CERTIFICATION OF APPROVAL

TESTING COCONUT SHELLS, SEASHELLS AND SARAWAK COAL AS CEMENT ADDITIVES FOR HPHT WELLS CEMENTING.

by

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CERTIFICATION

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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(YUNUS TAGAYEV)

ABSTRACT

Coconut shells, seashells and Sarawak coal (research materials) are tested as cement additives for high pressure and high temperature (HPHT) wells. For the cement design in HPHT conditions, pozzolanic additives such as fly ash and silica fume are used to enhance the compressive strength and the durability of cement sheath. Due to the increased demand by the construction industry, the cost of silica fume and fly ash are very high and the supply is not constant (Lake, 2006). Moreover, those supplementary additives are known as hazardous materials.

The scope of this project is to obtain powder from the research materials, blend them in the cement without any other additives and test three cement slurry parameters: compressive strength, fluid loss and rheology. Cement slurry is prepared and cured at 250F temperature and 3,000 psig pressure in HPHT cement curing chamber. Compressive strengths are measured at one and seven days' time. Slurry rheology and fluid loss were as well measured following API RP 10B specifications.

Results show that cement composition with seashell content has compressive strength values which are higher than the field requirements. 30% BWOC seashell has the compressive strength value of above 5,000 psi. Even though coal and coconut shell additives did not show good compressive strength results in high concentrations, they showed good rheology and fluid loss behaviors. API fluid loss of the cement slurry reduced with the increasing concentrations of coal and coconut shells to as low as 100 cc/min.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

The use of coconut shells, seashells and Sarawak coal as cement additives is a novel project for oil and gas industry. There are some studies done on the effects of coconut shells in concrete for civil constructions. However, seashells and coal are first to be tested with Portland cement for HPHT well conditions. These research materials exist in abundant quantity and low cost in Malaysia.

Current technologies allow the production of reservoirs that were once considered too expensive and risky to be commercially viable. These wells should be designed to withstand high temperatures and pressures, as well as frequently encountered corrosive gases such as H2S and CO2. Completions performed in HPHT reservoirs are some of the most expensive in the industry. Therefore, it is necessary to successfully cement the well casing on the primary cementing job and eliminate the need for remedial cementing. HPHT reservoirs are characterized by reservoir depths greater than 15,000 ft, reservoir pressure greater than 15,000 psi, and reservoir-fluid temperatures from 300 to 500°F. (Lake, 2006)

Laboratory tests conducted are to measure compressive strength and CO2 treatment effects of the Portland Class G cement with various proportions of new supplementary additives. Expected outcomes are similar to those supplements that are currently used in the industry.

Cementing, being the crucial stage of well construction, becomes much more challenging in HPHT wells especially in the presence of CO_2. Thickening time must be sufficient to displace the slurry long distance through the casing into the annulus. BHST and BHCT should be used in the testing of the thickening time at the cement lab.

This research paper encompasses studies and experimental analysis on cement strength and durability improvement in HPHT and CO_2 wells. High pressure and high temperature conditions are defined as the formations that either have pore pressures greater than 10,000 psi or temperature above 150° C. Elevated temperatures tend to degrade long term mechanical integrity of cement sheath. A latter phenomenon becomes more serious with the presence of CO_2 gases in wellbore area. Addition of pozzolanic supplement to a popular Portland cement of class G will enhance the compressive strength and CO_2 resistance of the set cement.

A pozzolan is a siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value, but which will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties. Fly ash and silica fume are the most common pozzolanic supplements used with Portland cement nowadays. The latter mentioned additives are by-products of heavy industrial plants. Moreover, there are potential sources of silica supplements from different types of waste products which are studied in this research. Potential materials being studied are coconut shells, seashells and Sarawak coal ash. The main job scope of this project is the investigation and preparation of pozzolanic supplements from those materials and experimentally testing until the set objectives are met are the main job scope of this project. In this final year project paper, optimized cement design for HPHT vertical wells with moderate CO_2 content is to be determined.

1.2 PROBLEM STATEMENT

Integrity of cement sheath in HPHT wells has been a concern for decades and many solutions for this problem were suggested. In HPHT conditions every small detail can cause huge consequences if not taken into deep consideration. CO_2 is of great concern when it comes to formations with high temperatures which accelerations chemical reactions with the set cement. CO_2 will eventually partially dissolve cement sheath increasing its permeability which can lead to poor zonal isolation, casing corrosion, gas migration to surface and other severe consequences.

To mitigate the above stated problem, supplementary cement additives are used together with Portland cement to increase the hardened cement's compressive strength and decrease permeability. Moreover, the use of such pozzolanic supplements has other major benefits from environmental perspective. There are cases that reported the replacement of 70% by weight of Portland cement with fly ash showed successful results. Latter statement tells us that by using more pozzolanic supplements the production of cement in the cement plants will decrease which leads to the reduction of CO_2 emission into the atmosphere.

However, it should be noted that the commonly used fly ash and silica fume additives are highly toxic and can cause severe damage if inhaled. This project is to improve the cement design for HPHT wells with CO_2 gases by researching and testing other "green" and more economical alternative sources of pozzolanic supplements from waste materials to replace the toxic ones currently in use. Moreover, abundance of alternative additives will further reduce the cement production in the plants, consequently reducing carbon dioxide emissions into the atmosphere.

1.3 OBJECTIVES OF STUDY

1. Investigate on the physical & chemical properties of coconut shells, seashells and Sarawak coal
2. Conduct laboratory tests on the compressive strength, fluid loss, slurry rheology of a hardened Portland cement with different proportions of each pozzolanic sample obtained above (from coconut shells, seashells and Sarawak coal ash). Based on the results, suggest optimum composition for the cement design in HPHT and CO_2 wells and determine the pozzolanic sample that showed the best results.

1.4 SCOPE OF STUDY

This study is focused on new cement additives derived from waste materials like coconut shells, seashells and Sarawak coal ash for the use in the cement design for HPHT and CO_2 wells. Initial studies on cementing design for HPHT wells and cement testing procedures should be covered.

Properties and characteristics of the research materials should be studied. This part includes being familiarized with pozzolanic properties of materials and their availability in nature. The way of extraction of pozzolans from the waste materials should be investigated and determined. Study also covers API RP 10B testing procedures for cement testing to measure the required properties of cement.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 POZZOLANIC SUPPLEMENTS TO IMPROVE COMPRESSIVE STRENGTH OF CEMENT

M.R. Islam et al. showed improvements in oil-well cement properties by using silica fume and fly ash supplementary additives. Compressive strength and durability test results indicate major increase in compressive strength with an increase in silica fume content from 0-30% by weight in the blends. Silica fume is a pozzolanic material with extremely small particle size that creates blocking effect which can be used to cope with the gas migration problems. Fly ash is relatively cheaper material, makes the blend economical.

In order to optimize the amounts of silica fume and fly ash to obtain sufficient compressive strength, series of slurries replacing up to 50% of cement with fly ash and silica fume blends were prepared. Table below shows the samples to be tested in the experiment:

TABLE 1: MIX PROPORTIONS AND FREE SILICA

Sample NO.	G-Type cement	H-Type Cement	Cement	Silica-fume	Fly Ash	water reducer	Free Silica (aprox.)
1	G(C)	H(C)	100%	0%	0%	0%	<2%
2	G(1)		50%	0%	50%	1%	17-20%
3	G(2)		50%	5%	45%	1%	22.5%
4	G(3)		50%	10%	40%	1%	25%
5	G(4)	H(4)	50%	15%	35%	1%	27.5%
6	G(5)	H(5)	50%	20%	30%	1%	30%
7	G(6)	H(6)	50%	25%	25%	1.5%	32.5
8	G(7)	H(7)	50%	30%	20%	2%	35%
9	G(8)		50%	35%	15%	2%	37.5%
10	G(9)		50%	40%	10%	3%	40%
11	G(10)		50%	45%	5%	3.5%	42.5%
12	G(11)		50%	50%	0%	3.5%	45%

Table 1 shows mixed proportions of silica fume and fly ash with Class G Portland cement. Sample 1 is a neat Portland cement without any additives. Dispersant was added with an amount of increase in silica fume to get optimum slurry rheology. Equipments used and testing procedures were conducted in full compliance to API requirements.

Compressive strength of all samples was measured after 1 and 14 days to test the durability of the cement as well. Figure 1 and 2 shows compressive strength measurements after 1 and 14 days respectively.

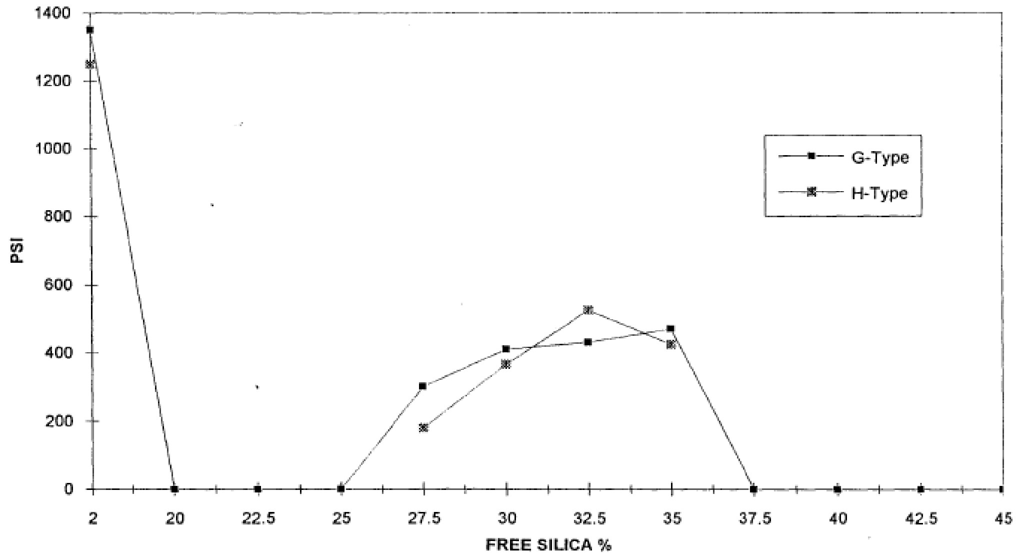


FIGURE 1: ONE-DAY COMPRESSIVE STRENGTH MEASUREMENT VS SILICA CONTENT

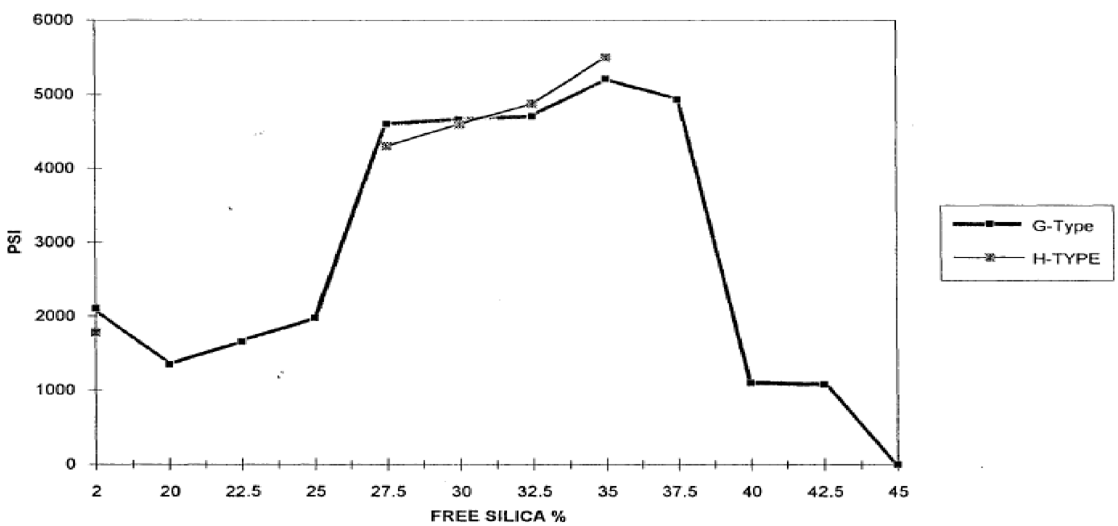


FIGURE 2: 14-DAY COMPRESSIVE STRENGTH MEASUREMENT VS SILICA CONTENT

This research done by M.R. Islam et al. shows that addition of silica to Class G Portland cement does improve the compressive strength of set cement. Measurements in the figures above show that Sample 1 which is neat cement achieves higher compressive strength in short time compared to the other mixed samples. However, 14-day compressive strength measurements show that the compressive strengths of samples having 30-35% silica BWOC are twice higher than that of Sample 1. Better corrosion resistance and less gas migration resulted in the use of silica fume and fly ash pozzolanic supplements (M.R Islam et al.).

2.2 IMPROVING CEMENT PROPERTIES FOR CO_2 ENVIRONMENT

An experiment done by Arina binti Sauki et al. investigates the effect of pressure and temperature on degradation of wellbore cement by CO_2.

Four major crystalline compounds in Portland cement are tricalcium silicate (Ca_3SiO_5), dicalcium silicate (Ca_2SiO_4), tricalcium aluminate ($Ca_3Al_2O_6$), and tetracalcium aluminoferrite ($Ca_4Al_2Fe_2O_{10}$). Silicates are the most plentiful phases in Portland cement comprising over 80 wt % of the cement, mostly in the form of tricalcium silicate. The compounds of Portland cement form C-S-H and $Ca(OH)_2$ hydration products upon mixing with water.

Portland cement tends to degrade once exposed to CO_2. The equations below shows the process of CO_2 attack on cement hydrate compounds:

CO_2 dissociation:

$$CO_2 + H_2O \rightarrow H_2CO_3 \qquad (1)$$

Cement Carbonation:

$$H_2CO_3 + Ca(OH)_2 \rightarrow CaCO_3 \qquad (2)$$

$$C\text{-}S\text{-}H + H_2CO_3 \rightarrow CaCO_3 + \text{amorphous silica} \qquad (3)$$

Calcium Carbonate dissolution:

$$CaCO_3 + H_2CO_3 \rightarrow Ca(HCO_3)_2 \qquad (4)$$

$$Ca(HCO_3)_2 + Ca(OH)_2 \rightarrow 2CaCO_3 + H_2O \qquad (5)$$

In Equation (1) carbonic acid is formed upon the dissolution of CO_2 in water. Carbonation of the cement takes place when the acid reacts with the $Ca(OH)_2$ in the cement as well as the C-S-H gels to form $CaCO_3$ in Equation (2) and (3). The latter reactions are fast and results in the densification of set cement as well as reduced permeability. Micro cracks can be developed due to an additional mass of $CaCO_3$ formed. However, the $CaCO_3$ can continue to react with fresh carbonic acid in Equation (4) which may lead to dissolution of $CaCO_3$. This reaction is a long term phenomenon and only happens when cement is surrounded by liquid water containing CO_2. As a result $CaCO_3$ is converted to water soluble calcium bicarbonate that will then coupled with the formation of water in Equation (5) to produce $CaCO_3$ and water.

As a result, the compressive strength of the set cement decreases and the permeability increases, leading to the loss of zonal isolation (Arina binti Sauki et al.).

Santra et al. proves that the depth of carbonation is directly proportional to the amount of pozzolanic supplement added to the cement. He conducts a research on the CO_2 reactivity with Portland cement following API RP 10B procedures. The cement samples with different proportions of pozzolanic supplements were cured for 15 days at 200 F and water pressure of 2000 psi. After that, the samples were put in CO_2 treatment test cells having 2000 psi of partial CO_2 pressure. The effects of CO_2 exposure on the samples were investigated after 15 and 90 days. The samples were immersed in fresh water during CO_2 treatment. Compositions of the test samples are shown in the table below:

TABLE 2: SLURRY COMPOSITIONS

Sample, No.	Cement, %	Silica Fume, %	Fly Ash, %	Ca/Si Ratio	Water/Solid by Weight	Water/Solids by Volume
1	100	0	0	3.12	0.45	1.42
2	83.3	16.7	0	1.46	0.47	1.19
3	71.4	28.6	0	0.95	0.48	1.11
4	62.5	37.5	0	0.71	0.53	1.11
5	55.6	44.4	0	0.56	0.56	1.15
6	50	50	0	0.47	0.58	1.13
7	83.3	0	16.7	2.22	0.46	1.18
8	71.4	0	28.6	1.72	0.45	1.05
9	62.5	0	37.5	1.41	0.46	0.98
10	55.6	0	44.4	1.19	0.45	0.92

In this experiment too, silica fume and fly ash pozzolanic supplement were used. It is repeatedly shown that water/solid ratio increases with the increasing amounts of silica fume. However, no dispersants were used in this experiment compared to the one done by M.R Islam.

Phenolphthalein dye test was used to provide visual indication of the depth of carbon penetration in the samples. For the samples with low or no amount of $Ca(OH)_2$ which is a sample with high content of pozzolans, phenolphthalein dye test cannot show any results due to no color changes. There TGA and XRD analysis were used to determine depth of carbonation for Samples 5 and 6 which have highest content of pozzolans.

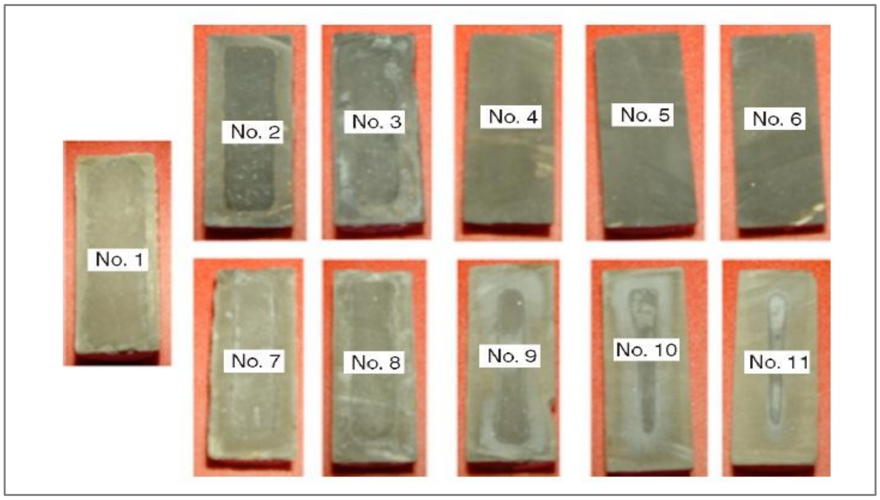


FIGURE 3: 15-DAY CO2 SAMPLES. PENETRATION DEPTH WAS MEASURED FROM THE DIFFERENT COLOR OF CARBONATED AND UNCARBONATED PORTIONS. FOR SAMPLES 5 AND 6 FULL-PENETRATION OF CO2 WAS CONFIRMED BY TGA AND XRD.

Figure 4 below shows that the depth of CO_2 penetration is increasing with an increasing amount of pozzolanic supplements.

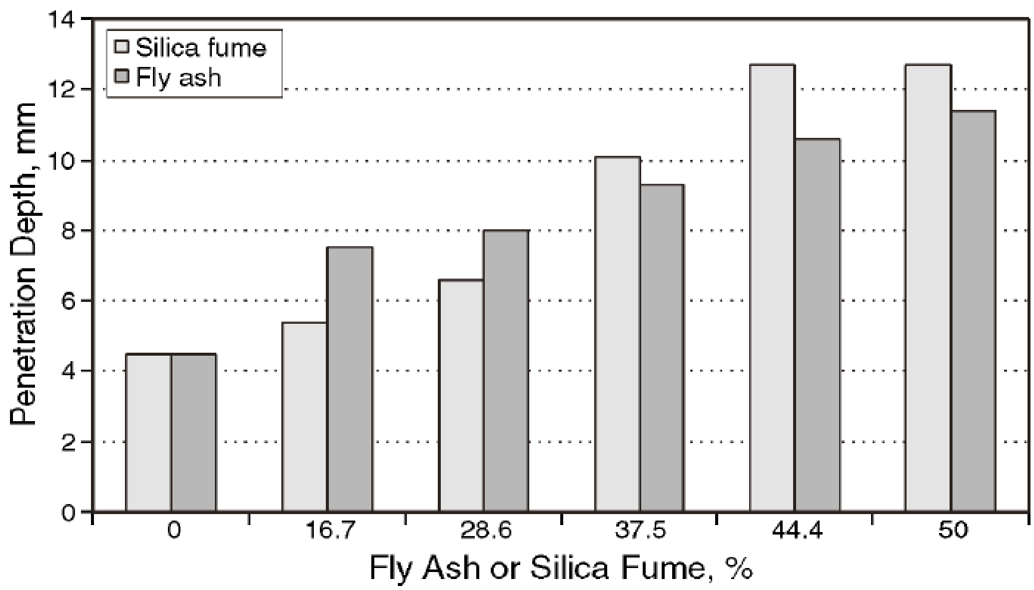


FIGURE 4: MEASURED PENETRATION DEPTH

Conclusion to be drawn from this research is that CO_2 penetration is dictated by two main factors: (a) degree of porosity/permeability (b) amount of $Ca(OH)_2$ present in the uncarbonated sample wherein the influences will be counteractive (Santra et al.). Given enough time, partial carbonation of Portland cement in down hole conditions is unavoidable. However, it should be noted that partial carbonation does not always lead to loss of overall mechanical integrity and loss of zonal isolation, as was confirmed by Carey et al. 2007.

The study suggests that, samples with higher pozzolanic content where CaCO3 formed is much lower after carbonation, will face less severe effects of dissolution simply because there is less material to dissolve, either by CO_2 or H_2CO_3.

SPE paper by N. Moroni, Eni E&P Division et al, discusses some holistic approach for cementing in CO2 environment. His research is very similar in objectives and procedures to the research work of Santra et al. Author improves the CO2 resistance of Portland cement by reducing the amount of cement hydration products which will react with CO2. Exact conclusions were drawn from the work of Santra et al. when they stated that due to the reaction between pozzolans and portlandite ($Ca(OH)_2$) there will be no or less material to be dissolved by CO2 or carbonic acid.

A.Brandi et al. experimentally compare the CO2 resistance of 35% silica flour BWOC and “pozzolan”. Pozzolan here referred to a specific marketing name given to the sample by the author. The difference in this research was the reduced water content of the cement slurry that enhanced its resistivity to CO2. The table below lists the slurry compositions of conventional and “pozzolan” cement design:

TABLE 3: CEMENT SYSTEM DESIGN, PROPERTIES, AND OXIDIC COMPOSITION

cement system	"pozzolan"	"conventional"
base blend	API class G + silica flour + pozzolan	API class G + 35% bwoc of silica flour
slurry density (lbs/gal)	15.0	15.0
water:solid (wt/wt)	0.55	0.72
portlandite (wt%)	not detected	not detected
Oxidic composition of set cement systems (calculated from EDS analyses)		
CaO (wt%)	46.6	47.5
SiO_2 (wt%)	40.8	39.8
C (wt%)	2.4	2.3
Na_2O (wt%)	0.6	0.6
MgO (wt%)	0.4	0.4
Al_2O_3 (wt%)	2.4	2.2
SO_3 (wt%)	3.1	2.8
Cl (wt%)	0.7	0.6
K_2O (wt%)	0.1	0.2
Fe_2O_3 (wt%)	2.9	3.4
total (wt%)	100.0	100.0

As it was mentioned before, water to solid ratio for "pozzolan" is much lower than for conventional design. Only this difference in the design of the cement slurry resulted in better performance of set cement in CO2 environment. The figure below shows the samples after CO2 treatment:

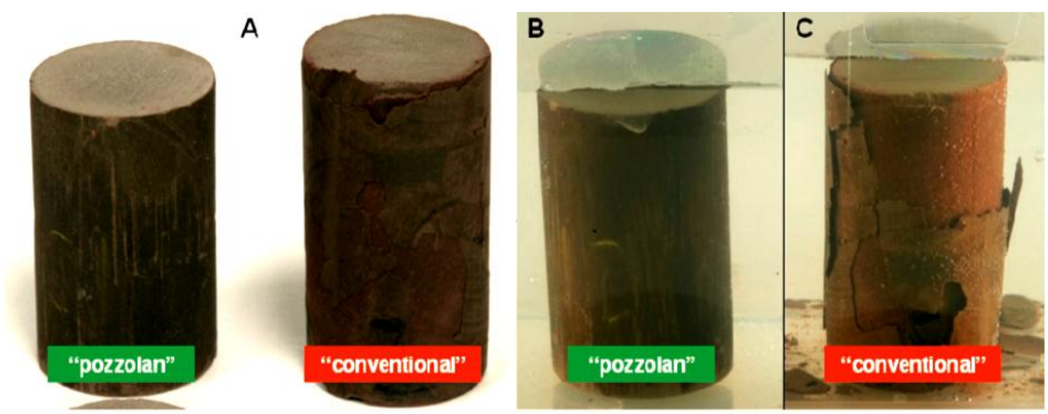
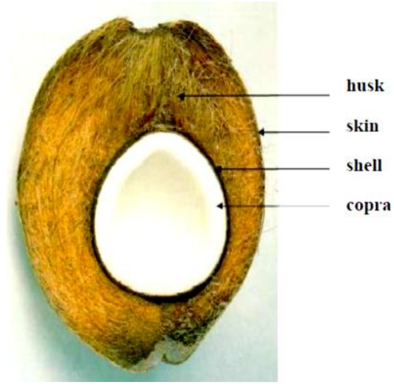


FIGURE 5: A) "POZZOLAN" (LEFT CYLINDER) AND "CONVENTIONAL" CEMENT SYSTEMS (RIGHT CYLINDER) AFTER RECOVERING FROM EXPOSURE TO CO2 LOADED WATER AT 300F AND 3,000 PSI FOR 6 MONTHS; SPECIMENS WERE TRIMMED AT THE BOTTOM AND TOP FOR TESTING WATER PERMEABILITY- ONLY TH

Two major conclusions for the design of cement for CO2 environments are: a) increase in pozzolanic supplements will decrease the amounts of CaCO3 produced after carbonation which is prone to dissolution by carbonic acid, b) reduction in the water to solid ratio of cement slurry will enhance the CO2 resistance of the set cement in downhole conditions.

2.3 PROPERTIES OF COCONUT SHELLS

Cocos Nucifera trees, otherwise known as coconut palm trees, grow abundantly along the coast line of countries within 15oof the equator. They prosper in sandy, saline soil and in tropical climates. A healthy coconut tree will produce approximately 120 watermelon-sized husks per year, each with a coconut imbedded inside. There are three constituents of the Cocos Nucifera that can be used for fuel: the husk, the coconut shell, and the coconut oil that is in the white coconut “meat” or copra as it is usually called. Thus, the coconut tree is a very abundant, renewable resource of energy. When coconuts are harvested, the husks are removed, thereby leaving the shell and the copra. Plate 1 shows the coconut with the husk being removed whereas plate 2 shows the different layers of the coconut fruit. These husks are considered as waste materials and are usually dumped into refuse bin. When consumers buy the coconut, they buy it with the shell and when it is to be consumed it is broken and the shell is removed. Large quantities of the shells can be obtained in places where coconut meat is used in food processing. The husk and the shell are both regarded as waste materials. These materials are then burnt into ashes in a furnace at a very high temperature to produce the coconut shell and husk ash. The coconut shell when dried contains cellulose, lignin, pentosans and ash in varying percentage. Table 4 shows the percentage composition of the shell whereas Table 5 shows the constituent in the coconut shell ash (Olugbenga O. Amu et al.).



A) B)

FIGURE 6: A) COCONUT WITH HUST BEING REMOVED; B) DIFFERENT PARTS OF THE COCONUT FRUIT

TABLE 4: COCONUT SHELL COMPOUND (DRY BASIS)

Compound	Percent
Cellulose	33.61
Lignin	36.51
Pentosans	29.27
Ash	0.61

TABLE 5: COCONUT SHELL ASH COMPOUND

Compound	Percent
K_2O	45.01
Na_2O	15.42
CaO	6.26
MgO	1.32
$Fe_2O_3 + Al_2O_3$	1.39
P_2O_5	4.64
SO_3	5.75
SiO_2	4.64

From the Tables 4 and 5 it can be seen that coconut shells possess very low percentage of ash. Coconut shell ash does not show a good property of pozzolan as its silica content is very low.

CHAPTER 3

METHODOLOGY

This project gives a lot more focus to the research stage than to experimental work. As soon as the initial analysis on choosing the best sources of pozzolanic supplement and their proper and economical extraction is complete, the laboratory testing is about following the standard procedures and obtaining the results. However, in order to obtain the results that will meet the objectives of the study, initial research should be reliable.

3.1 STUDY ON POTENTIAL WASTE SOURCES OF POZZOLANIC SUPPLEMENTS

Currently, coconut shells, seashells and Sarawak coal ash are being studied for the implementation as cement supplementary additives in HPHT and CO2 wells. This step includes a study about the physical and chemical properties of the materials, their abundance in nature. Until now, it was mentioned that coconut shells do not possess a good pozzolanic composition. However, it will be tested for further justification. Seashells are source of lime and major component for the production of Portland cement. Sarawak coal ash is the most promising source of pozzolan due to its popular co-by-product fly ash. Coal ash does have pozzolanic properties and will be further studied in this project.

3.2 EXPERIMENTAL WORKS

After the initial study and analysis on the pozzolan sources and their extraction methods, laboratory tests are to be conducted in compliance to API 10B standards. Three different pozzolans are to be mixed with Portland cement in various proportions. Water to solid ratio of the slurry is to be changed and tested accordingly. Compressive strength, rheology, pump ability, fluid loss of the new cement additives will be tested.

The flow chart in Figure 7 summarizes the project methodology

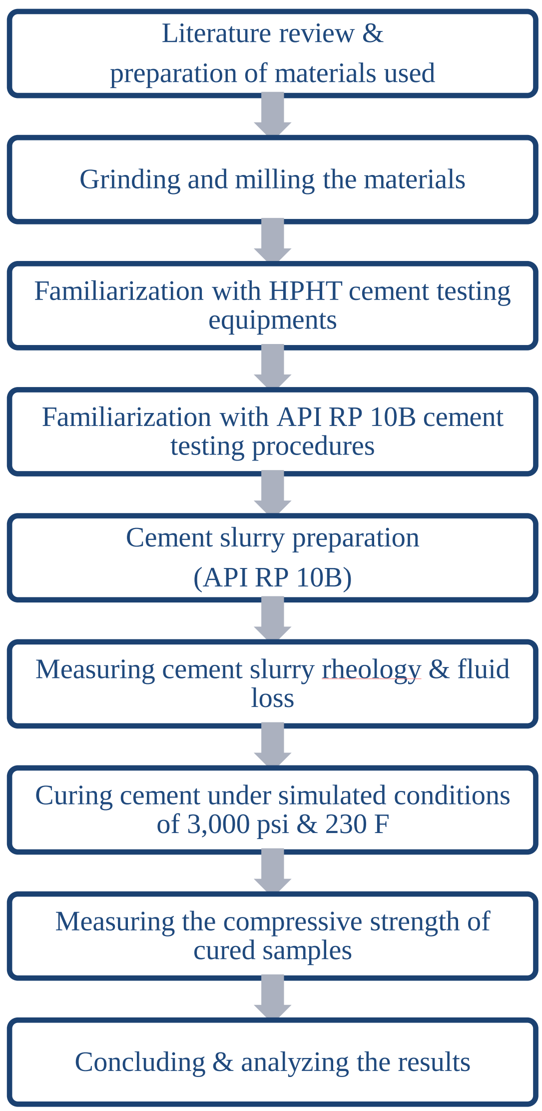


FIGURE 7: PROJECT METHODOLOGY

3.2.1 Grinding

Raw materials are grinded to small particle sizes to be mixed with cement. Small rock grinding machine is used to crush seashells and coal. Seashells formed flour like powder after grinding. For the coal, it was easily crushed and formed very light, fine dusty particles. Densities and particle sizes of these additives are to be determined.

3.2.2 Cement slurry preparation

Once the raw materials are grinded to the powder form, cement slurry can be prepared. Effects of these additives are going to be tested for compressive strength, fluid loss and slurry rheology. The prepared additives are to be added starting from 10% BWOC to 40% BWOC with 10% increments. The table below shows the cement slurry compositions:

TABLE 6: CEMENT SLURRY COMPOSITION

Sample No	Portland Cement	Seashell (%BWOC)	Sarawak Coal (%BWOC)	Coconut shell (% BWOC)	Water to solid ratio
1	API Class G	10	-	-	0.44
2	API Class G	20	-	-	0.44
3	API Class G	30	-	-	0.44
4	API Class G	40	-	-	0.44
5	API Class G	-	10	-	0.44
6	API Class G	-	20	-	0.44
7	API Class G	-	30	-	0.44
8	API Class G	-	40	-	0.44
9	API Class G	-	-	10	0.44
10	API Class G	-	-	20	0.44
11	API Class G	-	-	30	0.44
12	API Class G	-	-	40	0.44

Slurry Mixing

The cement slurry is prepared following the API RP 10 B specifications. The steps are as below:

- Slurry volume of approximately 600ml.
- Mix the water, cement and additives in API mixer at 4,000 rpm
- Shear at 12,000 rpm (35 sec)

The equipment used is constant speed mixer as shown in the figure below:

FIGURE 8:CONSTANT SPEED MIXER

3.2.3 CEMENT CURING

The cement is cured under simulated downhole conditions for 24 hours. HPHT curing chamber is used to cure the cement under 250 F temperature and 3000 psi oil pressure. The equipment used is shown in Figure 9:

FIGURE 9 : HPHT CEMENT CURING CHAMBER

This equipment has the capacity of curing 8 cement cubes at a time placed in 5x5x5cm moulds. Each sample should be 2 cured cement cubes for 2 and 7 days compressive strength testing. If 3 samples are cured at a time, it will take around 2 weeks for preparing 12 samples for testing.

3.2.4 Cement Testing Procedures

The objective of this project is to test the cement for fluid loss, rheology and compressive strength. The sequence of API governed test procedures for cement lab testing used by Baker Hughes is shown in Figure 10:

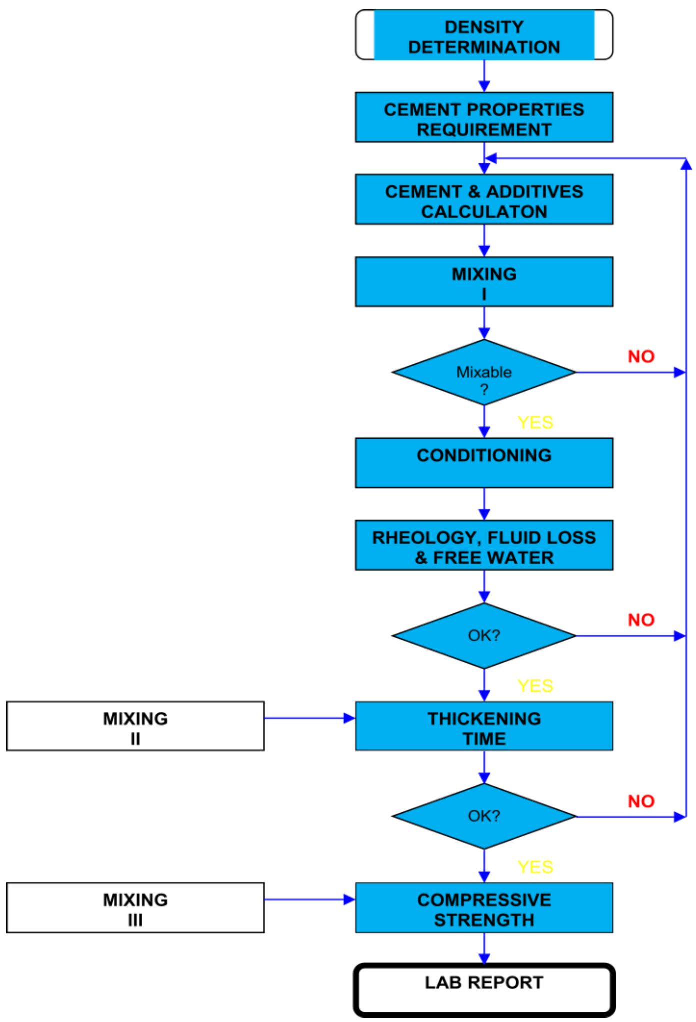


FIGURE 10: CEMENT TESTING FLOWCHART

Density determination and calculation of cement and additives are done using the excel spreadsheet provided by Baker Hughes. The slurry mixing procedure is already discussed earlier. So, the remaining three major cement tests namely fluid loss, rheology and compressive strength test procedure according to API RP 10 B is explained below.

3.2.5 COMPRESSIVE STRENGTH TESTS

12 samples of different cement compositions are to be cured under simulated downhole conditions using HPHT cement curing chamber. The cement is to be cured for 24 hours. Consecutively, the cement is to be tested for compressive strength after 1 and 7 days. The cubes are tested using Carver press as shown in the figure below:

FIGURE 11: CARVER PRESS COMPRESSIVE STRENGTH TESTER

The field requirement for the compressive strengths is show in the table below for different cases:

TABLE 7: COMPRESSIVE STRENGTH RANGES FOR DIFFERENT CASES

Function	Compressive Strength (psi)	Slurry Type/Operation
Axial Load Support	500 - 1000	Lead slurries
Drilling ahead	500 - 1000	Tail slurries
Perforating	1200 - 2000	Liner/Production casing slurries
Kick off plug (whipstock)	3000	Side track densitified slurries
Abandonment Plug	1000	Tagging/dressing off
Lost Circulation Plug	50	Thixotropic slurries

3.2.6 RHEOLOGY TESTS

Rheology is the study of flow and deformation of fluids. This parameter is measured using rotational viscometer as shown in Figure 12.

FIGURE 12: ROTATIONAL VISCOMETER

The experiment is conducted according to API RP 10 B procedures as stated below:

1. Prepare slurry as described in API slurry preparation (API specs 10B-2, sect 5).

2. Raise the table until the slurry has reached the line inscribed on the outside surface of the rotor. Lock the stage in position with the clamp screw. Switch the fann speed to **300 rpm**. Turn on the rotor and start the timer simultaneously.

3. When 60 seconds have elapsed, take a reading off the dial of the shear stress at 300 rpm.

4. After taking the 300 rpm reading, shift the instrument to the next lower speed of **200** rpm by moving and shifting the rod knob to the next lower range (knob fully extended) and motor speed toggle switch to the high position.

5. After 20 seconds at 200 rpm, take a dial reading.

6. After taking the 200 rpm shear stress reading (elapsed time of 80 seconds), shift the instrument to the next lower speed of **100 rpm** by moving the toggle switch to low speed setting.

7. After 20 seconds at 100 rpm, take a dial reading.

8. After taking the 100 rpm shear stress reading (elapsed time of 100 seconds), shift the instrument to the next lower speed of **6 rpm** by pushing the knob down to middle setting and toggle switch to high speed position.

9. After 20 seconds at 6 rpm, take a dial reading (elapsed time of 120 seconds), then switch the instrument low speed. (**3 rpm**)

10. After 20 seconds at 3 rpm, take a dial reading

3.2.7 FLUID LOSS TESTS

Fluid loss is the rate at which water comes out of cement when contacted with permeable formation. This property of cement is important to maintain the slurry pumpable and avoid dehydration.

FL test incorporates high pressure and specific filters to simulates wellbore pressure (the procedure govern by API 10B). 15.8 ppg slurry without FL additives has 1000 cc/30 min.

The testing procedures are as listed below:

1. Preheat the fluid loss cell to the test temperature.
2. Place the 325mesh screen and O-ring in the cell.
3. Pour in the precondition slurry and start the test as quickly as possible, but no more than 6 min shall elapse from the time of completion of condition to the start of the test.
4. Connect the nitrogen line and apply differential pressure of 1000psi ±50psi.
5. Open the top fluid loss cell valve to apply and maintain 1000psi differential pressure to the cell.

6. Open the bottom valve to start the test. Maintain at the specified temperature for the duration of the test.
7. Collect the filtrate and record the volume to an accuracy of ±1ml at 30s, 1min, 2min, 5min, 7.5min, 10min, 15min, 25min and 30min.
8. If nitrogen blows through at less than 30min, record the volume collected and time at which blowout occurs.
9. Calculate the ISO Fluid loss, expressed as milliliters per 30min.

Calculated ISO Fluid Loss = Vt(10.944)/Sqr(t)

Where

- Vt is the volume of filtrate collected at the time of the blowout.
- t is the time of the blowout, expressed in minutes.

The HPHT Fluid Loss Cell in Figure 13 is used to measure the fluid loss of the cement slurry.

FIGURE 13: STIRRED FLUID LOSS TESTER

CHAPTER 4

RESULTS & DISCUSSION

The planned project activities listed in the methodology section are successfully completed. Powders from coconut shells, seashells & Sarawak coal are prepared and used as cement additives. Different concentrations of these additives are tested for cement compressive strength, fluid loss and rheology. Following sections display and discuss the obtained results from laboratory tests of cement.

4.1 GRINDING & PULVERIZING THE MATERIALS

To blend the materials into cement, we need to turn it to powder first. Using pulverizer machine coconut shells were crushed into small particles. To crush seashells and coal grinding machine is used. After the materials are crushed, they we sieved through 120 mesh size screen to extract particle sizes larger than 125 microns. The latter is done to achieve good slurry mixing and workability. We can now proceed with testing the effects of these additives on the cement parameters stated in the project objective.

4.2 COMPRESSIVE STRENGTH RESULTS

HPHT cement curing chamber is used to prepare cement samples. Chamber was set for 24 hours curing time at 3,000 psi pressure and 250F temperature to simulate HPHT conditions. At temperatures higher than 230F cement sheath undergoes strength retrogression (PCSB SKG 10, Cementing). As this experiment was conducted at 250F temperature, degree of strength decline can be part of the discussion.

After 24 hours compressive strength of all 13 samples are tested. The remaining set of 13 samples is kept immersed in water (API guidelines) for a week until next compressive strength tests.

Table 1 shows the composition of the cement slurry for each sample. Basic composition is Portland API Class G cement, fresh water and the new additives. Water to solid ratio (by weight) was kept constant at 0.44.

TABLE 8: CONCENTRATION OF NEW ADDITIVES IN CEMENT SAMPLES

Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**
Additive	Seashell				Sarawak Coal				Coconut Shell				Neat Cement
Concentration (%BWOC)	10%	20%	30%	40%	10%	20%	30%	40%	10%	20%	30%	40%	

One-day and seven-day compressive strength results are shown in Figures 1 and 2 below.

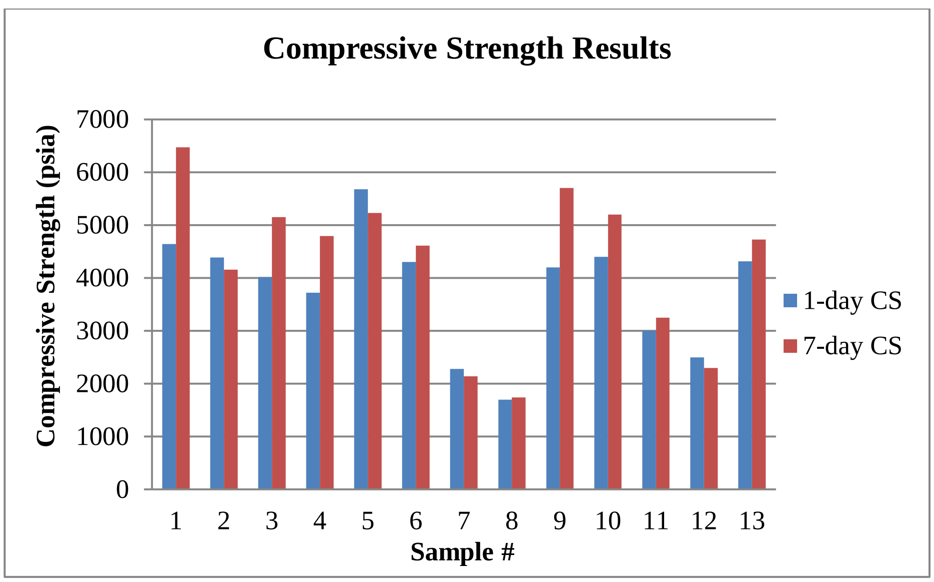


FIGURE 14: COMPRESSIVE STRENGTH RESULTS

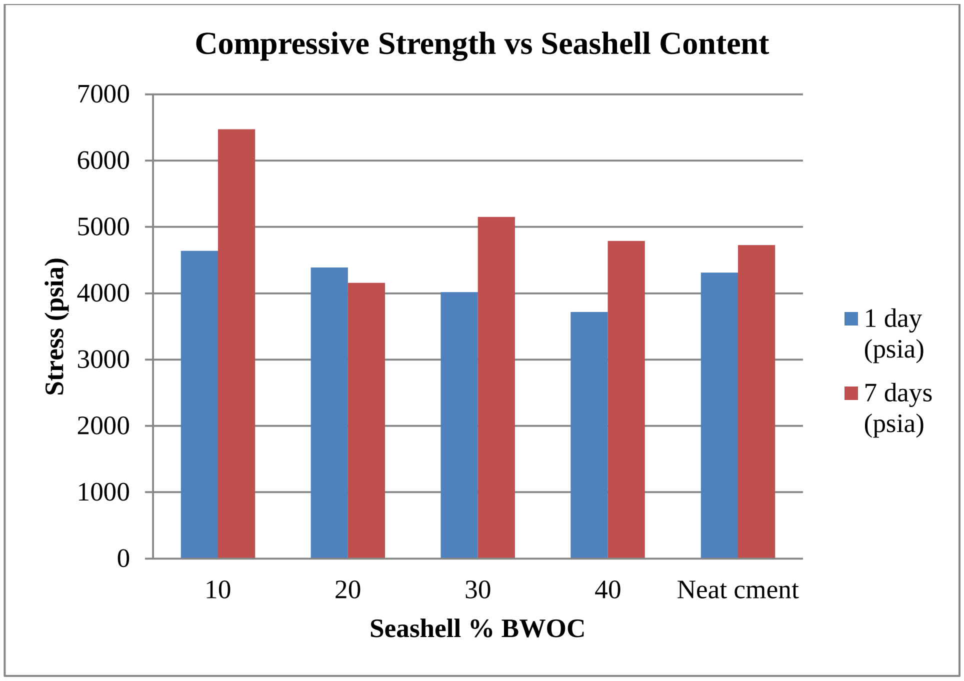


FIGURE 15: EFFECT OF SEASHELLS ON CEMENT COMPRESSIVE STRENGTH

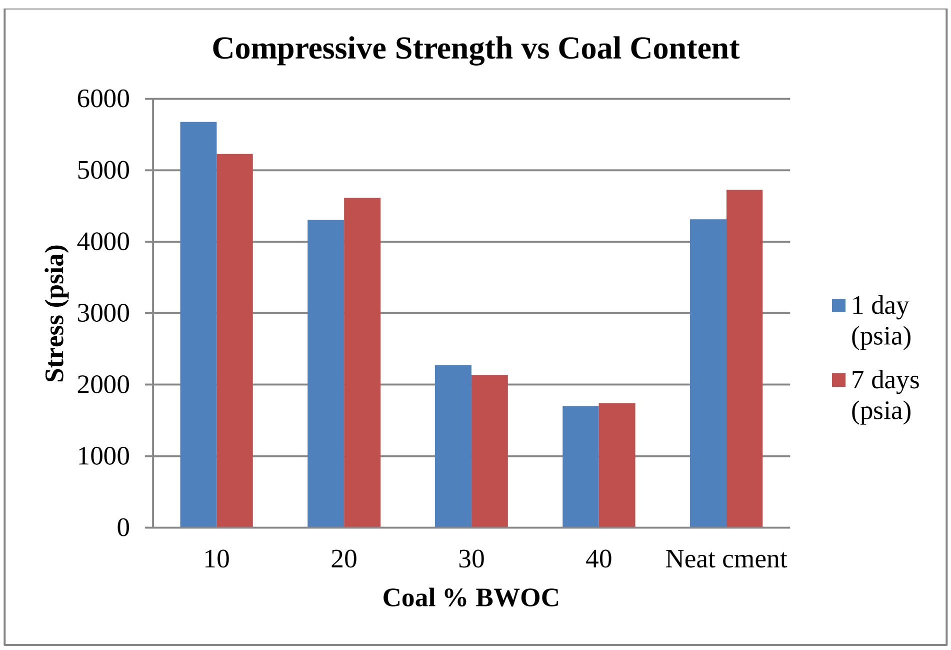


FIGURE 16: EFFECT OF SARAWAK COAL ON CEMENT COMPRESSIVE STRENGTH

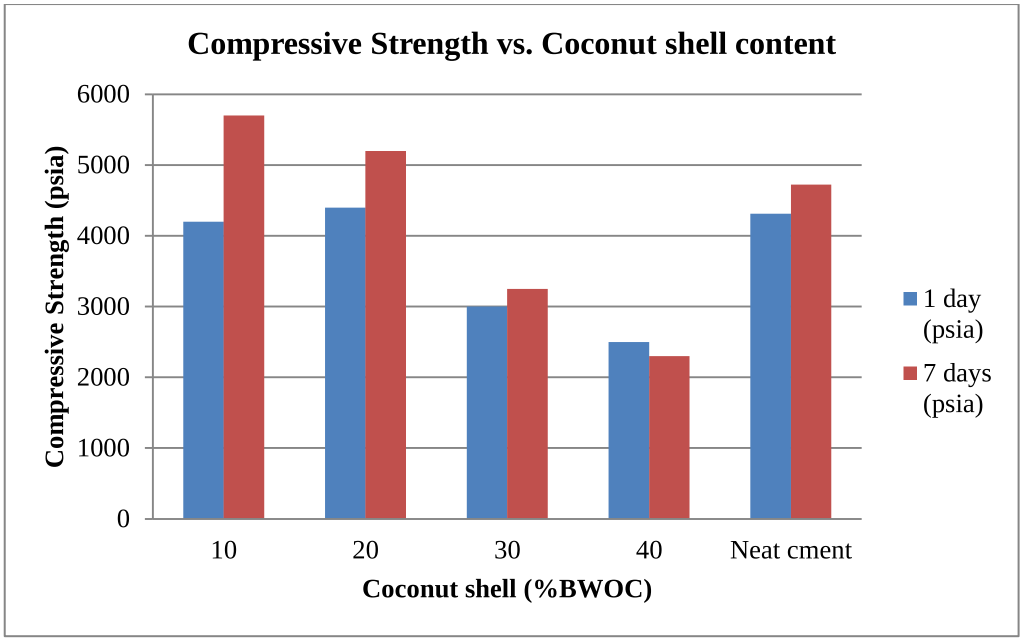


FIGURE 17: EFFECT OF COCONUT SHELL ON CEMENT COMPRESSIVE STRENGTH

4.2.1 INTERPRETATION & DISCUSSION

Among the additives used, seashell showed higher and stable results. Strength retrogression did not occur with seashell samples, indeed, seven-day compressive strength results show much higher readings than the one-day results. To compare seashell samples to the neat cement sample, it can be seen that at most concentrations of seashell CS results in higher values that neat Portland cement. According to the results, the optimum concentration of seashell is 10% BWOC however, concentrations up to 40% also shows compressive strengths which are within the range of field requirements (PCSB SKG 10, Cementing).

Coal, due to its comparably low density and hardness resulted in lower compressive strength results. Strength retrogression was observed at 10-30 % BWOC coal concentrations. As observed, cement slurries with increasing coal content resulted in problematic slurry mixing. As the result additional water was added to keep the slurry mixable. The latter will reduce the cement density which can contribute to the low CS values. Nevertheless, 10-20 % BWOC coal samples are within the industry range of CS requirements.

Coconut shell samples did not undergo strength retrogression. 10-20% BWOC samples showed high compressive strengths. 30-40% BWOC samples are below the minimum field requirements for compressive strength.

Overall, each of the additives showed better compressive strength values that neat cement at some concentrations. These values are higher than the CS values obtained using silica in the experiment of M.R. Islam (refer to literature review). His experiment was also conducted according to API RP 10 B specs but at slightly different downhole conditions. M.R. Islam used 200F curing conditions at which cement sheath does not undergo strength retrogression. However, silica increase compressive strength of cement by reacting with Portlandite and forming more C-S-H which also prevent degradation of cement sheath by CO_2 (Arina binti Sauki et al). For the additives used in this project, more study need to be done on the chemical processes taking place during the cement curing. This would give a deciding conclusion on the application of these new additives in the field.

4.3 Slurry Rheology Results

Rheology tests conducted on cement slurries to find out the effects of the additives on the slurry flow behavior. According API RP 10B specifications, rotational viscometer is used to measure the shear stresses of the cement slurry at different shear rates. Results are plotted in Table 2.

TABLE 9: VISCOMETER FANN READING RESULTS

Shear rate (rpm)	Fann Readings for samples											
	1	2	3	4	5	6	7	8	9	10	11	12
600	28	30	33	40	100	160	220	250	44	48	53	59
300	17	18	22	27	56	78	130	145	22	26	28	31
200	13	15	19	24	33	41	48	53	17	21	23	25
100	10	12	14	18	25	31	37	42	14	18	20	22
6	2	3	3	4	9	11	14	17	7	8	10	11
3	1	2	2	3	6	7	7	9	4	5	7	7

4.3.1 INTERPRETATION & DISCUSSION

Determining the rheology models (bingham or power law) is not the scope of this project. However, the latter can be determined by plotting shear rate versus shear stress values in Cartesian and log scales. Bingham will give straight line in a Cartesian scale whereas power law fluid will result in straight line in log scale.

Here we only make general conclusions about the flow behavior of the samples tested. Following the simple rule of thumb used in the field:

3 & 6 rpm < 5 = Solid settling

100 rpm > 20 = Gelation

300 & 600 rpm > 200 = Hard in mixing and pumping

The above conclusion is strongly possible and therefore always used by field engineers (PCSB SKG 10, Cementing). From that we can say that solid settling occurs in seashell samples. It was indeed observed that larger particles are left over in the mixer bowl.

In the samples with coal content gelation occurs. During the experiment coal is not mixed with cement homogenously (different colors were noted in the slurry). With increasing amount of coal, slurry became hard to mix and to pour (experimental observations). The latter observations are as well backed up by the Fann readings of 30-40% BWOC coal which indicates that the slurry is hard in mixing and pumping (rule of thumb).

Rheology results for the coconut shell samples are good except for gelation at 30-40% BWOC coconut shell concentrations.

Overall, the samples that are good in compressive strength resulted in good preliminary rheology measurements too. Samples 1-4 resulted in solid settling, 5-12 gelation and 7-8 hard in mixing and pumping. General but sufficient results are concluded about the effects of the new additives on the cement slurry rheology.

4.4 Slurry Fluid Loss Results

The third parameter measured in this project in fluid loss. Stirred cement fluid loss is used following API RP 10B fluid loss testing instructions. Fluid loss is an important parameter especially for HPHT cement where the well depth is high. Slurry fluid loss should have minimum requirement for successful well cementing without any cement bridging occurred.

Depending on the types of cement jobs, different fluid loss is required which can be attained by adding fluid loss additives into cement. However, in this experiment we want to see the effects of the new additives on the fluid loss without adding any fluid loss additives. The testing temperature is 220F and at 1000 psi differential pressure. Table 10 shows the field requirements for the cement fluid loss.

TABLE 10: MAXIMUM ALLOWABLE FLUID LOSS

Casing cementing	250 cc/30 min
Liner cementing	50 – 100 cc/30 min
Gas control	50 cc/30 min
Squeeze cementing	50 – 200 cc/30 min

Experimentally measured fluid loss values are plotted in Figure 5.

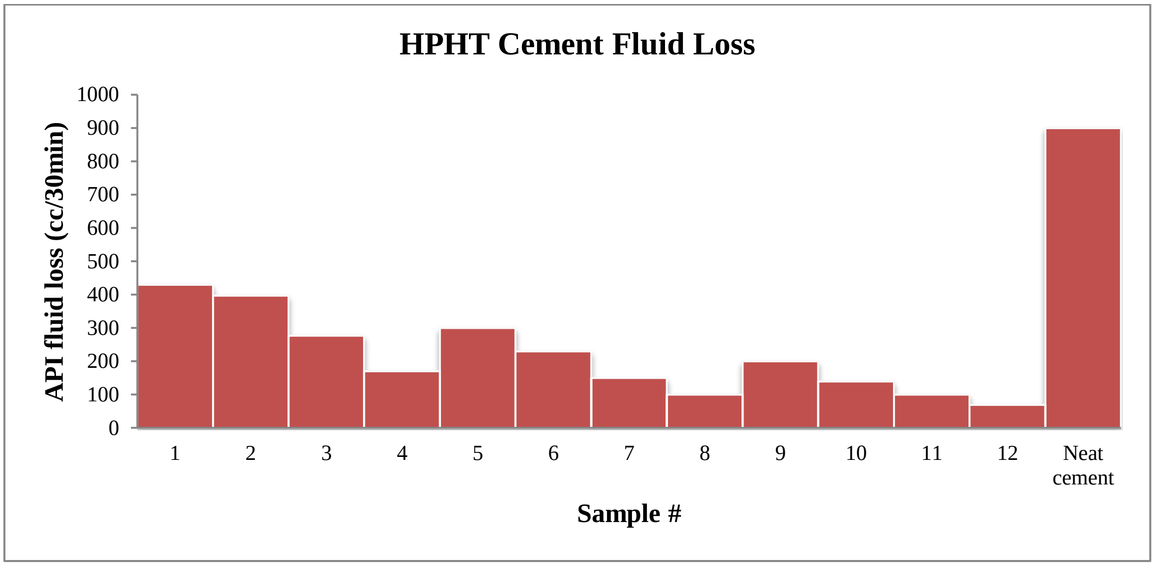


FIGURE 18: API FLUID LOSS MEASUREMENTS

4.4.1 Interpretation & Discussion

From the results shown in Figure 18, it can be seen that in many samples fluid loss is very low comparing to the neat cement. Fluid loss for sample 4 containing 40% BWOC seashell is suitable for casing cementing. And it can be seen that with increasing amount of coal fluid loss decreases. Despite that compressive strength of cement decreases with increasing coal content as shown before, coal is good additive to reduce slurry fluid loss.

Coconut additives show the strongest effect on the API fluid loss of the cement. Same as coal, coconut reduces the fluid loss with increasing concentrations, however more than coal does. For the test temperature of 220F these new additives showed good results in API fluid loss measurements. This project could be extended to test the combined effects of seashells and coconuts together as each of them improves compressive strength and fluid loss respectively.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

This section will sum up the project activities, results and findings. Moreover, some recommendations are provided to expand the scope of the project.

5. 1 CONCLUSION

The main objective of this project is to find alternative sources of additives to replace existing silica flour additives. As the silica flour additives are toxic and expensivel, new sources of additives are studied. The purpose of the silica flour is to enhance the compressive strength of HPHT cement. Therefore, the major parameter to be measured in this project is the compressive strength of cement resulting from the use of new additives.

This project measures the effects of coconut shells, seashells and Sarawak coal on the compressive strength, slurry rheology and fluid loss parameters of cement. Cement composition with seashell content showed good compressive strength values which are higher than the field requirements. 30% BWOC seashell has the compressive strength value of above 5,000 psi. We can now replace most of the Portland cement with seashell additive which will give higher strength and lower cementing costs.

Even though coal and coconut shell additives did not show good compressive strength results in high concentrations, they showed good rheology and fluid loss behaviors. API fluid loss of the cement slurry reduced with the increasing concentrations of coal and coconut shells.

Among the additives used, coal showed low compressive strength values and slurry that is hard in mixing and pumping.

5.2 Recommendations

1. The chemical reactions between the Portland cement and the new additives should be determined before the field use. Presence of weak components in the cement samples would have potential to degrade in high temperature environments.

2. The samples should be further tested for CO_2 exposure at HPHT simulated conditions.

3. Curing the samples in HPHT chamber for 7 and 14 days at temperatures above 230F would show more reliable results for strength retrogression of cement.

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