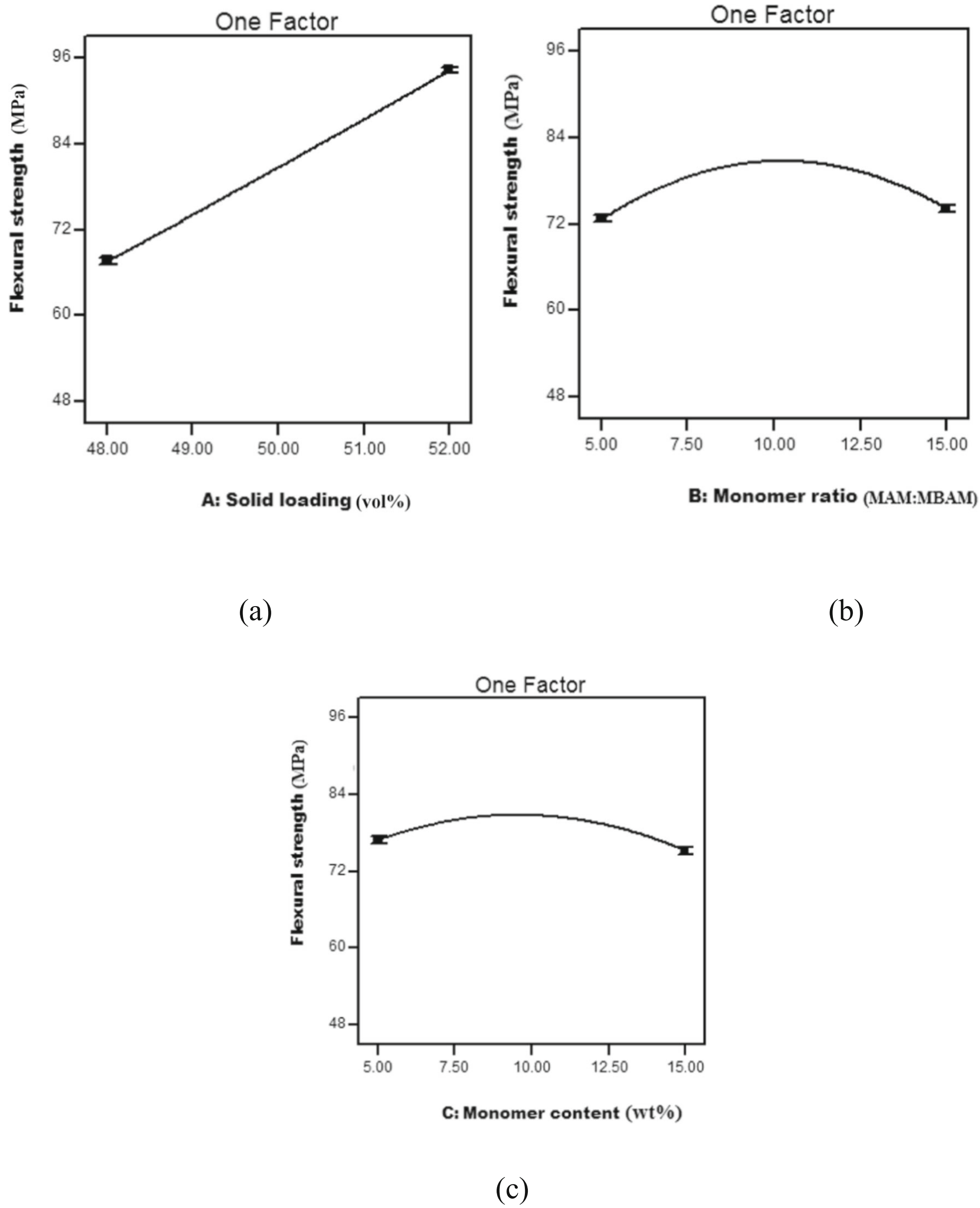

Fig. 3 The effect of three factors on Flexural strength (**a**) Solid loading, (**b**) Monomer ratio and (**c**) Monomer content

have precision, reliability and are satisfactory. The LOF data ($P > 0.05$) indicate that predicted models are significant and suitable for estimating the flexural strength, porosity and dielectric constant within the span of input parameters.

It is essential to know that the obtained model is giving an adequate approximation to the actual system. Model acceptability can be ensured by drawing diagnostic figures like predicted versus experimental. Figure 2a, b, c represents the difference between experimentally measured values and predicted values for flexural strength, porosity and dielectric constant respectively. The measured values were found relatively close to the straight line for three outputs. The graphs backed by Adj. R^2 values for three responses were 0.998, 0.994 and 0.990, which is near to 1 indicating a better compliance between observed and predicted values.

The response surface plots shown in Figs. 4, 6 and 8 depict the effects of 3-pairs of process parameters on three responses. Solid loading has a useful effect on flexural strength, dielectric constant and negative impact on porosity. The maximum value of flexural strength, dielectric constant and minimum value of porosity is obtained at 52 vol% of solid loading [28]. This is in compliance with the P-values attained for each factor from ANOVA. The influence of monomer ratio and monomer content varies from case to case. The flexural strength and dielectric constant improves up to a certain limit and then reduces with the addition of MC and increase in MR. Porosity reduces up to a certain stage and then enhances with the addition of monomer content and increase in monomer ratio. Understanding the connection between the procedure factors gives superior knowledge to the general procedure investigation. A variable may communicate with any or the greater part of alternate factors producing the likelihood of quality of an immense number of collaborations. The interactions between the factors are significant on the three responses.

The properties of the hybrid ceramic composites considerably depend on the solid loading and monomers. Figure 3 shows one factor effect of process variables on the flexural strength. The strength monotonically increases with SL, but in the case of MR and MC, it enhances up to a certain state and then reduces to an optimum point.

Figure 4 shows the interaction plots of process variables on flexural strength. From ANOVA Table 4, it is

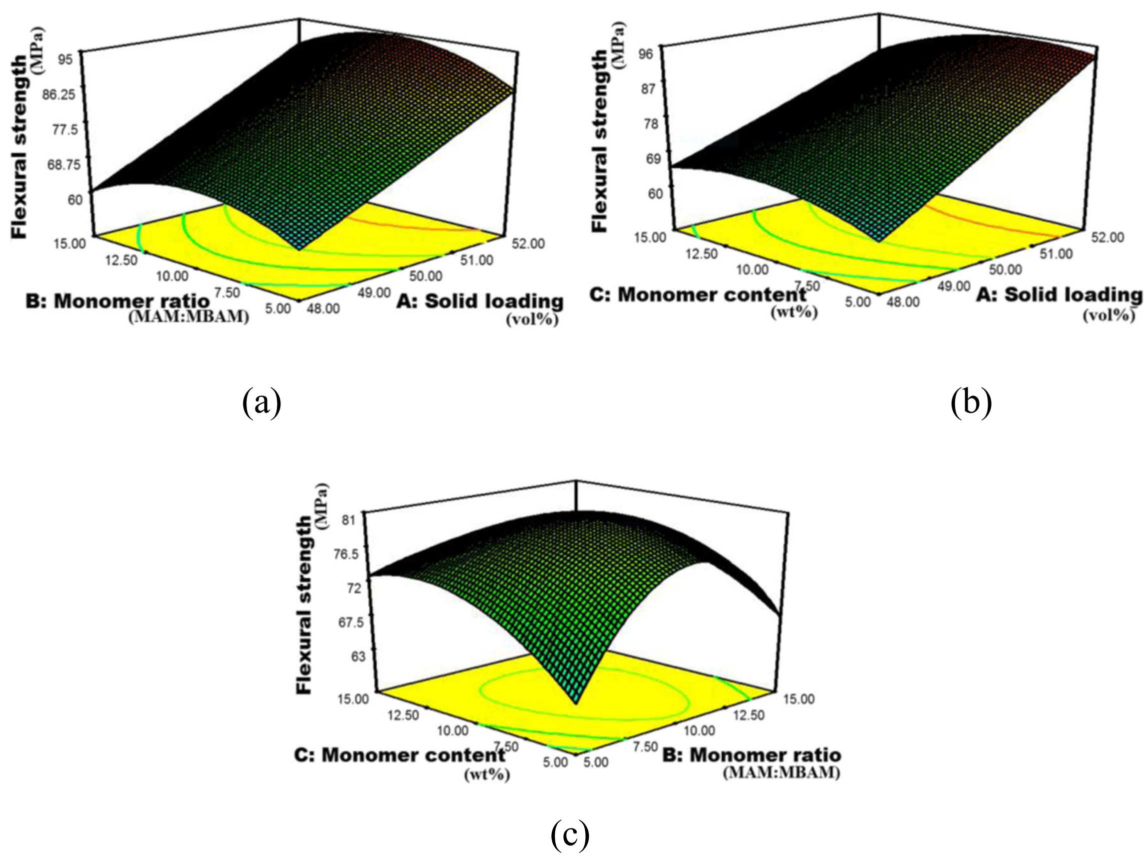

Fig. 4 Effect of interactions on flexural strength (**a**) solid loading and monomer ratio, (**b**) solid loading and monomer content and (**c**) monomer ratio and monomer content

observed that A, B, C, AB, AC, BC, B^2, C^2, ABC and B^2C were significant terms. From Fig. 3a, it is found that the flexural strength enhances with SL. It is observed that with increase in SL, there is enhancement in the density of fused silica ceramic composites which is favorable for the increase of flexural strength. However, the maximum flexural strength is obtained at 52%. The organic binder burnout plays a vital role in pore formations which affects the flexural strength of the sintered ceramics. If the MR is low, the flexural strength of dried samples remains low as the 3-dimesnsional network is too coarse that results in the non-uniform distribution of the SiO_2 particles that influences decrement in flexural strength. Maximum flexural strength is obtained at 52 vol% SL, 10:1 MR and 10 wt% MC.

Porosity of the ceramic composites mainly depends on solid loading, monomer ratio and monomer content. Solid loading has a negative impact on the porosity whereas monomer ratio and monomer content have an optimal point. Figure 5 shows the single factor influence on the response. The effect of SL on porosity is shown in Fig. 5a. The maximum porosity is obtained at 48% solid loading. Porosity decreases with increase of SL. The SL has more influence on density improvement in ceramic composites. The distance between the particles and shrinkage is less as the solid loading increases.

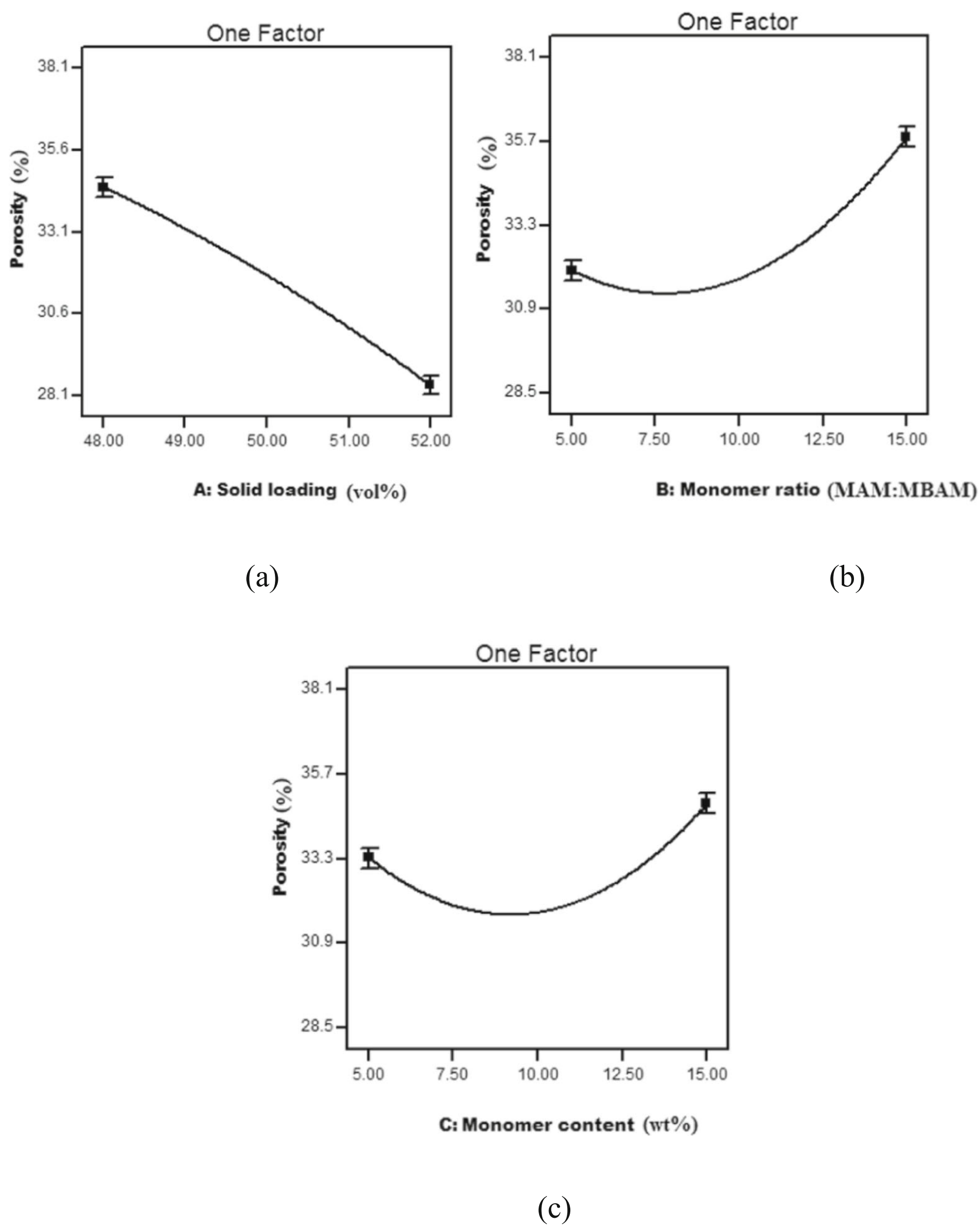

Fig. 5 The influence of three factors on porosity (**a**) Solid loading, (**b**) Monomer ratio and (**c**) Monomer content

Figure 5b, c show the influence of monomer ratio and monomer content.

Figure 6 represents the interaction plots of process parameters on porosity. From ANOVA Table 5 it is observed that A, B, C, BC, B^2, C^2, AC^2 and BC^2were significant terms. The monomer and cross linking agent can form macro molecular network to bind the ceramic particles together and also play a major role in the creation of the pores during the preparation of ceramic composites. The increase of monomer ratio makes the crosslink density of crosslinked polymer gels in dried sample increases; the dispersion of ceramic particles becomes more uniform, the dry shrinkage is minimum and thus the porosity of the sintered body improves. The maximum porosity is obtained at 48 vol% SL, 15:1 MR and 15 wt% MC.

Dielectric constant is a major performance measure for wave transparent applications along with mechanical and thermal properties. A very low value of dielectric constant (~10) [29] is required for missile radome applications. Figure 7 shows the single factor effect of variables on dielectric constant. The dielectric constant of materials mainly depends on the porosity. From the data, it is observed that the dielectric constant reduces with increase of porosity. SL has a positive impact on dielectric constant. The dielectric constant enhances up to a certain state and then reduces as well as indicating an optimum limit with increase in MR and MC.

Figure 8 shows the interaction plots of process variables on dielectric constant. The minimum value of the dielectric constant is found to be 4.02 at 48 vol% solid loading, 15:1 monomer ratio and 15 wt% monomer content. From ANOVA Table 6 it is found that A, B, C, BC, B^2, C^2, AC^2, B^2C and BC^2were significant terms.

4 Multi Objective Optimization

The multi objective optimization has been conducted on gelcasting process followed by mathematical modeling and analysis. The performance measures used for the optimization modeling are flexural strength, porosity and dielectric constant and the desirability approach has been implemented. "Desirability is an objective function D, introduced by Myers and Montgomery and its desirable value (d_i) ranges from 0 to 1, least to most desirable, respectively". Desirability function empowers adjusting among every single test reaction with extra

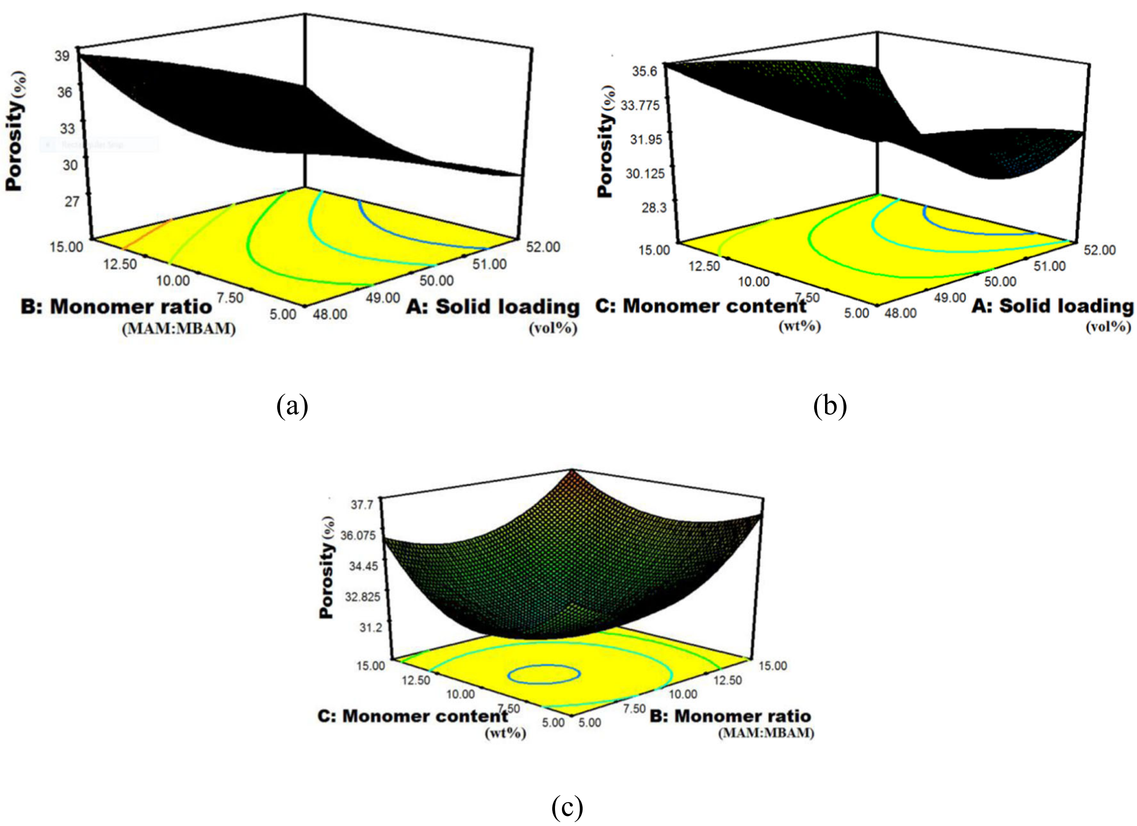

Fig. 6 Influence of interactions on porosity (**a**) solid loading and monomer ratio, (**b**) solid loading and monomer content and (**c**) monomer ratio and monomer content

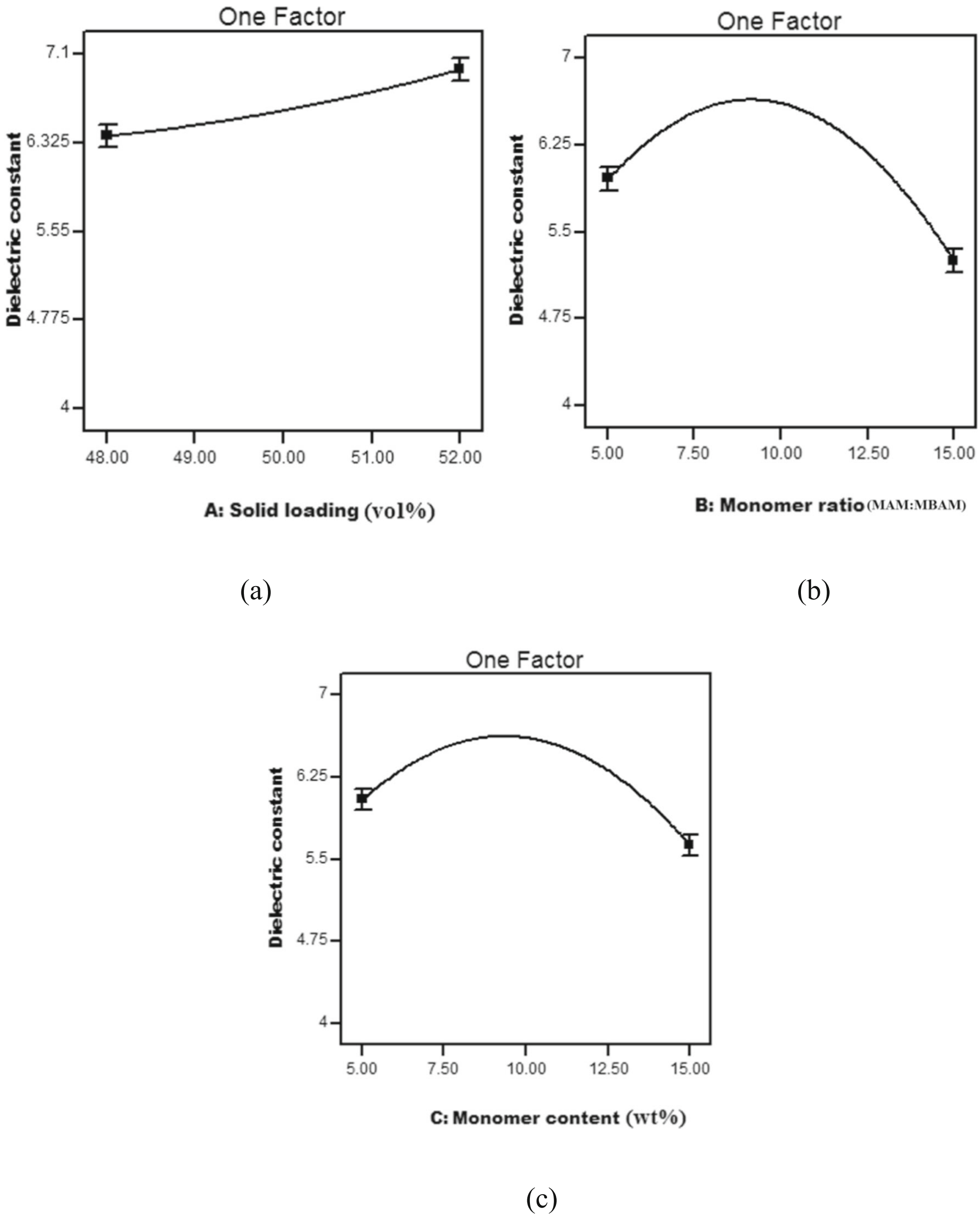

Fig. 7 The effects curves of three factors on dielectric constant (**a**) Solid loading, (**b**) Monomer ratio and (**c**) Monomer content

advantages such that the outcomes can be plotted. It has advantage over other multi model advancement procedures like linear programming. Gelcasting is a procedure that deals with identifying ideal working conditions to accomplish the best item quality for a specific application. On account of different strife destinations and communication impact of the procedures, gelcasting is a preferred manufacturing strategy as far as finding the ideal procedures factors and creating vigorous modern applications is concerned. It is required to achieve most extreme flexural strength and porosity while least dielectric constant.

The desirability function was utilized to change every reaction (y_i) into a solitary allure that shifts over the range $0 \leq d_i \leq 1$, where $d_i = 1$ when an objective fulfills the reaction necessity and $d_i = 0$ when the objective is

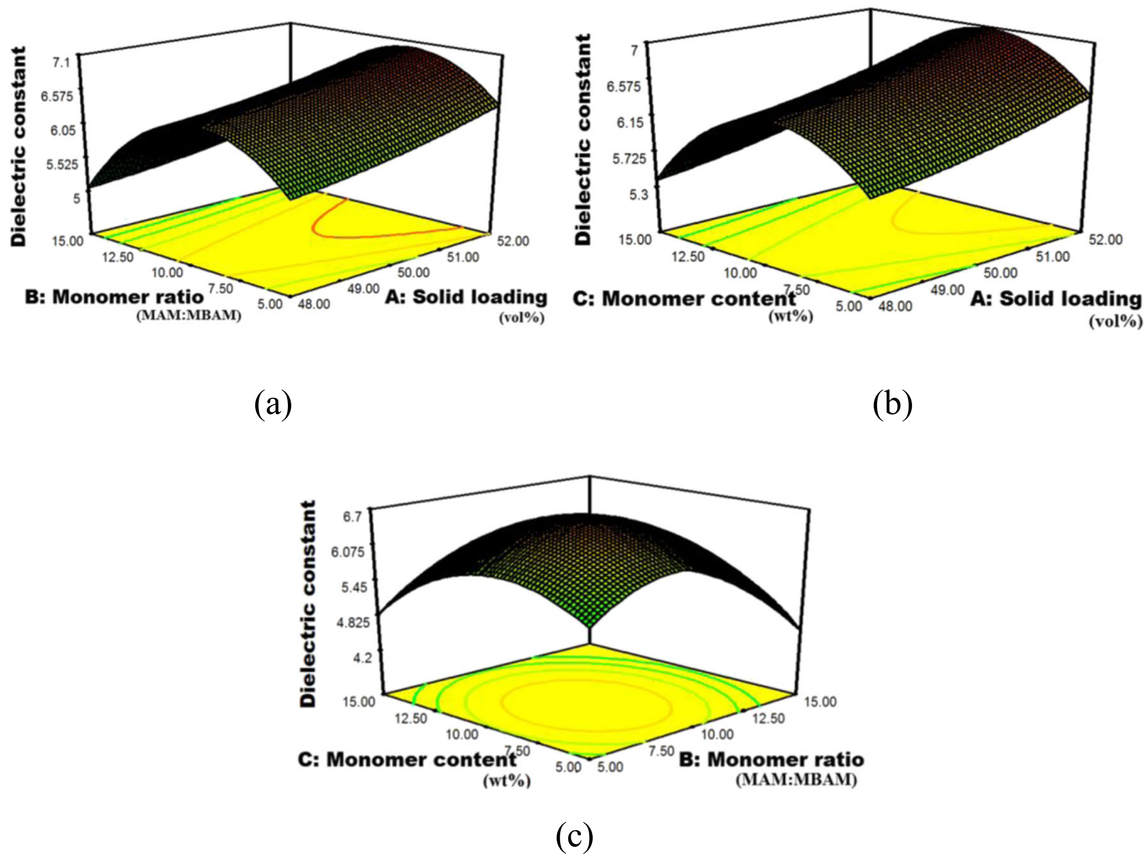

Fig. 8 Effect of interactions on dielectric constant (**a**) solid loading and monomer ratio, (**b**) solid loading and monomer content and (**c**) monomer ratio and monomer content

outside the acknowledged area. In the present paper, the least desirability function was chosen to limit the dielectric constant in Eq. 5, while the desirability from most extreme and the better one was chosen to boost flexural strength and porosity utilizing Eq. 6:

$$d = \begin{cases} 1, & y < T \\ \left(\frac{(U-y)}{(U-T)}\right)^r & T \le y \le U \\ 0 & y > U \end{cases} \quad (5)$$

where, U = upper bound, T = target, and r = desirability weight.

$$d = \begin{cases} 1, & y < L \\ \left(\frac{(y-L)}{(T-L)}\right)^r & L \le y \le T \\ 0 & L > T \end{cases} \quad (6)$$

where, L = lower bound, T = target, y = response and r = desirability weight.

The constraint for the optimization of the gelcasting process is shown in Table 7.

Once every reaction is changed, the individual desirabilities are then consolidated into a solitary reaction utilizing the general attractive quality capacity, which gives worldwide ideal of the multi-reaction streamlining,

Table 7 Constraints and rules applied on process variables and responses

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
A: Solid loading (vol%)	in range	48	52	1	1	3
B: Monomer ratio	in range	5:1	15:1	1	1	3
C: Monomer content (wt%)	in range	5	15	1	1	3
Flexural strength (MPa)	Maximize	48.12	95.12	1	1	3
Porosity (%)	Minimize	28.5	38.1	1	1	3
Dielectric constant	Minimize	4.02	6.982	1	1	3

Table 8 Results of confirmation test

Dependent variables	Optimum input parameters			Predicted values	Actual values	Error (%)
	Solid loading (%)	Monomer ratio	Monomer content (wt%)			
	52	15:1	5			
Flexural strength (MPa)				87.36	82.01	−6.523
Porosity (%)				35.66	34.5	−3.362
Dielectric constant				4.783	4.788	0.104

as communicated in Eq. 7. The factor settings with the most noteworthy worldwide attractive quality are chosen to be the ideal settings.

$$d = \begin{bmatrix} \left(\prod_{i=1}^{n} d_{i1}\right)^{1/n} \\ \left(\prod_{i=1}^{n} d_{i2}\right)^{1/n} \\ \left(\prod_{i=1}^{n} d_{i3}\right)^{1/n} \end{bmatrix} \tag{7}$$

Where, D is the overall desirability, n is the total number of the experimental responses and d_1, d_2, d_3... d_n are single desirability function for each response [30–33].

Alongside measurable approval and check of numerical models regarding a solitary reaction, the scientific models as far as multi objective optimization is concerned have additionally been affirmed by confirmation tests. Optimum settings of process variables were found by conducting the optimization, by maximizing flexural strength, porosity and minimizing dielectric constant. The optimum process parameters were found to be 52 vol% of solid loading, 15:1 of monomer ratio and 5 wt% of monomer content to meet the requirements of optimal responses. It is apparent from Table 8 that the actual (experimental) and predicted output values are firmly lining up with each other, which legitimize the ampleness of the RSM based optimization and forecast of flexural strength, porosity and dielectric constant. The percentages of residuals for these responses are 6.523%, 3.362% and 0.104% individually.

The overall desirability value of the optimal factors obtained was 0.7734 and represented in Fig. 9.

The outcomes show that the real qualities acquired through affirmation tests are near the anticipated qualities. The error percentage is computed by Eq. 8. The affirmation demonstrates that the exactness of the outcomes can be considered as evidence for the validity and sufficiency of the created mathematical models and test plan.

$$\textit{Prediction error}\ (\%) = \left[\frac{\textit{Experimental value}-\textit{Predicted value}}{\textit{Experimental value}}\right] X\ 100 \tag{8}$$

Figure 10 shows EDAX image optimum process conditions. It contains the Si, N, Al and O representing the major constituents of the ceramic composite.

Figure 11 shows the SEM image of the hybrid ceramic composite after applying the optimum conditions obtained from the desirability optimization through RSM. It is clear from the image that the obtained ceramic is denser and the particles are uniformly distributed. The properties of the hybrid ceramic composites obtained after optimization are suitable for the manufacturing of radomes for low speed missile applications.

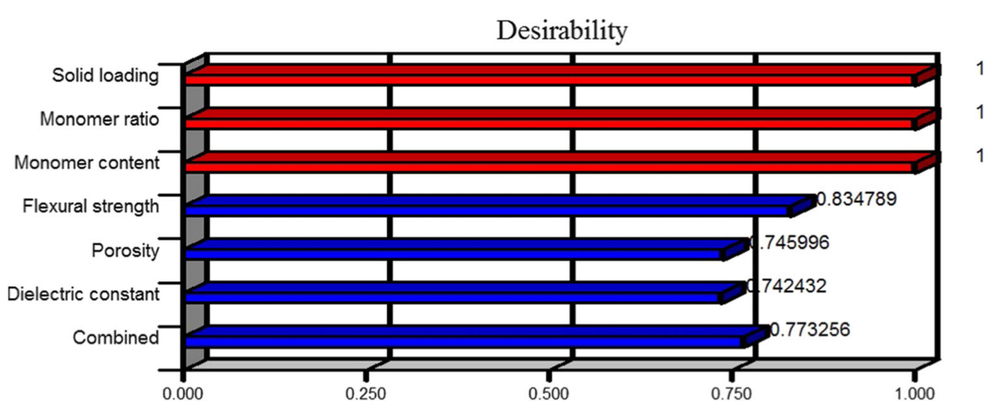

Fig. 9 Bar chart of the optimization

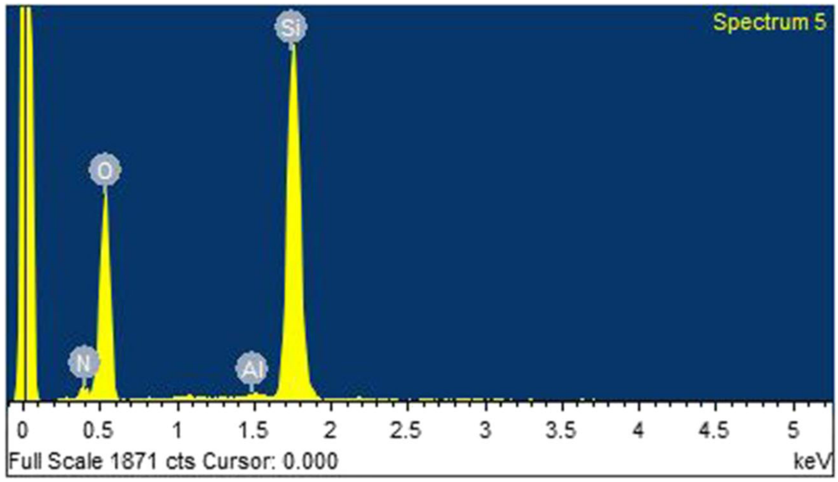

Fig. 10 EDAX at optimum process conditions

5 Conclusions

In the present study, mathematical and statistical modeling was applied to study the gelcasting process for the fabrication of fused silica hybrid ceramic composites. RSM was used to optimize the process. The concluding remarks are

- The mathematical models obtained through RSM for flexural strength, porosity and dielectric constant are acceptable due to their higher co. efficient of determination (R^2), which is close to one. Moreover, residual analysis of the results strengthens the acceptability of the generated mathematical models.
- ANOVA for flexural strength revealed that it is highly affected by solid loading, followed by monomer ratio and lastly by the monomer content.
- ANOVA for porosity revealed that it is most effected by solid loading and the monomer ratio followed by monomer content.
- ANOVA for dielectric constant showed that it is highly controlled by solid loading, monomer ratio and monomer content.
- Desirability based multi objective optimization has revealed that the flexural strength (maximized), porosity (maximized) and dielectric constant (minimized) were optimized when the solid loading was kept at 52 vol%, with monomer ratio maintained at 15:1 and monomer content maintained at 5 wt%.

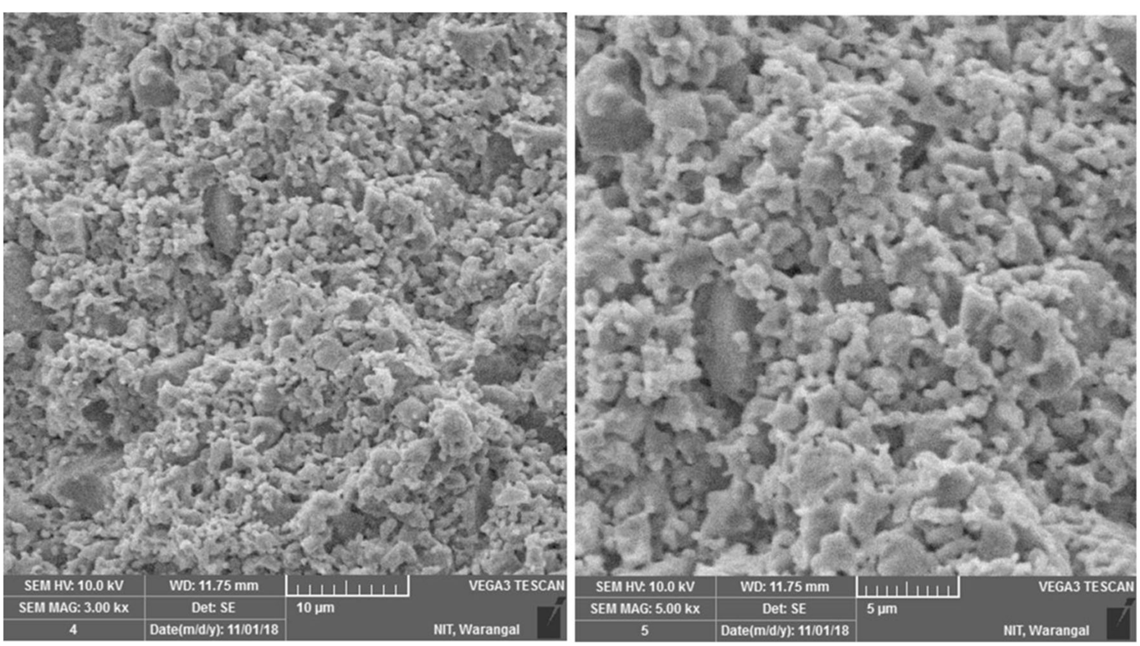

Fig. 11 SEM images at optimum process conditions

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