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Optimization of process parameters of Al-B_4C hybrid composites using response surface methodology

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Abstract. In this contemporary work, particulates of coconut shell ash and boron carbide were reinforced with an atomized aluminium powder hybrid composites was prepared by the powder metallurgy process. The process parameters in powder metallurgy influence the various material properties. Compaction pressure, sintering temperature and weight percentage of coconut shell ash were selected as influencing parameters. An empirical relationship has been formulated using response surface methodology. The properties such as hardness, relative density and percentage of porosity are considered a response. Variance of analysis was employed to determine the significance of process parameters on the responses and to determine the optimal combination of parameters. High harness value of 165.1 HV, maximum density of 0.819 g/cc and minimal porosity of 8.15 % was obtained for the optimum condition.

KEYWORDS: Powder metallurgy, Coconut shell ash, Response surface methodology.

1. INTRODUCTION

Aluminium remains the most utilized metallic alloy as matrix material in development of MMC's and it is the most reliable material. Aluminium based metal matrix composites (AMC) with discontinuous reinforcements have vastly been attracted by different industries due to their remarkable physical and mechanical properties such as superior wear resistance, high stiffness and durability, controlled co-efficient of thermal expansion, low density, high fatigue resistance and better stability at elevated temperature. In recent decades, the AMC applications have been extended from predominantly aerospace and automobile to defence, marine [1-7].

The major challenges faced in casting are the non-uniform distribution of reinforcements which causes agglomeration and clustering of particles that are inevitable. To overcome the problems in the casting process, powder metallurgy [PM] is one of the successful techniques. The advantages of PM include lower processing temperature compared to casting techniques, control of composition of the product, good distribution of reinforcing particles, can produce intricate shapes [8-11]. The PM parts can be batch produced to neat form, eliminating or reducing the need of subsequent machining. PM process wastes very little material i.e) approximately 97 % of starting powders is converted to product.

Different kinds of ceramic particles like SiC, Al_2O_3, TiC, B_4C, MgO, ZrO_2, TiO_2, fly ash., the arts are reinforced with aluminium and its alloys to improve the microstructure and mechanical properties. Among which boron carbide (B_4C) possesses unique characteristics such as high melting point, good chemical stability, neutron absorption capacity and high wear and impact resistance. Due to extreme hardness (ranks third after in hardness after diamond and cubic boron nitride) and low density (2.52 g/cm^3, even lower than Al), it could be used as replacement for SiC and Al_2O_3 where there is need for superior mechanical properties [12-15]. The B_4C strongly reacts with aluminium matrix and is widely used in the applications of nuclear industry as a storage tank material due to specific ability of B10 isotope to capture neutrons. Mohammed Sharifi et.al [16] evaluated the mechanical and tribological properties of B_4C reinforced aluminium Nano composites fabricated by powder metallurgy technique. The results indicate that there is an increase in hardness, compression strength, wear resistance and decrease in ductility when B_4C particles were reinforced into the matrix. Kanmani Subbu et.al [17] studied the workability and densification behaviour of atomised aluminium powder reinforced with varying percentage (2 %, 4 % and 6 %) of B_4C particle at a compaction pressure of 275 MPa, sintering temperature and time of 550^{0}C and 60 minutes respectively. The results shown that the highest relative density is attained at 2 weight percentage of B_4C reinforcement with matrix.

Aluminium hybrid composites are a new generation of MMC that have the potential of satisfying the recent demands of advanced engineering applications. There is a possibility of three broad categories of hybrid reinforcement. These are AMC hybrid with two synthetic ceramic materials; an agro waste combined with a ceramic material; and synthetic reinforcement combined with industrial waste. Anil Kumar Bodukuri et.al [18] fabricated MMC's having various combinations of Al-SiC-B_4C by powder metallurgy and studied the effect of hybrid reinforcement in it. Nowadays, research has been focussed on material possessing the advantages of low processing cost, accessibility, low density and reduced environmental pollution. In such cases, agro wastes are believed to be very promising material for the synthesis of AMC complementing synthetic reinforcement. Various researches using agro wastes as reinforcements in AMC's include bamboo leaf ash (BLA), rice husk ash (RHA), palm kernel shell ash (PKSA), bagasse ash (BA), bean shell waste ash (BSWA) have been studied [19-21]. The results indicate that agro waste improved the properties of AMC than unreinforced alloy at reduced production cost even at 50 % replacement of synthetic reinforcement [22].

Coconut shell ash (CSA) is one of the low cost agricultural waste having the presence of hard phases like MgO, Al_2O_3, Fe_2O_3, SiO_2, as major elements. Comparing to other agro waste material CSA is suggested because of its low density (0.47 g/cm^3) and maximum temperature withstand capacity of 1500^{0}C which finds application in the field of automobile [23]. Anish et.al [24] studied the compressive behaviour of SiC/Nano-coconut shell charcoal (NCSC) reinforced Mg composite processed through powder metallurgy route and concluded that inclusion of NCSC significantly enriched the density and porosity measurement shown marginal porosity.

In recent days, several statistical experimental methods are widely used for modelling or optimizing the complex and non-linear processes. Among them, response surface methodology (RSM) has been employed by many researchers because of its fewer experimental design compared to 'One factor' method. RSM is an efficient mathematical approach due to its accuracy towards modelling and analysing engineering problems [25-27]. RSM maps the relationship between one or more responses and a set of quantitative design variables for achieving either maximization or minimisation of quality characteristics. The response can be represented graphically either in three dimensional space or contour plots that help envision the pattern of response surface. Yan et.al [28] proposed to produce the 304L stainless steel/Cu composites by powder metallurgy and explored the effects of technological parameters on properties of composites using RSM. He concluded that RSM can be easily carried out to make the powder metallurgy process parameters in order to satisfy ouput responses.

When literature studies are analyzed, it is concluded that agricultural wastes facilitate application towards innovative and new spheres of economic concern and exploit their full potential as valuable resources. Very scarce researches have been focussed on using agricultural waste as hybrid reinforcement combined with ceramic material. In present work, Al/B_4C/CSA hybrid composites were prepared by powder metallurgy technique. To the author's best knowledge, there has been no paper published on it. In this study, it was aimed to determine the optimum process parameters using RSM.

2. MATERIALS AND METHODS

2.1. Material Selection.

In present work, the Al-B_4C-CSA is taken as the starting material. 99.72% purity of Atomised aluminium powder (density, **ρ**=0.8840 g/cc) having particle size of 325 mesh size is used as a matrix material. Boron carbide powder (**ρ**=2.51 g/cc) of 325 mesh size with 99.72% purity is selected as ceramic reinforcement. The coconut shell ash (CSA) powder with particle size of 44μm is considered as a hybrid reinforcing phase. Table 1, 2 shows the chemical arrangement of Atomised Al powder and CSA powder.

Table 1. Chemical composition of Atomised aluminium powder.

Element	Fe	Si	Mg	Mn	Cu	Zn	Ni	Ti	Cr	Others
Weight %	0.100	0.090	0.001	0.001	0.001	0.002	0.0030	0.0060	0.001	0.0752

Table 2. Chemical composition of CSA powder.

Element	Al_2O_3	Fe_2O_3	MgO	SiO_2	ZnO	CaO	K_2O	Na_2O	MnO
Weight	15.6	12.4	16.2	45.05	0.3	0.57	0.52	0.45	0.22

2.2. Design of experiment:

Design of experiment (DOE) is a systematic technique to determine the relationship between the factors distressing a process and output of that process. DOE selects a diverse experiment sets in which all factors are independent to each other despites being varied simultaneously. Powder metallurgy comprises of series of three major processes such as blending or milling of powders, compaction and sintering of green compact to obtain the required finished component. Each stage consists of several parameters to be maintained among which milling hour, compaction pressure and sintering temperature has major influence on the output variable [29, 30].

Box-Behnken Design (BBD), one of the most important experimental designs in the optimization process, has been extensively applied to develop response surface models. Box-Behnken Design usually have fewer design points than Central Composite Design (CCD), therefore they are less expensive to run with the same number of elements. Unlike CCD, Box-Behnken Design does not have axial points which are the primary benefit in addressing the focus of where the experimental limitations should be and in particular to avoid treatment combination that are extreme.

In this study, the effects of compaction pressure (MPa), sintering temperature (°C) and reinforcement percentage of CSA (%) were investigated and optimized. The coded and actual values are illustrated in table 3 which are selected based on the preliminary experiments and previous studies [17]. For statistical calculations, the variables were coded to lie ± for factorial points and 0 for centre points. Statistical software Design Expert 7.0 is used for statistical analysis and mathematical modelling. A three factor, three level BBD design with a total of 15 experimental runs was developed using it.

Table 3. Coded and actual values of design factors

Independent factors	Levels		
	-1	0	+1
A: Compaction pressure (MPa)	250	275	300
B: Sintering temperature (^{0}C)	500	550	600
C: CSA reinforcement (%)	2	4	6

2.3 Preparation of hybrid composites:
The approximate quantities of Al, B_4C and CSA were weighed individually. The 2% composition of B_4C [17] is kept constant and that of CSA reinforcement is varied as of table 3.The matrix and reinforcements were first milled in a planetary ball mill (figure 1) using a tungsten carbide vial and balls of diameter 10 mm. The rotation speed of 150 rpm and the milling time of 1 hour were carried out during the process to ensure the proper mixing. The milled powders were cold pressed into a two piece die set of D3 tool steel having an inner diameter of 25 mm and 25 mm length for 15 min under varying compaction pressure as of DOE. After compaction, the green compacts produced were sintered in muffle furnace using predetermined temperatures for a waiting period of one hour. The sintered samples were furnace cooled and homogenized to room temperature.

Figure 1. Planetary Ball Mill setup.

2.4. Evaluation of properties:
Hardness value for each specimen was evaluated by using Vickers micro-hardness tester as per ASTM: E384-10. Prior to the test, the test samples surface were polished by smooth emery paper. The test was carried out under a load of 300 g for a period of 10 sec. The testing of composite was shown in figure 2. Each specimen was investigated at three different positions to avoid the possible effects of indenter resting on hard reinforcement particles and the averages of all the readings are described. The density and porosity of the sintered hybrid composite were measured using Archimedes principle, distilled water was utilized as the immersion fluid. The mass of each sample was quantified in air and then in water. The samples were weighed using an electronic balance with an accuracy of ± 0.001 mg. Theoretical

density was calculated by using the rule of mixtures principle. The experimental density, theoretical density and porosity percentage of composite was measured by the following formulae,

$$Experimental\ density, \rho_{exp} = \frac{\rho\ of\ fluid \times m_a}{m_a - m_w} \quad (1)$$

$$Theoretical\ density, \rho_{th} = (\rho_m + v_m) + (\rho_R + V_R) \quad (2)$$

$$Porosity\ (\%) = \frac{\rho_{th} - \rho_{exp}}{\rho_{th}} \times 100 \quad (3)$$

Where, m_a – mass of the sample in air, m_w – mass of sample in water, ρ_m - density of matrix, ρ_R - density of reinforcement, V_m – volume fraction of matrix, V_R - volume fraction of reinforcement.

Figure 2. Hardness testing of prepared composites

3. RESULTS AND DISCUSSIONS

Using the RSM, the 15 experimental combinations and corresponding values of responses based on BBD are summarized in table 4. The experimental designs were prepared for the formation of linear models for hardness, density and porosity. The linear model generated by the design can be represented by the following equation [31]:

$$y = a_0 + \sum_{i=1}^{n} a_i x_i + z \quad (4)$$

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Where, y is predicted response, a_0 is constant coefficient, a_i is constant coefficient estimated from regression and z is the unwanted signal. Analysis of variance (ANOVA) is a statistical technique used to determine the present contribution of each parameters over the output responses. The significance of each coefficient of the linear backward reduction model with an exit value of 0.100 was performed with F- test and its associated probability, p values in which the conclusions were obtained at 95% confidence level. Applying the 5% significance level, a value of "Prob- F" less than 0.050 indicate model terms are important.

Table 4. Design layout matrix and experimental results

Run	Process parameters			Responses		
	A	B	C	HARDNESS (HV)	DENSITY (g/cc)	POROSITY (%)
1	275	550	4	86.7	0.7473	16.96
2	250	550	6	113.6	0.7315	17.96
3	275	550	4	87.6	0.5766	35.93
4	250	500	4	72.2	0.5803	35.52
5	250	550	2	46.2	0.3196	64.81
6	275	600	2	45.7	0.4679	48.49
7	250	600	4	69.9	0.6567	27.03
8	275	600	6	165.1	0.819	8.15
9	275	550	4	64.2	0.6468	28.13
10	275	500	2	38.9	0.4179	53.98
11	300	550	2	78.4	0.5305	41.59
12	275	500	6	158.1	0.7714	13.49
13	300	500	4	98.5	0.716	20.45
14	300	600	4	82.3	0.646	22.81
15	300	550	6	124.4	0.8146	8.65

3.1. Effect of process parameters on Hardness:

The reduced linear regression equation for hardness in terms of actual factors is:

$$Hardness = 0.78667 + 22.000 \times CSA\ reinforcement \qquad (5)$$

Table 5 shows the ANOVA results of the regression model. The model F-value of 43.12 implies the model is significant. In this case, compaction pressure is significant model term. The "Lack of Fit F-value" of 2.23 implies the Lack of Fit is not significant relative to pure error. There is a 34.98 % chance that a "Lack of Fit F-value". The "pred R-squared" of 0.6716 is in sensible agreement with the "Adj R-squared" of 0.7505. "Adeq precision" measures the signal to noise ratio. The ratio of 12.716 in model indicates an adequate signal and desirable.

Table 5. ANOVA results for Hardness

Source	Sum of Squares	df	Mean Square	F Value	p-value, Prob > F	
Model	15488	1	15488	43.12	< 0.0001	significant
C-CSA Reinforcement	15488	1	15488	43.12	< 0.0001	
Residual	4669.24	13	359.17			
Lack of Fit	4317.7	11	392.52	2.23	0.3498	not significant
Pure Error	351.54	2	175.77			
Cor Total	20157.24	14				

Figure 3a, 3b demonstrates the surface and contour plots at centre point of sintering temperature (550^0C) with varying compaction pressure and reinforcement percentage of CSA. From the surface plot it is clear that hardness value is constant at minimal percentage of CSA reinforcement (2%) and increasing compaction pressure. The most important factor is the CSA reinforcement. The hardness value is in proportional to the CSA reinforcement. The increase in CSA reinforcement percentage lead to increase in hardness value. The reason for rise in hardness is due to the existence of hard ceramic particles and metal oxides like Al_2O_3, SiO_2, MgO, Fe_2O_3, etc in the CSA and activated carbon in it is also the primary reason for increase in hardness.

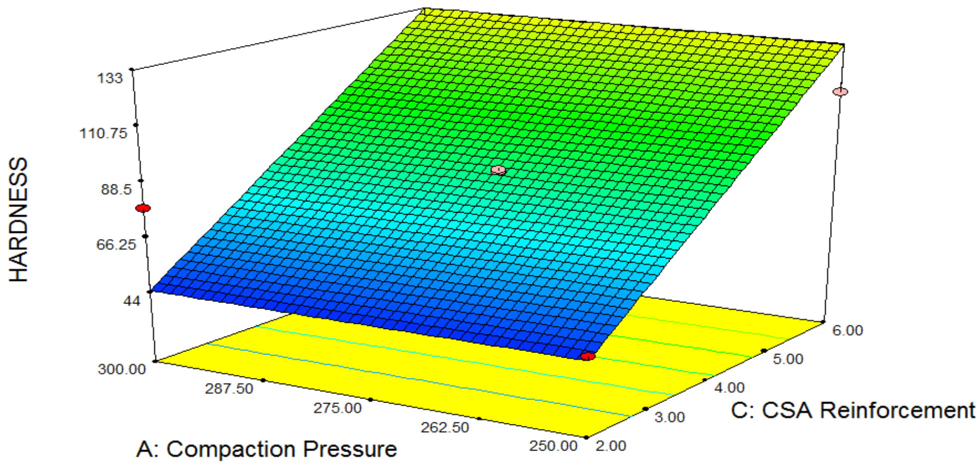

Figure 3: a) Surface plot for hardness

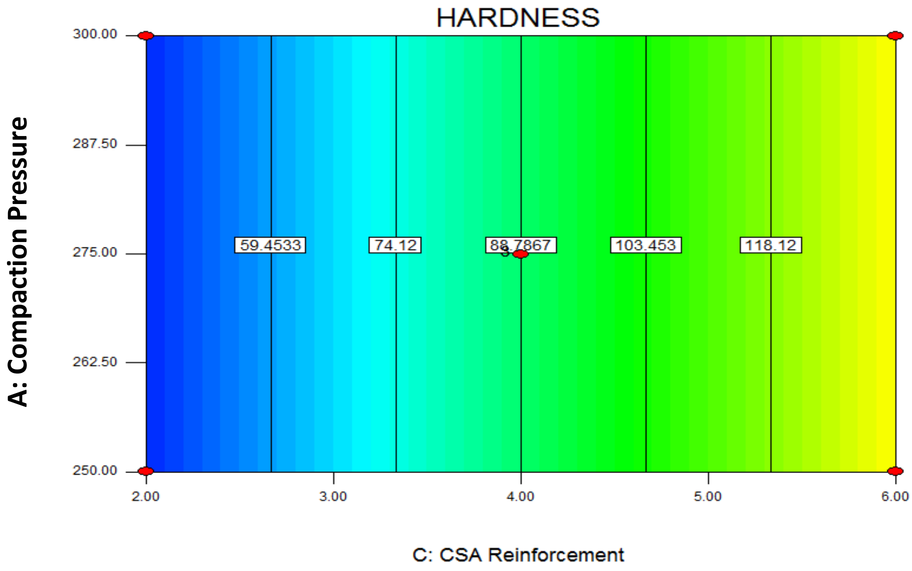

Figure 3. b) contour plot for hardness

3.2. Effect of process parameters on density

The mathematical equation eliminating the insignificant coefficients proposed for density in terms of actual values is,

$$Density = -0.29680 + 0.002095 \times Compaction\ pressure + 0.087537 \times CSA\ reinforcement \quad (6)$$

The results for analysis of variance (ANOVA) for reduced model was shown in table 6. The model F-Value of 41.99 implies the model is significant. The factors compaction pressure and CSA reinforcement are significant model terms. The "Lack of Fit F-value" of 0.32 implies the lack of fit is not significant relative to pure error. There is 91.25 % chance that a "Lack of Fit F-value". The predicted R-squared value of 0.8108 is in reasonable agreement with the adjusted R-squared value of 0.8541. The adequate precision ratio measured the signal to noise ratio of 18.035 which indicates an adequate signal and the model is desirable.

Table 6. The results of ANOVA for density

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	0.27	2	0.13	41.99	< 0.0001	significant
A-Compaction Pressure	0.022	1	0.022	6.9	0.0221	
C-CSA Reinforcement	0.25	1	0.25	77.08	< 0.0001	
Residual	0.038	12	3.18E-03			
Lack of Fit	0.023	10	2.35E-03	0.32	0.9125	not significant
Pure Error	0.015	2	7.36E-03			
Cor Total	0.31	14				

The three dimensional response surface plot and contour plots of density with compaction pressure and CSA reinforcement are depicted in the figure 4a, figure 4b. It can be observed that density increased with compaction pressure as with the reinforcement percentage of CSA. The lower density is obtained at the lower percentage of CSA (2 %) and low compaction pressure (250 MPa). The combination of higher percentage of CSA (6%) and higher compaction pressure (300 MPa) exhibited higher density. The justification is that, compaction produces adhesion and bonding of powder particles to improve green strength and facilitates plastic deformation of powder particles that increases compact density which is in accordance with [32].

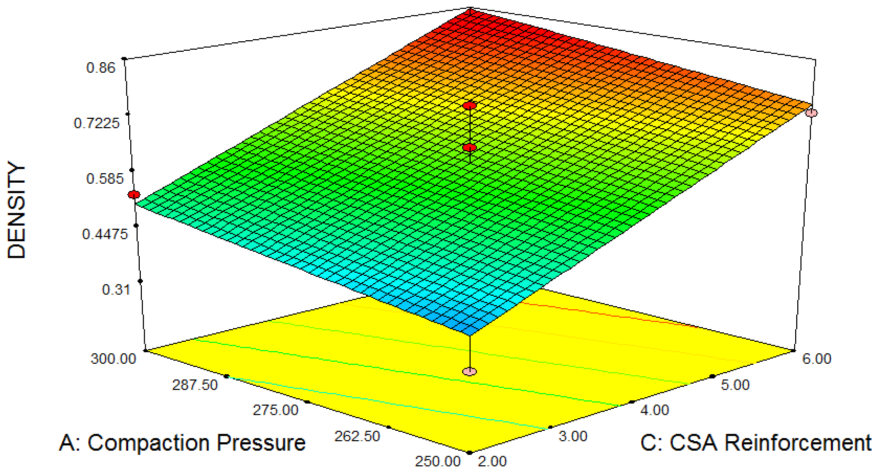

Figure 4. a) Surface plot for density

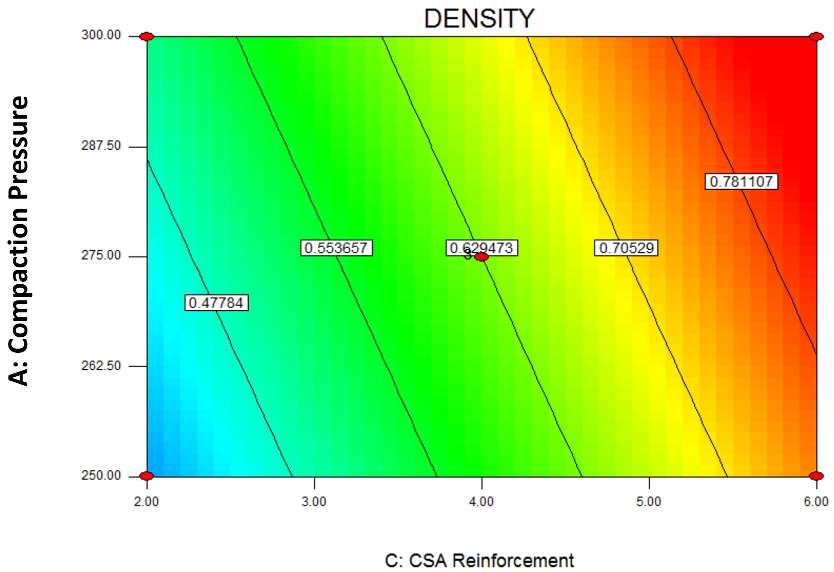

Figure 4. b) Contour plot for density

3.3. Effect of process parameters on porosity

Eliminating the insignificant process variable, the final relationship between the response and the significant variables for the reduced linear model generated for porosity can be represented by equation 7 in terms of actual factors is,

$$Porosity = +141.0042 - 0.2591 \times Compaction\ pressure - 10.03875 \times CSA\ reinforcement \quad (7)$$

The ANOVA table for porosity is shown in table 7. The model F-value of 48.36 indicates that the model is significant. The significant model terms are compaction pressure and reinforcement percentage of CSA. The "Lack of Fit F-value" of 0.29 implies the lack of fit is not significant relative to pure error. There is 92.95 % chance that a "Lack of Fit F-value". The predicted R^2 value of 0.8332 is in reasonable agreement with the adjusted R^2 value of 0.8712. The adequate precision ratio of 19.754 indicates an adequate signal and the model is desirable.

Table 7. ANOVA results for porosity

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	3560.51	2	1780.26	48.36	< 0.0001	significant
A-Compaction Pressure	335.66	1	335.66	9.12	0.0107	
C-CSA Reinforcement	3224.85	1	3224.85	87.61	< 0.0001	
Residual	441.71	12	36.81			
Lack of Fit	259.89	10	25.99	0.29	0.9295	not significant
Pure Error	181.82	2	90.91			
Cor Total	4002.22	14				

It is obvious from the 3D surface graphs and 2D contour graphs shown in figure 5a, figure 5b that porosity decreased with increase in percentage of CSA reinforcement and increase in compaction pressure. The composites produced by the percentage of combinations of low percentage of CSA reinforcement and low compaction pressure exhibited maximum amount of porosity in it, whereas both at higher values depicted minimal porosity. The purpose for minimal amount of porosity can be attributed to filling of pores which lead to better packing and shrinkage of voids.

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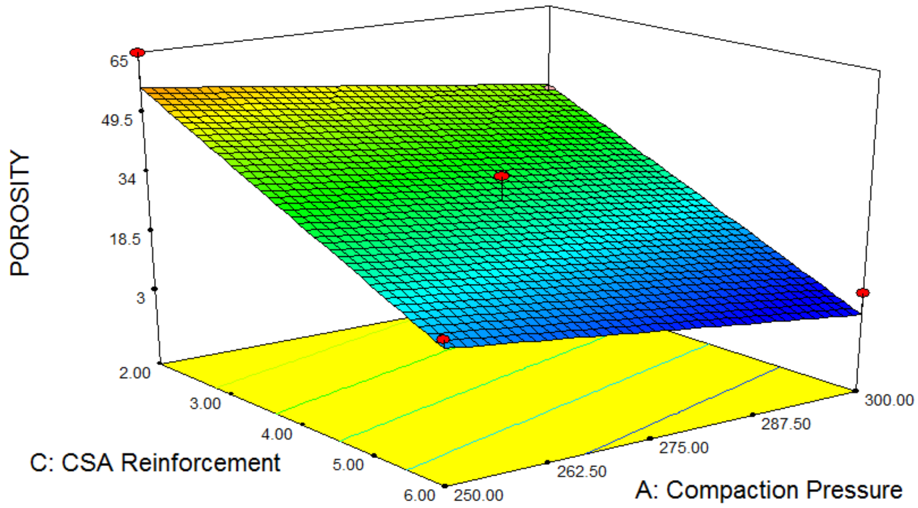


Figure 5. a) Surface plot for porosity

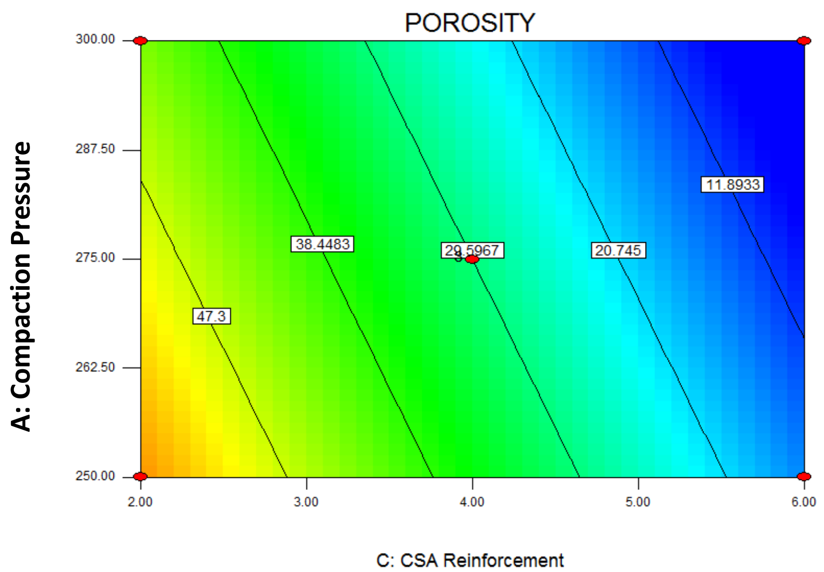
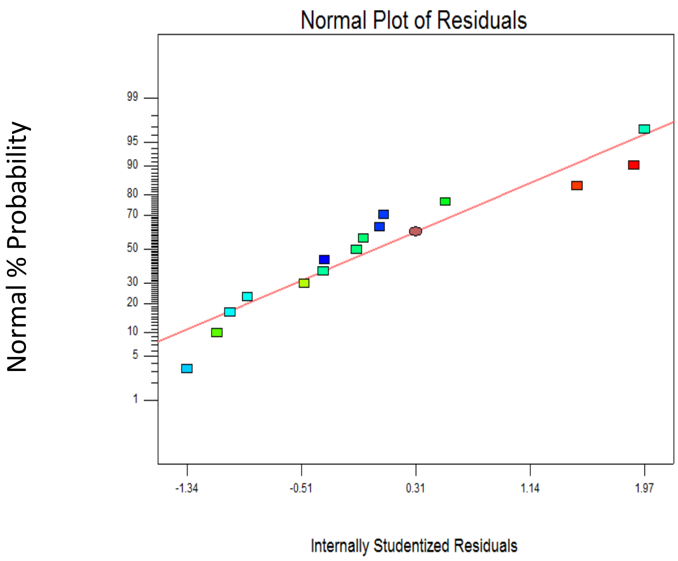


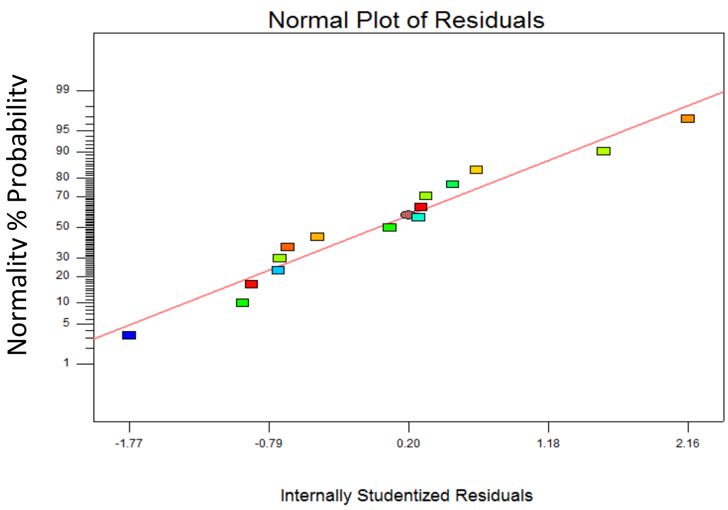
Figure 5. b) Contour plot for porosity

3.4. Normality of data

Normal probability plot is a diagnostics plot which indicates whether the residual follow a normal distribution. The normal probability of plot of residuals (difference between predicted and measured value) for responses are shown in figure 6. It illustrates that points distributed on the plot are approximately lying along the straight line with no reasonable outliers which shows that dispersal of regression residuals is at standard ranges. Thus, it can be contingent that regression model is acceptable.


Normal Plot of Residuals
Normal % Probability
99
95
90
80
70
50
30
20
10
5
1
-1.34
-0.51
0.31
1.14
1.97
Internally Studentized Residuals

(a)


Normal Plot of Residuals
Normality % Probability
99
95
90
80
70
50
30
20
10
5
1
-1.77
-0.79
0.20
1.18
2.16
Internally Studentized Residuals

(b)

(C)

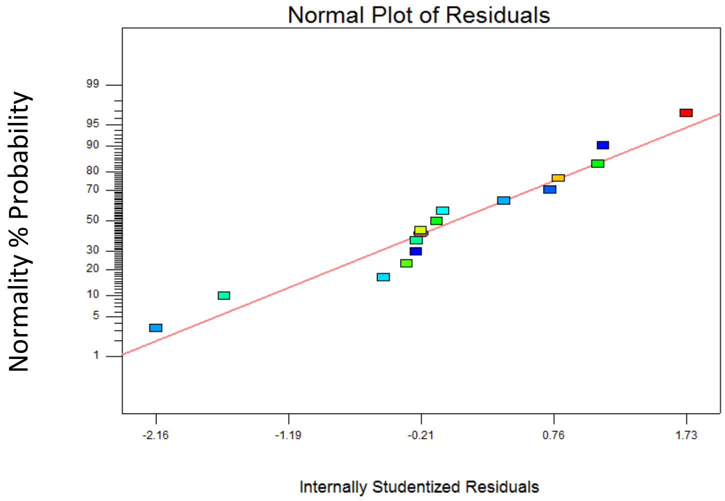


Figure 6. Normal probability plot for a) hardness b) density c) porosity

3.5. Verification of optimized condition and predictive model

The optimization model examines for a combination of factor levels that simultaneously fulfil the necessities placed on each of responses and factors. The preferred goal of each factor and response is chosen. In the present investigation to find a beneficial and desired set of combinations that will meet all goals, the factors were set within range. The objective of the function is maximum hardness and density at a minimum porosity and the goals were chosen. The 30 set of optimum values were found. Among them, the solution containing the desired function such as high hardness, high density and low porosity is selected for the validation of response surface linear regression in order to authenticate the mathematical model with the prediction interval of 95 %. The results of conformation test and their comparisons with predicted values are enumerated in table 8. The results shows that residual and percentage error are minor and percentage error between actual and predicted values for all responses lies within 95 % prediction interval.

Table 8. Confirmatory test

Process parameters			For Hardness (HV)				For Density (g/cc)				For Porosity (%)				Desirability
A	B	C	Actual	Predicted	Residual	Error (%)	Actual	Predicted	Residual	Error (%)	Actual	Predicted	Residual	Error (%)	
299.88	549.65	6	132.787	131.65	1.37	0.856	0.85667	0.834	0.023	1.129	3.07297	3.025	0.0489	1.561	0.906

4. CONCLUSIONS

Al-B_4C-CSA composites with varying percentage of CSA and process parameters like compaction pressure and sintering temperature have been successfully fabricated by powder metallurgy technique. Moreover the use of RSM to study the effect of design factors on hardness, density and porosity of prepared hybrid composite was explored in the study and the conclusions are drawn as follows,

1. Hardness increased with increase in reinforcement percentage of CSA. The highest hardness value of 165.1 HV was obtained at composite having 6 % reinforcement of CSA.
2. Increase in percentage of CSA and compaction pressure increased the density of composite as compaction produces binding of powder particles.

3. The porosity and density are inversely proportional. The porosity decreased with increase in compaction pressure and CSA percentage of reinforcement. Higher contents of CSA, the particles are close to each other and the compaction causes powder particles to contact, filling the empty gaps. No direct effect of sintering temperature on responses is detected.
4. In the developed linear model, the predicted R^2 values of all the responses are in sensible covenant with the adjusted R^2 values.
5. In RSM, the lack of fit for responses were studied and the P-value of lack of fit is not significant which implies that the proposed linear model fit the experimental data and the independent parameters have considerable effects on responses.
6. Optimum process parameters for higher hardness, high density and minimal porosity are determined. The optimal values obtained was 299.18 MPa compaction pressure, 549.65^{0}C sintering temperature and 6 % reinforcement of CSA which has composite desirability of 0.906.
7. The predicted and experimental values are satisfactory which are fairly close to each other indicating the model is adequate.
8. The Proposed hybrid composite could be used in the applications of disc brake and brake pad.

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