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Statistical assessment of physicomechanical properties of refractory ceramics based on petroleum waste sludge -bauxite compositions

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Statistical assessment of physicomechanical properties of refractory ceramics based on petroleum waste sludge -bauxite compositions

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Abstract: The aim of this work is to apply the analysis of variance (ANOVA) statistical program to the experimental data of high-quality refractory ceramics prepared from various compositions of petroleum waste sludge (PWS) and raw bauxite mineral to reach a precise and conclusive decision on a statistical basis to the optimum mix that is thought to be more suitable for use in refractory applications. Seven ceramic mixes were prepared from various proportions of PWS and bauxite varying between 0 and 100 wt. % via solid state technique with heat treatment at different degrees of firing reached 1600 °C. The physicomechanical properties namely, linear change, mechanical strength, bulk density, as well as apparent porosity were tested according to the international standards. The One-way ANOVA proves that there is statistically difference regarding linear shrinkage (p = 0.01) and mechanical strength (p < 0.001) for six groups of firing temperature [F (5, 24) = 15.87, p < 0.001]. There was also a statistically significant difference in both bulk density of the ceramic bodies for the six groups [F $(5, 24) = 12.5$, $p = 000$ and the apparent porosity in mean apparent porosity [F $(5, 24) = 21.538$, $p = 0.000$]. Thus, the Oneway ANOVA results are compatible with the results shown in our previous published data. Moreover, the test added a good value by showing CM4 almost like CM3 and economically it is much better to utilize it instead of CM3 in industrial applications.

Keywords: ANOVA, bauxite, petroleum waste sludge, mechanical, density, porosity, shrinkage.

1. Introduction

Refractories are defined as materials that can stand heat at high temperature and mainly contained alumina oxide (A_1Q_3) and silica oxide $(SiO₂)$ to form a group of aluminosilicate fireclay bricks, this chemical composition serves as a basic for classification of refractories [1]. Refractories are classified as non-metallic, heterogeneous, porous and inorganic materials composed of additives, thermally stable mineral combinations and a binder phase [2-4]. The physical characteristic of refractory is one of the major and essential properties that must be considered in material assortment to produce refractories [5]. Consequently, refractories with particular set of properties are prudently chosen for a precise purpose to meet the exact service conditions and other special requirements. The cost effectiveness of refractories considerably influences cost of refractory product. Therefore, proper selection of refractory materials is extremely essential to ensure low production costs and durability of refractory product. The combination of these properties was to maintain reliability and standards of refractories in the thermal industry. The physical properties include apparent porosity, bulk density, firing shrinkage, water absorption [6].

 Recently, many researchers and industrialist paid attention to waste management to overcome the associated ecological and healthy problems resulting from their steady accumulation as by-products during various industries. [7-14]. Several attempts scientifically and economically were made to develop different methods to make use of these wastes [16,17]. Avoiding the serious environmental risk arises from gases and solid seep to environment without any treatments that can reflected in climatic change [18-20]. Saudi Arabia is one of the largest producing oil countries in the world, the oil industries accompanied with huge industrial wastes during the extraction, manufacture processes. The petroleum sludge (solid wastes) that resulting from oil well drilling, collection, transportation as well as refining processes in the form of complex emulsion

containing different hydrocarbon compounds, heavy metals, water, and solid materials. Using chemical process could remove the hydrocarbons, while the heavy metals and solid particles are still problematic after the industrial manufacturing. The huge accumulation of such remnants (petroleum sludge) causing severe damage to the environment through air pollution, soil and water. Many studies were done to make use of these wastes in different ways, majority of these studies were concentrated on extracting and recovering the hydrocarbons materials [15].

 On the other hand, huge reservoir of bauxite is available in Saudi Arabia, especially in Al-Zubaira region, east of KSA however its use is still limited to the field of aluminium metal industries $[21]$. In our previous research work $[22]$ we have studied the suitability of using PWS and bauxite to produce high-quality ceramics. From the preliminary reading of the physicomechanical and refractory properties results, ceramic mix composition prepared from 40 wt. % PWS and 60 wt.% bauxite was considered as the optimum ceramic mix. These data were published in Ceramics International Journal [22]. However, a credible decision for accurate selection of the most suitable batch composition could not be concluded due to the relative variance in the obtained data. So, some statistical calculations are needed urgently for reaching a more suitable decision from economical point of view. Analysis of variance (ANOVA), a statistical tool applied to the data analysis that have a great utility and flexibility for the experimental data that can be applied in order to determine what experiments should be carried out to help in designing decisions effectively based on the differences between several different groups of treatments and multiple comparisons between the group means using t-tests [23].

 In the present work, we extend our evaluation to the prepared ceramic bodies through statistical studies for the experimental data physicomechanical properties to aid the understanding of chemical processes and contribute to make reliable decisions. To perform this analysis, One-way analysis of variance (ANOVA), is applied. This procedure allows to test the possible differences in physicomechanical properties according to the treatments used, considering that the data are functions.

2. Materials and Methods

2.1. Material

PWS was provided by petroleum company, while bauxite mineral was collected from Al-Zuberia region east KSA. Detailed chemical and mineralogical studies were presented in our previous study [22].

2.2. Experimental

2.2.1. Compositions of the prepared ceramic mixes:

The compositions of the prepared ceramic mixes are given in table (1).

Table 1: Compositions of the ceramic mixes [22]

2.2.2. Physicomechanical properties:

Linear shrinkage (LS, %), bulk density (BD, $g/cm³$), apparent porosity (AP, %) as well as cold crushing strength (CCS., kg/cm²) were tested according to the international standard specifications of refractories [24, 25].

2.2.3. Statistical calculations

A One-way Analysis of variance is a method to test the difference of three or more means at the time. There are many assumptions, among them, the true populations must be normally or approximately, the samples must be independent, and the variance of the populations must be equal. The null hypothesis is the all population means are equal the alternative hypothesis is that at least one mean is different. The test model is

$$
Y_{ij} = \mu_i + \varepsilon_{ij} \qquad i = 1, \dots, I; j = 1, \dots, J, \qquad \varepsilon_{ij} \text{ i.i.d } N(0, \sigma^2),
$$

Table 2: ANOVA table

Where SSB, is the difference between groups, SSW, is the difference within groups, SST, the total of difference, df is the degree of freedom [26,27].

3. Results 3.1. Obtained results

3.1.1. Descriptive statistics for linear shrinkage (%):

The values of linear shrinkage obtained from the experimental data [22] are given in the table 3.

Temperature		Linear shrinkage $(\%)$								
(oC)	CM1	CM2	CM3	CM4	CM5					
800	2.250	2.900	3.150	3.300	3.750					
1000	2.700	3.100	3.500	3.800	3.950					
1200	2.650	2.900	3.100	3.400	3.810					
1400	2.980	3.220	3.450	3.760	3.930					
1500	3.100	3.200	3.600	3.750	3.980					
1600	2.900	3.000	3.200	3.350	3.400					

Table 3: LS of ceramic bodies fired at different firing temperatures [22]

*N.B. Ceramic bodies prepared from CM6 and CM7 batches failed to withstand more than 1300 °C so they were excluded from further study.

Table 4, provides some very useful descriptive statistics for the samples, including; mean, standard deviation and 95% confidence intervals for the dependent variable linear shrinkage (%) for each separate group (CM1, CM2, CM3, CM4, and CM5), as well as for all combined groups (Total). The experiment repeated 30 times, we conducted it with equal replications, six times for each batch. CM5 has the highest mean of linear shrinkage $(\%)$, $(M = 3.8, SD = 0.22)$, followed by CM4 $(M = 1.8, SD = 0.22)$ 3.56, SD = 0.23), CM1 has the smallest mean of $(M = 2.7, SD = 0.3)$, the overall mean is $(M = 3.3, SD = 0.4)$.

Batches	N	Mean	Std.	Std.		95% Confidence Interval for	Minimum	Maximum
			Deviation	Error		Mean		
					Lower Bound	Upper Bound		
CM1	6	2.7633	.30310	.12374	2.4453	3.0814	2.25	3.10
CM2	6	3.0533	.14236	.05812	2.9039	3.2027	2.90	3.22
CM3	6	3.3333	.20897	.08531	3.1140	3.5526	3.10	3.60
CM4	6	3.5600	.23281	.09504	3.3157	3.8043	3.30	3.80
CM5	6	3.8033	.21649	.08838	3.5761	4.0305	3.40	3.98
Total	30	3.3027	.42789	.07812	3.1429	3.4624	2.25	3.98

Table 4: Descriptive statistics for linear shrinkage $(\%)$

Table 5, shows the descriptive statistics, including the mean, standard deviation and 95% confidence interval for the linear shrinkage (%) in different levels of firing temperatures (800 °C to 1600 °C), we measured the linear shrinkage 30 time, five for each temperature, the overall sample mean ($M = 3.3$, $SD = 0.43$), the 1500 °C shows the highest mean ($M = 3.53$, $SD = 0.43$) 0.37) while 800 °C has the lowest mean (M = 3.1, SD = 0.55), it is clear that the mean increases with increasing temperature until 1500 oC then it decreases at 1600 $^{\circ}$ C.

Table 5: Descriptive statistics for linear shrinkage (%)

3.1.2. Descriptive statistics for mechanical strength (Kg/cm²):

The mechanical strength (CCS kg/cm²) values obtained from the experimental data [22] are given in the table 6.

Table 6: CCS of ceramic bodies at different firing temperatures [22]

Table 7, shows the descriptive statistics for the effect of different batches of compositions on the mechanical strength of the ceramic bodies (CCS ($Kg/cm²$)), including the mean, standard deviation and 95% confidence intervals for separate group $(CM1, CM2, CM3, CM4, and CM5)$, CM3 composition shows the largest mean of CCS $(kg/cm²)$, $(M=595, S=285)$, while CM1 shows the lowest one, $(M = 335, S = 148)$, and the total mean is $(M = 460, S = 213)$.

Batches	N	Mean	Std.	Std.		95% Confidence Interval for	Minimum	Maximum
			Deviation	Error	Mean			
					Lower Bound	Upper Bound		
CM1	6	335,0000	148.42507	60.59428	179.2374	490.7626	140.00	510.00
CM2	6	403.3333	168.12694	68.63753	226.8949	579.7717	190.00	600.00
CM3	6	595.0000	284.72794	116.23969	296.1964	893.8036	210.00	910.00
CM4	6	510.8333	215.50909	87.98122	284.6704	736.9963	195.00	710.00
CM5	6	460.5000	195.06281	79.63406	255.7941	665.2059	178.00	640.00
Total	30	460.9333	213.11709	38.90968	381.3541	540.5126	140.00	910.00

Table 7: Descriptive statistics for CCS (Kg/cm²)

Table 8, contains descriptive statistics of cold crushing strength (CCS, $kg/cm²$) of the ceramic bodies at different firing temperatures, these are mean, standard deviation and 95% confidence intervals, (800 $^{\circ}$ C to 1600 $^{\circ}$ C), the experiment done 30 times, repeated equally for all firing temperatures, among them 1500 °C has the largest mean of CCS (kg/cm²), (M = 674, S= 150), 800 °C shows the lowest mean ($M = 182$, S = 26), as we can see from the table the mean of CCS (kg/cm²) increases with increasing the firing temperature until 1500 °C, then it decreases at 1600 °C. The total mean of CCS (kg/cm²) at all firing temperatures is $(M = 461, S = 213)$.

Table 8: Descriptive statistics for CCS (Kg/cm²)

3.1.3. Descriptive statistics for bulk density (g/cm3):

Table 9 shows the bulk densities (BD) values from experimental data [22] for prepared ceramic bodies fired at different temperatures.

Table 10, shows the descriptive statistics for the effect of different batches composition on the bulk density (g/cm^3) of the ceramic bodies prepared including the mean, standard deviation and 95% confidence intervals for separate batches (CM1, CM2, CM3, CM4, and CM5), the number of trails is 30, six trails for each batch, the results show that CM3 composition has the largest mean of bulk density ($g/cm³$), (M = 3.15, S = 0.22), while CM5 have the lowest mean of bulk density ($g/cm³$), (M $= 2.85$, S = 0.14), and the total mean is (M = 2.99, S = 0.24).

Batches	N	Mean	Std. Deviation	Std. Error	95% Mean	Confidence Interval for	Minimum	Maximum
					Lower Bound	Upper Bound		
CM1	6	2.8950	.29992	.12244	2.5803	3.2097	2.40	3.20
CM2	6	3.0433	.26220	.10704	2.7682	3.3185	2.55	3.29
CM3	6	3.1583	.21995	.08979	2.9275	3.3892	2.75	3.38
CM4	6	3.0000	.23143	.09448	2.7571	3.2429	2.70	3.27
CM5	6	2.8567	.14989	.06119	2.6994	3.0140	2.65	3.08
Total	30	2.9907	.24663	.04503	2.8986	3.0828	2.40	3.38

Table 10: Descriptive statistics for Bulk density (g/cm³)

Table 11, gives the descriptive statistics of bulk density $(g/cm³)$ of the ceramic bodies at different firing temperatures, these are, the mean, standard deviation and 95% confidence intervals, for each separate temperature (800 $^{\circ}$ C to 1600 $^{\circ}$ C), the total number of trails is 30, repeated equally for all firing temperatures, among them 1500 °C has the largest mean of bulk density $(M = 3.24, S = 0.11)$ whereas 800 °C shows the lowest mean $(M = 2.6, S = 0.13)$, as we can see from the table (11) the mean increases with increasing the firing temperatures until 1500 °C, then it decreases at 1600 °C (M = 3, S = 0.14). The total mean of bulk density at different firing temperatures is $(M = 2.99, S = 0.25)$.

Table 11: Descriptive statistics for bulk density (g/cm3)

3.1.4. Descriptive statistics for apparent porosity (%):

Table 12, shows the apparent porosity (AP) percentages from experimental data [22] for ceramic bodies at different firing temperature.

Temperature	Apparent porosity $(\%)$								
$(^{\circ}C)$	CM1	CM2	CM3	CM4	CM5				
800	17.03	15.66	13.01	14.66	16.08				
1000	12.93	11.43	10.22	11.96	13.11				
1200	11.86	10.35	08.93	10.77	12.07				
1400	09.04	07.93	06.31	08.50	10.21				
1500	07.33	06.18	04.02	07.03	08.96				
1600	08.91	07.96	05.16	08.19	09.86				

Table 12: AP of the ceramic bodies at different firing temperature [22]

The sample characteristics of the effect of different batches on the apparent porosity of the ceramic bodies are shown in table 13, the descriptive statistics are, the mean, standard deviation and 95% confidence intervals for different batches (CM1, CM2, CM3, CM4, and CM5), as we can see, the highest mean of apparent porosity was in CM5 ($M = 11.5$, $S = 2.62$), while CM3 has the smallest one ($M = 7.9$, $S = 3.39$), the total mean is ($M = 10.188$, $S = 3.22$).

Batches	N	Mean	Std.	Std.	95% Confidence Interval for		Minimum	Maximum
			Deviation	Error	Mean			
					Lower Bound	Upper Bound		
M1	6	11.1833	3.52994	1.44109	7.4789	14.8878	7.33	17.03
M ₂	6	9.9183	3.38236	1.38084	6.3688	13.4679	6.18	15.66
M ₃	6	7.9417	3.39515	1.38606	4.3787	11.5047	4.02	13.01
M4	6	10.1850	2.83872	1.15890	7.2059	13.1641	7.03	14.66
M ₅	6	11.7150	2.62379	1.07116	8.9615	14.4685	8.96	16.08
Total	30	10.1887	3.22971	.58966	8.9827	11.3947	4.02	17.03

Table 13: Descriptive statistics for apparent porosity (%)

Table 14, shows the descriptive statistics of apparent porosity at different firing temperature, the total sample size (number of trails) is 30, divided equally for different temperature, the statistics are, the mean, standard deviation and 95% confidence intervals, for each separate temperature (800 $^{\circ}\text{C}$, 1000 $^{\circ}\text{C}$, 1200 $^{\circ}\text{C}$, 1400 $^{\circ}\text{C}$, 1500 $^{\circ}\text{C}$, 1600 $^{\circ}\text{C}$), from the result, 800 $^{\circ}\text{C}$ group has the largest mean comparing to other groups ($M = 15.29$, $S = 1.5$), while 1500 °C shows the minimum mean ($M =$ 6.7, S = 1.8), the total mean was ($M = 10.188$, S = 3.2).

Table 14: Descriptive statistics for apparent porosity (%)

3.2. Discussion

There was statistically significant difference between groups in comparing the effect of different batches on the linear shrinkage (%) of the ceramic bodies at the ($p < 0.01$) as determined by One-way ANOVA [F (4, 25) = 19.588, $p = 000$], (table 15). A Tukey post hoc test (table 16) revealed that there is statistically difference in linear shrinkage (%) between CM1 and CM3 (P = 0.002) as well as between CM1 and CM4 (P= 000), also between CM1 and CM5 (p = 000). In addition, the test stated that there is statistically difference between CM2 and CM4 ($p = 006$), as well as between CM2 and CM5 ($p =$ 000), the test also shows that there is statistically difference in linear shrinkage (%) between the CM3 and CM5 ($p = 0.01$). However, there were no difference between (CM1 & CM2), (CM2 & CM3), (CM3 & CM4), finally (CM4 and CM5) (p > 0.05). A One- way between groups, ANOVA was performed to compare the effect of different firing temperature on linear shrinkage (%). There wasn't any significant effect of temperatures on linear shrinkage (%) at the $(P < 0.05)$ level, [F (5, 24) $= 0.964$, p $= 0.459$], table 17.

 A One-way between groups analysis of variance was conducted to explore the impact of the firing temperatures on mechanical strength of the prepared ceramic bodies. There was a statistically significant difference (table 18) at the (p < 0.001) level in mechanical strength for five groups of firing temperatures $[F (5, 24) = 15.87, p < 0.001]$. Post-hoc comparison (table 19) using the Tukey HSD test indicated that the mean strength at 800 °C (M = 182.6, SD = 26.4) was significantly different from 1200 °C (M= 480, SD = 116.4), 1400 °C (M = 570, SD = 130.38), 1500 °C (M = 674, SD = 150.4), and 1600 ^oC (M = 610, SD = 142.3), in addition the test revealed that the mean strength of 1000 ^oC (M = 249, SD = 48) was statistically different from 1200 °C (M = 480, SD = 116.4), 1400 °C (M = 570, SD = 130.38), 1500 °C (M = 674, SD = 150.4), and 1600 $\rm{^{\circ}C}$ (M = 610, SD =142.3). There was no statistically significant difference in mean strength between the firing temperatures (1200 °C & 1400 °C), (1200 °C & 1500 °C) and (1200 °C & 1600 °C), (table 20).

ANOVA

ANOVA

Table 17: The Contract of the Table 19:

ANOVA

ANOVA

Table 20: CCS (Kg/cm²)

 A One-way between groups ANOVA was performed to compare the impact of firing temperature on bulk density of the ceramic bodies. Temperatures divided into five groups (800 $^{\circ}$ C, 1200 $^{\circ}$ C, 1400 $^{\circ}$ C, 1500 $^{\circ}$ C, 1600 $^{\circ}$ C). There was a statistically significant difference in bulk density (table 21) of the prepared ceramic bodies for the six groups [F $(5, 24)$ = 12.5, $p = 000$. Post-hoc comparisons using the Tukey HSD test (table 22) indicated that the mean bulk density for 800 °C $(M = 2.6100 SD = 0.13)$ was significantly different from 1200 °C (M = 2.99, SD = 0.15), 1400 °C (M = 3.1, SD = 0.12), 1500 ^oC (M = 3.24, SD = 0.11) and 1600 ^oC (M = 3.08, SD = 0.14). In addition, the test raveled that the mean bulk density for 1200 °C (M = 2.99, SD = 0.15) was significantly different from 800 °C (M = 2.6100 SD = 0.13) only, but it differed from the other firing temperatures. In addition to that the test stated that the mean bulk density for $1500 \degree C$ (M = 3.24 SD = 0.11) was significantly different from 1000 °C ($M = 2.87$ SD = 0.17), but the test showed significant difference between other groups. A One-way ANOVA (table 23) was conducted also to compare the effect of different batches on the bulk density of the ceramic bodies, the test shows that there wasn't any significant effect of different batches on the mean bulk density at the P < 0.05 level, [F (4, 25) = 1.54, p = 0.22].

ANOVA

Table 21: BD (g/cm^3)

	Sum of Squares	df	Mean Square		Sig.
Between Groups	.275		.255	12.519	.000
Within Groups	489	24	.020		
Total	.764	29			

ANOVA

A one-way between subject's ANOVA was conducted to compare the effect of firing temperatures (800 $^{\circ}$ C - 1600 $^{\circ}$ C) on apparent porosity of the ceramic bodies. Temperatures divided into five groups (800 °C, 1200 °C, 1400 °C, 1500 °C, 1600 $^{\circ}$ C). There was a statistically significant difference (table 24), in apparent porosity of the prepared ceramic bodies for the six groups $[F (5, 24) = 21.5, p = 000]$. Tukey HSD test (table 25) indicated that the mean apparent porosity for 800 °C (M = 15.2) SD = 1.5) was significantly different from 1000 °C (M= 11.9, SD = 1.17), 1200 °C (M = 10.78, SD = 1.26), 1400 °C (M = 8.39, SD = 1.43), 1500 °C (M = 6.7, SD = 1.8) and 1600 °C (M = 8.0, SD = 1.8). In addition, the test revealed that the mean apparent porosity for 1000 °C (M= 11.9, SD = 1.17) was significantly different from 1400 °C (M = 8.39, SD = 1.43) and 1500 ^oC (M = 6.7, SD = 1.8) and 1600 ^oC (M = 8.0, SD = 1.8). However, there was no statistically significant difference in mean apparent porosity between 1000 °C and 1200 °C. In addition to that the test stated that the mean apparent porosity at 1200 °C $(M = 10.78, SD = 1.26)$, was significantly different from 1500 °C (M = 6.7, SD = 1.8), and 1600 °C (M = 8.0, SD = 1.8), but showed significant difference between mean apparent porosity at 1200 °C (M = 10.78, SD = 1.26) and 1000 °C (M= 11.9,

 $SD = 1.17$) and 1400 °C (M = 8.39, SD = 1.43). The test showed the evidence of significance difference in mean apparent porosity between 1400 °C (M = 8.39, SD = 1.43), 1500 °C (M = 6.7, SD = 1.8), and 1600 °C (M = 8.0, SD = 1.8). A One-way ANOVA was conducted also to compare the effect of different batches on the apparent porosity of the ceramic bodies, the test shows that there wasn't significant effect of different batches (table 26) on the mean apparent porosity at the level (P < 0.05) level, $[F(4, 25) = 1.25, p = 0.313]$.

 These results support our conclusions on the previous work [22] regarding the improvement in physicomechanical properties of the prepared ceramic bodies especially the mixes CM3 and CM4 at 1500° C due to the presence of recognized assemblage of minerals namely; mullite $(3Al_2O_3.2SiO_2)$, aluminate, barium aluminate $(BaO.Al_2O_3)$ and corundum (Al_2O_3) system [22].These formed minerals (proved before with XRD and SEM [22]) are characterized with good mechanical properties,(they interacted together forming a compact rod-like crystals of mullite interacted with patch crystals of barium aluminate while the hexagonal plate-like turned together from one side with the other minerals on the other leading to a well compact microstructure and hence less pores and cavities in the matrix causing on improvement in volume stability (low linear shrinkage), a relatively higher bulk densities, lower apparent porosity and hence good recognized mechanical strength [28-35].

ANOVA

ANOVA

4. Conclusion:

Based on the detailed statistical studies both CM3 and CM4 mixes show outstanding physicomechanical behavior, however the statistical variance calculation between CM3 and CM4 is not significant, this is also true between temperatures 1500° C and 1600 \degree C. So, from economical point of view M4 mix (50% bauxite + 50 % PWS) could be selected as the optimum mix regarding the physicomechanical properties.

Multiple Comparisons

*. The mean difference is significant at the 0.05 level.

Table 19: CCS (Kg/cm²)

) Tukey HSD

*. The mean difference is significant at the 0.05 level.

$$
86 \leq \text{Exp}
$$

Multiple Comparisons

*. The mean difference is significant at the 0.05 level.

*. The mean difference is significant at the 0.05 level.

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