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Factorial Model Prediction for Performance of Ordinary Portland Cement Blended with Metakaolin and Sawdust Ash for Oil Well Cementing

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Article Info

Abstract

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Factorial model exhibits good iteration and response information for analysis and mathematical prediction model; which may be manipulated using any design of experiment (DOE) software such as Minitab for accurate and high correlation analysis as well as correlated predictions. In this study, the viability of using Sawdust Ash (SDA) and Metakaolin (MK) as a blend to ordinary Portland cement (OPC) was carried out using a mathematical arrangement of a factorial model allowing combinations and percentages blend of the OPC with Metakaolin for 10%,12.5% and, 15% whereas Sawdust Ash at 0%, 5% 10% which made up for 10 to 25% of both pozzolan incombination to modify the properties of ordinary Portland cement (class B), mitigate against the pollution challenges and also provide an efficient, cheaper oil well-cementing material. Materials for the study were characterized based on physical, chemical, and pozzolanic test properties. Slurry property testing was based on the mix formulation generated from Minitab software for the Design of the Experiment (DOE) using two domains three interactive factors (2x3) level factorial model. The slurry was tested for thickening time, free fluid, fluid loss, slurry density, and rheology parameters respectively at 45-65oC conditioning temperature using a sensitometer following the specifications of the America Institute of Petroleum (API) SPEC 10A and 10B). Metakaolin blend with OPC alone at 15% where detrimental to rheology but the incorporation of Sawdust Ash up to 10% with Metakaolin at both 10% and 15% improved the slurry performance as shown by the various factorial outputs of coefficients regarding free water (0.032-1.435%) which was far below the 5.9% maximum and fluid loss decrement with API RP-10B of 50 to 250ml for liner cementing, increment of thickening time as well as modulating the rheology characteristic to make the formulation pumpable. Results also showed that a blend of oil well-cementing material for blend samples (75% OPC, 10% MK, 10% SDA), (75% OPC, 15% MK, 10% SDA), and (90% OPC, 10% MK, 10% SDA) had an optimal performance with respect to both slurry and mechanical properties; giving an indication of the effectiveness of the factorial model for studying the interaction of material when in underuse and overuse.

1. Introduction

Factorial design (FD) is a model method that monitors the interactions of multiple factors as well accommodate the outcome of both main and interaction effects of the experimental factors or variables [1]. The number of experimental runs performed for the model development for full factorial design is governed by the Equation 1.0.

$$\mathbf{N} = \mathbf{L}^{\mathbf{K}} \tag{1.0}$$

Where K denotes experimental variable referred to as factors, L is the number of domain levels of variables, N is the total number of experimental runs, While K can be represented as X_n, X_{n+1}, X_{n+2} ..., and X_K regarding various experimental factors Experimental runs based on two (2) level is attributed to 2^{K} level domain '-'(-1) to indicate low level and '+' (+1) for high level which can be percentage of additive of constituent in an experimental sample or percentage of replacement of constituent in an experimental sample. The response variable for any experimental design for two (2) level factorial parameters from full factorial design for either four factors (2^4) yield sixteen (16) experimental runs generated for four (4) factors whereas eight (8) experimental runs for three (3) factors interaction formulations (2^3) [2]. The quantity of each of the variables under low and high domain related to the response variable for the experiment is as well as the desired test outcome. Running the full complement of all possible factor combinations means that all the main and interaction effects can be estimated. This study focuses on the pozzolanic interaction of the blending of ordinary portland cement (OPC) with Metakaolin for 10%,12.5% and, 15% whereas sawdust ash at 0%, 5% 10% which made up for 10 to 25% combine replacement. The model allows underuse and addition in excess of pozzolan samples (Metakaolin and Sawdust Ash) from 5% - 10%; following factorial standard runs.

The locally obtained material were tested for various pozzlanic characteristic while the experimental samples were tested through laboratory simulated main properties which include; thickening time, fluid loss, free water, slurry density and rheology for the slurry material use in oil-well cementing operation in accordance to [3].

The mechanical properties were studied within the required and essential test parameter mainly the compressive strength; for curing period of 8hours, 24 hours, required by [4] as well as 7, and 28days necessary for ascertaining the immediate and post effect of pozzolan on the cement cake. The cube density, Water absorption and static elastic modulus were other mechanical properties considered.

1.1.Theoretical Background

Oil well-cementing operation is a sensitive operation that needs prescient design information for onsite formulation. It makes use of a tested and viable model of higher accuracy of interaction and regression coefficient to predict the various desired required properties of cementing material for a successful well-cementing operation [5]. Therefore, the factorial model is considered based on their viability and validity in different fields and oil well fields. The modern techno-economic challenges regarding the development of the world economy require the activation of works to search, investigate and develop oil and gas fields. The development of wells is a time-consuming process, which consists of a large number of operations using special equipment, and is accompanied by multi-million expenses hence require a highly effective model. As the depth of drilling increases, the wellbore conditions pose more challenges on the stages of construction and subsequent operation of the wells. One of the important requirements before introducing wells into operation is the strengthening of the casing columns and insulation of layers by injecting grouting [6]. Furthermore, one of the criteria considered in choosing an oil-well cement slurry for individual wells is the physical and performance requirements of the slurry which include the thickening time, rate of fluid loss, amount of free fluid, slurry density,

and rheology of the slurry. Increased attention in modern oil-well cement materials calls for reduction in the water separation of the slurry, which must be kept to zero free water with regards to requirements of the American Petroleum Institute (API) Specification [7]. A consideration for complex behavioural requirement of oil well cement slurry design demands for the use of factorial design model method which has been successfully employed in various studies such as to develop a model to predict the compressive strength of oil well cement, predict the rheology of oil well cementing material containing chemical additives. The model allows effects of different factors which can be the constituent or additives to be usually considered individually as well as their interaction with each other during the development. The model has a high efficiency with correlation coefficient of 99.8%, standard error of 0.1325, and accuracy of 99.8% [2]. The model can be employed to determine the behaviour of cement slurry when any of slurry material or additives are in under-dosed or added in excess of the required quantity. The mathematical model with r repetitions per cell in a completely randomized design according to Equation 2 yielding Equation 3.

 $Yijk = \mu + Ai + Bj + ABij + Ck + ACik + BCjk + ABCijk + Eijkv$ (2)

Where i, j, k = (0,1) and r = (1,2,...,r) repetitions, μ = interactive intercept coefficient while E = experimental error coefficient [8]

Thus for X_1 , X_2 , and X_3 factors the model becomes

 $Y = b0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 - b_{123}X_1X_2X_3 + e$ (3) Where b0 corresponds to μ and e corresponding the interactive errors for b1,b2 and b3 factors. The model pseudo effect for 2^3 level experimentation and its domain is as shown in the Table 1.

Exp	Ι	\mathbf{X}_1	X 2	X 3	X_1X_2	$X_1 X_3$	$\mathbf{X}_{2} \mathbf{X}_{3}$	$X_1 X_2 X_3$	Yield (%)
•									
1	+	-	-	-	+	+	+	-	\mathbf{Y}_1
2	+	+	-	-	-	-	+	+	Y ₂
3	+	-	+	-	-	+	-	+	Y3
4	+	+	+	-	+	-	-	-	Y4
5	+	-	-	+	+	-	-	+	Y ₅
6	+	+	-	+	-	+	-	-	Y ₆
7	+	-	+	+	-	-	+	-	Y_7
8	+	+	+	+	+	+	+	+	Y ₈

Table 1 Effect interaction of three various factor Model matrix and the yield response

Source: [9]

Therefore, for the experimental design, factorial model of 2^{K} ($2^{3}x2$) level was eminent for this work.

2.0 Materials and Methods

2.1 Experimental Material: The following materials were obtained, prepared and employed in conformation to the requirements and specifications of the experimental process. Sawdust ash obtained from sawdust in timber market Umuahia as well as kaolin for Metakaolin obtained from Umuariaga Oboro Ikwuano, Abia state; were prepared from standard methods within the specified temperature of calcination and heating time of 90 min for Metakaolin before being employed to the experimental module. Ordinary Portland cement for the research of grade 42.5 OPC class B were obtained conforming to the classifications of [9] and American Petroleum Institute [4]. The water for the study was clean tap water from the laboratory conforming to the standard spelt in [11 and 4]

2.2 Methods: For this study, two sets (2) of samples were prepared. One set made from factorial model experimental runs as generated in Minitab design of experiment (DOE) for slurry properties of Metakaolin –Sawdust Ash (MKSDA) blended OPC as shown in Table 2 and the other set of control sample containing pure class B cement in accordance to [4] mix specifications.

The optimum mix ratio for the oil well cementing material sample experimentation was formulated using output of the Minitab randomised factorial 2^3 level experimentation design following the domain of experiment which have various combinations and percentages blend of the ordinary portland cement (OPC) with Metakaolin for 10%, 12.5% and, 15% whereas sawdust ash at 0%, 5% 10% which made up for 10 to 25% pozzolan incorporation in low and high level in the experimentation at absolute volume density of the individual pozzolan material. The model design allows for slight under use as well as excess dose of the combine percentage blending with Metakaolin and sawdust ash; aligning with the factorial design by [2] For the batching process, the water cement ratio W/C was maintained at 0.46 based on API standards API SPEC 10B for all the sample to be mixed as reported by [6] for Mk dosage below 30%. The pseudo mix form the compressive strength was equivalent to the replacement combination with water /cement ratio maintained at 0.46 (46%) based on the formulated factorial design of three factors-two replica (2^3x^2) factor level design with two centre points randomized to take care of statistical fringes [2] the resultant design is as shown in the Table 3. Where N1 to N18 represents the run order for the randomised blended cementing sample from the factorial design, while the C1 and C2 run orders represent the control sample with only class B ordinary portland cement. The subsequent testing of the slurry for various led down simulated test by API 10A&B for slurry required to be used in well bore; included thickening time, fluid loss, free water, rheology, and slurry density. The mechanical specimen testing experimentation were mainly for cube density, water absorption and compressive strength at 8 hours, 24 hours, 7 and 28 days curing periods respectively using 160 samples of 100 mm³ cubes.

Table 2: Randomized Minitab Experimental formulation for a 2³x2 level factorial design Full Factorial Design

Factors:3Base Design:3, 8Runs:18Replicates:2Blocks:noneCenter pts (total):2

All terms are free from aliasing

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StdOrder	RunOrder	Blocks	Cement	Metakaolin(MK)	Sawdust ash (SDA)
11	1	1	-1	1	-1
3	2	1	-1	1	-1
9	3	1	-1	-1	-1
12	4	1	1	1	-1
16	5	1	1	1	1
14	6	1	1	-1	1
10	7	1	1	-1	-1
2	8	1	1	-1	-1
15	9	1	-1	1	1

	17	10	1	0	0	0
	1	11	1	-1	-1	-1
	13	12	1	-1	-1	1
	18	13	1	0	0	0
	7	14	1	-1	1	1
	8	15	1	1	1	1
	5	16	1	-1	-1	1
ľ	4	17	1	1	1	-1
ľ	6	18	1	1	-1	1

Table 3: Factorial Pseudo and Actual Mix Ratio for the Formulation Generated from Minitab De	OF
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StdOr der	RunOrder	Blocks	OPC	Metakaolin (MK)	Sawdust Ash (SDA)	Equivalent OPC (%)	Equivalent MK (%)	Equivale nt SDA (%)
11	1	1	-1	1	-1	90	15	-
3	2	1	-1	1	-1	90	15	-
9	3	1	-1	-1	-1	90	10	-
12	4	1	1	1	-1	75	15	-
16	5	1	1	1	1	75	15	10
14	6	1	1	-1	1	75	10	10
10	7	1	1	-1	-1	75	10	-
2	8	1	1	-1	-1	75	10	-
15	9	1	-1	1	1	90	15	10
17	10	1	0	0	0	82.5	12.5	5
1	11	1	-1	-1	-1	90	10	-
13	12	1	-1	-1	1	90	10	10
18	13	1	0	0	0	82.5	12.5	5
7	14	1	-1	1	1	90	15	10
8	15	1	1	1	1	75	15	10
5	16	1	-1	-1	1	90	10	10
4	17	1	1	1	-1	75	15	-
6	18	1	1	-1	1	75	10	15
C1						100	-	-
C ₂						100	-	-

3.0 Result and Discussion

This research results were presented in phases of the experimental methodology regarding material experimentation testing, slurry phase experimentation and mechanical property experimentation phase respectively.

3.1 Material Characterization: From the material test experimentation, the results obtained physically characterized sawdust ash as the lightest of the three cementitious material and their particle size distribution compared to that of fly ash and silica fume respectively shown in Figure 1. This potentially present it as a possible constituent of tail cementing slurry which is required to have lower specific gravity nevertheless. The character of metakaolin (MK) makes it a positive blending material in both case of lead or tail cementing. On the other hand, results from the chemical and pozzolanic experimentation which showed a combine percentage composition of the major oxides including: [Silica (SiO2), Aluminium Oxide (Al2O₃), Iron oxide (Fe₂O₃) and Potassium Oxide (K₂O)] for Metakaolin to be 84.26% which is above the stipulated minimum index of 70% of (SiO2), (Al2O₃), (Fe₂O₃) as reported by [15] thus classifies it as Class N pozzolan according to ASTM C618-15. Sawdust ash for this investigation had slightly lower composition of about 70.02% slight below 75.9% as per British

Standard pozzolan activity index; classing it according to ASTM as Class C pozzolan as shown in Table 3. The pozzolanic activity of Metakaolin was paramount as compared to that of sawdust as only 1.1 ml of acid was used up after 40 min with regards to 1 gram of Metakaolin in solution of 1 mole of calcium hydroxide whereas 6ml titrated after 120min of reaction for same solution containing 20 grams of sawdust ash. This shows a quicker reaction or use up of detrimental $Ca(OH)_2$ in the cement matrix by metakaolin as shown in Table 4. Hence its potential positive blend in cementing for geothermal and deep well where $Ca(OH)_2$ by product is more.

Table 3. Chemical characterization of sample Metakaolin(MK) and Sawdust Ash (SDA)

Oxide/ Sample	Silica (Aluminuim	Calcuim	Magnesium	Iron oxide	Potassium Oxide
	SiO ₂)	Oxide	Oxide	Oxide	(Fe ₂ O ₃)%	(K ₂ O) %
	%	(Al ₂ O ₃) %	(CaO) %	(MgO)%		
Metakaolin	59.11	16.18	1.44	1.03	6	1.53
(MK)						
					8	
					2	
Sawdust Ash (SDA)	61.58	5.96	18.63	3.18	2.48	16.33

Table 4 Summary of Pozzolan Test Result of SDA and MK

Sample	Duration	volume	Titration	Sample	volume of H ₂ SO ₄	Shake/Titration
МК	Of	of HCl	Duration	SDA	Used (ml)	Duration (min)
	Heating	Used	(min)	-		,
	(min)	(ml)		Α	24.6	30
Α	16	15.2	10	В	11.5	60
в	16	11.4	20	С	7.4	90
С	16	4.8	30	D	6.0	120
D	16	1.1	40	U	0.0	120



Figure 1. Particle Size Distribution of Metakaolin and Sawdust Ash as compared to other pozzolan

3.2 Slurry Experimental Result: For the result obtain from slurry experimentation in accordance to American Petroleum Institute spec 10A&B for both the control samples C1 and C2 as well as blended N_1 - N_{18} showed that thickening time, the time to reach 100 Bearden unit of consistency (Bc) were more than stipulated 90 minutes minimum for all samples; but the higher values of time for run samples N_4 ,

and N_{17} as well as N_1 , and N_2 which have a high level (15%) of Metakaolin compared to control samples and those containing sawdust ash at high level (10%) as shown in Figure 2. This is a clear indication that Metakaolin (MK) retard the slurry more from thickening than when incorporated alongside sawdust ash (SDA) hence potential in creating more handling and pumping time of the slurry to its target depth. This is in agreement with the output of analysis following the factorial model fit in the Table 5 which yielded probability "P" value less than 0.05 P significant value as well as F values less than the critical value as stated by [5] as an indication of positive interaction with Metakaolin as shown in Figure 2. The following regression model with respect to Equation 3. Corresponding to the yielded coefficient of effect as in Equation 4.

Thickening time = $107.913 + 2.15X_1 + 5.90X_2 - 2.588X_3 + 3.037X_1X_2 + 3.90X_1X_3 - 2.85X_2X_3 - 0.963X_1X_2X_3 + 0.3626$ (4) Where: X₁ -Cement, X₂ -Metakaolin (MK), X₃-Sawdust Ash (SDA)

1 actorial 1 It. Estimated Effects and Coefficients for unexeming this	Table	5	Factorial	Fit:	Estimated	Effects	and	Coefficients	for	thickening	time
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Term	Effect	Coef	StDev Coef	 	P
Constant		107.913	0.3626	297.63	0.000
cement	4.300	2.150	0.3626	5.93	0.000
metakaolin	11.800	5.900	0.3626	16.27	0.000
sawdust	-5.175	-2.588	0.3626	-7.14	0.000
cement*metakaolin	6.075	3.037	0.3626	8.38	0.000
cement*sawdust	7.800	3.900	0.3626	10.76	0.000
metakaol*sawdust	-5.700	-2.850	0.3626	-7.86	0.000
cement*metakaol*sawdust	-1.925	-0.963	0.3626	-2.65	0.026
Ct Pt		-5.913	1.0877	-5.44	0.000

Pareto Chart of the Standardized Effects (response is thickeni, Alpha = .05)



Figure 2 Interactive Effect Significant Plot for Thickening Time of Slurry

The result of slurry density shows a yield density and specific gravity within the range of 1.71-1.785. Within the range for class A and B cement as stipulated and reported by [3] also were in agreement with the range reported by [12]. (1.45g/cm3-1.84g/cm3). Metakaolin increased the density of the slurry at both high level and low level (15% and 10%) blending from the mean value of 1.75 up 1.785g/cm3 accounting for 2% increase. Nevertheless, incorporating sawdust ash reduced the sample density from control 1.75 to 1.71g/cm3 accounting for 2.3% decrement as compared to that of control samples. Also, The result of free water at conditioned temperature range of $65-45^{\circ}C$ static for 2 hours shows that the controlled samples C_1 and C_2 had the highest values of free water (1.123 and 1.435%) respectively whereas the lowest free water was observed by sawdust ash incorporated samples at (10%) and 5% blend N_9 and N_{14} (0.120%, 0.032%). This is far below the 5.9% maximum free water stipulated by [4 and 10] and a clear indication of the interactive positive effect of sawdust ash as shown in Table 6 with its p value less than 0.05 and interactive plot in Figure 3. Hence potential in reducing excess free water during slurry pumping. The following regression model with respect to Equation 3 corresponding to the yielded coefficient of effect is given in Equation 6.

Term	Effect	Coef	StDev Coef	Т	Р
Constant		0.3463	0.02072	16.71	0.000
cement	0.1663	0.0831	0.02072	4.01	0.003
metakaol	-0.2252	-0.1126	0.02072	-5.44	0.000
sawdust	-0.1245	-0.0623	0.02072	-3.00	0.015
cement*metakaol	-0.0340	-0.0170	0.02072	-0.82	0.433
cement*sawdust	-0.2528	-0.1264	0.02072	-6.10	0.000
metakaol*sawdust	-0.0463	-0.0231	0.02072	-1.12	0.293
cement*metakaol*sawdust	0.2650	0.1325	0.02072	6.40	0.000
Ct Pt		0.0138	0.06215	0.22	0.830

Table 6 Fractional Factorial Fit: Estimated Effects and Coefficients for free water

Pareto Chart of the Standardized Effects



Figure 3 Interactive Effect Significant Plot for free Fluid (water) of Slurry

As regards fluid loss, samples blended with sawdust ash in combination with Metakaolin such as samples N_1 and N_{18} (204.483 and 197.898 ml/30minutes) kept the fluid loss within the maximum range of API RP-10B (50 to 250ml /30minutes) as compared to samples with only Metakaolin blend as well as control sample C_1 and C_2 which had the highest mean fluid loss of (484.970 and 508.645 ml/30min) at 10 and

11minutes blow out. Thus, presenting sawdust ash (SDA) blend with Metakaolin (MK) as a decent fluid loss and blowout control pozzolan reducing the slurry fluid loss by about 57% as agreed with the P values were below 0.05 as well as their F values which were outside the critical region as shown and interaction plot showed in Figure 4.

The following regression model with respect to Equation 3. corresponding to the yielded coefficient of effect in Table 7 is given in Equation 7.

 $\begin{aligned} \text{Fuid loss} &= 298.90 + 10.83X_1 - 6.700X_2 - 18.54X_3 + 3.76\ X_1X_2 - 6.89X_1X_3 + 64.55X_2X_3 + \\ & 11.93\ X_1X_2X_3 + 5.273 \end{aligned} \tag{7}$

Where: X1 -Cement, X2 -Metakaolin (MK), X3-sawdust ash (SDA)

Table 7 Fractional Factorial Fit: Estimated Effects and Coefficients for fluid loss

Term	Effect	Coef	StDev Coef	Т	P
Constant		298.90	5.273	56.68	0.000
cement	21.66	10.83	5.273	2.05	0.070
metakaol	-13.39	-6.70	5.273	-1.27	0.236
sawdust	-37.09	-18.54	5.273	-3.52	0.007
cement*metakaol	7.52	3.76	5.273	0.71	0.494
cement*sawdust	-13.78	-6.89	5.273	-1.31	0.224
metakaol*sawdust	129.11	64.55	5.273	12.24	0.000
cement*metakaol*sawdust	23.85	11.93	5.273	2.26	0.050
Ct Pt		40.17	15.819	2.54	0.032

Pareto Chart of the Standardized Effects

(response is fluid Io, Alpha = .05)



Figure 4 Interactive Effect Significant Plot for fluid loss of Slurry

In terms of rheology performance of the slurry samples, which is function of slurry plastic viscosity, yield point of shear stress and corresponding gel strength at 10 seconds as well as 10 minutes gel strength after 20minutes conditioning at 60°C. The plastic viscosity where below 100 mPa.s (cp) which according to the report of [13] regarded as the maximum limit for all cementing slurry to be kept pumpable; with exception of samples N_4 and N_{17} (129cp, 132cp) which contain only cement combination of Metakaolin at high level replacement. The result from the gel strength at 10 seconds and 10 minutes as displayed in Figure 5 shows control samples C_1 and C_2 to have lowest value of

gel strength compared to other samples. The result also shows Metakaolin interactive effect at all levels of combination with Sawdust Ash in improving the rheology components. Also, for yield point shear stress, samples with MK N₁,N₂,N₈, as well as N₁₇ had higher values (76.097, 68.702, 66.078,and 63.454) compared to the samples containing combinations of Sawdust Ash at 10% (N₁₃, N₁₀, N₃,N₅,N₁₈) (28.865,33.635, 52.481, 57.252, 57.968). Hence, a clear indication of the detrimental effect of Metakaolin at high level blend (15%) on the cementing slurry in absence of saw dust ash in the blend.



Figure 5. Plot of Gel Strength of Slurry at 10 Seconds and 10 Minutes

The factorial fit analysis output for both plastic viscosity, yield point and 10 seconds gel strength as tabulated in Table 8, Table 9 and Table 10 as well as Figure 6, 7 and 8 respectively showing that Metakaolin interactions at all levels of combination with only cement or with cement and sawdust ash increased the plastic viscosity and yield point; following the P values less than 0.05 but the yield point indicated strongly that Metakaolin was the main cause of increased viscosity according to the plot from Figure 8.

The following factorial regression model with respect to equation 3.4 corresponding to the yielded coefficient of effect is given in Equation 8, 9, and 10;

Plastic Viscosity = $58.31 + 15.19X_1 + 13.50X_2 - 11.63X_3 + 5.25X_1X_2 - 8.25X_1X_3 - 8.44X_2X_3 - 9.94X_1X_2X_3 + 1.155$ (8)

Yield Point = $59.19 + 1.58X_1 + 2.74X_2 - 3.82X_3 - 3.82X_1X_2 + 2.03X_1X_3 - 1.22X_2X_3 + 1.70 X_1X_2X_3 + 0.7813$ (9)

 $\begin{array}{l} 10 \; \text{Sec Gel Strength} = 30.375 + 2.00 X_1 + 2.625 X_2 - 4.75 X_3 + 3.00 X_1 X_2 - 2.125 \; X_1 X_3 - 3.250 X_2 X_3 - 2.875 \; X_1 X_2 X3 + 0.6719 \quad (10) \end{array}$

Where: X₁ -Cement, X₂ -Metakaolin (MK), X₃-Sawdust Ash (SDA)

Term	Effect	Coef	StDev Coef	Т	P
Constant		58.31	1.155	50.50	0.000
cement	30.37	15.19	1.155	13.15	0.000
metakaol	27.00	13.50	1.155	11.69	0.000
sawdust	-23.25	-11.63	1.155	-10.07	0.000
cement*metakaol	10.50	5.25	1.155	4.55	0.000
cement*sawdust	-16.50	-8.25	1.155	-7.14	0.000
metakaol*sawdust	-16.88	-8.44	1.155	-7.31	0.000
cement*metakaol*sawdust	-19.88	-9.94	1.155	-8.61	0.000
Ct Pt		-29.94	3.464	-8.64	0.000

Table 8 Factorial Fit: Estimated Effects and Coefficients for plastic

Pareto Chart of the Standardized Effects





Figure 6 Pareto and interaction effect Plot of Plastic Viscosity Table 9 Factorial Fit: Estimated Effects and Coefficients for yield point

Term	Effect	Coef	StDev Coef	Т	P
Constant		59.19	0.7813	75.76	0.000
cement	3.16	1.58	0.7813	0.7813 2.02	
metakaol	5.49	2.74	0.7813	3.51	0.007
sawdust	-7.63	-3.82	0.7813	-4.89	0.000
cement*metakaol	-6.56	-3.28	0.7813	-4.20	0.002
cement*sawdust	4.06	2.03	0.7813	2.60	0.029
metakaol*sawdust	-2.44	-1.22	0.7813	-1.56	0.152
cement*metakaol*sawdust	3.40	1.70	0.7813	2.17	0.058
Ct Pt		-27.94	2.3439	-11.92	0.000





Term	Effect	Coef	StDev Coef	Т	Р
Constant		30.375	0.6719	45.21	0.000
cement	4.000	2.000	00 0.6719 2.98 25 0.6719 3.91		0.016
metakaol	5.250	2.625	0.6719	0.6719 3.91	
sawdust	-9.500	-4.750	0.6719	-7.07	0.000
cement*metakaol	6.000	3.000	0.6719	4.47	0.002
cement*sawdust	-4.250	-2.125	0.6719	-3.16	0.011
metakaol*sawdust	-6.500	-3.250	0.6719	-4.84	0.000
cement*metakaol*sawdust	-5.750	-2.875	0.6719	-4.28	0.002
Ct Pt		-0.375	2.0156	-0.19	0.857

Table 10 Factorial Fit: Estimated Effects and Coefficients for 10	sec	gel
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Figure 8 Pareto Interaction Effect Plot for 10 Seconds Gel Strength

3.3 Mechanical Test Result: The result of the third segment of the experimentation "the mechanical tests"; shows that the cube density were influenced by the blend of SDA and MK at higher replacement levels (10% and 15%). The density of the samples where within the range of (1746.667 – 1906.667 kg/m3) while samples blended with MK alone had lower density of less than 5.7% as compared to the controlled samples, on the other hand, the samples water absorption after 28 days curing were within the range (1-3%) as stipulated by [11] as shown in the Figure 9 indicating a well compacted micro matrix for samples with SDA. Hence will imply resistance to fluid migration.



Figure 9. Sample Cube Density and water Absorption The average strength in agreement with factorial fit probability values [OPCMk(0.35),OPCMKSDA(0.068), Mk(0.23) OPC (0.24) and SDA (0.022)] showing the interaction of Sawdust Ash-Metakaolin blend to be influential on the early strength of the cubes shown in Table 11 with probability value less than 0.05. from the interactive Figure 10 and F statistical probability in Table 12 sawdust ash shows more influence in improving the strength at 28 days when in high level blend with Metakaolin at all levels of OPC incorporation. This could be attributed to the pozzollan filler tendency of the two combine materials in the micro matrix of the cemented cake. More strength increment is expected following the report of [13,14,15,and 16] displayed interactive plot shown in Figure 11.

The resultant factorial regression model following the coefficient of the fit is for 24 hours and 28 days strength is given by Equation 11, and 12:

 $\begin{array}{l} 24 \ hours \ strength = 11.7250 - 0.2563X_1 + 0.0063X_2 + 0.4562X_3 - 0.2500X_1X_2 - 0.1875\ X_1X_3 - 0.4375X_2X_3 - 0.1563\ X_1X_2X_3 + 0.08260 \end{array} \tag{11}$

 $\begin{array}{l} 28 \ days \ strength = 30.1864 - 0.2239 X_1 - 0.498 X_2 + 0.4828 X_3 + 0.1698 X_1 X_2 + 0.0052 \ X_1 X_3 - 0.1864 X_2 X_3 - 0.3636 \ X_1 X_2 X_3 + 0.1753 \end{array} \tag{12}$

Where: X₁ -Cement, X₂ -Metakaolin (MK), X₃-Sawdust Ash (SDA) Table 11 Factorial Fit: Estimated Effects and Coefficients for 24hrs compressive strength

Term	Effect	Coef	StDev Coef	Т	P
Constant		11.7250	0.08260	141.95	0.000
cement	-0.5125	-0.2563	0.08260	-3.10	0.013
metakaol	0.0125	0.0063	0.08260	0.08	0.941
sawdust	0.9125	0.4562	0.08260	5.52	0.000
cement*metakaol	-0.5000	-0.2500	0.08260	-3.03	0.014
cement*sawdust	-0.3750	-0.1875	0.08260	-2.27	0.049
metakaol*sawdust	-0.8750	-0.4375	0.08260	-5.30	0.000
cement*metakaol*sawdust	-0.3125	-0.1563	0.08260	-1.89	0.091
Ct Pt		0.3250	0.24780	1.31	0.222







Term	Effect	Coef	<u>StDev Coef</u>	T	P
Constant		30.1864	0.1753	172.15	0.000
cement	-0.4479	-0.2239	0.1753	-1.28	0.234
metakaol	-0.4396	-0.2198	0.1753	-1.25	0.242
sawdust	0.9646	0.4823	0.1753	2.75	0.022
cement*metakaol	0.3396	0.1698	0.1753	0.97	0.358
cement*sawdust	0.0104	0.0052	0.1753	0.03	0.977
metakaol*sawdust	-0.3729	-0.1864	0.1753	-1.06	0.315
cement*metakaol*sawdust	-0.7271	-0.3636	0.1753	-2.07	0.068
Ct Pt		0.3636	0.5260	0.69	0.507

Fable	12 Factorial	Fit: Estimated	Effects and	Coefficients for	or 28days	s Com	pressive	Strength
								~

Pareto Chart of the Standardized Effects

(response is 28 days c, Alpha = .05)



Figure 11. Interaction effect of material for 28 days strength

4.0 Conclusion

Oil well-cementing operation is a sensitive operation that needs prescient design information for onsite formulation. It makes use of a tested and viable model of higher accuracy of interaction and regression coefficient to predict the various desired required properties of cementing material for a successful well-cementing operation. Factorial model has been successfully deployed in investigating and predicting the engineering performance of Ordinary Portland cement when blended with Metakaolin and Sawdust Ash identifying and estimating significant interactive parameters.

The advantages of factorial design (FD) and its role in additives performance of cement, which was aimed at the development of a mathematical model to predict slurry performance and compressive strength using three different constituents for improving compressive strength for oil well-cementing employed factorial design.

Hence, Sample formulations [N18 (75%OPC, 10%MK, 10% SDA), N15 (75%OPC, 15%MK, 10%SDA), and N12 (90% OPC, 10%MK, 10%SDA)] had optimal performance with respect to both slurry and mechanical properties.

Sawdust Ash serve as restrictive blend in modifying the detrimental effect of Metakaolin which is a more active pozzolan when in excess or alone in the replacement blend; as well as reduction in OPC dependency which will in turn lower the foot print of cement production on CO_2 emission.

Reference

- Cheong Y., and Gupta R., (2005). Experimental Design and Analysis Methods of Assessing Volumetric Uncertainties. In: SPE 80537 paper first presented at the 2003 SPE Asia Pacific Oil and gas conference and exhibition, Jakarta, Indonesia, pp 9–11.
- [2] Falode, O. A., Salam, K. K., Arinkoola, A. O., and Ajagbe, B. M. (2013). Prediction of Compressive Strength of Oil Field Class G Cement Slurry Using Factorial Design. *Petrol Vol 20, pp 1-8*
- [3] API, (2010), 10A, "Specification for Cements and Materials for Well Cementing,"
- [4] Torbjorn, L., Elisabeth, S., Lisbeth, A., Bernt T. C., Asa, N., and Jarle, P., (1998). Experimental Design and Optimization: Rolf Bergman Pharmacia and Upjohn Structure-Property Optimization Center, F3A-1, SE-751 82 Uppsala, Sweden Pharmacia and Upjohn Lund Research Center, P.O. Box 724, SE-220 07 Lund, Sweden.
- [5] Magarini, P., Lanzetta, C., and Galletta, A., (1999), Drilling Design Manual. Eni-Agip Division. pp: 97-107.
- [6] Qosai, S. and Radi, M., (2018) Benefits of Using Mineral Additives, as Components of the Modern Oil-Well Cement Case Studies in Construction Materials Vol 8, p455–458
- [7] ASTM C150, (2007) Standard Specification for Portland cement
- [8] EN 196-1, Methods of testing cement Part 1: Determination of Strength
- [9] EN 196-2, Methods of testing cement Part 2: Chemical Analysis of Cement
- [10] EN 196-7, Methods of Testing Cement Part 7: Methods of Taking and Preparing Samples of Cement
- [11] Seifert, E., Abramo, L., and Thelin, B. (1998). Experimental Design and Optimization, Chemometrics and Intelligent Laboratory Systems 42 p 3–40 Ž. Experimental Elsevier Science, PII: S0169- 7439 98 00065-3
- [12] API, (2013), 10B-2, "Recommended Practice for Testing Well Cements." p. 124.
- [13] Schlumber (2013) Oil Field Review, A Journal, Communicates Technical Advances In Finding And Producing Hydrocarbons To Consumers, Employees And Other Oilfield Professionals. Volume 25 number 2 issn 0923-1730 www.slb.com/oilfieldreview
- [14] Broni-bediako, E., Joel, O. F., and Ofori-sarpong, G. (2016). Oil Well Cement Additives: A Review of the Common Types Oil and Gas Research Oil Well Cement Additives: A Review of the Common Types. <u>https://doi.org/10.4172/ogr.1000112</u>
- [15] Ettu, L. O., Ezenkwa C. S., Awodiji .C. T. G., Njoku. F. C., and Opara H., (2016), Tensile Strengths of Concrete Containing Sawdust Ash from Different Calcination Methods, *International Journal of Engineering Research* and Technology IJSRSET | Volume 2 | Issue 4 | 2016 [(2)4: 349-355]
- [16] Explore Production Technology vol.3, pp297-302.. https://doi.org/10.1007/s13202-013-0071-0
- [17] Hemant, C., (2011), "Effect of Activated Flyash in Metakaolin Based Cement" National Conference on Recent Trends in Engineering & Technology 13-14, BVM Engineering College, Gujarat, India.
- [18] Pacheco, T. F., Arman, S., and Said, J., (2011) Using Metakaolin to Improve the Compressive Strength and the Durability of Fly ash Based Concrete, Invaco2: International seminar, innovation & valorization in civil engineering & construction materials, Rabat-Morocco, ISBN 987-9954-30-595-9, pp 23-25 ,.
- [19] Dojkov, I., Stoyanov, S., Ninov, J., and Petrov, B., (2013) Consumption Of Lime By Metakaolin, Fly Ash And Kaoline In Model Systems, *Journal of Chemical Technology and Metallurgy*, 48,pp. 54-60.