



Influence of Metakaolin and Nano-clay on Compressive Strength and Thickening Time of Class G Oil Well Cement

Journal:	<i>Songklanakar Journal of Science and Technology</i>
Manuscript ID	SJST-2019-0380
Manuscript Type:	Original Article
Date Submitted by the Author:	27-Aug-2019
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Keyword:	G-class cement, supplementary material, compressive strength, , thickening time, Box-Behnken Design

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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) dosage was evaluated in the presence of Nanoclay (NC). The variables were randomized in a Box-Behnken Design (BBD) experiment using 5 - 15 wt. % MK by weight of cement and 5-15 wt. % NC by weight of MK. The CS and thickening time were assessed and optimized using Response Surface Methodology (RSM). The result shows 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. TT reduction of 103 minutes was recorded when MK was increased to 15 wt.% in the slurry with 5wt.% NC. At optimum condition, 10.78

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4 wt. % MK and 13.73 wt. % NC resulted in CS and TT of 3029 ± 2.65 psi and 410 ± 1.25
5
6 minutes, respectively.
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10 **Keywords:** G-class cement, supplementary material, compressive strength, thickening
11 time, Box-Behnken Design
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14 **1. Introduction**

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16 The oil well cementing was primarily introduced in the late 1920s because drilling
17 fluid alone cannot prevent the well bore from collapsing (Joshi & Lohita, 1997). Among
18 other reasons for cementing oil wells include: protecting oil producing zones against salt
19 water, protecting the casing from collapse under pressure, protecting well casings from
20 corrosion, reducing the risk of ground water contamination by hydrocarbons or salt water
21 and to provide zonal isolation. There are two critical conditions which a successful oil
22 well cementing should satisfy: ability to remain pumpable for sufficient time to ensure
23 proper placement in the well bore and ability to build, and maintain sufficient mechanical
24 strength to provide adequate support for the casing.
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37 Ordinary Portland Cements (OPCs) has been used as oil well cement for many
38 years. However, OPCs was reported to undergo strength reduction, increased porosity
39 with severe durability at elevated temperature, in acid rich, geothermal and deep oil wells
40 environments (Ma, Chen, & Chen, 2014; Won, Lee, Na, Lee, & Choi, 2015). Thus, a
41 special class of cements, the oil well cements (OWCs) emerged. OWCs are classified into
42 grades depending on their C_3A (Tricalcium Aluminate) content. The detailed classes are
43 available elsewhere (API Specification 10A, 2010). Of all these classes, classes A, G and
44 H are the three most commonly used for cementing oil and gas wells. While class A is
45 used in milder, less demanding well conditions, classes G and H are usually specified for
46 deeper, hotter and higher pressure well conditions (Eric, Joel, & Grace, 2016).
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4 The use of supplementary cementitious materials (SCMs) for class G cements has
5 received increased attention over the last few decades. These materials react with
6 $\text{Ca}(\text{OH})_2$ released during cement hydration and convert it into high crystallinity calcium
7 silicate hydrates Yuhuan, Jiapei, Shenglai, Huajie, & Chenxing, 2016). Buntoro &
8 Rubiandini, (2000) studied the mechanical properties of cement slurry with 35 wt.% silica
9 flour. The results after 3 days of curing show higher shear bond strength and compressive
10 strength. Li, Sun, and Li, (2010) compared the performance of the mechanical properties
11 of MK cement with alkali-treated slag cement; the result obtained showed that cement
12 pastes containing MK exhibited better CS at high temperatures. The behaviour of blended
13 cement mortars containing nano-MK at elevated temperatures was investigated by Morsy,
14 Al-Salloum, Abbas, and Alsayed, (2012). Their conclusion was that at low temperature
15 of 25 °C, 5 wt% nano-MK gave optimal mortar and at temperature of 80 °C, 15 wt% nano-
16 Mk produced better CS. Shatat (2013) studied the hydration behaviour and mechanical
17 properties of blended cement containing various amounts of rice husk ash in the presence
18 of MK. They recorded better mechanical behaviour than OPC with ternary blends of
19 cement with 5–10 wt% rice husk ash and 15–20 wt% MK. Nadeem, Memon, and Lo,
20 (2014) evaluated the fly ash and MK concrete at elevated temperatures using stiffness
21 damage test. The hardened cement blend showed better mechanical behaviour than OPC.

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46 Mechanical properties of OWCs depend also on the slurry density. The various
47 weighing agents and extenders control the compressive strength and the time after initial
48 mixing when the cement can no longer be pumped. High specific gravity and finely
49 divided solid materials such as barites, bentonites and micro-sands are used to increase
50 the density (Halliburton, 2009). Micro-sand according to Chenevert and Shrestha, (1991)
51 are capable of reducing the total chemical shrinkage thereby reduce gas leakages, reduces
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4 the free water and prevent environmental problems. For field application, it is expected
5 that an additive should influence only the property for which purpose it is added.
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7 Experimental Design has been successfully deployed to resolve how factors interact in
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9 mixtures and has assisted in solving many engineering problems (Salam, Arinkoola, Oke,
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11 and Adeleye, 2014; Arinkoola & Ogbe, 2015). Additives with multiple and conflicting
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13 effects pose serious challenges as improvement of one property could result in negative
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15 effects on one or more properties of the slurry. The objective of this present study is to
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17 investigate and optimize the couple effects of ternary blends of class G cement with 5–
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19 15wt% MK and 5–15wt% NC on CS and TT properties using experimental design and
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21 response surface methodology.
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27 **2. Materials and Methods**

28 **2.1 Materials**

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31 The class G oil well OPC used was obtained from SOWSCO Oil Well Service (Nig.) Ltd,
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33 Port Harcourt, Nigeria. **Table 1** shows the various oxides and physical properties of the
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35 cement. The kaolin clay from where MK was synthesized was locally sourced from a clay
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37 deposit site in Okpella, Etsako East LGA of Edo state, Nigeria (latitude 7.120 N,
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39 longitude 6.280 E). The Nanoclay (1.31 ps) used was a product of Nanocor, inc.
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41 purchased from SIGMA ALDRICH (M) Sdn. Bhd, Malaysia. It made up of
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43 montmorillonite clay surface modified with 15 – 35wt% Octadecylamine and 0.5 - 5 wt%
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45 aminopropyltriethoxysilane. Other chemical additives such as fluid loss control additive
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47 (FLA-001), dispersing agent (polynaphthalene sulfate), retarder, gas block additive
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49 (SWLGX3) and antifoam were provided by SOWSCO, Nigeria.
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55 **2.2 Synthesis of Metakaolin**

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4 The oven dried kaolin clay sample was grounded using electric grinder (Marlex
5 Appliances PVT Limited) and screened to fine powder ($\sim 20 \mu\text{m}$) before it was subjected
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7 to calcination in a muffle furnace (NYC-12 model) operating at $750 \text{ }^\circ\text{C}$ for 2 h. The
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9 chemical composition of MK produced is shown in **Table 2**.

13 **2.3 Cement slurry design**

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15 Slurry formulation was done according to API standard RP 10B-2012. The NC and MK
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17 were varied randomly in the cement slurry within the range 5-15 wt.%. While the dosage
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19 of MK was determined by weight of cement (BWOC), the NC dosages were measured
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21 by weight of MK (BWOMK). **Table 3** shows the fixed concentration of additives and
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23 matrix for nine pastes randomized according to BBD (Stat Ease Design Expert Version
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25 11). The fluid loss control additive (12-18 wt%), retarder and dispersing agent (7 wt%)
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27 were dissolved in 380 ml of water and transferred into the cup of waring blender. Then,
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29 the blended cement was added within 15 s to the aqueous solution with a stirring rate of
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31 4000 rpm and mixed for 35 s at 12000 rpm. After being prepared, cement slurry was
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33 placed into 5 cm cubes for CS test and pressurized consistometer for consistency
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35 measurement.

41 **2.4 Compressive strength test**

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43 The test condition and well specification for CS test is presented in **Table 4**. The CS of
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45 different pastes was measured using Ultrasonic Compressive Analyzer (Chandler
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47 Engineering model 4265 UCA). The cement slurry was placed in an autoclave unit in the
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49 UCA at BHST of $80 \text{ }^\circ\text{C}$ and pressure of 3500 psi then sonic wave was transmitted through
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51 it. The CS developed after 24 h for two samples was recorded automatically and
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53 transferred to data acquisition software (Chandler Engineering Model 5270). The average
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55 value of the CS was recorded.
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2.5 Thickening time

The thickening time is related to pumpability time under 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 runs and the average values recorded as shown in **Table 4**.

3. Results and Discussion

3.1 Compressive strength

The result obtained for the CS of all cement slurries tested with different dosage 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 minimum CS of 500 psi, a period of 11:05 h, 15:48 h, 12:10 h and 10:04 h when 0, 5, 10 and 15 wt% MK dosages was used, respectively. This result indicated that 15 wt% MK substitutions in the presence of 5wt% NC had accelerated the attainment of minimum CS about 1 hour earlier than the control with 0 wt.% MK. Similar result 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, maximum CS obtained for various dosages of MK and NC ranges between 2560-3100 psi as against 1,997 psi obtained for the control paste. The highest CS was recorded at 15 wt% each of MK and NC while the lowest CS was recorded at 5 wt% MK and NC. However, CS increases non-uniformly with NC dosages for specific dosage of MK. For example, with 15 wt. % MK

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4 in the cement paste as shown in figure 1(a-c), CS of 3045, 3090 and 3100 psi were
5 recorded for 5, 10 and 15 wt% NC, respectively. Similarly, for a fix amount of NC in the
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9 mix, the cement samples with MK replacement was observed to acquire remarkably
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11 higher CS than the sample with no MK substitute. This observation could be attributed to
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13 higher rate of dissolution and hydration of MK which make more silica to be available
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15 for the pozzolanic reaction. It was observed therefore that both MK and NC show positive
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17 effects on the CS of cement.
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20 21 **3.2 Thickening time**

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24 The result obtained for the TT measured at 70 Bc consistency for 10 cement slurries
25 (including the control) that were tested containing different dosages of MK and NC are
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27 shown in figures 2–4. TT for MK and NC laden cements ranges between 334 – 492
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29 minutes at 70 Bc. When this is compared with the control slurry with 408 minutes TT, it
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31 was clear the addition of certain wt% MK and NC increased the thickening time. This is
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33 desirable because there would be enough time for pumping and setting of cement. It is
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35 expected that within the first 30 minutes of pumping, the consistency trend of cement
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37 should be located below 30 Bc (API RP 10B, 2012). Quick survey of figures 2-4 shows
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39 some high degree of compliance below 30 Bc including the control experiment. However,
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41 noticeable is the inconsistency and initial fluctuation observed especially in figure 3 (c)
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43 and figure 4 (a) after the first 30 minutes. The initial fluctuation in figure 2(b) and figure
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45 4 (c) can be due to calcium ion (Ca^{2+}) chelation. According to Huajie, Yuhuan, Jay, and
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47 Zhonghou, (2015), MK has the ability to chelate Ca^{2+} generated by the hydration of
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49 cement. The chelation of Ca^{2+} can cause the consistency wave at thickening initial stages.
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51 Comparing all the slurries with the control experiment, longer TT is guaranteed even with
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53 minimal amount of MK and NC. However, a much higher TT was recorded with 5wt%
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each of MK and NC. But 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 % each of MK and NC. 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, whilst too long a TT leads to costly delays (Billingham, Francis, King, and Harrisson, 2005).

4. Optimization study

4.1 Analysis of Variance (ANOVA)

For optimization study, building objective function is necessary. This was achieved using the Analysis of Variance (ANOVA) as presented in Table 5. The F-value of 865.49 and 67.32 obtained for CS and TT implies the selected quadratic models are significant. There is only a 0.01% and 1.47% chances that these F-values could occur due to noise. The values of the P-statistics if less than 0.005 indicate high degree of significance of model terms. Since response surface methodology was adopted, only terms that satisfied this condition were selected and therefore MK, NC, MK*NC, MK² and NC² are significant model terms.

For both responses, the predicted R² of 0.9911 for CS and 0.8793 for TT are in reasonable agreement with the Adjusted R² 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 equation 1 and 2.

$$CS (psi) = 1890.556 + 132.2111 * MK + 27.3111 * NC - 1.46 * (MK * NC) - 3.78 * MK^2 \quad (1)$$

$$TT \text{ (mins)} = 497 - 2.91 * MK - 0.114 * NC + 0.206 * (MK * NC) - 0.423 * MK^2 - 0.22 * NC^2 \quad (2)$$

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

4.2 Main effect 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 behaviour (i.e. TT decreased with increase in MK while CS increased with increase in MK quantity). It is obvious from figure 5 that as MK or NC increases, the TT reduces. This reduction in TT is desirable since too long thickening time leads to costly delays and increases cementing costs. However, very short thickening times leads to premature setting of cement in the casing or pumping equipment (Coveney, Fletcher, and Hughes, 1996). The dominant effect of MK on TT was obvious with a sharp TT reduction when equal dosages of factors are used. MK and CS exhibit a non-linear relationship as evidence on figure 5. As MK dosage increases, the CS of the cement increases disproportionately. On the other hand, NC shows a linear relationship with CS. As NC dosage increases, the CS of the cement increases almost linearly. The curvature produced by the MK is an indication of existence of optimality. This may be attributed as the main reason why only 5-15wt% cement substitution is being reported mostly in the literature. Beyond the optimum amount, further increase in MK could lead to a reduction in CS and perhaps an increase in TT. Though the use of MK up to 20wt% have been reported (Yuhuan Jiapei, Shenglai, Huajie, and Chenxing, 2016).

4.3 Interaction effect of MK and NC on CS and TT

Figure 6 shows the simultaneous increase in dosage of MK and NC on TT and CS at test conditions. It was observed that increase in MK dosage from 5 to 15 wt% led to a decrease in TT from 470 to 367 min when the NC is fixed at 5 wt% in the cement slurry. When the dosage 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 (red and black color), it was inferred that, further increase of NC beyond 15 wt% could lead to further reduction of TT. However, the same could not be said of MK as the two curves tend towards approaching each other at some MK dosage beyond the studied maximum limit of 15 wt%. The interaction of the two variables on CS indicated that both parameters contributed positively to the cement strength as indicated on the CS curves. Higher dosage of NC implies higher CS of cement. At maximum NC dosage of 15 wt% (red color), varying MK dosage between 5 and 14 wt% increased the CS from 2757 to 3103 psi. It was noticed that MK dosage beyond about 14 wt% produced no significant increase in CS which is a very good rationale for optimization study.

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 3-Dimensional plot in figure 7. At this point, 10.78 wt% MK and 13.73 wt% NC produced at optimum, CS and TT of 3036 psi and 403 minutes, respectively. The optimum points were validated experimentally. The experiments were replicated twice with average and standard deviation of 3029 ± 2.65 psi and 410 ± 1.25 minutes for CS and TT, respectively.

5. Conclusions

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4 The couple effects of ternary blends of class G cement, MK and NC was
5 investigated experimentally and components of the mixture optimized for effective CS
6 and TT using response surface methodology. Based on the result and analysis performed,
7 the following conclusions were drawn: the compressive strength of cement pastes
8 increased with increase in the dosage of MK and NC. Although CS increases linearly with
9 dosages of NC, the effect is less pronounced when compared with MK. MK effect on CS
10 is hyperbolic and outstanding within some limits. Beyond 13 wt% dosage, MK shows no
11 significant increment of CS in the presence of NC. MK and NC are good reducer of TT
12 and are therefore applicable for cementing if extremely high temperature is expected.
13 They both exhibited disproportionate relationship with TT. This reduction in TT is
14 desirable since too long thickening time delays the process and increase operating costs.
15 The modeling of effect of MK and NC on CS and TT revealed a synergy between the
16 variables. Since MK is not expensive, readily available and just little dosage of NC was
17 tested in this present study to produce tremendous increase in CS with a reduction of TT,
18 it is therefore recommended to investigate the ternary blends at much higher temperature
19 and pressure.

20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 **6 Acknowledgements**

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44 The authors express gratitude to management of SOWSCO Well Services (Nigeria)
45 Limited, plot 212 Trans Amadi Industrial Layout Phase II, Port-Harcourt, Rivers State,
46 for making provisions for chemicals and for the permission granted to make use of their
47 cement laboratory.
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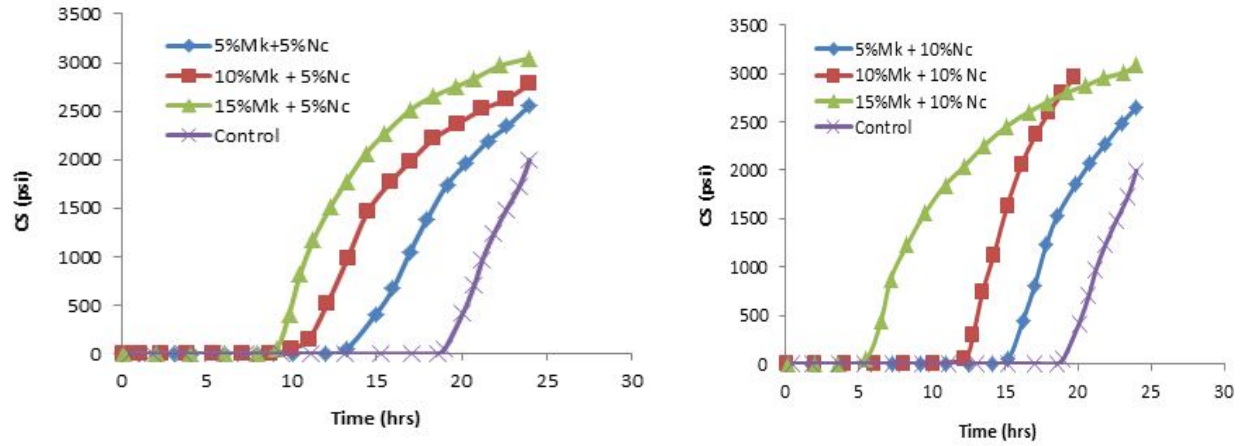
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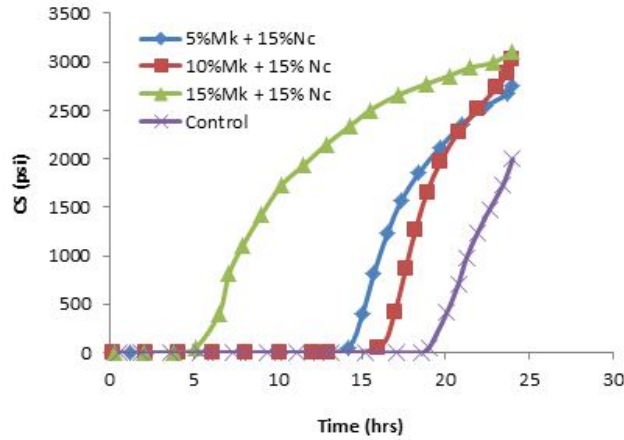
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(a) 5wt% Nanoclay

(b) 10wt% Nanoclay



(c) 15wt% Nanoclay

Figure 1: Effect of MK dosages on compressive strength of cement slurry in the presence of different amount of nano-clay

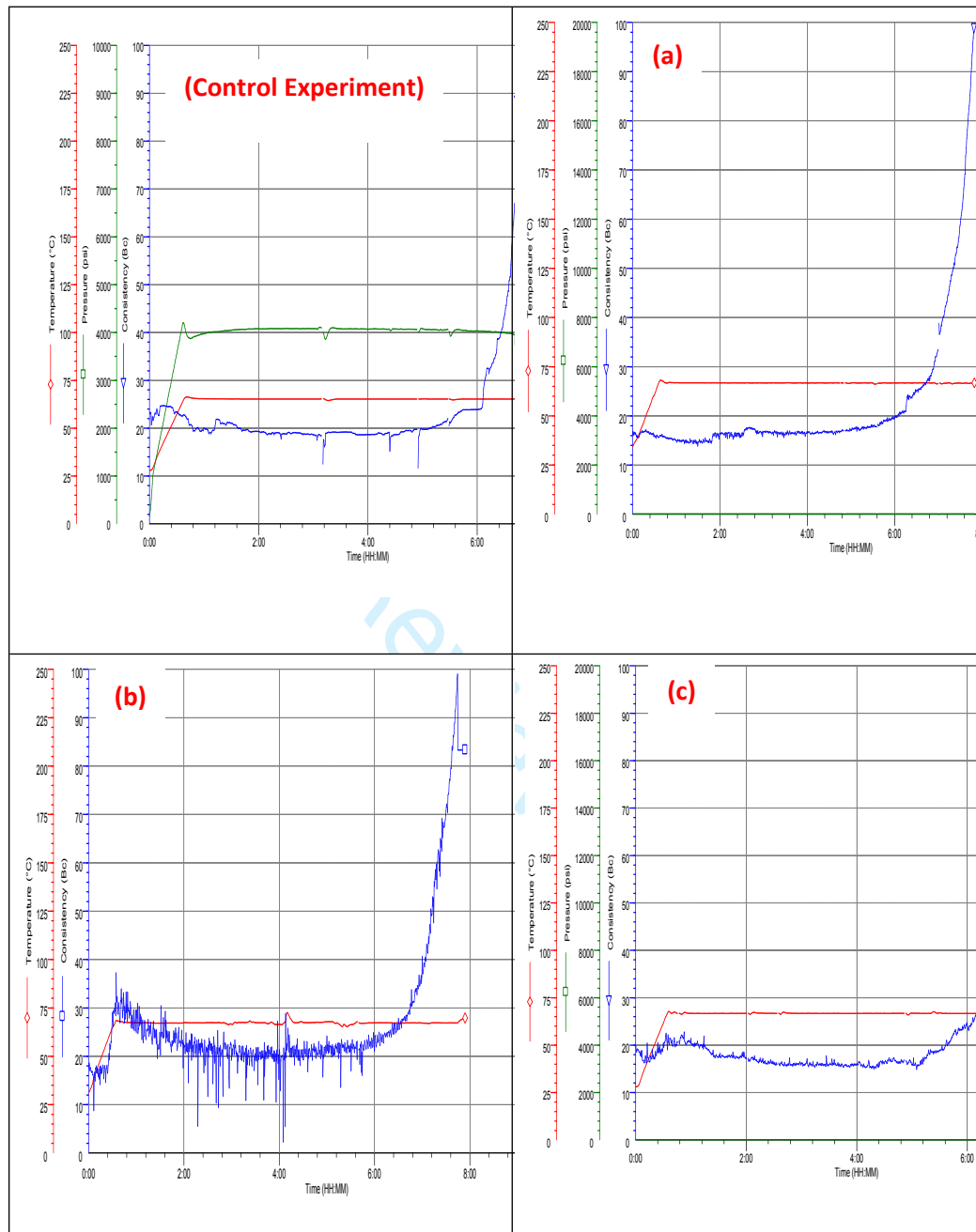


Figure 2: The thickening time of cement slurry with 5% MK (a) 5 % NC (b)10 % NC (c)15

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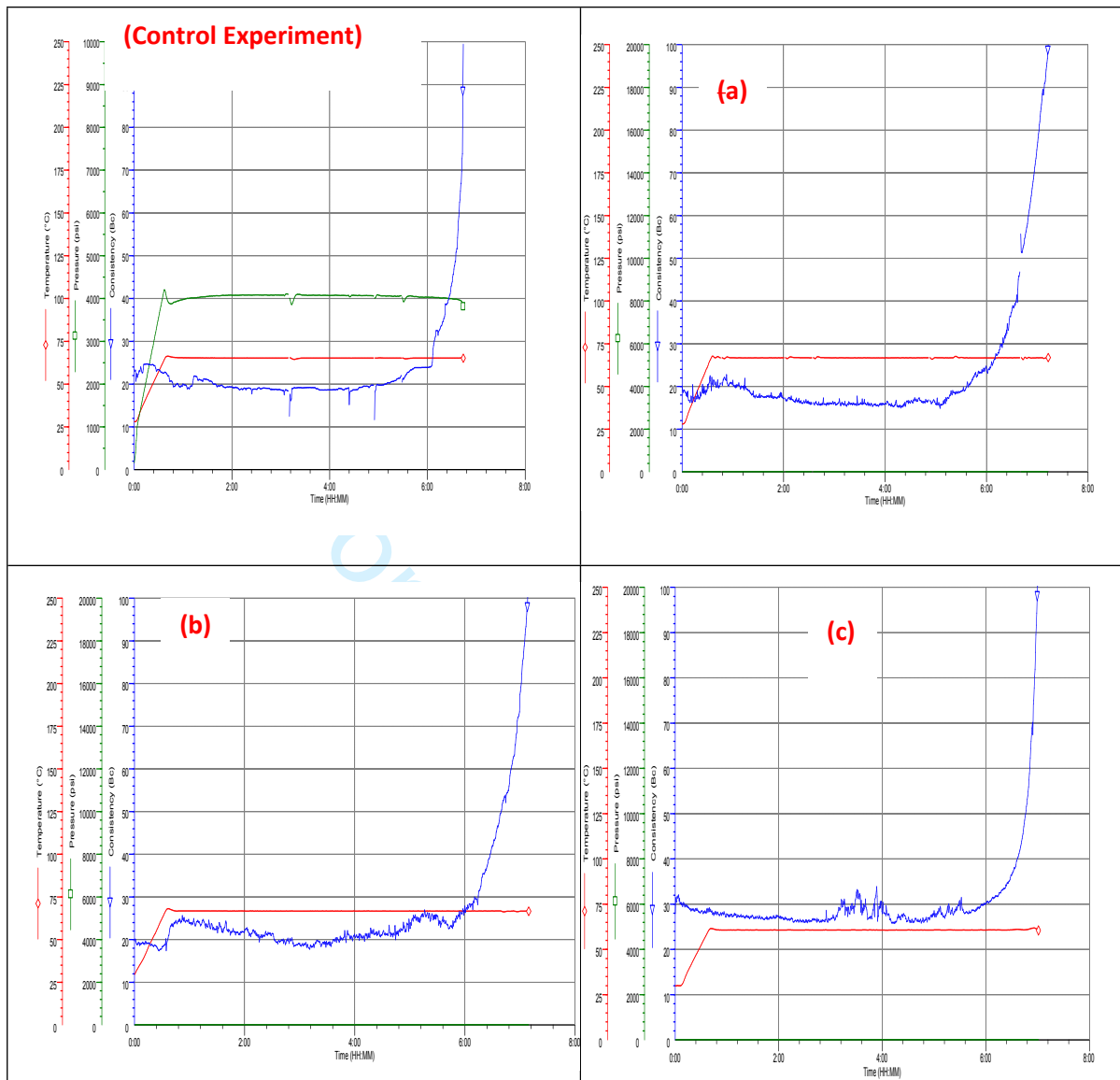


Figure 3: The thickening time of cement slurry with 10 % MK (a) 5 % NC (b) 10 % NC (c) 15%NC.

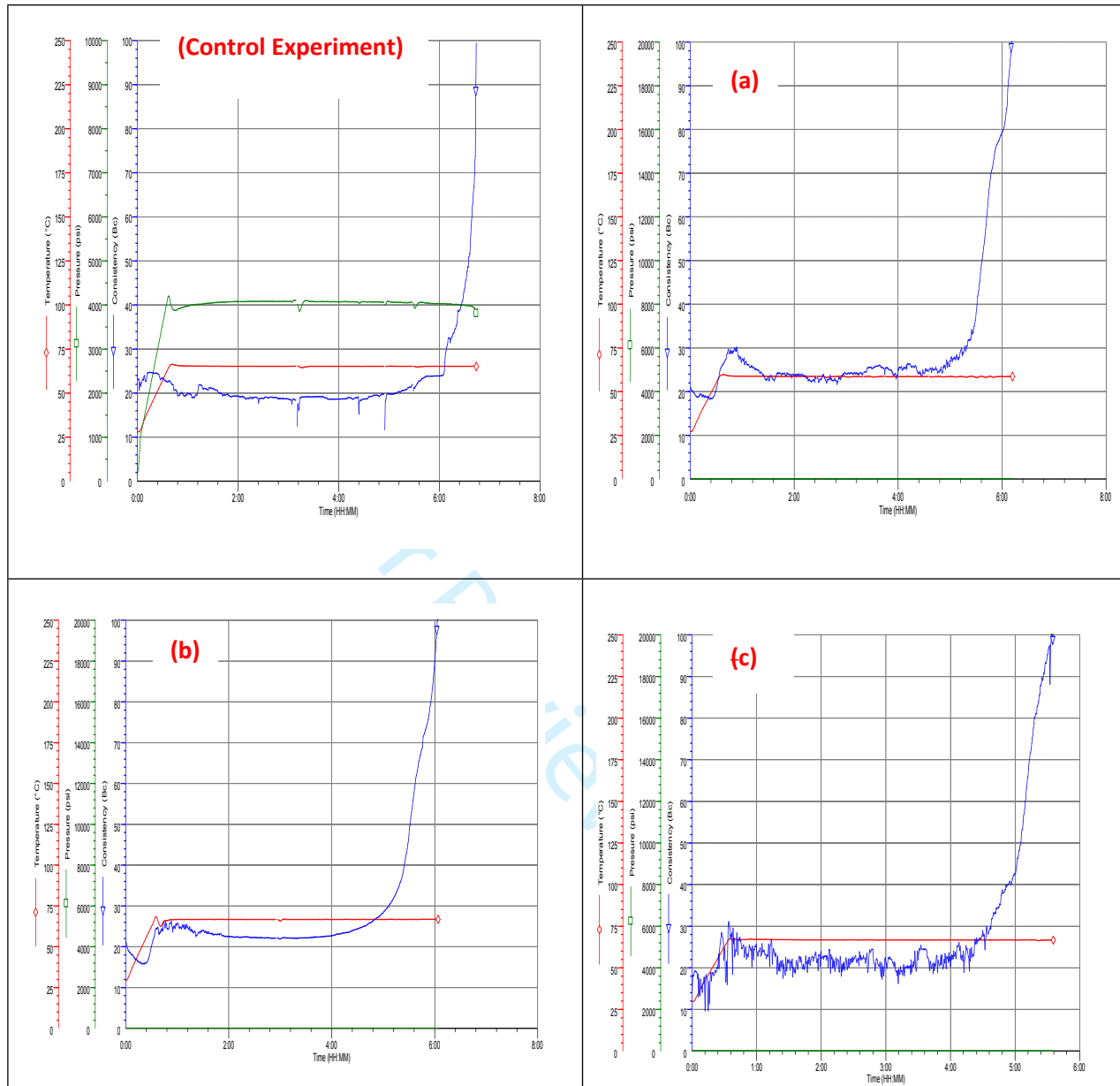


Figure 4: The thickening time of cement slurry with 15 % MK (a) 5 % NC (b) 10 % NC (c) 15 % NC.

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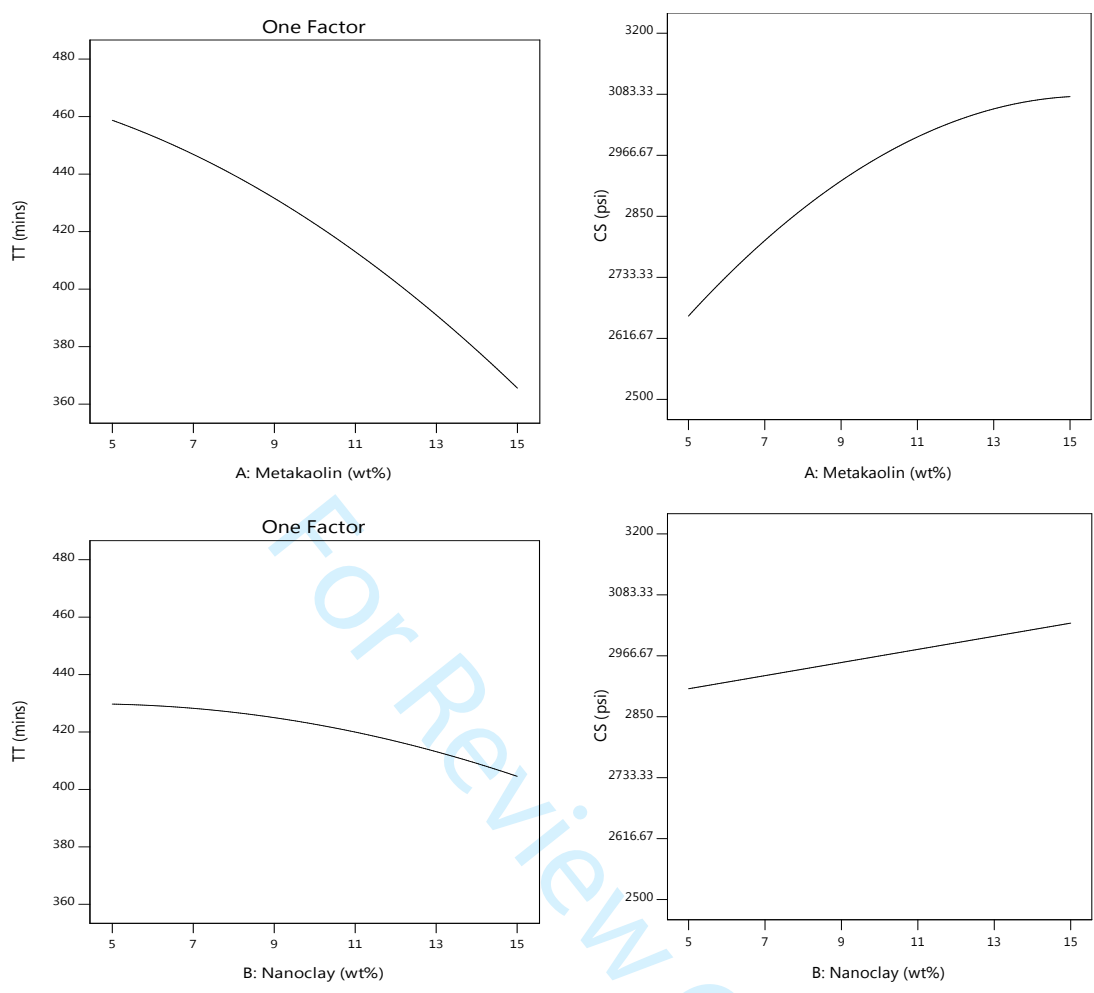
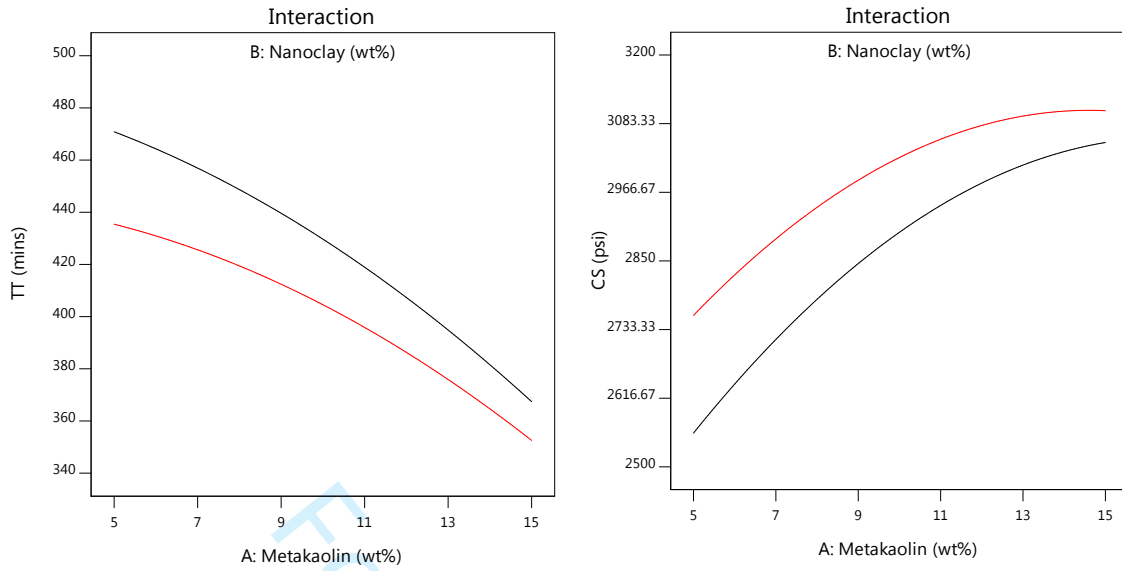


Figure 5 Factor effects on TT and CS of partially substituted cement



Red colour- maximum, Black colour- minimum

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

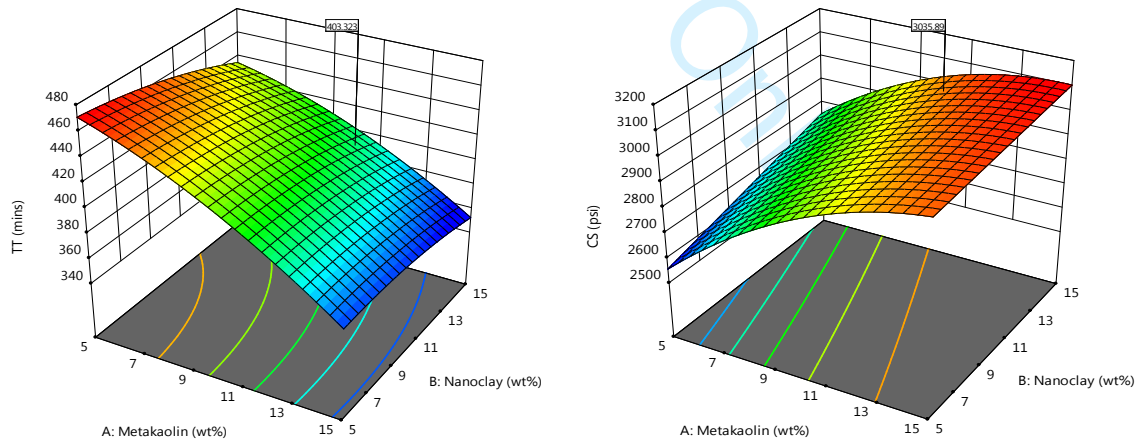


Figure 7 Surface plot of showing the interaction of variables at optimal TT and CS

Oxides	wt%
CaO	64.2
SiO ₂	19.4
Al ₂ O ₃	5.5
Fe ₂ O ₃	4.5
MgO	2
SO ₃	2.8
P ₂ O ₅	0.1
K ₂ O	0.6
Ignition loss	0.19
Density (g/cm ³)	3.16
Specific surface area (M ² /kg)	335.3

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

Oxides	Wt%
CaO	0.27
SiO ₂	53.68
Al ₂ O ₃	37.39
Fe ₂ O ₃	4.94
MnO	0.06
SO ₃	0.40
P ₂ O ₅	1.19
K ₂ O	1.25

Ignition loss	1.20
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Table 2: x-Ray Fluorescence (XRF) Analysis of MK

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

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

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)

Table 4 Test specification

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

Table 5: ANOVA for CS and TT model selection

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