Experimental analysis of *Mucuna solannie* as cement extender additive for oil and gas well cementing

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Abstract

Due to the difficulty that the compressive strength of cement slurries formulated with bentonite are not stable at elevated temperature conditions, in addition to other properties at high temperatures, *Mucuna solannie* commonly known as “Ukpo” was examined as an alternative. API standard procedures were employed throughout the laboratory measurements to determine overall rheological properties, compressive strength, thickening time, and free water of the extenders both at 150 °F (65.6 °C) and 200 °F (93.3 °C) BHCT. *Mucuna solannie* results gave compressive strength at 24 h as 952 psi (6.56E+6 Pa) and 900 psi (6.21E+6 Pa), free water values of 0 and 0.2 ml, yield point values of 67 lb/100ft² (32.08 Pa) and 66 lb/100ft² (31.60 Pa), and 10 min gel strength of 16 lb/100ft² (7.66 Pa) and 22 lb/100ft² (10.53 Pa). Bentonite additive gave 24 h compressive strength as 620 psi (4.27E+6 Pa) and 565 psi (3.9E+6 Pa), free water of 4.4 and 4.8 ml, yield point of 56 lb/100ft² (26.81 Pa) and 46 lb/100ft² (22.02 Pa), and 10 min gel strength of 16 lb/100ft² (7.66 Pa) This showed that *Mucuna solannie* is a better alternative cement extender than Bentonite, especially where optimum free water and compressive strength are needed. Although it is found to be lacking in efficient plastic viscosity and thickening time, it can be resolved by the use of additives such as dispersant and accelerator to complement its properties.

Keywords Bentonite · Cement slurry extender · *Mucuna solannie* · Compressive strength · Free water · Rheological properties · Thickening time

Abbreviations

API American Petroleum Institute  
BHCT Bottom hole circulating temperature  
BHST Bottom hole static temperature  
BVOB By volume of blend  
BWOC By weight of cement  
HPHT High pressure high temperature  
OWC Oil well cement  
RPM Revolutions per minute

Units/Conversion

| Unit |
|------|
| 10 s gel strength |
| 10 min gel strength |
| Plastic viscosity |
| Yield point |
| 1 lb/100ft² |
| 1 Pa s |
| °C |
| 5 (°F − 32) |
| 1000 cP |

Introduction

Drilling and completion operations cannot be complete without cementing especially in the unconsolidated or stubborn formations. Well cementing occurs when either or combination of oil well slurry type like neat slurry, uniform cement slurry, or combination of tail slurry and lead slurry (two-stage cementation) is placed in the annulus between the well casing and the geological formations surrounding the wellbore to provide zonal isolation of the formation fluids in the well (Oriji and Dulu 2014). It has been used as the primary sealant in oil and gas wells throughout the world and is manufactured to meet specific chemical and physical standards set up by the API (Arin and Irawan 2003; Salam et al. 2013). In most cases, additives must be added to cement slurries to achieve desirable cement slurry properties. There are many cementing additives depending on the objectives of the cementer. Notable additives used in oil and gas industry include: accelerators: to increase the hydration
process and the development of strength, retarders: to slow the hydration process to keep the cement slurry workable for a definite period of time. Also, since some formations might not support long columns of cement slurries due to low fracture gradient, extenders, which allow addition of more water and lighten the slurry, are used to reduce the slurry density which is a function of hydrostatic pressure. They are also used to increase the yield of cement slurry. Economics is also impacted positively since extenders reduce the amount of cement required, bringing considerable savings. Microspheres, foamed cement, sodium silicate, pozzolans, solid hydrocarbon, diatomaceous earth, and the most common bentonite are examples of materials used as extenders (Shuker et al. 2014). The bentonite could be blended before mixing or pre-hydrated in mix water. The disadvantage of bentonite is reduction of cement strength and thickening time if added in high concentration. Also, at temperature above 230 °F, there is strength retrogression. Others are anti-foam and the dispersants to remove foam and to reduce viscosity of cement slurry, respectively. These additives have proven to be very effective and provide the necessary mechanisms to control adverse and negative behaviors of the cement slurries, especially at the reservoir zone. Extenders are mostly applied for cement lead slurry design, particularly during the cementing of intermediate casing that has a long column of formation that requires longer thickening time and lower slurry density. Several studies on evaluation of certain factors that affects the physical and chemical properties of cement slurry when pumped-in-hole have been made. An evaluation on thickening time, that is, the time after initial mixing to the time immediately cement slurry is pumped behind casing (when the cement slurry can no longer be pumped) is common. This is very crucial in the prediction and effectiveness of a cementing operation to obtain good cement bond behind casing. Billingham et al. (2005) and Salam et al. (2013) reinstated that if the thickening time is too short, the cement fails to reach its required placement, while if it is also too long, a thickening time leads to costly delay on wait-on-cement. An increase in temperature, pressure, or free water will each reduce the thickening time. These conditions will be simulated when the cement slurry is being formulated and tested in the laboratory before the operation is performed (Heriot Watt 2005). Operational problems as a result of short thickening time are dramatic, because cement can set prematurely in the casing or pumping equipment (Coveney et al. 1996; Salam et al. 2013).

It is also found that sugar acts as a retarder of cement slurries when added in small concentration and as an accelerator when added in high concentration ( Bermudez 2007). Generally, Mucuna solannie “Ukpo” contains 20.0–25.4% crude protein, 43.0–49.0% carbohydrate, 5.05–7.0% fat, 25.0–27.4% crude fiber, and about 6.4–14% moisture (Enwere 1998). Mucuna solannie which has a high content of carbohydrate is a tropical leguminous crop belonging to the sub-family, Papilionaceae, of flowering plants. They are mostly climbing wooden vines that twine through the rain forest trees like botanical boa constrictors (Ukachukwu and Obioha 2000; Onyemah 2017). Their seed pods are covered with dense whiskers—like hairs, which can be very painful when come in contact with the skin. This serves as a protection from predators (Ukachukwu and Obioha 2000; Onyemah 2017). This is known to originate from Asia and was later introduced into the Western hemisphere via Mauritius (Nkpa 2004). It has a specific gravity between 0.5 and 1.0 compared with that of Bentonite extender of 1.14.

Uwaezuoke et al. (2017) and Igwilo et al. (2017) studied the effects of temperature on mud based mud properties like Mucuna solannie mud and Detarium microcarpum, Brachystegia eurycoma, and Pleurotus mud. They discovered that rheological properties decrease with increase in temperature, while fluid loss increases with increase in temperature. Both drilling mud and cement slurry are all pseudo-plastic fluids: They have similar behavior in terms of rheological properties and fluid loss (free water) properties. Around 212 °F (100 °C), the cement paste begins to hydrate (loses chemically combined water of hydration), which gradually weakens the paste and paste-aggregate bond. As cement sets, it undergoes a transition from liquid to solid state. The volume occupied by set cement is a smaller volume than the liquid slurry. This shrinkage can lead to a reduction in the cement pressure which if lower than the formation fluid pressure can lead to gas migration into cement sheet. Cement slurry is usually designed to achieve overpressure compared to the formation pore pressure. Conversely, too much overpressure can lead to loss of cement mix water into permeable rock layers. Also, cement shrinkage leads to volumetric reduction and can consequently lead to de-bonding between cement and casing or the formation. The shrinkage can also result in tensile cracks and consequently increased permeability to provide a migration path for fluids. Additionally, circumferential fractures may be created because of cement shrinkage leading to gas accumulation after the cement has set.

Ki-Bong and Takafumi (2017) conducted experiments under typical summer and winter weather conditions, and temperature histories at different locations in the walls were recorded, and the strength developments of concrete at those locations were measured. The main factors investigated that influence the strength developments of the obtained samples were the bound water contents, the hydration products and the pore structure.

Shahriar (2011) investigated the mechanisms underlying the effects of chemical admixtures on the rheology of OWC slurry at different temperatures using an advanced shear-stress/shear-strain-controlled rheometer. It was found that the rheological properties of OWC slurries are highly dependent on the temperature, water/cement ratio and the
admixture used. Rheological behavior of cement is important for the drilling process: it will be optimum to predict correctly about slurry placement. Oil well slurries depend on its homogeneity of additive concentrations, quality and quantity to contribute the placement and success of a well drilling cementing operation (Memon et al. 2013).

The aim of this study is to evaluate the application of *Mucuna solannie* in cement slurry formulation as an extender because of its special properties to improve compressive strength, rheological properties, thickening time and free water of the cement. The main motivations to carry out this study are grouped into three. These include the fact that:

1. *Mucuna solannie* is available and contains high soluble fiber and carbohydrate which would most likely result in improved value of free water, yield good compressive strength and good retarding characteristic.
2. It is environmentally friendly.
3. It will improve the durability of cement sheet, especially at high temperature, since its application in drilling fluids resulted in trends in consonance with those observed when temperature effects on drilling fluids were carried out, signifying temperature stability (Uwaezuoke et al. 2017).

The cost comparison of *Mucuna solannie* with bentonite, both in their market values and their applications, has been presented in a study (Igwilo et al. 2020).

**Materials and methodology**

This section is divided into eight parts, namely: (a) collection of the *Mucuna solannie*, (b) physical pre-treatment of the *Mucuna solannie*, (c) cement slurry formulation, (d) conditioning of cement slurry, (e) cement slurry rheological measurements, (f) free water measurement, (g) thickening time measurement, and (h) curing of cement slurry and compressive strength measurement. These measurements were carried out in accordance with API specifications (API RP 10B-2 2013). The bentonite used for the test was pre-hydrated for about 30 min in the mix water. The mixing container with the pre-hydrated bentonite was placed in the mixer base and stirred at 4000 RPM ± 200 RPM prior to adding cement at a uniform rate which lasted for 15 s. The mixing container was covered and mixing continued at 12000 RPM ± 500 RPM for 35 s ± 1 s. Also, for slurry with *Mucuna solannie*, it was added with the cement, and stirred at the recommended RPM using the same procedure. It dissolves and mixes easily in water just like cement.

**List of apparatus/materials**

Apparatuses and materials used were Electronic weighing device, Variable speed mixer, Atmospheric pressure consistometer, HPHT consistometer, mold, Variable speed viscometer, Measuring cylinders, Spatula, Stop watch, Bentonite, *Mucuna solannie* powder, Dyckerhoff G cement, Defoamer, Fresh water.

The equipment and materials used for these experimental studies were all sourced from the drilling fluid and cementing technology laboratory of Weafri Well Services Company, Delta State of Nigeria.

**Collection of Mucuna solannie**

The *Mucuna solannie* utilized for this study were obtained from Onitsha International market, Anambra state, Nigeria.

**Processing of Mucuna solannie**

The sorted seeds were cracked open with a sledge hammer to allow for water to seep into the seed as shown in Fig. 1. The water helps in softening the seed and allow for easy removal from the seed coat. The seeds were then boiled, allowed to cool, and thereafter dried in a moisture extractor oven at 149 °F (65 °C). The dried seeds were afterward pulverized into flour. The flour was screened through a standard test BS sieve (0.63 mm) to obtain flour particle size as shown with other samples in Fig. 2.
Atmospheric pressure conditioning

The procedure is limited to a maximum temperature of 190 °F (87.8 °C). If the boiling point of water at the test location is less than 212 °F (100 °C), the temperature is adjusted accordingly. The procedure for conditioning is stated, thus:

Within 1 min after mixing, the slurry container of the atmospheric pressure consistometer was filled to the marked line. The test fluid is heated to ambient temperature to the mean temperature. The slurry container preheated to the decisive temperature is placed in the atmospheric consistometer cell. After the slurry reached test temperature, the test temperature was held for ± 30 min to allow the test fluid temperature to reach equilibrium. The paddle was removed and the test was stirred briskly with a spatula to ensure consistency.

Cement slurry properties measurement

Compressive strength

Two-inch cube molds were used. The molds and plate contact surfaces were cleaned and dried and lightly coated with release agents. After 5 min of the last slurry mixing, the molds used to hold the cement slurry were filled and covered with the top plate. The cement mould was placed in a curing vessel at the desired test temperature 80 ± 5 °F (26.7 ± 2.8 °C). The setup was heated and pressured according to the test schedule.

Table 1 Cement slurry design parameters using Bentonite and Mucuna solannie extenders at 150 and 200 °F BHCT

| Parameter                        | Value                        |
|---------------------------------|------------------------------|
| Vertical depth (TVD)            | 13,730 ft (4184.9 m)         |
| Temperature gradient (TG)      | 1.1 °F/100 ft (1.1 °F/30 m)  |
| BHST                            | 230 °F (110 °C)              |
| BHCT                            | 150 °F (65.6 °C)             |
| Ambient temperature             | 80 °F (26.7 °C)              |
| Initial pressure                | 1029 psi (7.0947E+6 Pa)      |
| BHP (final pressure)            | 9371 psi (6.461E+7 Pa)       |
| Ramp time                       | 48.19 min                    |
| Ramp rate                       | 2.12 °F/min                  |
| API schedule                    | 9.20 M                       |
| Consistometer                   | PCON-004                     |

Table 2 Bentonite and Mucuna solannie extenders slurry compositions

| Slurry name | Bentonite                     | Mucuna solannie               |
|-------------|-------------------------------|--------------------------------|
|             | Lead slurry                   | Lead slurry                   |
| Cement sack weight | 94 lb/sk (42.6 kg/sk) | 94 lb/sk (42.6 kg/sk) |
| Slurry weight | 12 lb/gal (1440.6 kg/m³) | 12 lb/gal (1440.6 kg/m³) |
| Slurry yield | 2.397 ft³/sk (0.06788 m³/sk) | 2.357 ft³/sk (0.06674 m³/sk) |
| Cement type | Conventional cement           | Conventional cement           |
| Mix water type | Fresh water                  | Fresh water                  |
| Mix water required | 14.175 gal/sk (0.053658 m³/sk) | 13.741 gal/sk (0.052015 m³/sk) |
| Mix fluid required | 14.334 gal/sk (0.054260 m³/sk) | 14.033 gal/sk (0.053121 m³/sk) |
| Slurry composition | Dyckerhoff G cement + 0.0313 gal/sk (0.00011848 m³/sk) ASP-742 + 3% BWOC Bentonite | Dyckerhoff G cement + 0.0313 gal/sk (0.00011848 m³/sk) ASP-742 + 3% BWOC Mucuna solannie |
Curing time

The curing time began with the recording of the transit time and the application of temperature and pressure and continued until the test was terminated. Recording of the transit time data was done within 5 min after the application of temperature and pressure.

Thickening time

The slurry consistency at which the thickening time test was terminated was documented and reported. The procedure employed to estimate the thickening time is stated, thus:

The filled slurry container was placed on the drive table in the pressure vessel. The slurry container was rotated and the potentiometer was secured and monitored to measure the consistency and engage the paddle shaft drive bar. The vessel was filled with oil. The paddle shaft was not rotating at this point. The head assembly was secured in the pressure vessel, the thermocouple was inserted through its fitting and the threads were partially engaged. After the pressure vessel was completely filled with oil, the threads of the thermocouple were tightened. The thickening time test was initiated by applying the initial pressure and starting the temperature ramp. The results of the thickening time test for the two slurry samples, formulated sample with 3% BWOC Mucuna solannie and formulated sample with 3% BWOC Bentonite as control, at 150 °F (65.6 °C) and 200 °F (93.3 °C) are shown in Figs. 3, 4, 5 and 6.

Rheological properties

The rotor, bob, and cup was preheated to the test temperature. The cement slurries were prepared and conditioned as stated in required schedule. The rotor, bob, and cup were dried immediately prior to the test and all reassembled on the variable speed viscometer. The conditioned test slurry was poured into the preheated viscometer cup to a level adequate to raise the fluid to the scribed mark on the rotor without the rotor or bob touching the bottom of the cup. The initial dial reading was taken after 10 s of continuous rotation at 3 RPM. The remaining dial readings at other speeds were taken: first at ascending order and then in descending order. The highest speed used was 300 RPM. The ratio of the dial readings was also taken during ramp up to ramp down at each speed. The ratio was used to qualify the fluid properties.

Free water test

The slurry was stirred to achieve consistency. The slurry samples were conditioned for 20 min in the atmospheric consistometer. The conditioned cement slurry was poured into a 250 ml measuring cylinder and allowed to stand for 2 h. The free water deposited at the top of the slurry was measured after the 2 h stand time had elapsed. The free water was decanted to separate it from the slurry and the volume was measured and recorded.

Results and discussion

The direct results obtained from cement slurries measurements using Mucuna solannie and Bentonite extenders, tested at 150 °F (65.6 °C) and 200 °F (93.3 °C) BHCT with the required equipment and apparatuses are shown as: thickening time charts (Figs. 3, 4, 5 and 6), compressive strength chart (Figs. 8, 9, 10 and 11), rheological and the free water data shown in Table 4. The analyses of the cement properties were then presented in Figs. 7, 12, 13, 14, 15 and 16.

The thickening time evaluated as presented in Fig. 7 showed that Mucuna solannie has a slightly irregular and inefficient thickening time as compared with that of

| Additives | Function          | Concentration              | Weight (g) | Volume (ml or cm³) |
|-----------|-------------------|---------------------------|------------|--------------------|
|           | Bentonite/Mucuna  |                           | Bentonite  | Mucuna solannie    |
|           | solannie extender |                           | Bentonite  | Mucuna solannie    |
| Dyckerhoff G cement | Cement          | 100% BVOB                | 377.66     | 384.11             |
| ASP-742   | Defoamer          | 0.0313 gal/sk (0.00011848 m³/sk) | 0.95       | 0.97               |
| Bentonite | Extender          | 3%BWOC                   | 11.33      | 11.52              |
| Mucuna solannie | Mix water       | 14.175 gal/sk (0.053658 m³/sk) | 474.40     | 467.74             |
| Fresh water | Mix water       | 14.334 gal/sk (0.054260 m³/sk) | 486.35     | 480.23             |
Bentonite. It took Bentonite slurry formulations 2 h 30 min and 1 h 44 min to achieve a Bearden consistency of 40 at 150 °F (65.6 °C) and 200 °F (93.3 °C), while it took Mucuna solannie 10 h 1 min and 9 h 19 min to achieve a Bearden consistency of 40 at the same BHCT. The excessive thickening time of the slurry designed with Mucuna solannie is because of its high rheological properties values and its retarding property. In this case, dispersant is needed to improve the flow behavior index so that the slurry can easily be pumped during field application. Thinning will viscosity and adding accelerator might shorten the thickening time based on the volume of cement slurry to be pumped. This
would help the cement slurry to solidify at the requested annular space between the casing and the hole. Also, the thickening time of the Bentonite slurry sample can be prolonged by including the retarding additive into the formulation. It depends on the slurry volume to be placed in the annulus. Having enough thickening time is very imperative when designing the lead slurry because of high volume of cement slurry to be pumped.

The compressive strength of cement sheet result shown on Figs. 12 and 13, prepared with both *Mucuna solannie* and Bentonite extenders were measured at 12, 18 and 24 h under 150 °F (65.6 °C) and 200 °F (93.3 °C) BHCT respectively. Set cement with *Mucuna solannie* extender developed higher compressive strength at 18 and 24 h, but lower value at 12 h wait-on-cement. The compressive strength developed

![Fig. 5 Thickening time chart for Bentonite at 150 °F (65.6 °C)](image1)

![Fig. 6 Thickening time chart for Bentonite at 200 °F (93.3 °C)](image2)

![Fig. 7 Cement slurry thickening time for Mucuna solannie and Bentonite extenders](image3)
by cement prepared with *Mucuna solannie* was better than that of the Bentonite, except at 12 h, because of its retarding property. The effective free water control property is due to high crude fiber content of *Mucuna solannie* that gave rise to zero free water, is one main reason of having a good compressive strength after cementing. Similarly, from Fig. 14, it is deduced that the compressive strength of cement with bentonite reduced with increased BHCT, while temperature did not appreciably alter the compressive strength of the *Mucuna solannie* cement formulation. *Mucuna solannie* formulation exhibited higher stability compared with that of bentonite.

The main reason for determination of rheological properties is to predict plastic viscosity, gel strength and yield
point value of the cement samples in order to qualitatively evaluate the suitability of *Mucuna solannie* for cementing operations in this case. From Fig. 15, it can be seen that cement formulated with bentonite has lower plastic viscosity and also yield point values compared to that of the slurry formulated with *Mucuna solannie*. Cement formulated with *Mucuna solannie* recorded a high plastic viscosity of 89 cP ($8.9 \times 10^{-2}$ Pa s) as compared to 11 cP ($1.10 \times 10^{-2}$ Pa s) for that of Bentonite, showing its high resistance to flow, although it has a better transport capacity, if optimum flow behavior index can be established. It will require a significant amount of thinner to work effectively, even though Dyckerhoff cement, which was the base cement used are usually designed for high temperature/high pressure wells.

It was also observed that the gel strength was progressive for the *Mucuna solannie* sample when compared with cement sample formulated with Bentonite. This indicates that the gelation of the cement is operating with a slightly steady strength with time for both samples, although that of the Bentonite sample is better. However, both extenders are
suitable for cementing operations due to the appreciable gel strength values obtained.

The free water measurements were carried out for 2 h in accordance with API specifications. The result from Fig. 16 depicts that the free water volume collected from the Bentonite cement sample are 4.4 and 4.8 ml at 150 °F (65.6 °C) and 200 °F (93.3 °C) BHCT, while no free water loss was recorded for that of the Mucuna solannie cement sample. This could be attributed to the fact that the water may not have been completely integrated or hydrated in the Bentonite cement sample, thereby forming unstable cement slurry. Formation of free water on top of the cement slurry typically is due to the non-uniform dispersion of cement particles. Higher free water volumes indicate that the cement slurry failed to achieve stable hydration and consistency. Both formulations showed increase in free water with increase in temperature. This is because higher temperature makes reactions faster, leaving extra water to settle out as free water. Therefore, Mucuna solannie cement sample is considered to be a better alternative than the Bentonite cement sample by achieving stronger water integration within all components mixed. It is imperative to note that free water problems would not be encountered if the cement design formulated with Mucuna solannie is used for cementing operations as the water, Mucuna solannie and cement properly integrate and achieve the desired consistency required. A good free water value gives good compressive strength for set cement.

**Conclusion**

This study through the experimental measurements of cement slurry using Mucuna solannie provided information about the use of Mucuna solannie as an extender. This implies that Mucuna solannie cement slurry formulation

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**Table 4** Slurry rheological properties of *Mucuna solannie* extender and Bentonite extender at 150 °F (65.6 °C) and 200 °F (93.3 °C) BHCT

| Rotational speed (RPM) | Mucuna solannie and Bentonite for 150 °F (65.6 °C) BHCT | Mucuna solannie and Bentonite for 200 °F (93.3 °C) BHCT |
|-----------------------|--------------------------------------------------------|------------------------------------------------------|
|                       | Dial readings (Mucuna solannie) | Dial readings (Bentonite) |
|                       | Dial readings (Mucuna solannie) | Dial readings (Bentonite) |
| 3                     | 14                           | 10                        | 18                        | 8                        |
| 6                     | 22                           | 12                        | 24                        | 9                        |
| 30                    | 55                           | 52                        | 56                        | 39                       |
| 60                    | 77                           | 56                        | 81                        | 45                       |
| 100                   | 92                           | 59                        | 104                       | 49                       |
| 200                   | 131                          | 64                        | 147                       | 53                       |
| 300                   | 156                          | 66                        | 180                       | 54                       |
| 10 s gel at 77.6 °F (25.3 °C) | 13                       | 13                        | 15                        | 10                       |
| 10 min. gel at 190 °F (87.8 °C) | 16                       | 16                        | 22                        | 16                       |
| Free water (ml)        | 0                            | 4.4                       | 0.2                       | 4.8                      |
is a good choice as cement extender, provided dispersant and accelerator are inclusive in the design, to reduce its excessive rheological properties and improve its poor thickening time to be suitable for oil and gas cementing operations. Based on this study, the conclusions are that *Mucuna solannie* is a promising cement slurry extender especially where a high compressive strength is needed. Similarly, *Mucuna solannie* has a good thermal stability in high temperature conditions considered. Performance of *Mucuna solannie* showed stability of compressive strength at increased temperature compared with bentonite formulation. Hence, stability of *Mucuna solannie* has once again been highlighted by the trend observed in the curves of compressive strength variation with time. Also, it gave very minimal free water, even at low concentration. However, it found lacking in effective rheological properties.
and thickening time. These can be resolved by the use of dispersant and accelerator to complement these properties.

**Compliance with ethical standards**

**Conflict of interest** No conflict of interest has been declared by the authors.

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