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Compositions Optimization of Antang Corundum For Developing Advanced Ceramic

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Abstract - The research aims to study and optimize the formulation of materials required for advanced ceramic production using response surface methodology (RSM). In this research effort, the five (5) process independent variables studied with their corresponding levels are: *Antang* corundum powder, A (92.2 – 100 %W); polyvinyl alcohol, B (0 – 5 %W); CaO, C (0 – 2.3 %W); MgO, D (0 – 0.5 %W);and the sintering temperature, E (1200 – 1500 °C).The mechanical property responses determined were density, *ρ*, compressive strength, *C/S,*flexural strength, *F/S*; which are key characteristics of ceramics for armour applications. The optimized density, compressive strength and flexural strength of the sintered *Antang* corundum are 3.45 g/cm^3 g, 1982 MPa and 295 MPa respectively; while the respective RSM prediction values are 3.46 g/cm^3 g, 1979 MPa and 286 MPa. The optimum material compositions from the experiment were Corundum, 95.66 %W; PVA, 2.78 %W; CaO, 1.28 %W; MgO, 0.28 %W at a Sintering Temperature of 1500 °C.On comparing the determined optimum mechanical responses of the sintered *Antang* ceramic with the maximum RSM prediction values, there is high level of assurance in using RSM for the formulation processing ceramic armour development.

Keywords: Armour; Corundum; Response surface methodology; Optimization; Ceramic

Introduction

Recently, ceramics are incorporated into more effective lightweight armour systems. Specifically, low density, high hardness, high rigidity and compression strength of ceramics make it popular and suitable for armour systems; including aircraft structures and military vehicles (Silva *et al.*, 2014; Liaghat *et al*, 2013; Medvedovski, 2006; Zhang *et al.*, 2006; Fawaz *et al.*, 2004; Tolochko, 2011). Composite materials used for protection from high-energy bullets (that is threat projectile that contains a steel core rather than only lead) are mostly combined with a ceramic strike face (Cooper and Gotts, 2003) in order to enhance the system's ballistic effectiveness.

The leading ceramic materials used commercially in the development of ballistic armours are alumina (Al_2O_3), boron carbide (B_4C) and silicon carbide (SiC) (Dateraksa *et al.,* 2012). Advanced high-alumina ceramics are generally produced via dry pressing, injection or hydrostatic molding, hot pressing, extrusion and so on (Badmos and Ivey, 2001). Badmos and Ivey (2001)employed both slip casting and dry pressing techniques for alumina composition preparation. In studying the research results in terms of hardness property, they discovered that the dry pressed samples marginally showed higher hardness values than the slip cast samples.

The performance characteristics of an advanced ceramics largely depends on the starting material compositions, the production technique adopted and the sintering temperature (Badmos and Ivey, 2001). It is on this note that the research seeks to formulate a beneficiated alumina (via froth floatation) for the development of ceramic for armour application and compare with an established ceramics of CoorsTek Ceramics; AD-series. CoorsTek Ceramics is well known in the field of ballistics and it has been effectively used by researchers: Swab *et al*., 2014; Serjouei *et al*., 2014; Bourne *et al*., 2007; Sarva *et al*., 2007; Fawaz *et al*., 2004; Shih *et al*., 1998; Bless *et al*., 1996. CoorsTek AD-85 alumina was specifically referred as the material has alumina content (Al_2O_3-85%) close to the main starting material (*Antang* corundum alumina). The objectives of this work is to formulate and produce advanced ceramic using beneficiated corundum against synthesized alumina; evaluate the mechanical properties and compare it with the established CoorsTek ceramics; and compare the developed ceramics properties with the prediction values of Response surface method.

Materials and Methods

Corundum alumina sourced from *Antang* in Kaduna-Nigeria and purified via froth floatation process is the major component; its chemical composition is presented in Table 1. Polyvinyl alcohol, PVA, served as the binder (produced by Sigma-Aldrich, UK). MgO (produced by Tateho, Japan) additive was used as a grain growth inhibitor. Also, CaO (manufactured by Yida, China) serves as a stabilizer and reduces sintering temperature (Iatsenko *et al*., 2015). The sintering temperature was varied between 1200 - 1500°C for studying the effect of temperature on the developed ceramics.

Table 1: Chemical composition of the beneficiated *Antang* corundum

Compounds	Concentrations (%)
Al_2O_3	65.4
SiO_2	7.9
P_2O_5	0.02
SO_3	0.03
K_2O	0.13
CaO	0.566
TiO_2	2.04
V_2O_5	0.11
Cr_2O_3	0.161
MnO	0.092
Fe_2O_3	16.09

Design-Expert software (Version 6.0.6) installed on an *ACER* Laptop running windows 10 operating system was utilized for the design of experiments. The option "new design" was clicked upon to design new experiments for the materials formulation. The Response Surface option of the software was selected and then D-Optimal was selected from the drop icon as the tool that allows up to five (5) components desired for this experiment. The response surface methodology (RSM) was used in material formulations and studying the effect of interactions of the five independent variables; details in Table 2. The effects of the variables (A: Corundum, B: PVA, C: CaO, D: MgO and E: Temperature) in percentage weight (A-D) were studied on three responses (density, compressive strength, flexural strength). This resulted in 31 experiment runs in Table 3.

Blending, Pressing and Sintering

The *Antang* corundum powder, calcium oxide, magnesium oxide and binder were weighed and thoroughly blended in percentage weight shown in Table 3 to form nearly dry free flowing powders using pestle and mortar.

Dry pressing method was adopted for this experimentation. The blended mixture was then filled in the prepared metal moulds and compacted uniaxially under 110 MPa using Universal Testing Machine (WA-300B by Okhard Machine Tools, 2014 at Elizade University, Nigeria).

The pressed green body were preliminary fired at temperature of 200°C in order to reduce the risk of cracking by using Laboratory dry oven (DHG-9101-2A by Searchtech Instruments, 2014 at Elizade

University). The samples were then sintered, using a Brother Furnace (Model number: XD-1700M) at Elizade University. The samples were heated at temperature range of 1200-1500°C for 60 minutes dwelling time to attain the ceramic bond which gives the desired mechanical characteristics. The samples were then allowed to cool before further studies.

Table 2: The level and range of actual independent variables selected for materials formulation process in Response Surface Methodology

Nomenclature	Process Factors	Unit	Low Level (-1)	High Level (+1)
A	Corundum	% Weight	92.2	100
B	PVA	% Weight	0	5
C	CaO	% Weight	0	2.3
D	MgO	% Weight	0	0.5
E	Temperature	°C	1200	1500

Mechanical Characterizations

Density - In accordance to ASTM C 20-87, density of the sintered samples were determined by dimensional analysis using Equation 3.1 (Papitha *et al*., 2013). After sintering, the samples were first weighed and the volume was measured by displacement method using graduated cylinder (Sutcu, 2004).

$$\rho = \frac{M}{V} \tag{1}$$

Where, $M \text{ and } V$ are the mass and volume of the sample respectively.

Compressive Strength Test

This was carried out in accordance to ASTM C773-88 (Kutz, 2002; CoorsTek, 2008).The experiment involved subjecting the sintered *Antang*corundum sample to uniaxial compression force using the Universal Testing Machine. The samples were then placed between the compression anvils to commence uniaxial compression testing.

The cold compressive strength, CCS (MPa), was calculated by Equation 2 (Ekka, 2011):

$$CCS = \frac{F}{A} \tag{2}$$

Where, F is the maximum or yield force; A is the area normal to the applied force, A = w * b

Flexural Strength Test

Testing in flexure (bend test) was done by a three-point loading of a test specimen using ASTM C1161-94. Figure 3.1 shows the specimen configuration used for the experiments. The flexural strength,σ_{fs} from the applied load F was calculated using Equation 3 given by Unaler (2005):

$$\sigma_{fs} = \frac{3FL}{2bh^2} \tag{3}$$

Where,

L is the span length ; *b* is width and *h* is height.

Table 3: Design of experiment for material formulation

	Factor 1	Factor 2	Factor 3	Factor 4	Factor 5
Run	**A: Corundum**	**B: PVA**	**C: CaO**	**D: MgO**	**E: Temp**
	% Wt	% Wt	% Wt	% Wt	°C
14	92.2	5	2.3	0.5	1200
22	92.2	5	2.3	0.5	1500
30	92.2	5	2.3	0.5	1500
6	92.7	5	2.3	0	1500
13	92.7	5	2.3	0	1200
4	93.44	5	1.28	0.28	1350
3	94.5	5	0	0.5	1500
19	94.5	5	0	0.5	1200
31	94.5	5	0	0.5	1200
5	94.7	2.5	2.3	0.5	1500
8	94.7	2.5	2.3	0.5	1200
15	95	5	0	0	1200
21	95	5	0	0	1500
29	95	5	0	0	1500
1	95.2	2.5	2.3	0	1200
27	95.2	2.5	2.3	0	1500
12	95.66	2.78	1.28	0.28	1500
17	96.1	2.5	1.15	0.25	1350
18	96.35	2.5	1.15	0	1350
20	97.2	0	2.3	0.5	1200
26	97.2	0	2.3	0.5	1350
23	97.25	2.5	0	0.25	1350
11	97.5	2.5	0	0	1200
9	97.7	0	2.3	0	1500
10	97.7	0	2.3	0	1200
2	99.5	0	0	0.5	1500
25	99.5	0	0	0.5	1200
28	99.5	0	0	0.5	1500
7	100	0	0	0	1500
16	100	0	0	0	1200
24	100	0	0	0	1500

Results and Discussion

Mechanical properties characterization results

Table 4 presents the materials formulation and the results of mechanical characterization on the sintered *Antang* ceramic. Density, compressive strength and flexural strength properties determined from the sintered samples as the responses from the various independent material variables formulations were discussed.

Density

It is observed from Table 4 that the sintered *Antang* corundum have density values in the range of 2.93 - 3.61 g/cm^3. This is in agreement with the work of Medvedovski (2006) that recorded 3.52 – 3.56 g/cm^3. Figiel *et al.* (2011) recorded density ranging between 3.54 – 3.92 g/cm^3, the high value range was achieved as researchers employed different non-conventional methods of sintering. Also, Bakhsh *et al.* (2014) was able to attain 3.86 g/cm^3 of density in their work, this is expected as nano alumina powder with dispersed carbon nanotubes were used as raw materials. As it is the trend in other researches (Islam *et al.*, 2012; Mongkolkachit *et al.*, 2010), the density of this sintered *Antang* corundum generally increases with the increase in sintering temperature. Sample run 26 without the PVA produced the highest value of density as there was no binder to be burnt out which consequently create voids or deliberate pores for filters (Ma and Zhao, 2012; CaO *et al.*, 2006) that lower the density. Reducing the quantity of the binder, PVA, is observed to increase the density of the sintered sample; compare samples runs 3, 19, 31 with runs 5, 8. More often, MgO and CaO are also observed to play significant role in producing maximum densification of the sample at high content concentrations.

In this research work, the optimum density obtained in the run 12, 3.45 g/cm^3, compete favourably with CoorsTEK (2008) AD-85 of density 3.42 g/cm^3 that serves as control. This showed that the density is one of the good application property in this work.

Compressive Strength

For the experimental results of compressive strength, values ranging between 1673 and 1985 MPa (Table 4) was obtained. The compressive strength of the samples majorly increases with increase in the sintering temperature; this is in agreement with Iatsenko *et al.* (2015). The common trend observed in this research work is that the compressive strength also increases with the increase in the binder, PVA; this is in agreement with the work of Cao *et al.* (2007).

Aside the sample compositions that have only MgO as the additive (runs 2, 25, 28), presence of MgO with other additives (CaO and PVA) produced enhanced compressive strength of values ranging from 1711 – 1985 MPa. The significant of binder in production of high compressive strength is also observed by comparing groups of sample runs 2, 25, 28 with runs 3, 19, 31. The samples (runs 15, 21, 29) without MgO but with the same PVA content with samples (runs 3, 19, 31) present higher values of compressive strength.

It is also observed that CaO in the range of 1.15 – 1.28 %W of the sample formulation produced high values of compressive strength in the range of 1843 – 1982 MPa. This confirms the optimum quantity of CaO required for the armour ceramic formulation.

The optimum compressive strength obtained in the targeted run 12 is 1982 MPa. This result surpasses the CoorsTEK (2008) AD-85 of 1930 MPa that serves as control.

Flexural Strength

For the experimental results of flexural strength, values ranging between 191 and 295 MPa (Table 4) was obtained. It is observed that the flexural strength of the sintered *Antang* corundum is significantly increased with increase in temperature with exception of few sample groups (runs 14, 30; 6, 13; 5, 8; 1, 27) that shows decrease in flexural strength as temperature rises. This exceptional behaviour could have arisen from poor formulation mixture or sintering process.

The absence of all the additives in the sintered sample significantly drops the average value of flexural strength (198.33 MPa) recorded for runs 7, 16, and 24 with 201 MPa, 197 MPa and 197 MPa respectively. However, the inclusion of only 0.5 %W of MgO is observed to slightly improve the flexural strength average value of samples runs 2, 25, 28. Presence of CaO only (runs 9, 10) also proofs to increase the values of flexural strength but its effect is less than that of samples with MgO inclusion only. This means that MgO is more important in the armour ceramic formulation for better flexural strength characteristics. In comparing with other samples with only one additive, the flexural strength is observed to be best on the inclusion of PVA only as it is shown in runs 15, 21, 29.

Formulating the sample with maximum additives inclusion (runs 14, 22, 30) show higher values of flexural strength compared to inclusion of only individual additive. Sample run 12 with 2.78 %W PVA, 1.28

%W CaO and 0.28 %W MgO provided the highest value (295 MPa) of flexural strength in all the formulations. This is the optimum flexural strength obtained and it has a unit less than that of CoorsTEK (2008) AD-85 with 296 MPa; the difference can be traced to the differences of formulations and processing routes. Also, the result is five units less than the work of Medvedovski (2006) that recorded 300 MPa but it is far higher than the extruded alumina for high temperature application by Mongkolkachit *et al*., (2010) that has maximum value of 83.01 MPa.

Table 4: Experimental Design Formulations and the Mechanical Characterization Studied

S/N	Run	**A**	**B**	**C**	**D**	**E**	*ϱ*	*C/S*	*F/S*
		% W	% W	% W	% W	°C	g/cm³	MPa	MPa
1	14	92.2	5	2.3	0.5	1200	3.38	1883	286
2	22	92.2	5	2.3	0.5	1500	3.42	1940	270
3	30	92.2	5	2.3	0.5	1500	3.39	1875	285
4	6	92.7	5	2.3	0	1500	3.4	1910	264
5	13	92.7	5	2.3	0	1200	3.31	1900	272
6	4	93.44	5	1.28	0.28	1350	3.4	1979	287
7	3	94.5	5	0	0.5	1500	3.52	1863	242
8	19	94.5	5	0	0.5	1200	3.5	1855	231
9	31	94.5	5	0	0.5	1200	3.51	1859	233
10	5	94.7	2.5	2.3	0.5	1500	3.57	1850	218
11	8	94.7	2.5	2.3	0.5	1200	3.6	1985[b]	289
12	15	95	5	0	0	1200	3.44	1699	260
13	21	95	5	0	0	1500	3.54	1731	260
14	29	95	5	0	0	1500	3.35	1725	263
15	1	95.2	2.5	2.3	0	1200	3.43	1873	264
16	27	95.2	2.5	2.3	0	1500	3.55	1840	208
17	12	95.66	2.78	1.28	0.28	1500	3.45	1982	295[b]
18	17	96.1	2.5	1.15	0.25	1350	3.51	1969	277
19	18	96.35	2.5	1.15	0	1350	3.51	1843	200
20	20	97.2	0	2.3	0.5	1200	3.46	1781	205
21	26	97.2	0	2.3	0.5	1350	3.61[b]	1851	221
22	23	97.25	2.5	0	0.25	1350	3.45	1711	221
23	11	97.5	2.5	0	0	1200	3.37	1673[a]	261
24	9	97.7	0	2.3	0	1500	3.55	1799	207
25	10	97.7	0	2.3	0	1200	3.4	1777	209
26	2	99.5	0	0	0.5	1500	3.44	1700	212
27	25	99.5	0	0	0.5	1200	2.93[a]	1778	191[a]
28	28	99.5	0	0	0.5	1500	3.44	1675	208
29	7	100	0	0	0	1500	3.47	1770	201
30	16	100	0	0	0	1200	3.45	1764	197
31	24	100	0	0	0	1500	3.45	1758	197

Note: A- Beneficiated *Antang* Corundum, B- PVA, C- CaO, D- MgO, E- Temperature; *ϱ*- density, *C/S*- compressive strength, *F/S*- flexural strength (Minimum [a]; Maximum [b])

Statistical Analysis for Mechanical Properties

Mathematical models for the sintered *Antang* corundum ceramic

This work presents multiple analyses carried out using response surface analysis to fit experimental data mathematical models aiming at obtaining optimal properties for the studied response variables. Then, the relationship between the five independent variables as presented in Table 3 are also studied on the six responses.

The RSM adopted allowed the development of models presented in Equations 4 – 6where each of the response was assessed as a function of the independent variables. In the equations, density, ϱ; compressive strength, C/S; flexural strength, F/S are the response functions. The input variable parameters are corundum, polyvinyl alcohol (PVA), calcium oxide (CaO), magnesium oxide (MgO) and the sintering temperature designated respectively as A,B, C, D and E. According to Keshani *et al.* (2010), the models show the relationship between the responses and the variable parameters and can be used to estimate and compare both the predicted and experimental data.

$$\rho = \begin{pmatrix} 4.46 + 2.56B + 0.072C - 0.045D + 0.13E - 0.65A^2 \\ +0.22C^2 - 0.16D^2 - 0.45E^2 + 0.045AB + 0.03AC - \\ 0.027AE - 0.19BC + 0.048BD - 0.051BE + 0.065CD \\ -0.12CE - 0.061DE - 2.41B^3 - 0.53AB^2 + 0.45AD^2 \end{pmatrix} \quad 4)$$

$$\frac{C}{S} = \begin{pmatrix} 2255.39 + 101.73A + 789.15B + 45.81C + 24.27D + 66.15E - \\ 331.6A^2 + 2.82B^2 - 4.63D^2 - 104.7E^2 + 15.45AB - 2.17AD + \\ 1.47AE - 46.18BC + 11.95BD - 17.76BE - 16.93CD - 79.05CE \\ -12DE - 697.49B^3 - 202.41AB^2 + 174.89AD^2 - 94.62AE^2 \end{pmatrix} \quad (5)$$

$$\frac{F}{S} = \begin{pmatrix} 309.38 - 4.01A + 28.24B + 12.85C + 0.93D + 1.47E \\ +3.36A^2 - 42.35B^2 - 16.59C^2 - 9.33D^2 - 14.82E^2 \\ -1.29AB + 3.15AC - 3.87AD + 2.35AE + 4.49BC - \\ 2.47BD - 0.23BE + 7.2CD + 0.71CE + 5.5DE \end{pmatrix} \quad (6)$$

Density

The statistical significance of the model Equation 4 for the density of the *Antang* corundum was checked through the analysis of variance (ANOVA) and this is presented in Table 5.

Table 5: ANOVA for Antang corundum density response surface model

Source model	Sum of squares	DF	Mean square	F-Value	Prob > F	
Model	0.43	20	0.021	38.12	< 0.0001	significant
B	0.061	1	0.061	109.32	< 0.0001	
C	0.063	1	0.063	111.66	< 0.0001	
D	0.018	1	0.018	32.12	0.0002	
E	0.014	1	0.014	24.13	0.0006	
A^2	0.039	1	0.039	70.39	< 0.0001	
C^2	0.015	1	0.015	27.15	0.0004	
D^2	0.029	1	0.029	52.24	< 0.0001	
E^2	0.082	1	0.082	147.21	< 0.0001	
AB	0.030	1	0.030	53.68	< 0.0001	
AC	8.878E-003	1	8.878E-003	15.85	0.0026	
AE	0.014	1	0.014	24.13	0.0006	
BC	0.061	1	0.061	109.30	< 0.0001	
BD	0.035	1	0.035	62.75	< 0.0001	
BE	0.029	1	0.029	52.19	< 0.0001	
CD	0.038	1	0.038	68.35	< 0.0001	
CE	0.012	1	0.012	22.22	0.0008	
DE	0.069	1	0.069	122.65	< 0.0001	
B^3	0.061	1	0.061	109.27	< 0.0001	

AB^2	0.025	1	0.025	44.48	< 0.0001	
AD^2	0.022	1	0.022	38.62	< 0.0001	
Residual	5.601E-003	10	5.601E-004			
Lack of Fit	3.551E-003	5	7.101E-004	1.73	0.2806	not significant
Pure Error	2.050E-003	5	4.100E-004			
Cor Total	0.43	30				

In this statistical analysis, Table 5, the model F-value of 38.12 has a probability (p) value "Prob > F" of less than 0.05 which indicates that the model terms are significant as presented by other researchers (Balasubramania *et al.*, 2012; Yamin *et al.*, 2013; Arunachalam and Annadurai, 2011; Keshani *et a*l., 2010) that adopted this method. The high value coefficient of determination ($R^2 = 0.9871$), which is higher than 0.80 (Keshani*et al.*, 2010), shows that the cubic polynomial model was highly significant and sufficient.

Surface Plots of Variable Parameters and Responses Studied for Density

From Figure 1, factors A, B are varied simultaneously while C, D, E factors are fixed at 1.69 %W, 0.23 %W and 1300 °C respectively. The 3D plot shows that the density initially decreases with increase in binder PVA between 0 - 2.5 %W, while about 94.15-96.10 %W of *Antang* corundum produces the peak value for density.

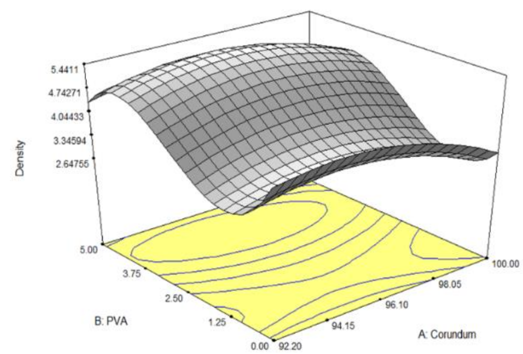

Figure 1. 3D-Surface plots for density of sintered corundum as a function of PVA and Corundum

Compressive Strength

Table 6 presents the analysis of variance (ANOVA) for compressive strength of the sintered *Antang* corundum using the Design Expert software. The RSM predicted as sum of constant, five linear terms in A, B, C, D, E first-order effects, nine interaction effects (AB, AD, AE, BC, BD, BE, CD, CE, DE), seven second-order effects (A^2, B^2, D^2, E^2, AB^2, AD^2, AE^2) and a cubic effect of B^3.As shown in Table 4.4, F-value of 34.41 has a probability (p) value of <0.0001 which indicates that the model terms are significant in agreement with other researchers (Bamgboye and Adejumo, 2015; Sanda *et al.*, 2015; Balasubramania *et al.*, 2012; Yasin *et al.*, 2013; Keshani *et a*l., 2010).

3D-Surface Plots for Compressive Strength

Figure 2 shows the 3D-Surface plots for compressive strength of sintered alumina corundum as a function of interaction effects of independent variables. From Tabel 4 factors A, B are varied simultaneously while C, D, E factors are fixed at 1.69 %W, 0.23 %W and 1300 °C respectively. The 3D plot shows that the compressive strength increases with the binder PVA between 0.5- 3.4 %W, while the compressive strength increases gradually with corundum composition up to 96.1 %W before gradually fall.

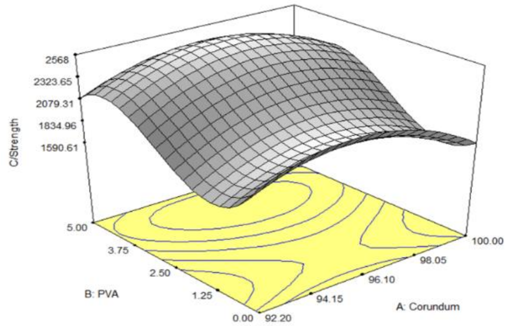

Figure 2. 3D-Surface plots for compressive strength of the ceramic as a function of PVA and Corundum

Table 6: ANOVA for compressive strength from *Antang* corundum material formulation

Source model	Sum of squares	DF	Mean square	F-Value	Prob > F	
Model	2.588E+005	22	11765.39	34.41	< 0.0001	significant
A	2194.06	1	2194.06	6.42	0.0351	
B	1708.06	1	1708.06	5.00	0.0559	
C	10360.55	1	10360.55	30.30	0.0006	
D	4620.25	1	4620.25	13.51	0.0063	
E	13819.13	1	13819.13	40.42	0.0002	
A^2	6738.91	1	6738.91	19.71	0.0022	
B^2	7.09	1	7.09	0.021	0.8891	
D^2	12.95	1	12.95	0.038	0.8505	
E^2	1554.30	1	1554.30	4.55	0.0656	
AB	2823.19	1	2823.19	8.26	0.0207	
AD	29.21	1	29.21	0.085	0.7775	
AE	31.95	1	31.95	0.093	0.7677	
BC	2682.74	1	2682.74	7.85	0.0232	
BD	2169.22	1	2169.22	6.34	0.0359	
BE	1917.43	1	1917.43	5.61	0.0454	
CD	1918.38	1	1918.38	5.61	0.0453	
CE	14719.83	1	14719.83	43.05	0.0002	
DE	2141.92	1	2141.92	6.26	0.0368	
B^3	1433.56	1	1433.56	4.19	0.0748	
AB^2	21950.44	1	21950.44	64.20	< 0.0001	
AD^2	3722.85	1	3722.85	10.89	0.0109	
AE^2	6641.75	1	6641.75	19.43	0.0023	
Residual	2735.27	8	341.91			
Lack of Fit	936.27	3	312.09	0.87	0.5161	not significant
Pure Error	1799.00	5	359.80			
Cor Total	2.616E+005	30				

Flexural Strength

Table 7 presents the analysis of variance (ANOVA) for flexural strength using the DesignExpert software. The RSM predicted as sum of constant, five linear terms in A, B, C, D, E first-order effects, ten interaction effects (AB, AC, AD, AE, BC, BD, BE, CD, CE, DE) and five second-order effects (A^2, B^2, C^2, D^2, E^2).

3D-Surface Plots of Variables and Responses Studied for Flexural Strength

Figure 3 shows the 3D-Surface graphics for flexural strength of sintered alumina corundum as a function of interaction effects of independent variables. From Figure 3, factors C, B are varied simultaneously while A, D, E factors are fixed at 92.47 %W, 0.23 % W and 1300°C respectively. The 3D plot also displays that the flexural strength increases with the increase in binder PVA but with less flexural strength increase with CaO content increment.

Table 7: ANOVA for flexural strength from Antang corundum material formulation

Source model	Sum of squares	DF	Mean square	F-Value	Prob > F	
Model	33863.61	20	1693.18	21.35	< 0.0001	significant
A	377.82	1	377.82	4.76	0.0540	
B	13715.65	1	13715.65	172.91	< 0.0001	
C	3223.36	1	3223.36	40.64	< 0.0001	
D	19.06	1	19.06	0.24	0.6346	
E	37.92	1	37.92	0.48	0.5051	
A^2	15.67	1	15.67	0.20	0.6662	
B^2	2276.50	1	2276.50	28.70	0.0003	
C^2	543.04	1	543.04	6.85	0.0258	
D^2	185.43	1	185.43	2.34	0.1573	
E^2	353.39	1	353.39	4.46	0.0610	
AB	28.08	1	28.08	0.35	0.5651	
AC	213.42	1	213.42	2.69	0.1320	
AD	213.01	1	213.01	2.69	0.1323	
AE	81.55	1	81.55	1.03	0.3345	
BC	346.02	1	346.02	4.36	0.0633	
BD	106.66	1	106.66	1.34	0.2732	
BE	0.92	1	0.92	0.012	0.9163	
CD	939.18	1	939.18	11.84	0.0063	
CE	8.73	1	8.73	0.11	0.7470	
DE	457.70	1	457.70	5.77	0.0372	
Residual	793.23	10	79.32			
Lack of Fit	620.73	5	124.15	3.60	0.0931	not significant
Pure Error	172.50	5	34.50			
Cor Total	34656.84	30				

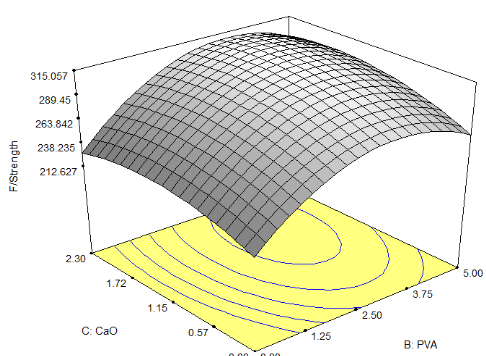

Figure 3. 3D-Surface plots for flexural strength of sintered corundum as a function of CaO and PVA

From Table 4, sample run 12 represents the best combined mechanical properties among others. This can be attributed to the right combination of the raw materials as well as adequate sintering temperature which is also supported by the significance result output of the DesignExpert software. The optimized formulation of run 12 (AC-R12) required five formulated independent variables: A: Corundum, 96.1 %W; B: PVA, 1.25 %W; C: CaO, 1.72 %W; D: MgO, 0.38 %W and E: Temperature, 1500 °C.

The three studied responses of the sintered *Antang* corundum ceramic results and the predicted properties are presented in Table 8 with close values.

Table 8: Comparing the actual and predicted values of the Sintered *Antang* Corundum

Properties	*AC-R12*	*Predicted*
Density(g/cm^3)	3.45	3.46
Compressive Strength (MPa)	1982	1979
Flexural Strength (MPa)	295	286

Despite the lower Al_2O_3 content of the *AC-R12*, its density of 3.45 g/cm^3 compete favourably with the work of Figiel *et al.* (2011) and Tolochko (2011). The flexural strength of 295 MPa also agrees with and exceeds Silva *et al.* (2014) that recorded 225 MPa.

Conclusions

The work investigated the effect of each independent variable polyvinyl alcohol, magnesium oxide, calcium oxide and sintering temperature on the formulation of advanced ceramic from the beneficiated *Antang* corundum for armour applications. Summarily, Design Expert software 6.0.6 was successfully used in the formulation and analyzing the essential armour mechanical characterization which projects run 12 as the best material formulation. The compositions optimization of *Antang* corundum for developing advanced ceramic were Corundum, 95.66 %W; PVA, 2.78 %W; CaO, 1.28 %W; MgO, 0.28 %W at sintering temperature of 1500 °C; which produced comparable properties with the control AD-85. The research applied uniaxial dry pressing method successfully, on the beneficiated *Antang* corundum, for developing the ceramic component of composite armour system and found that an optimum sintering temperature of 1500 °C for effective material treatment.

From the research result, the mechanical properties of the developed *Antang* corundum were found close to and some instances surpassed that of the CoorsTek AD-85 selected as the reference alumina ceramic. Also, it is observed that the values of both actual mechanical properties and the predicted values are closely related; this confirms the effectiveness and reliability of the RSM analysis using DesignExpert software.

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