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Original Article

Influence of Metakaolin and nano-clay on compressive strength and thickening time of class G oil well cement

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Abstract

In this research, the Compressive Strength (CS) and Thickening Time (TT) of oil well cement with different Metakaolin (MK) dosages was evaluated in the presence of Nanoclay (NC). The experiments followed a randomized Box-Behnken Design (BBD) using 5 - 15 wt. % MK by weight of cement and 5-15 wt. % NC by weight of MK. The CS and TT were assessed and optimized using Response Surface Methodology (RSM). The results show that CS increases linearly with NC and hyperbolically with MK. Cement slurries with 5–15 wt.% NC shorten TT by about 35 minutes in the presence of 5 wt.% MK. A TT reduction of 103 minutes was recorded when MK was increased to 15 wt.% in the slurry with 5wt.% NC. At the optimum conditions with 10.78 wt. % MK and 13.73 wt. % NC, CS and TT were 3029±2.65 psi and 410±1.25 minutes, respectively.

Keywords: g-class cement, supplementary material, compressive strength, thickening time, Box-Behnken Design

1. Introduction

Oil well cementing was introduced in the late 1920s primarily because drilling fluid alone cannot prevent the well bore from collapsing (Joshi & Lohita, 1997). Other reasons for cementing oil wells include: protecting oil producing zones against salt water, protecting the casing from collapse under pressure, protecting well casings against corrosion, reducing the risk of groundwater contamination by hydrocarbons or salt water, and zonal isolation. There are two critical conditions that a successful oil well cement should satisfy: ability to remain pumpable for a sufficient time to ensure proper placement in the well bore, and ability to build and maintain sufficient mechanical strength to provide adequate support for the casing.

Ordinary Portland Cements (OPCs) have been used as oil well cements for many years. However, OPCs are reported to undergo strength loss with increased porosity and severe loss of durability at elevated temperatures, in acid rich, geothermal and deep oil well environments (Ma, Chen, & Chen, 2014; Won, Lee, Na, Lee, & Choi, 2015). Thus a special class of cements, the oil well cements (OWCs),

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emerged. OWCs are classified into grades depending on their C_3A (Tricalcium Aluminate) contents. The details of this classification are available elsewhere (API Specification 10A, 2010). Of all these classes, the classes A, G and H are the three most commonly used for cementing oil and gas wells. While class A is used in milder, less demanding well conditions, classes G and H are usually specified for deeper, hotter and higher pressure well conditions (Eric, Joel, & Grace, 2016).

The use of supplementary cementitious materials (SCMs) for class G cements has received increased attention over the last few decades. These materials react with $Ca(OH)_2$ released during cement hydration and convert it into high crystallinity calcium silicate hydrates (Yuhuan, Jiapei, Sheng lai, Huajie, & Chenxing, 2016). Buntoro & Rubiandini, (2000) studied the mechanical properties of cement slurry with 35 wt.% silica flour. The results after 3 days of curing show higher shear bond strength and compressive strength. Li, Sun, and Li, (2010) compared the mechanical properties of MK cement with alkali-treated slag cement; the results obtained showed that cement pastes containing MK exhibited better CS at elevated temperatures. The behavior of blended cement mortars containing nano-MK at elevated temperatures was investigated by Morsy, Al-Salloum, Abbas, and Alsayed, (2012). Their conclusion was that at the low temperature of 25 ^{o}C, 5 wt% nano-MK gave optimal mortar; and at a temperature of 80 ^{o}C, 15 wt% nano-Mk produced better CS. Shatat (2013) studied the hydration behavior and mechanical properties of blended cement containing various amounts of rice husk ash in the presence of MK. They recorded better mechanical behavior than that of OPC with ternary blends of cement containing 5–10 wt% rice husk ash and 15–20 wt% MK. Nadeem, Memon, and Lo, (2014) evaluated fly ash and MK containing concrete at elevated temperatures using stiffness damage test. The hardened cement blend showed better mechanical behavior than OPC.

Mechanical properties of OWCs depend also on the slurry density. The various weighing agents and extenders control the compressive strength and the time after initial mixing when the cement can no longer be pumped. High specific gravity and finely divided solid materials such as barites, bentonites and micro-sands are used to increase the density (Halliburton, 2009). Micro-sands, according to Chenevert and Shrestha, (1991) are capable of reducing the total chemical shrinkage thereby reduce gas leakage, while reducing the free water and preventing environmental problems. For field applications, it is expected that an additive should influence only that property for control of which it is added. Experimental Design has been successfully employed to resolve how components interact in mixtures and has assisted in solving many engineering problems (Arinkoola & Ogbe, 2015; Salam, Arinkoola, Oke, & Adeleye, 2014). Additives with multiple and conflicting effects pose serious challenges, as improvement of one property of the slurry could result in negative effects on another one. The objective of this present study was to investigate and optimize the coupled effects on CS and TT properties in ternary blends of class G cement with 5–15wt% MK and 5–15wt% NC, by using experimental design and response surface methodology.

2. Materials and Methods

2.1 Materials

The class G oil well OPC used was obtained from SOWSCO Oil Well Service (Nig.) Ltd, Port Harcourt, Nigeria. Table 1 shows the various oxides in and physical properties of the cement. The kaolin clay from which MK was synthesized was locally sourced from a clay deposit site in Okpella, Etsako East LGA of Edo state, Nigeria (latitude 7.120 N, longitude 6.280 E). The Nanoclay (1.31 ps) used was a product of Nanocor, Inc. purchased from SIGMA ALDRICH (M) Sdn. Bhd, Malaysia. It was made up of montmorillonite clay that was surface modified with 15 – 35wt% octadecylamine and 0.5 - 5 wt% aminopropyltriethoxysilane. Other chemical additives, such as fluid loss control additive (FLA-001), dispersing agent (polynapthalene sulfate), retarder, gas block additive (SWLGX3) and antifoam were provided by SOWSCO, Nigeria.

2.2 Synthesis of Metakaolin

The oven dried kaolin clay sample was ground using an electric grinder (Marlex Appliances PVT Limited) and screened to fine powder (~20 µm) before it was subjected to calcination in a muffle furnace (NYC-12 model) at 750 ^{o}C for 2 h. The chemical composition of MK produced is shown in Table 2.

2.3 Cement slurry design

Slurry formulation was done according to API standard RP 10B-2012. The NC and MK contents in the cement slurry were randomized within the range 5-15 wt.%. While the dosage of MK was determined by weight of cement (BWOC), the NC dosages were relative to the weight of MK (BWOMK). Table 3 shows the fixed concentrations of additives and matrix for nine pastes randomized according to BBD (Start Ease Design Expert Version 11). The fluid loss control additive (12-18 wt%), retarder and dispersing agent (7 wt%) were dissolved in 380 ml of water and transferred into the cup of waring blender. Then, the blended cement was added within 15 s to the aqueous solution with a stirring rate of 4000 rpm and mixed for 35 s at 12000 rpm. After this the cement slurry was placed in 5 cm cube molds for CS test and a pressurized consistometer was used for consistency measurement.

2.4 Compressive strength test

The test conditions and well specifications for CS test are presented in Table 4. The CS of different pastes was measured using an Ultrasonic Compressive Analyzer (Chandler Engineering model 4265 UCA). The cement slurry was placed in an autoclave unit in the UCA at BHST of 80 ^{o}C and pressure of 3500 psi, then a sonic wave was transmitted through it. The CS developed after 24 h for two samples was recorded automatically and transferred to data acquisition software (Chandler Engineering Model 5270). The average value of the CS was recorded.

Table 1. Chemical composition and physical properties of Class G cement

Oxides	Wt%
CaO	64.2
SiO_2	19.4
Al_2O_3	5.5
Fe_2O_3	4.5
MgO	2
SO_3	2.8
P_2O_5	0.1
K_2O	0.6
Ignition loss	0.19
Density (g/cm^3)	3.16
Specific surface area (M^2/kg)	335.3

Table 2. x-Ray Fluorescence (XRF) Analysis of MK

Oxides	Wt%
CaO	0.27
SiO_2	53.68
Al_2O_3	37.39
Fe_2O_3	4.94
MnO	0.06
SO_3	0.40
P_2O_5	1.19
K_2O	1.25
Ignition loss	1.20

Table 4. Test specifications

Characteristics	Test conditions
Bottom Hole static Temperature (BHST)	80 °C (176 °F)
Bottom hole Circulation Temperature (BHCT)	66 °C (150 °F)
Bottom Hole pressure	2.41E7 pa. (3500 psi)
Well depth	1524 m (5000 ft)
Casing diameter	0.33m (13 3/8 in)

2.5 Thickening time

The thickening time is related to pumpability time under the well conditions of temperature and pressure (Salam et al., 2014). The test was performed using a High Pressure High Temperature (HPHT) consistometer (Chandler Engineering, model 7720). The unit of consistency is Bearden (Bc). For each run, the test was terminated when the slurry achieves a consistency of 100 Bc. The slurry container which is equipped with a stationary paddle assembly is rotated at a speed of 150 rpm. The experiment was repeated twice for each run and the average values recorded are shown in Table 4.

3. Results and Discussion

3.1 Compressive strength

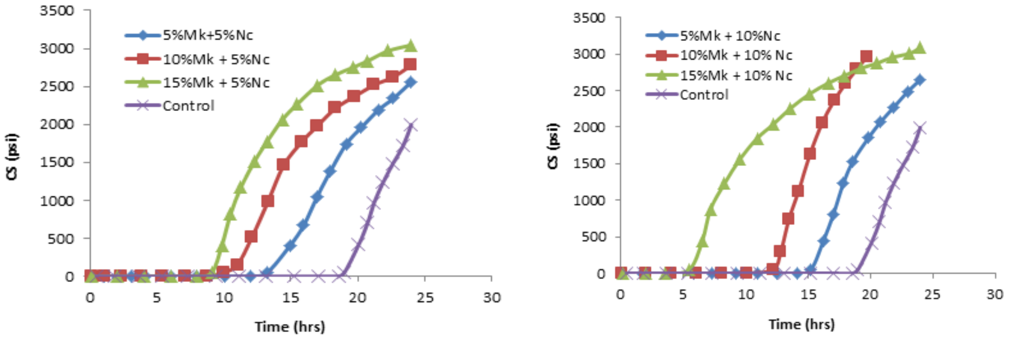
The results obtained for the CS of all cement slurries tested with different dosages of MK and NC after 24 h are shown in Figure 1. For 5 wt% NC in the cement slurry, it took the cement paste to attain the minimum CS of 500 psi periods of 11:05 h, 15:48 h, 12:10 h and 10:04 h when 0, 5, 10 and 15 wt% MK dosages were used, respectively. This indicates that 15 wt% MK substitution in the presence of 5wt% NC accelerated the attainment of minimum CS to about 1 hour earlier than for the control with 0 wt.% MK. A similar effect was observed when the NC dosage was increased to 10 and 15 wt.%. The increment is attributed to the released of calcium hydroxide and silica for pozzolanic reaction (Nadeem, Memon, and Lo, 2013). After 24 h curing time, the maximum CS obtained for the various dosages of MK and NC ranged within 2560-3100 psi, while 1,997 psi was obtained for the control paste. The highest CS was recorded at 15 wt% of MK and NC each, while the lowest CS recorded was at 5 wt% MK and NC. However, CS increases non-uniformly with NC dose at a specific dose level of MK. For example, with 15 wt. % MK in the cement paste, as shown in Figure 1(a-c), CS of 3045, 3090 and 3100 psi were recorded for 5, 10 and 15 wt% NC, respectively. Similarly, for a fixed amount of NC in the mix, the cement samples with MK replacement were observed to acquire remarkably higher CS than the sample with no MK substitute. This observation could be attributed to the higher rate of dissolution and hydration of MK, which makes more silica available for the pozzolanic reaction. It was observed, therefore, that both MK and NC show positive effects on the CS of cement.

3.2 Thickening time

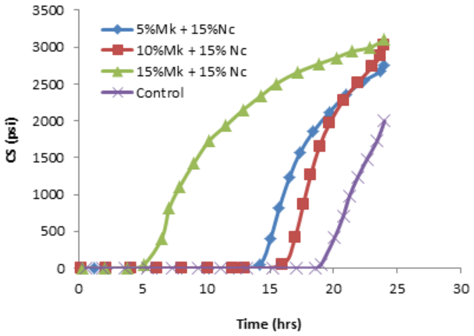
The results obtained for the TT measured at 70 Bc consistency for 10 cement slurries (including the control) that were tested, containing different doses of MK and NC, are shown in Figures 2–4. TT for MK and NC laden cements ranged in 334 – 492 minutes at 70 Bc. When this is compared

Table 3. Box–Behnken design matrix for substitution of cement using MK and NC

Run	A:Metakaolin (wt.%)	B:Nanoclay (wt.%)	Fluid loss (ml)	Dispersant (ml)	CS (psi)	TT (mins)
1	-1	0	7	12	2651±1.12	463±2.10
2	-1	1	7	12	2761±0.78	432±1.32
3	0	1	7	12	3024±1.02	408±1.21
4	-1	-1	7	12	2560±2.10	470±0.65
5	0	0	7	12	2964±1.02	421±0.23
6	0	-1	7	12	2779±0.87	428±0.05
7	1	1	7	18	3100±0.67	334±0.04
8	1	-1	7	18	3045±1.13	370±1.01
9	1	0	7	18	3090±1.03	363±0.45



(a) 5wt% Nanoclay (b) 10wt% Nanoclay



(c) 15wt% Nanoclay

Figure 1. Effects of MK dose on compressive strength of cement slurry in the presence of various amounts of nano-clay

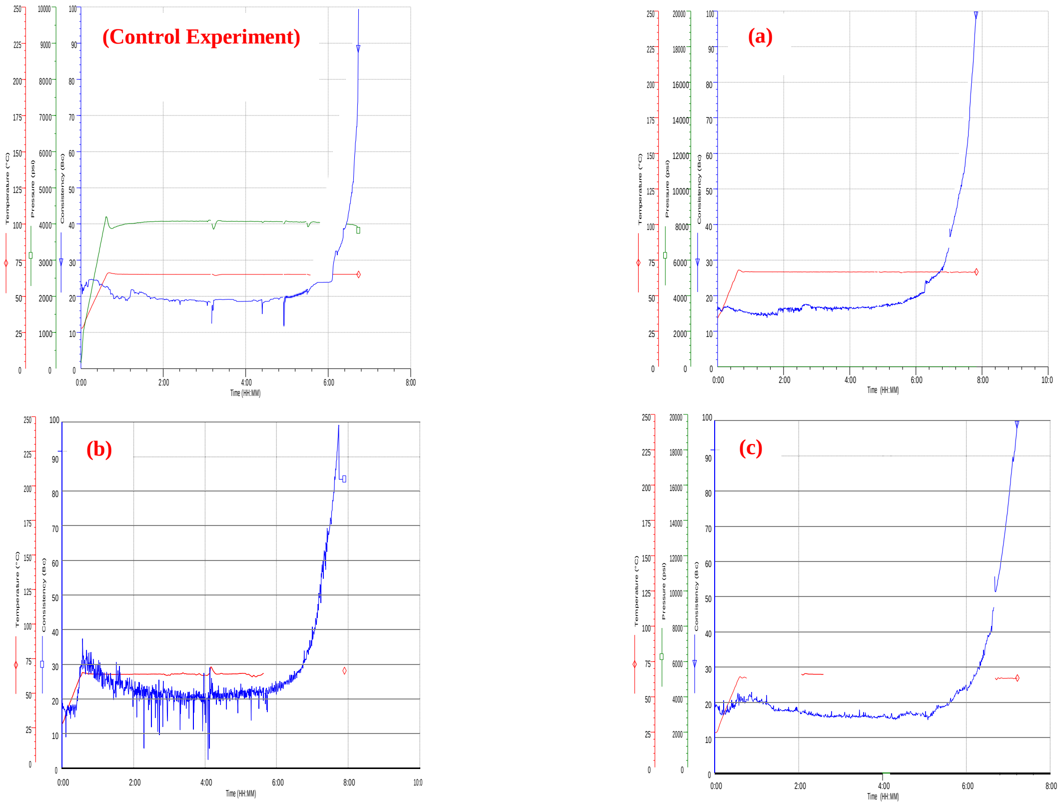


Figure 2. Time profiles of thickening of cement slurry with 5% MK, (a) 5 % NC, (b) 10 % NC, and (c) 15 % NC

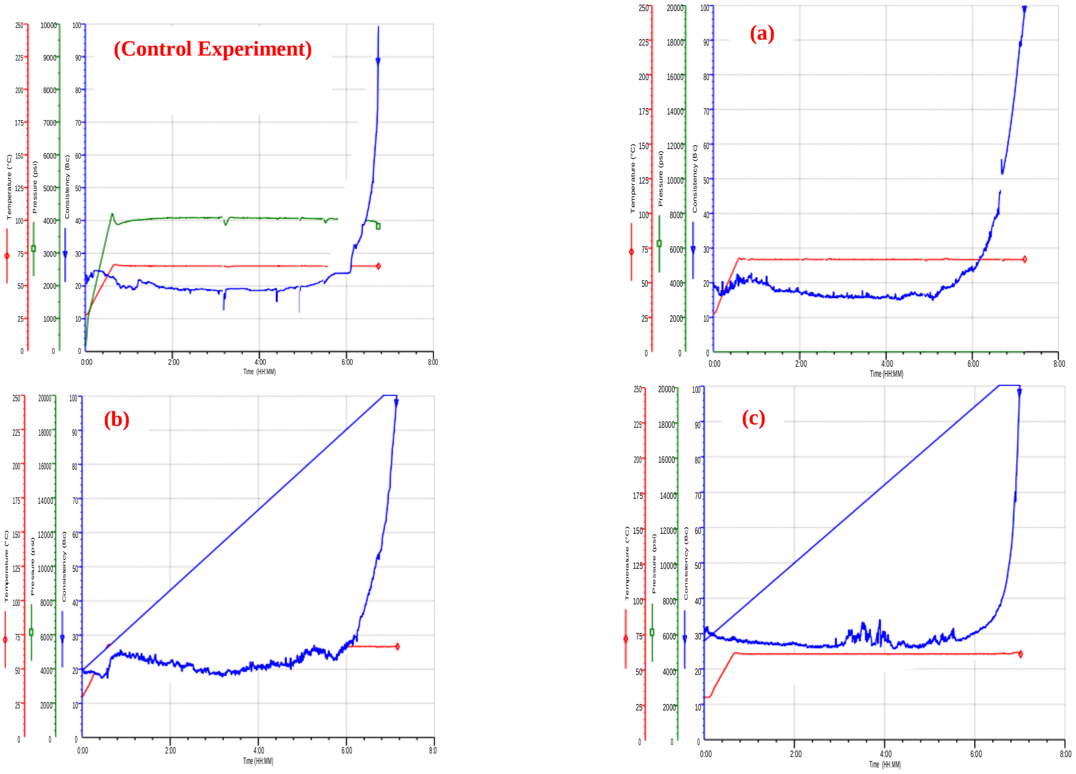


Figure 3. Time profiles of thickening of cement slurry with 10 % MK, (a) 5 % NC, (b) 10 % NC, and (c) 15 % NC

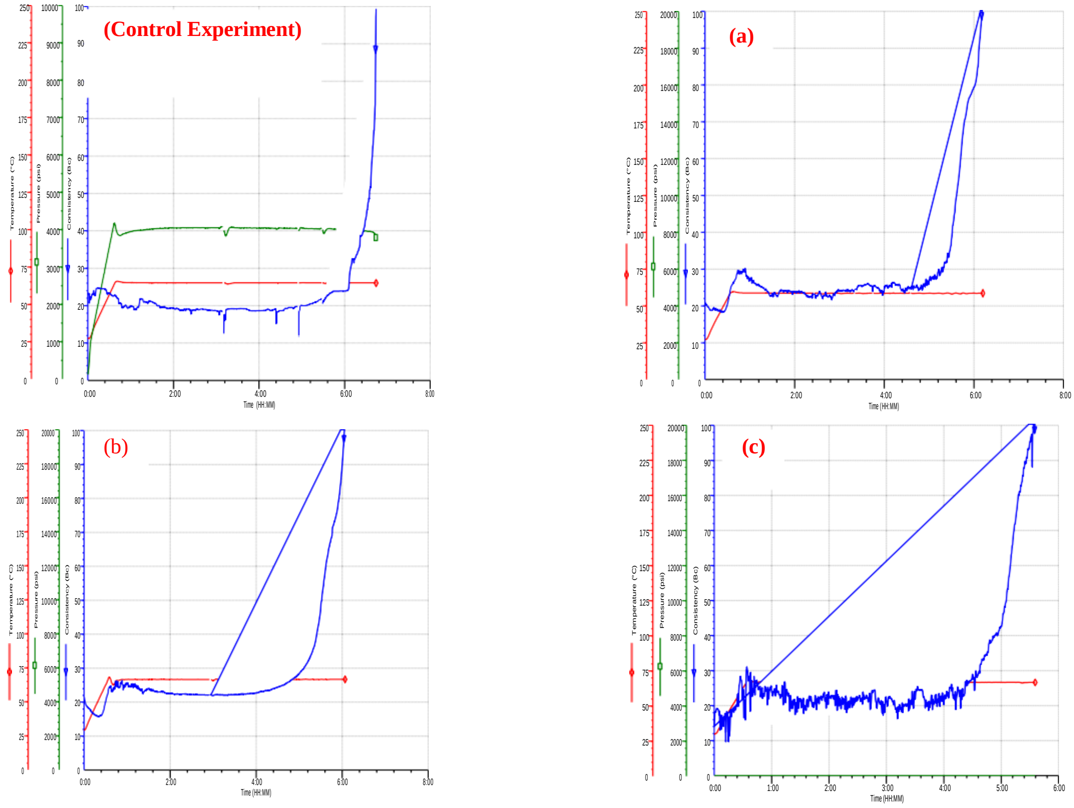


Figure 4. Time profiles of thickening of cement slurry with 15 % MK, (a) 5 % NC, (b) 10 % NC, and (c) 15 % NC

with the control slurry with 408 minutes TT, it is clear that the addition of certain wt% of MK and NC increased the thickening time. This is desirable because there would be enough time for pumping of cement before it sets. It is expected that within the first 30 minutes of pumping, the consistency of cement should be below 30 Bc (API RP 10B, 2012). A quick survey of Figures 2-4 shows a high degree of compliance below 30 Bc, including the control experiment. However, noticeable is the inconsistency and initial fluctuations observed especially in Figure 3 (c) and Figure 4 (a) after the first 30 minutes. The initial fluctuations in Figure 2(b) and Figure 4 (c) can be due to calcium ion (Ca^{2+}) chelation. According to Huajie, Yuhuan, Jay, and Zhonghou, (2015), MK has the ability to chelate Ca^{2+} generated by the hydration of cement. The chelation of Ca^{2+} can cause a consistency wave at initial thickening stages. Comparing all the slurries with the control experiment, a longer TT is guaranteed even with minimal amounts of MK and NC. However, a much higher TT was recorded with 5wt% of MK and NC each. However, higher CS was obtained with values above 5 wt%. Thus, to ensure adequate CS and TT, the two variables need to be optimized. Similarly, the shortest TT was recorded with 15 wt % of MK and NC each. For sustainable substitution of cement, accurate control of the TT is necessary. If the TT is too short, the cement fails to reach its required placement, while too long a TT leads to costly delays (Billingham, Francis, King, and Harrisson, 2005).

4. Optimization Study

4.1 Analysis of variance (ANOVA)

For the optimization study, building an objective function by use of numerical models is necessary. The numerical modeling used Analysis of Variance (ANOVA), as presented in Table 5. The F-values 865.49 and 67.32 obtained for CS and TT indicate that the selected quadratic models are significant. There is only a 0.01% and 1.47% chances that these F-values could occur randomly. The value of P-statistic below 0.005 indicates high degree of significance of a model term. Since the response surface methodology was adopted, only terms that satisfy this condition were selected, and therefore MK, NC, MK*NC, MK^2 and NC^2 are the significant model terms.

For both responses, the predicted R^2 of 0.9911 for CS and 0.8793 for TT are in reasonable agreement with the Adjusted R^2 of 0.9980 for CS and 0.9793 for TT, since the difference is less than 0.2 in both cases. The equations in terms of actual factors are presented in equations 1 and 2.

$$CS(psi) = 1890.556+132.21111*MK+27.3111*NC-1.46*(MK*NC)-3.78*MK^2 \quad (1)$$

$$TT(mins) = 497-2.91*MK-0.114*NC+0.206*(MK*NC)-0.423*MK^2-0.2*NC^2 \quad (2)$$

Equations 1 and 2 can be used to make predictions about the response for given levels of each manipulated factor. Here, the levels should be specified in the original units for each factor.

4.2 Main effects of MK and NC on CS and TT

Figure 5 shows the effects of different dosages of MK and NC on CS and TT of cement pastes. For each of the variables investigated, TT and CS exhibited distinct and opposite behaviors (i.e. TT decreased with MK, while CS increased with MK). It is obvious from Figure 5 that as MK or NC increases, the TT decreases. This reduction in TT is desirable since too long thickening time leads to costly delays and increases cementing costs. However, a very short thickening time leads to premature setting of cement in the casing or pumping equipment (Coveney, Fletcher, & Hughes, 1996). The dominant effect of MK on TT was obvious with a sharp TT reduction when equal doses are used. MK and CS exhibit a non-linear relationship evidenced in Figure 5. As the MK dose increases, the CS of the cement increases disproportionately. On the other hand, NC shows a linear relationship with CS. As NC dose increases, the CS of the cement increases almost linearly. The curved response to MK indicates existence of an optimum. This may be the main reason why only 5-15wt% cement substitution is mostly reported in the literature. Beyond the optimum amount, further increase in MK could reduce CS and perhaps increase TT. As an exception, the use of MK up to 20wt% has also been reported (Yuhuan Jiapei, Shenglai, Huajie, & Chenxing, 2016).

4.3 Interaction effects of MK and NC on CS and TT

Figure 6 shows effects of simultaneous increases in doses of MK and NC on TT and CS at test conditions. It is observed that increasing MK dose from 5 to 15 wt% decreased TT from 470 to 367 min when the NC was fixed at 5 wt% in the cement slurry. When the dose of NC was increased to 15 wt%, TT decreased from 435 to 353 when MK dosage was increased from 5 to 15 wt%. From the two curves on the TT plot (in red and black), it is inferred that a further increase of NC beyond 15 wt% could further reduce TT. However, the same could not be said of MK, as the two curves tend to approach each other at some MK dosage beyond the studied maximum limit of 15 wt%. The interaction of the two variables on CS indicates that both parameters contributed positively to the cement strength. Higher dosage of NC implies higher CS of cement. At maximum NC dosage of 15 wt% (red color), varying the MK dosage from 5 to 14 wt% increased the CS from 2757 to 3103 psi. It is noted that an MK dosage beyond about 14 wt% produced no significant increase in CS, which is a very good motivation for optimization.

Table 5. ANOVA for CS and TT model selection

Properties	Model	Adj. R-square	Pred. R-square	DF	F-value	p-value	SSE	MSE
CS (psi)	Quadratic	0.998	0.9911	4	865.49	<0.0001	3.13E+05	78311.11
TT (mins)	Quadratic	0.9793	0.8793	5	67.32	0.0147	10386.16	2077.23

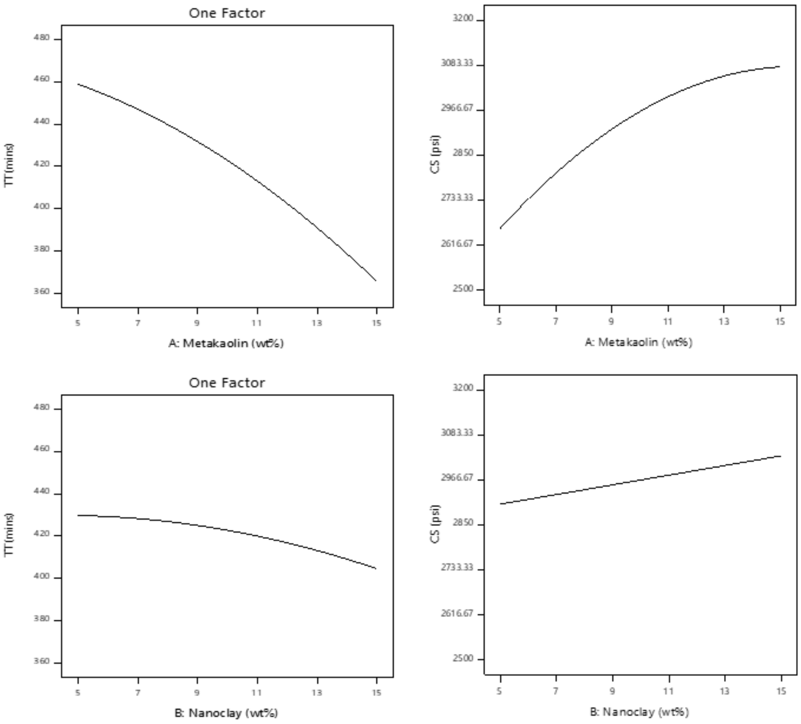
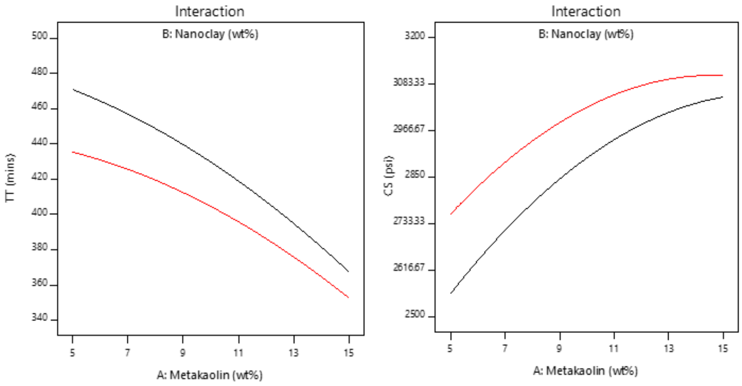


Figure 5. Factor effects on TT and CS of partially substituted cement



Red colour-maximum, Black colour-minimum

Figure 6. The factor effects of MK and NC on TT and CS of partially substituted cement

4.4 Optimization studies

The numerical optimization using desirability function as available in the Design Expert version 11 was employed to minimize TT and maximize CS of the cement slurry within the selected ranges of 5 – 15 wt% for MK and NC. After 100 iterations, the solution with desirability of 1 was selected as optimum. The solution points are shown on a 3-Dimensional plot in Figure 7. At this point, 10.78 wt% MK and 13.73 wt% NC that produced the optimum, CS and TT of 3036 psi and 403 minutes were model predicted, respectively. The optimum point was also validated experimentally. The experiments were replicated twice with averages and standard deviations of 3029 ±2.65 psi and 410±1.25 minutes for CS and TT, respectively.

5. Conclusions

The coupled effects in ternary blends of class G cement with MK and NC were investigated experimentally and blend ratio of the mixture was optimized for effective CS and TT using response surface methodology. Based on such analysis, the following conclusions are drawn: the compressive strength of cement paste increased with MK and NC dose levels. Although the CS increases linearly with NC dose, the effect is less pronounced when compared with effects of MK. MK effect on CS is hyperbolic and outstanding within some limits. Beyond 13 wt% dose, MK shows no significant increase of CS in the presence of NC. MK and NC are good reducers of TT and are therefore applicable cementing if strongly elevated temperatures are expected.

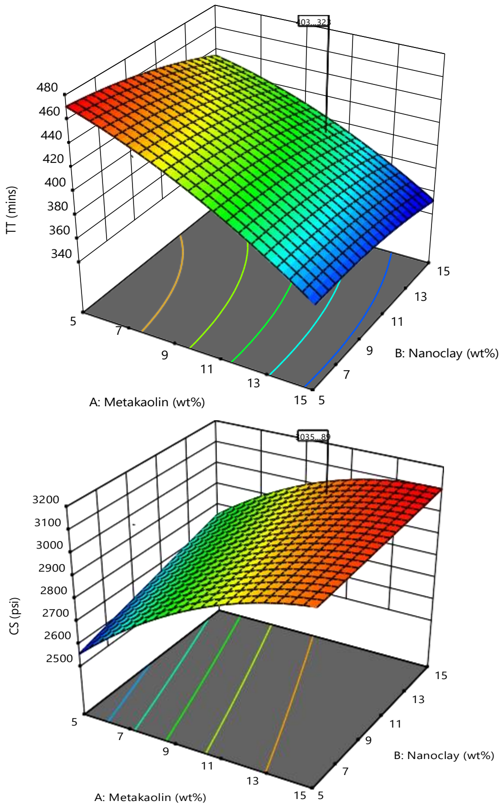


Figure 7. Surface plots showing interactions of factors at optimal TT and CS

They both exhibited disproportionate relationship with TT. This reduction in TT is desirable, since too long thickening time delays the process and increases operating costs. The modeling of effects of MK and NC on CS and TT revealed a synergy between these manipulated variables. Since MK is not expensive, is readily available, and only very low doses of NC were tested in this present study, while they produced tremendous increases in CS with a reduction of TT, it is therefore recommended to investigate similar ternary blends at much higher temperatures and pressures.

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