

An undergraduate Technical Research Submitted to the College of
Engineering Architecture and Technology
Palawan State University
Puerto Princesa City

As Partial Fulfilment of the Requirements
For Degree of
Bachelor of Science in Petroleum Engineering

October 2018

**COCONUT SHELL ACTIVATED CARBON (CSAC) AS A
POTENTIAL TREATMENT FOR CALCIUM ION
CONTAMINATION IN WATER-BASED DRILLING MUD**

By

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ABSTRACT

This study is entitled “*Coconut Shell Activated Carbon (CSAC) as a Potential Treatment to Calcium Ion Contaminated Water-Based Drilling Mud*”. The CSAC functions as a treatment that is most-known in water-treatment plants for its ability to calcium and magnesium ions in water. For this reason, the proponents aim to determine its potential to adsorb calcium-ion in water-based drilling mud, along with the evaluation of the changes in the properties of a drilling mud with the presence of CSAC.

The proponents prepared four samples, each with three trials. The samples were composed of 350 mL Distilled Water, 21 grams of Bentonite Clay, 0.3 grams of Portland Cement for the first sample, and the rest of the three samples were then added CSAC with the amount of 6, 9, 12 grams, respectively. To identify the changes in the rheological properties of the mud, such as the density, viscosity and cake thickness, the proponents prepared a controlled setup which does not contain Portland Cement and CSAC. The settling time for adsorbing was allowed for 1 hour before undergoing rheological property readings. All three trials from each sample were then mixed and proportioned to be submitted to the Optimal Laboratory for Calcium-ion Concentration Test.

For the experimental setups using 30, 60 and 150 rpm, it was shown that there is a significant difference in the viscosity, density, and cake thickness of the mud. The viscosity and density significantly increased, and the cake thickness decreased. It was also shown that the Calcium-ion contamination decreased as the amount of CSAC increased.

APPROVAL SHEET

This is to certify that we have supervised the preparation and read the research paper prepared by **DE LOS SANTOS, KELSEY MARIE E., ONG, REGIENA MURRAE C., and TRESPECES, GABRIEL DOMINIC B.**, entitled **COCONUT SHELL ACTIVATED CARBON (CSAC) AS A POTENTIAL TREATMENT TO CALCIUM ION CONTAMINATED WATER-BASED DRILLING MUD** and that the said paper has been submitted for final examination by the Oral Examination Committee.

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As members of the Oral Examination Committee, we certify that we have examined this paper and hereby recommend that it be accepted as partial fulfilment of the undergraduate requirement for the Degree **Bachelor of Science in Petroleum Engineering**.

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ACKNOWLEDGEMENT

The completion of this undertaking would not have been possible without the participation and assistance of these sets of individuals. Their contributions are deeply recognized and appreciated, and we, the researchers, are beyond grateful for each of their roles.

To Mr. Jonas George S. Arcilla, P. Geol., our research adviser, our deepest and sincerest thanks for your guidance as our mentor. Your ideas and support into providing the necessary material that we originally needed for our study motivated us so much. Your advises gave us hope and direction as we struggled in finishing and analysing our results.

To our beloved research readers, Engr. Manuel L. Cabiguen , Jr., Engr. Allan Jay L. Javier, and Engr. Ryan Kristoffer C. Aguirre, for their perceptive comments and recommendations into polishing our study.

To Sir Russell Fernandez, for analysing our experimental results in rush hours, and opening insightful comments with regards to the statistical analysis of our data.

To our parents, family, and friends, for their undying moral and financial assistance in making this thesis study possible, and motivating us to be one step ahead and be more of what we are in the present. All of you made us not only better students, but also better individuals as we apply everything we have learned in the industry.

And above everything, we are thankful for our Supreme Being, the author of knowledge and wisdom, for providing us strength and positivity in finishing this research.

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Chapter 1

INTRODUCTION

Even in the most planned wells there will always be a problem that will arise. One of the most common problems that the industry is facing today is the contamination of our drilling mud while drilling. Contaminant can be any unwanted material that can cause changes in our drilling mud properties. The sources of calcium ions are gypsum, anhydrite, cement, lime, seawater, and hard-brackish makeup water. (NETWAS GROUP OIL, 2017)

Sodium carbonate or more commonly termed as soda ash in drilling, is the most common treatment to most types of calcium ion contamination in freshwater and seawater muds by eliminating calcium ions by converting to insoluble carbonate, and should be added prior to the addition of bentonite or polymer to the fluid system. (Halliburton, 2011)

The activated carbon and sodium carbonate exhibit a different chemistry in calcium-ion treatment. The capability of an activated carbon especially the coconut shell activated carbon (CSAC) was proven to be used as softener for hard water, effectively reducing the calcium and magnesium ion content of the water. According to the study of Rolence, C., “Water hardness removal by coconut shell activated carbon”, it has been concluded that CSAC is effective to reduce water hardness up to 94%. Studies have yet to be conducted on its potential as a treatment for water-based drilling mud contamination.

Activated carbon works by entrapping and adsorbing chemicals in its small pores. Many kinds of activated carbon are already produced using variety of woods and husks and one of them is the coconut shell. Coconut shell is organic in nature; it is one of the two agricultural commodities that earn the Philippines more than a billion dollars in export revenues wherein the average production of coconut in the last three (3) years is 14.9 billion according to the Philippine Coconut Authority.

In a tropical country like the Philippines, Coconut tree is one of the most common trees there is. The availability of the coconut shell is vast, thus, it is a convenient choice for this research study as an additive for water-based drilling mud treatment. This study provides innovation to the use of coconut shell activated carbon's properties to the oil and gas industry. The initial findings in this study will serve as a backbone to more potential innovative studies in regards to the use of coconut shell activated carbon to drilling. Its potential effectiveness can bring an increase in profit and other opportunities to coconut farmers in the country.

The proponents of this study aim to know the potential of Coconut Shell Activated Carbon (CSAC) as a drilling mud treatment in calcium-ion contaminated mud. Density, viscosity and calcium ion contents are the parameters that were measured. The purpose of this study is merely for experimental evaluation, and any conditions in drilling conditions were not necessary as well.

The type of drilling mud used in the study was mid-viscous water-based mud (6% w/v or 21g bentonite clay per 350mL of water) which is adopted from API SP 13A-18th Edition. This mixture is considered as spud mud and is only composed of distilled water and bentonite.

The test procedures are based on the American Petroleum Institute Recommended Practice Standard Procedure for Field Testing Drilling Fluids (API RP 13B-4TH Edition and API RP 13A-18TH Edition) and the Calcium-ion determination is tested in the Optimal Laboratory in Bgy. San Manuel, Puerto Princesa City, Palawan.

During the experiment the average ambient temperature ranging from 25 to 30 degrees celsius and average ambient pressure would be 14.7 psia (1 atm). The ambient temperature and pressure were constant all throughout the experiment. The samples are mixed thoroughly with accordance to the API Standards abovementioned.

Chapter 2

REVIEW OF THE RELATED LITERATURE

2.1 DRILLING FLUIDS

Drilling fluids are usually in the form of liquid and gaseous fluids with mixtures of additives (such as solid suspensions, mixtures and emulsions of liquids, gases and solids) and often used in rotary drilling operations of subterranean wells and are often referred to as drilling mud. Drilling fluids are essential in drilling as they carry cuttings from the wellbore to the surface, control formation pressures, suspend and release cuttings, seal permeable formations, maintain wellbore stability, minimize reservoir damage, cool, lubricate and support the bit and drilling assembly, transmit hydraulic energy to tools and bit, ensure adequate formation evaluation, control corrosion, facilitate cementing and completion, and minimize impact on the environment. (DRILLING FLUIDS ENGINEERING MANUAL, 2006)

There are three-types of drilling fluids, based on composition; (1) water-based, (2) gas-liquid mixture, and (3) gaseous.

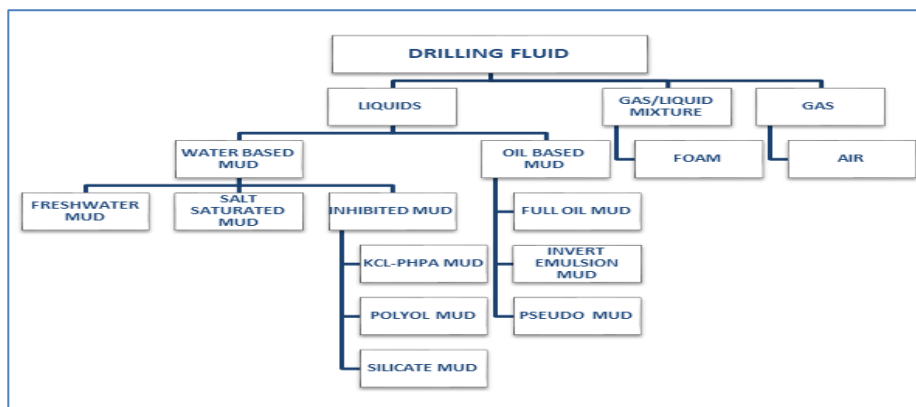


Figure 2.1.1 Types of Drilling Fluids

2.1.2 WATER-BASED DRILLING MUD

Water-based drilling mud is the extensively and inexpensive drilling fluids used. It is created by mixing water and clay at a desired amount, with the use of the rightful additives that are a must-require with accordance to the wellbore condition and stabilization. Fresh water is used as the base for most of these muds, but in offshore drilling operations salt water is more readily available. (Rolence, 2014)

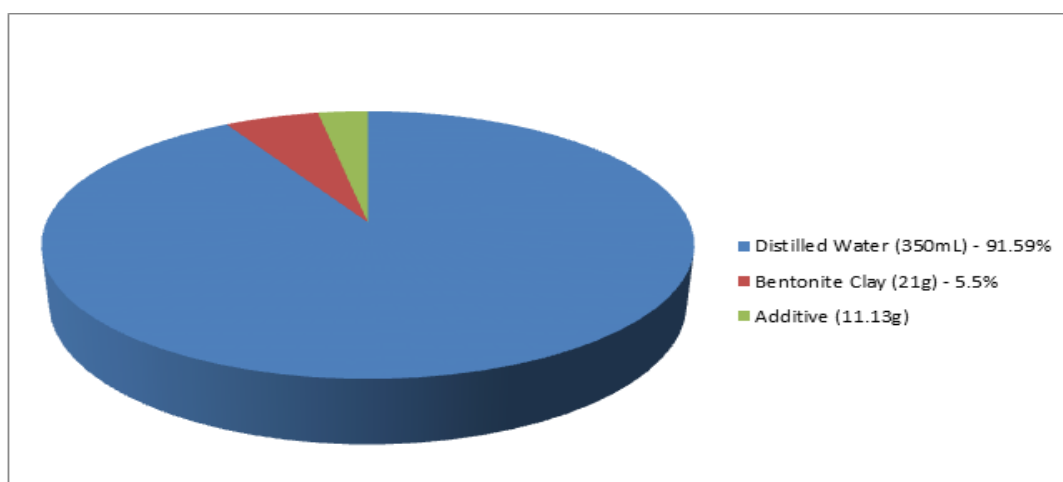


FIGURE 2.1.2.1 API Specification 13A 18th Edition – Standard Composition of Water-Based Drilling Mud

The figure above is based from the American Petroleum Specification, where the additive is the coconut shell activated carbon. The standard amount of distilled water to be used by bentonite clay is 350ml per 21g. The additive that must be used is 3 percent by weight (w/v), which makes it to 11.13g by conversion. The said additive abovementioned is the minimum amount of additive that can be used in a standard water-based drilling mud. It may or may not be used as well. The standard

measurement of the rheological properties of water-based drilling mud is based on **API-RP-13B-1 Specification**.

2.2 ACTIVATED CARBON

Activated carbon is a non-hazardous carbon-bearing product with a porous structure and a large internal surface area, and is manufactured from coconut shell, peat, hard and soft wood, lignite coal, bituminous coal, olive pits and various carbonaceous materials. Its chemical structure is defined from crude form of graphite, with an amorphous structure. Activated carbon is mostly known for its treatment property that is based primarily on its adsorption potential. Its internal surface area ranges from 500 to 1500m²/g which is highly qualified for adsorption. Impregnation of chemicals to activated carbon will enhance its properties as well. Activated carbon works by entrapping and adsorbing chemicals in its small pores. Many kinds of activated carbon are already produced using variety of woods and husks and one of them is the coconut shell.

Activated carbon is available in three forms, such as Powdered Activated Carbon, and Extruded Activated Carbon. Each are available in many sizes depending on the application and requirements and certain form and size.

Powderized Activated Carbons (PAC) are defined by the ASTM as particles passing through an 80-mesh sieve (0.177mm) and smaller. PAC typically has a diameter less than 0.1mm and an apparent density ranging between 23 and 46 lb/ft³, depending on the material used and manufacturing process. It is used by water treatment plants on either a full time basis or as needed for taste and odor control or

removal of organic chemicals as it adsorbs gas and solid particles. In water treatment, the dosages of PAC can range between 100 mg/L depending on the type and concentrations of organic compounds presents. Dosages of 1 to 20 mg/L are typical for nominal taste and odor control. Its application generates additional sludge which is not able to be regenerated as is spent GAC. (E.P.A., 2018)

Granular Activated Carbon (GAC) is known to be a good adsorbent due to its high surface area and became common in the 29th century as an agent for water purification when the process of activation of charcoal was applied. The thermal activation of charcoal improves its pore volume, surface area, and structure – which makes it a good agent for water treatment. It has strong attraction to organic compounds and other non-polar contaminants (such as calcium-ion) and adsorbs them onto the surface of the activated carbon. Each carbon source produces a granular activated carbon and has a unique pore structure consisting of micro-pores, meso-pores and macro-pores. Micro-pores are typically of molecule size around 5-1000 Angstroms, which is by conversion, 1/10,000,000 of a millimetre. (WATER QUALITY ASSOCIATION, 2013)

Extruded Activated Carbons, also known as “pressed pellets”, are mainly made by mixing pulverized anthracite or charcoal with a suitable binder which are extruded at high pressure into a shape of cylinder. (DESOTEC, 2017)

Among the three forms of activated carbon, PAC and GAC are often used in water-treatment plants. The GAC's greater surface area than PAC enables it to adsorb more particles. However, the researchers resorted into using PAC due to the unavailability of GAC.

2.2.1 ACTIVATED CARBON PREPARATIONS AND ADSORBING CAPACITY

From the study conducted by Das D., et. al., activated carbon prepared from green coconut shells by chemical activation method for characterization and the following physical properties were as well determined: 1) bulk density, 2) moisture content, 3) volatile matter content, 4) ash content, 5) surface area, and 6) porosity. The activated carbon was used as adsorbent for the removal of carbon dioxide from flue gas as well as various inorganic contaminants from waste water. Removal efficiency of Methylene blue was studied by changing various parameters such as adsorbent dosage and contact time. This study showed that for the removal of various pollutants from flue gas and waste water treatment activated carbon can be used as a good adsorbent .

From the study conducted by Hao Y., et. al., the three kinds of activated carbon prepared using coconut shell indicate that the carbons were mainly amorphous and only a few impurity groups were adsorbed on their surfaces. According to the texture property test, it revealed that the activated carbons displayed different texture properties, especially on the micropore size distribution. The adsorption capacities of the activated carbons were investigated by adsorbing CH₄, CO₂, N₂ and O₂ at 25 °C in the pressure range of 0-200 kPa. The results reveal that all the activated carbons had high CO₂ adsorption capacity, one of which had the highest CO₂ adsorption value of 2.55 mmol/g at 200 kPa. And the highest adsorption capacity for CH₄ of the activated carbons can reach 1.93 mmol/g at 200 kPa. In the pressure range of 0-200 kPa, the adsorption capacities for N₂ and O₂ were increased linearly with the change of pressure

and K-AC is an excellent adsorbent towards the adsorption separation of greenhouse gases.

From the study conducted by Liu, X., et. al., the activated carbons prepared were processed using pyrolysis activation. Activation temperature and time on pore structure and adsorption properties were taken into consideration for investigation. The results showed that the prepared carbon has mainly micropore under the condition of activation temperature at 900 degrees Celsius for 4 hours. Prepared carbon had a surface area of 994.42 m²/g with the iodine adsorption of 1295 mg/g and methylene blue adsorption of 135 mg/g. The results of N₂ adsorption showed that the average pore size of the activated carbon is around 2 nm. The micropore volume is 0.430 3 cm³/g and total pore volume of 0.503 9 cm³/g. This indicated that ratio of micropore is 85.39%. The results of CO₂ dynamic adsorption showed that the adsorption quality is up to 56.61 mg/g. With the increase of activation temperature and activation time, the yield of activated carbons is reduced at different degree, in the preparation of activated carbons from coconut shell by carbonization.

Water hardness is characterized by the amount of dissolved minerals such as, calcium and magnesium in the water, with a few metal impurities. According to Rolence C., the use of Coconut Shell Activated Carbon (CSAC) has a removal efficiency at nearly neutral pH of 6.3 for synthetic and field collected water samples were 60% and 55% respectively. Hence, the removal efficiency increases with the increase in contact time and adsorbent dose. The various adsorbent factors were the adsorbent dose, initial pH, contact time, and temperature. (ROLENCE, 2014)

In water-based drilling mud sampling mixture, the given water-based drilling mud additives do not have a significant reaction to the hard water if a treatment is to be added.

The type of activated carbon that was used in the experimental procedure was Powderized Activated Carbon instead of the Granular Activated Carbon due to its unavailability.

2.2.2 COCONUT SHELL ACTIVATED CARBON

Activated Carbon is known to be more adsorbent to organic materials. It can be extracted from coconut shell, pear, hard and soft wood, and other organic materials. Hence, a study conducted by Mishra, S., “*Characterization of Activated Carbon of Coconut Shell, Rice Husk and Karanja Oil Cake*”, shows comparative characteristics of organic materials where activated carbon is extracted from. The characterization of the adsorbents includes estimation of proximate analysis of the moisture content, ash content, of the adsorbents includes estimation of proximate analysis of the moisture content, ash content, volatile matter content and fixed carbon content) along with bulk density, BET surface area, SEM, porosity, pH, iodine number and methylene blue number. Hence the adsorption capacity of the activated carbon was determined by its potential to remove methylene blue at the maximum.

Table 2.2.2.1 Summarized characterization results of Activated Carbon extracted from Coconut Shell, Rice Husk, and Oil Cake

Activated Carbon Source	% moisture content	% Ash Content	% Fixed Carbon	Bulk Density	Surface Area (m ² /g)	Iodine Number (mg/g)	Pore Volume (cc/gm)
COCONUT SHELL	2.22	1.5	88.922	0.58	1000.15	986	0.07
RICE HUSK	1.07	6.25	78.06	0.121	289.1	178	0.0537
OIL CAKE	1.46	3.92	76.08	0.352	179.3	258	0.0592

Table 2.2.2.2. Adsorption Capacity of 3 types of Activated Carbon extracted from Coconut Shell, Rice Husk, and Oil Cake

Activated Carbon Source	Initial Dye Concentration (mg/Lt)	Final Dye Concentration (mg/Lt)	% of Removal of Blue Methylene	Adsorbed (mg/g-Lt)
COCONUT SHELL	50	2	96	80
RICE HUSK	50	19	62	51.67
OIL CAKE	50	8.5	83	69.17

The fixed carbon content from the coconut shell activated carbon is higher than rice husk and oil cake. It shows that the moisture content in coconut shell activated carbon is higher, while the volatile and ash content are the least – which is due to the high fixed carbon, which is a preferred adsorbent in comparison to the other two. A high bulk density also indicates a better flow consistency and packaging quantity of solid fuel material.

Iodine number is a measure of the micro-pore content of the activated carbon. It has a direct relationship with the micro porosity of the sample, thus, a higher iodine

number signifies higher micro-porosity of the sample. Pore volume, obtained from the table above, shows that the activated carbon from coconut shell has a higher exhibit than the other two.

The two abovementioned show that the activated carbon from Coconut Shell has a better adsorption capacity than rice husk and oil cake.

2.3 DRILLING FLUID CONTAMINANTS

A mud is said to be contaminated when a foreign material enters the mud system and causes undesirable changes in mud properties, such as density, viscosity, and filtration. Generally, water-based mud systems are the most susceptible to contamination. Mud contamination can result from overtreatment of the mud system with additives or from material entering the mud during drilling. The most common contaminants to water-based mud systems are solids (added, drilled, active, inert); gypsum/anhydrite (Ca^{++}); cement/lime (Ca^{++}); makeup water (Ca^{++} , Mg^{++}); soluble bicarbonates and carbonates (HCO_3^- , CO_3^{--}); soluble sulfides (HS^- , S^{--}); and salt/salt water flow (Na^+ , Cl^-).

Table 2.3.1. Common drilling fluid contaminants along with their source and common treatment.

CONTAMINANTS	DESCRIPTION/SOURCE/TREATMENT
SOLID CONTAMINATION	Solids are materials that are added to make up a mud system (bentonite, barite) and materials that are drilled (active and inert). Excess solids of any type are the most undesirable contaminant to drilling fluids. They affect all

	mud properties. It has been shown that fine solids, micron and submicron sized, are the most detrimental to the overall drilling efficiency and must be removed if they are not a necessary part of the mud makeup. The removal of drilled solids is achieved through the use of mechanical separating equipment (shakers, desanders, desilters, and centrifuges). Shakers remove solids in the size of cuttings (approximately 140μ or larger). Desanders remove solids in the size of sand (down to 50μ). Desilters remove solids in the size of silt (down to 20μ). When solids become smaller than the cutoff point of desilters, centrifuges may have to be used. Chemical flocculants are sometimes used to flocculate fine solids into a bigger size so that they can be removed by solids-removal equipment. Total flocculants do not discriminate between various types of solids, while selective flocculants will flocculate drilled solids but not the added barite solids. As a last resort, dilution is sometimes used to lower solids concentration.
BIOCARBONATE AND CARBONATE CONTAMINATION	The contaminant ions (CO_3^-, HCO_3^-) are from drilling a CO_2-bearing formation, thermal degradation of organics in mud, or over treatment with soda ash and bicarbonate. These contaminants cause the mud to have high yield and gel strength and a decrease in pH. Treating the mud system with gypsum or lime is

	recommended.
HYDROGEN SULFIDE CONTAMINATION	The contaminant ions (HS^- , S^{2-}) generally are from drilling an H_2S -bearing formation. Hydrogen sulfide is the most deadly ion to humans and is extremely corrosive to steel used during drilling operations. (It causes severe embrittlement to drillpipe.) Scavenging of H_2S is done by use of zinc, copper, or iron.
SALT/SALTWATER FLOWS	The ions, Na^+Cl^- , that enter the mud system as a result of drilling salt sections or from formation saltwater flow cause a mud to have high yield strength, high fluid loss, and pH decrease. Some actions for treatment are dilution with fresh water, the use of dispersants and fluid-loss chemicals, or conversion to a mud that tolerates the problem if the cost of treatment becomes excessive.
CALCIUM ION CONTAMINANTS	The calcium ion is a major contaminant of water-based drilling fluids. Calcium can enter the mud as part of the makeup water or formation while drilling cement, anhydrite or gypsum. Calcium contamination drastically changes the nature of freshwater, clay-based systems.

2.3.1 CALCIUM-ION CONTAMINATION

The sources of calcium ions are gypsum, anhydrite, cement, lime, seawater, and hard/brackish makeup water. The calcium ion is a major contaminant to freshwater-based sodium-clay treated mud systems.

The calcium ion tends to replace the sodium ions on the clay surface through a base exchange, thus causing undesirable changes in mud properties such as rheology and filtration. It also causes added thinners to the mud system to become ineffective. The treatment depends on the source of the calcium ion. For example, sodium carbonate (soda ash) is used if the source is gypsum or anhydrite. Sodium bicarbonate is the preferred treatment if the calcium ion is from lime or cement. If treatment becomes economically unacceptable, break over to a mud system, such as gypsum mud or lime mud, that can tolerate the contaminant. (MITCHELL, 2006)

Calcium ion contaminant to drilling mud affects the solubility of drilling mud as it gradually changes the flocculation chemistry of calcium ions. This contaminant in water-based drilling fluids dramatically increase filtration volume loss and worsens rheological properties, especially in high temperature boreholes. Calcium can enter the mud as part of the makeup water or formation while drilling cement, anhydrite or gypsum. Calcium contamination drastically changes the nature of freshwater, clay-based systems. (LIU, 2009)

According to Donald Price of Netwas Group Oil, small quantity of CaSO_4 can affect the rheological properties. The intensity of flocculation depends on clay content. When CaSO_4 causes the calcium concentration to increase above 200 mg/L, fluid loss may become difficult to control and will have the viscosity fluctuate drastically. He also

stated that when treating calcium contamination with soda ash; always take care not to over treat the mud.

2.3.1.1 FORMATION CONTAMINATION

Anhydrite is one of the most common calcium-based rock drilled, along with gypsum and limestone, and causes calcium ion contamination that can lead to increased fluid loss, yield point, and gel strengths. It is an anhydrous calcium sulphate (CaSO_4) and is typically interbedded with halite gypsum, and limestone. Anhydrite highly influences the type of mud selected as it would chemically react with selective ions and cause flocculation. (Rolence, 2006)

2.3.1.2 CEMENT CONTAMINATION

In most drilling operations, cement contamination often occurs when casing strings are cemented and the plugs are drilled out. The extent of contamination and its effect on mud properties depend on solid content, type and concentration of deflocculants, and the quantity of cement incorporated. Freshwater bentonite systems are flocculated by cement, resulting in increased rheology and fluid loss. The severity of the flocculation depends upon the quantity and quality of solids present and the solubility of Calcium Ion. Therefore, when cement contamination reaches a level where it is no longer practical to treat out, it may be desirable to convert the system to a calcium-based mud. It may be possible to isolate the contaminated fluid and dump it. (NETWAS GROUP OIL, 2012)

Portland cement (Type 1) is a cement that is manufactured from limestone and clay and is the most common type of cement in general use around the world as a basic

ingredient of concrete, mortar, and grout. Portland cement is our main source of calcium-ions, since it is the most convenient source of abundant Calcium-ion concentration available. Portland Cement is composed of **46.0975%** Calcium Ions, having 53.9025% occupied by metals and other elements such as Silicon Dioxide, Iron (III) Oxide, Sodium Oxide, Potassium Oxide, and Magnesium Oxide.

2.4 CALCIUM-ION DETERMINATION

The most common methods in determining calcium-ion in water samples are by EDTA Complexometric Titration and Spectroscopy.

2.4.1 EDTA COMPLEXOMETRIC TITRATION

EDTA Complexometric Titration is a form of volumetric analysis in which the formation of a colored complex is used to indicate the end point of the titration. The test measures the amount of the soluble magnesium and calcium ions in the mud sample/filtrate. It is then simultaneously measured; as a result, it is called total hardness. The mud sample/filtrate. It is then simultaneously measured; as a result, it is called total hardness. The calcium ion content of the filtrate can also be measured separately, thus when it is subtracted from the total hardness, the magnesium-ion content is determined as well. In this process, there are five reagents to be used, such as: Calmagite as the indicator solution, Ammonium Buffer Solution, EDTA Titration Solutions (#205-06 & #205-10), and Deionized Water. (OFI Testing Equipment – Total Hardness Titration Kit No. 145-10)

2.4.2 ATOMIC ABSORPTION SPECTROSCOPY

The AA Spectrophotometer method allows calcium and magnesium to be determined more reliably and quickly than that of the titration method. The preparation of the sample is usually simple and rapid, and concentrations of calcium and magnesium ion at the parts per million level (ppm) are easily measured. Interference by counter ions or complexing ions is minimal, except for phosphate ion suppression of calcium, which can be countered by addition of lanthanum (III) ion to the solutions to be measured. For calcium, the absorption is measured at a wavelength of 422.7 nanometres, and magnesium absorption is measured at a wavelength of 285.2 nanometres. (*page 30 Chemistry 321L Manual, 2012*)

Our mud filtrates were tested and analysed at the Optimal Laboratories, Inc. at Bgy. San Manuel, Puerto Princesa City, Palawan. Optimal Laboratories used the method of Direct Air-Acetylene Flame Method with the instrumentation of Flame Atomic Absorption Spectrophotometry in analysing our samples.

2.5 RELATED STUDIES

2.5.1 Water Hardness Removal By Coconut Shell Activated Carbon

Based on the results obtained in the study of Rolence C., it is clear that CSAC is effective in water softening. Coconut Shells are available in coast regions where hardness problem is prevailing, then, CSAC adsorbents are expected to be economically feasible for removal of hardness from groundwater. Langmuir adsorption isotherm model indicated that the adsorption of hardness ions onto CSAC is favorable. Although the removal at pH 12 is high, this will necessitate the use of other pH

correcting reagents which will make the process uneconomical and leads to the introduction of chemical contaminants in softened water. Therefore, adsorption at neutral pH, though less effective than at a pH of 12, is preferred due to economic and water safety reasons.”

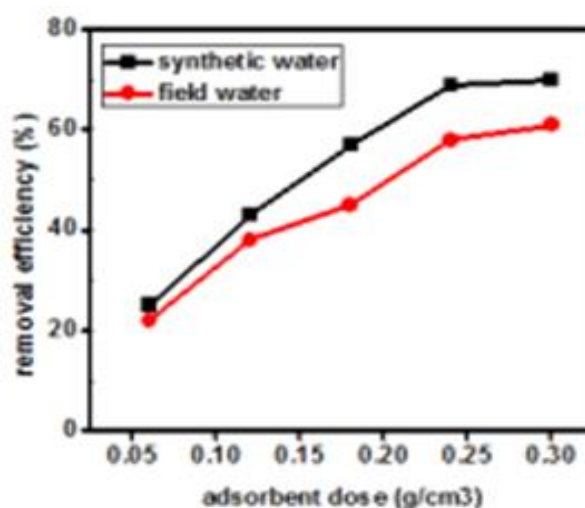


Figure 2.5.1.1 Effect of adsorbent dose on hardness removal by CSAC: contact time: 4 hours, hardness 864mg/L, pH: 6.3, temperature: 303K

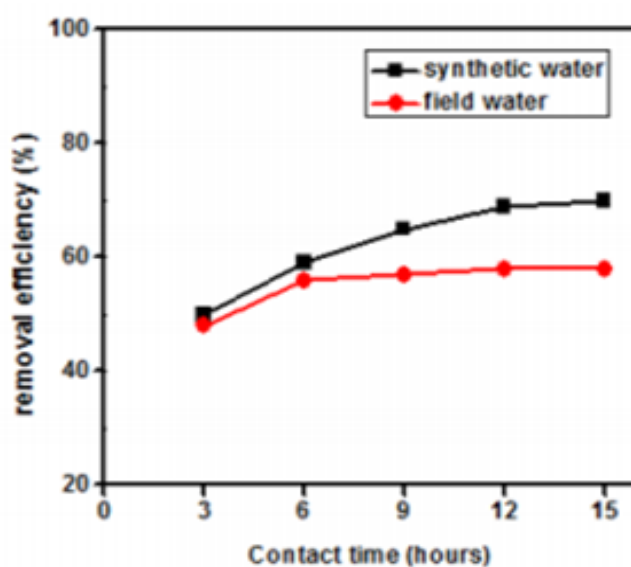


Figure 2.5.1.2 Effect of contact time on hardness removal by CSAC: temperature : 303K, hardness 864mg/L, CSAC dose : 0.16g/cm³, pH : 6.3

2.5.2 Adsorption Efficiencies Of Calcium (II) Ion And Iron (II) Ion On Activated Carbon Obtained From Pericarp Of Rubber Fruit

From the data obtained from a study conducted by Panumati, S. et. Al., “Adsorption efficiencies of calcium (II) ion and iron (II) ion on activated carbon obtained from pericarp of rubber fruit”

Fraction (L)	[Ca ²⁺] (ppm)	Efficiency (%)
0.10	0.00	100.0
0.20	0.00	100.0
0.25	0.00	100.0
0.30	0.52	97.8
0.40	5.37	77.2
0.50	8.26	64.9
0.60	8.57	63.6
0.70	11.57	50.9
0.75	11.98	49.1
0.80	12.60	46.5
0.90	13.22	43.9
1.00	14.46	38.6
1.25	15.70	33.3
1.50	17.35	26.3
1.75	17.77	24.6
2.00	17.77	24.6
2.25	17.87	24.1
2.50	18.80	20.2
2.75	19.21	18.4
3.00	19.63	16.7
3.25	19.83	15.8
3.50	20.04	14.9
3.75	20.04	14.9
4.00	20.04	14.9
4.25	20.04	14.9
4.50	20.56	12.7
4.75	20.87	11.4
5.00	20.87	11.4

Figure 2.5.2.1 The volumes of collected Ca²⁺ (in L) solution after flowing through the column including their Ca²⁺ ion concentrations (in ppm) and the efficiencies (in %)

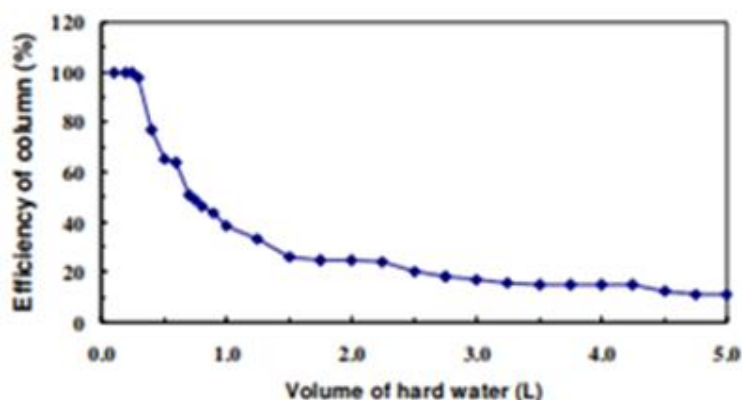


Figure 2.5.2.2 Efficiency of the column (in %) in relation to the volumes of collected solution of Ca^{2+} (in L).

The graph shown in the figure above is the adsorption efficiencies and collected volumes of Ca^{2+} ion solution plot. It was found that the lowest adsorption efficiency of 15g activated carbon for Ca^{2+} ion with a flow rate of 26 mL/min was 11.4% and the passed through volume of solution was 4.75 L. Therefore, we can use this result to estimate the expiry date of an activated carbon filter in the same way.

2.5.3 Effect of Activated Charcoal on the Rheological and Filtration properties of Water-based Drilling Fluids

According to the study conducted by Mahto, V., “Effect Of Activated Charcoal On The Rheological And Filtration Properties Of Water Based Drilling Fluids”, it is proven that adding activated carbon in our water based mud can decrease fluid loss and filter cake thickness that can be an advantage for our wellbore.

Table 2.5.3.1 Composition of Base Drilling Fluids

Constituents	Unit	Compositions of Drilling Fluids			
		A	B	C	D
Xanthan Gum	g/l	1	2	3	4
Polyanionic cellulose	g/l	3	3	3	3
Pregelatinized starch	g/l	30	30	30	30
Potassium Chloride	g/l	50	50	50	50

Table 2.5.3.2 Rheological Properties and Filtration Property of Base Drilling Fluids

Properties	Unit	Base Drilling Fluids			
		Mud A	Mud B	Mud C	Mud D
Apparent Viscosity	cp	20	30	32	38
Plastic Viscosity	cp	12	16	18	18
Yield Point	N/m ²	8	14	14	20
Initial Gel Strength	N/m ²	1.25	2.5	4	5
Final Gel Strength	N/m ²	1.75	4	6	7.5
API Fluid Loss	ml	20	20	20	20
Cake Thickness	cm	0.24	0.24	0.24	0.24

Table 2.5.3.3 Rheological Properties and Filtration Property of Developed Water Based Drilling Fluids after Addition of 30 G/L (3% W/V) Activated Charcoal in Base Drilling Fluids

Properties	Unit	Drilling Fluid Combinations			
		Mud A + 3% Activated Charcoal	Mud B + 3% Activated Charcoal	Mud C + 3% Activated Charcoal	Mud D + 3% Activated Charcoal
Apparent Viscosity	cp	21	31	33	40
Plastic Viscosity	cp	14	18	18	20
Yield Point	N/m ²	7	13	15	20
Initial Gel Strength	N/m ²	1.3	2.5	4.5	6
10 Min. Gel Strength	N/m ²	2.5	4	6.5	9
API Fluid Loss	ml	5.5	5.5	5	5
Cake Thickness	cm	0.025	0.025	0.025	0.025

2.6 STATISTICAL ANALYSIS

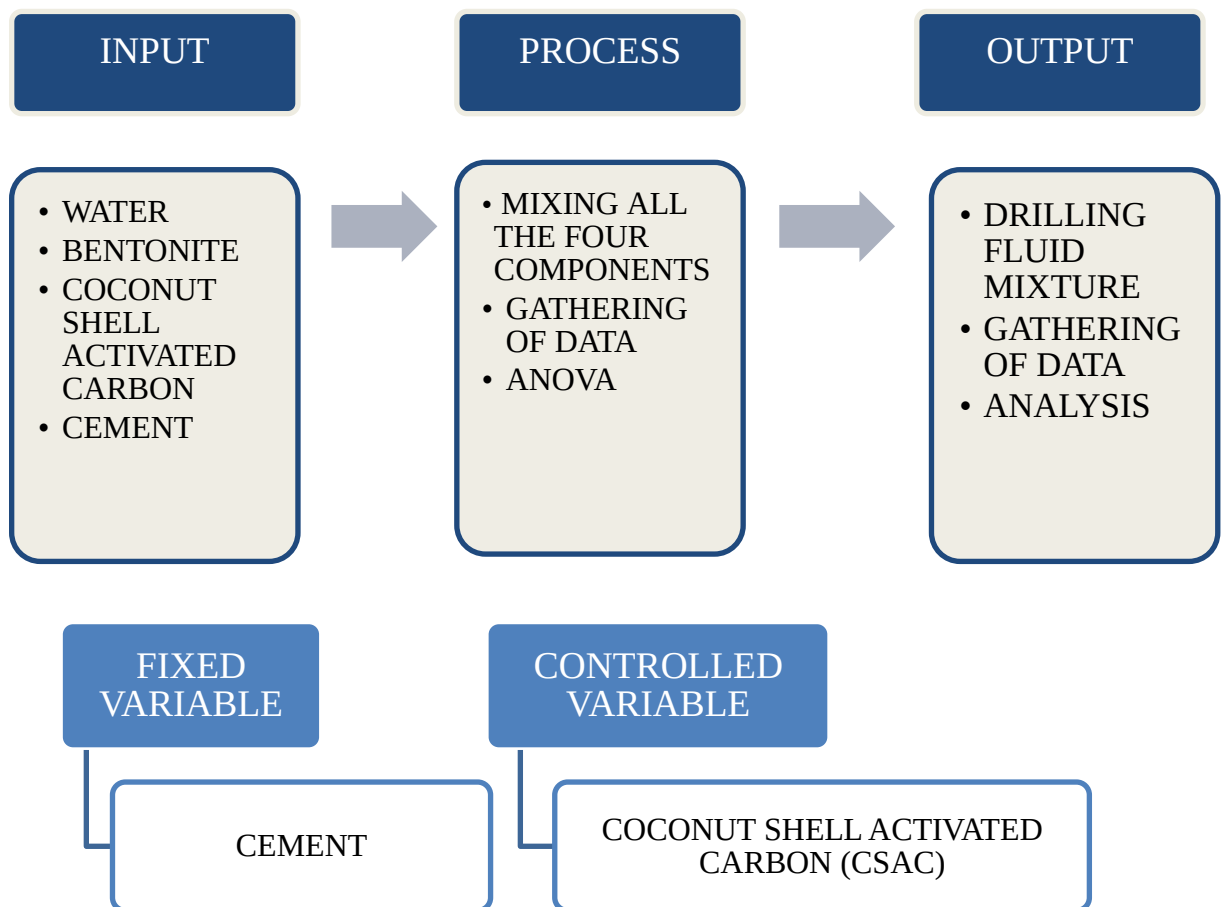
In the analysis of our density and viscosity the researchers used One-Way Anova in analyzing the result. The calcium-ion contamination was subjected to a regression analysis. ANOVA is short for “**AN**alysis **Of** **V**ariance”. The main purpose of an ANOVA is to test if two or more groups differ from each other significantly in one or more characteristics. For some statisticians the ANOVA doesn’t end there – they assume a cause effect relationship and say that one or more independent, controlled variables (the factors) *cause* the significant difference of one or more characteristics. The way this works is that the factors sort the data points into one of the groups and therefore they cause the difference in the mean value of the groups.

Regression analysis, on the other hand, is a powerful statistical method that allows you to examine the relationship between two or more variables of interest. While there are many types of regression analysis, at their core they all examine the influence of one or more independent variables on a dependent variable.

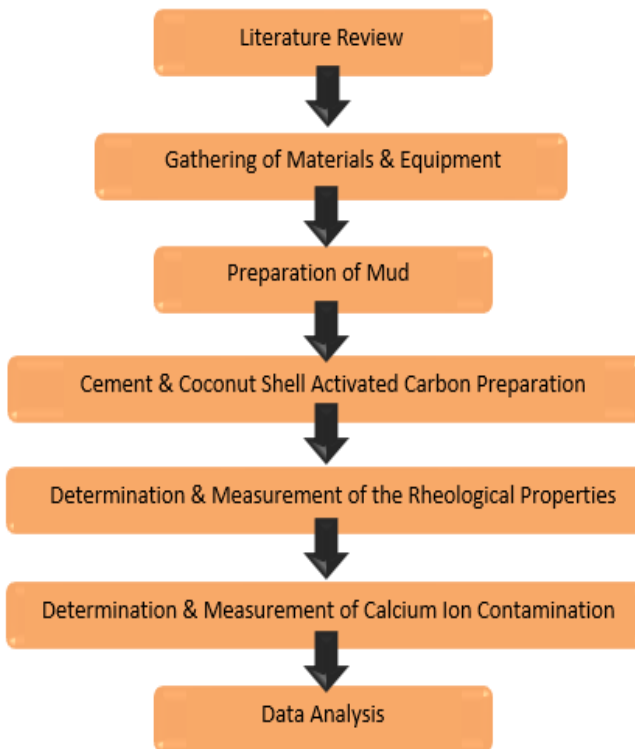
Chapter 3

THEORETICAL FRAMEWORK**3.1 CONCEPT OF THE RESEARCH**

Water-based drilling mud was prepared with Coconut Shell Activated Carbon and Portland cement based on the API standards for drilling mud. Parameters such as viscosity and density were determined. The drilling mud was tested based on API recommended methods.

3.2 RESEARCH PARADIGM

RESEARCH PROCESS FLOWCHART



3.3 DEFINITION OF TERMS

ACTIVATED CARBON - charcoal that has been heated or otherwise treated to increase its adsorptive power.

ANHYDRITE - a white mineral consisting of anhydrous calcium sulfate. It typically occurs in evaporite deposits.

ANOVA - used to determine significant difference between groups of data.

BENTONITE - a kind of absorbent clay formed by the breakdown of volcanic ash, used especially as a filler.

CALCIUM - is the fifth most abundant element in seawater and is very reactive by itself in nature. It has a positive charge of 2 and it's a white metallic element that burns with a brilliant light

CARBON - the chemical element of atomic number 6, a nonmetal that has two main forms (diamond and graphite) and that also occurs in impure form in charcoal, soot, and coal.

CEMENT – the binding material in sedimentary rocks that precipitate between grains from pore fluids. Calcite and Quartz are common cement / forming minerals.

DENSITY - degree of consistency measured by the quantity of mass per unit volume.

MUD ADDITIVE - material added to drilling fluid to enhance its properties.

MUD BALANCE - measures mud weight and specific gravity

MUD WEIGHT - density of mud, often in lb/ft³.

VISCOSITY - a quantity expressing the magnitude of internal friction, as measured by the force per unit area resisting a flow in which parallel layers unit distance apart have unit speed relative to one another.

WATER-BASED MUD - is a type of drilling mud consisting mainly of water, which has additives to modify it and make it more effective.

3.4 RESEARCH HYPOTHESIS

1) The addition of Coconut Shell Activated Carbon is not effective as a potential treatment for Calcium Ion Contaminated Water-based Drilling mud but is proven to be significant in reducing Calcium Ion Contamination.

2) There is a significant difference in the parameters obtained on the water-based drilling mud with the addition of Coconut Shell Activated Carbon.

3.4 CALCULATIONS

CALCULATION FOR TOTAL HARDNESS

- ❖ Cement: 0.3 g
- ❖ Calcium Ion Content in Cement: 46.09%
- ❖ Bentonite Density: $0.593\text{g/cm}^3 = 593\text{g/L}$
- ❖ Water: 0.35 L
- Calcium Ion Content
 - ➔ $0.3\text{ g} (0.4609) = 0.13827\text{ g} = \mathbf{138.27\text{ mg Ca}^{2+}}$
- Mud Volume
 - ➔ $(0.35\text{ L of Water}) + [(21\text{ g})/(593\text{g/L})\text{ of Bentonite}] = \mathbf{0.3854\text{L mud}}$
- **TOTAL HARDNESS**

The total hardness is obtained through the ratio of calcium ion content and the mud volume

 - ➔ $138.27\text{ mg Ca}^{2+} : 0.3854\text{ L Mud}$
 - ➔ **$358.770\text{ mg Ca}^{2+}/1\text{L Mud}$**

**** The calculation above is based from API SP 13-A 18th EDITION and 2.3.1:**

Calcium Ion Contaminantion

Chapter 4

MATERIALS AND METHODS**4.1 MATERIALS, TOOLS AND INSTRUMENTS**

Table 4.1.1 Equipment and Apparatuses used to perform the experiment

EQUIPMENT / APPARATUS	SPECIFICATIONS	SOURCE
Viscometer	Brookfield D2VT (up to 200 RPM)	PSU Malampaya Laboratory
Mud Balance	OFITE Metal Mud Balance 4-Scale: #115-00	PSU Malampaya Laboratory
Electronic Balance	Kern Electronic Scales, 3.5kg Capacity Type C – Type G – British 3 – pin	PSU Malampaya Laboratory
Beaker (250 mL)	PYREX®Volumetric Glassware	PSU Malampaya Laboratory
Glass Stirring Rod	PYREX®Volumetric Glassware	PSU Malampaya Laboratory
Standard API Filter Press	OFITE Filter Press with CO ₂ Pressure Assembly: #140-10	The Proponents
Blender (agitator)	FUKUDA FBL-158Pib 3-Speed Blender (30W, 60Hz, AC220V~240v)	The Proponents
Atomic Absorption Spectrometer (AAS)		Optimal Laboratories, Inc.

Table 4.1.2 Materials and reagents used to perform the experiment

MATERIALS	SPECIFICATIONS	SOURCE
Distilled Water (5.25 L)	Wilkins Distilled Drinking Water	SM Mall Puerto Princesa City
Bentonite Powder (315 g)	Water District Bentonite	Puerto Princesa City Water

	Powder (315 g)	District
Coconut Shell Activated Carbon (81 g)	Powdered Coconut Shell Activated Carbon	Millen Organics Trading -- Quezon City
Cement (3.6)	Portland Cement Type 1	Johndee Merchandising – Puerto Princesa City, Palawan

4.2 RESEARCH METHOD AND DESIGN

RESEARCH PROCEDURE

A. Acquisition of Coconut Shell Activated Carbon

The Powdered Coconut Shell Activated Carbon (500g) was ordered from Millen Organics Trading located in Quezon City in the month of March.

B. Acquisition of Cement

The Portland cement type 1 (1kg) was bought from Johndee Merchandising.

C. Bentonite-water Suspension

The bentonite clay in this research is supplied by Puerto Princesa City Water District.

A base bentonite-water suspension is prepared for the purpose of this study. The mud is based on the API standard for mid-viscous drilling mud. Bentonite concentration is six percent weight by volume (w/v) as a base composition.

Standard Procedure in formulating raw drilling mud:

- 1) Weigh 21g of bentonite using electronic balance and measure 350 mL distilled water using a beaker.
- 2) Add the bentonite slowly to the water and continuously stir it to prevent aggregation of the clay.
- 3) Mix the mixture using blender for 30 minutes.
- 4) Put the Mud in a clean plastic container and let it settle for 24 hours.

We have 2 Set-up. First set-up will be the Raw Drilling Mud Composition and the Second set-up will be the Drilling Mud Composition together with Cement and Coconut Shell Activated Carbon.

Table 4.2.1 Raw Drilling Mud Composition (SET-UP 1)

Raw Drilling Mud	DRILLING MUD COMPOSITION	
	Distilled Water	Bentonite Clay
	350 mL	21g

Table 4.2.2 Drilling Mud Composition with Coconut Shell Activated Carbon and Cement (SET-UP 2)

SAMPLE NUMBER	DRILLING MUD COMPOSITION			
	Distilled Water	Bentonite Clay	Cement (46.09% calcium)	Coconut Shell Activated Carbon (3% by weight from raw drilling mud)
1	350 mL	21 g	0.3 g	0 g
2	350 mL	21 g	0.3 g	6 g
3	350 mL	21 g	0.3 g	9 g
4	350 mL	21 g	0.3 g	12 g

D. Measurement of Rheological Properties

The Quantification of the parameters were patterned after the API Recommended Practice (American Petroleum Institute, 2009).

SETUP 1

- 1) From a clean container, transfer the Mud in a beaker.
- 2) Measure and Record their Specific Gravity using the mud balance. Calculate for Density.

- 3) Immerse the rotary sleeve #2 of the Viscometer (Brookfield DV2T) in the indicated line in the Mud.
- 4) After, subject the mixture to a speed of 30, 60 and 150 rpm. Record their corresponding viscosity readings.
- 5) Repeat steps 1-4 for trials 2 and 3.

SETUP 2

For concentration of 0 g Coconut Shell Activated Carbon (Trial 1)

- 1) Add 0.3 g of Cement to the mud slowly. Mix it for 30 minutes using blender to ensure a homogenous mixture.
- 2) Put the mixture in the beaker.
- 3) Measure and Record their Specific Gravity using the mud balance. Calculate for Density.
- 4) Immerse the rotary sleeve #2 of the digital viscometer in the indicated line in the bentonite-water suspension.
- 5) After, subject the mixture to a speed of 30, 60 and 150 rpm. Record their corresponding viscosity readings.
- 6) Repeat Steps 1-5 for trials 2 and 3.
- 7) Mix all three (3) trials
- 8) Filter out the mixture using API Filter Press (Ofite). Measure and record filtrate and Cake Thickness.

9) Use Atomic Absorption Spectroscopy to measure the amount of Calcium ions in the mixture. Record the Result.

For concentrations of 6 to 12 g Coconut Shell Activated Carbon (Trial 2-4)

- 1) Add 6 grams Coconut Shell Activated Carbon and pour it in the Mud slowly.
- 2) Mix the mixture using blender for 30 minutes.
- 3) Add 0.3 g of Cement and add it to the mud slowly. Mix it for 30 minutes using blender to ensure a homogenous mixture.
- 4) Put the mixture in the beaker.
- 5) Measure and Record their Specific Gravity using the mud balance. Calculate for Density.
- 6) Immerse the rotary sleeve #2 of the digital viscometer in the indicated line in the bentonite-water suspension.
- 7) After, subject the mixture to a speed of 30, 60 and 150 rpm. Record their corresponding viscosity readings.
- 8) Repeat steps 1-7 for trials 2 and 3.
- 9) Mix all three (3) trials
- 10) Filter out the mixture using API Filter Press (OFITE). Measure and record their filtrate and Cake Thickness.
- 11) Use Atomic Absorption Spectroscopy to measure the amount of Calcium ions in the mixture.
- 12) Repeat for steps 1 to 11 for increasing increments of 3 g Coconut Shell Activated Carbon (6g-12g)

Table 4.2.3 DATA PRESENTATION**A) Data Analysis of Rheological Properties of Mud**

VISCOSITY (cp)	30 rpm			
	Trial 1	Trial 2	Trial 3	Average Viscosity
RAW DRILLING MUD	X	X	X	X
SAMPLE 1	X	X	X	X
SAMPLE 2	X	X	X	X
SAMPLE 3	X	X	X	X
SAMPLE 4	X	X	X	X

VISCOSITY (cp)	60 rpm			
	Trial 1	Trial 2	Trial 3	Average Viscosity
RAW DRILLING MUD	X	X	X	X
SAMPLE 1	X	X	X	X
SAMPLE 2	X	X	X	X
SAMPLE 3	X	X	X	X
SAMPLE 4	X	X	X	X

VISCOSITY (cp)	150 rpm			
	Trial 1	Trial 2	Trial 3	Average Viscosity
RAW DRILLING MUD	X	X	X	X
SAMPLE 1	X	X	X	X
SAMPLE 2	X	X	X	X
SAMPLE 3	X	X	X	X
SAMPLE 4	X	X	X	X

DENSITY (kg/L)				
	TRIAL 1	TRIAL 2	TRIAL 3	Average Density
RAW DRILLING MUD	X	X	X	X
SAMPLE 1	X	X	X	X
SAMPLE 2	X	X	X	X
SAMPLE 3	X	X	X	X
SAMPLE 4	X	X	X	X

B) Data Analysis of Cake Thickness, Filtrate Volume and Calcium Ion Content after Filtration

	Raw Drilling Mud	Sample 1 (0.3g cement)	Sample 2 (0.3g cement & 6g CSAC)	Sample 3 (0.3g cement & 9g CSAC)	Sample 4 (0.3g cement & 12g CSAC)
Cake Thickness (mm)	X	X	X	X	X
Filtrate Volume (mL)	X	X	X	X	X
Calcium Ion Content (mg/L)	X	X	X	X	X

Chapter 5

RESULTS AND DISCUSSION

This chapter presents the results, the analysis of data, and the interpretation of results addressing the objectives of the study.

5.1 DATA GATHERING OF RHEOLOGICAL PROPERTIES OF MUD

Table 5.1.1 Values obtained for Viscosity at 30 rpm

VISCOSITY (cp)	30 rpm			
	Trial 1	Trial 2	Trial 3	Average Viscosity
RAW DRILLING MUD	23	28	24	25
SAMPLE 1 (0.3g cement)	248	248	221	239
SAMPLE 2 (0.3g cement & 6g CSAC)	361	305	349	338.33
SAMPLE 3 (0.3g cement & 9g CSAC)	421	501	405	442.33
SAMPLE 4 (0.3g cement & 12g CSAC)	434	498	430	454

Table 5.1.2 Values obtained for Viscosity at 60 rpm

VISCOSITY (cp)	60 rpm			
	Trial 1	Trial 2	Trial 3	Average Viscosity
RAW DRILLING MUD	14.50	12	14	13.5
SAMPLE 1 (0.3g cement)	116.5	110	104.60	110.366
SAMPLE 2 (0.3g cement & 6g CSAC)	192	202	187	193.66
SAMPLE 3 (0.3g cement & 9g CSAC)	195	236	208	213
SAMPLE 4 (0.3g cement & 12g CSAC)	241	196	250	229

Table 5.1.3 Values obtained for Viscosity at 150 rpm

VISCOSITY (cp)	150 rpm			
	Trial 1	Trial 2	Trial 3	Average Viscosity
RAW DRILLING MUD	11.40	9	11.40	10.6
SAMPLE 1 (0.3g cement)	57.80	59.30	55	57.37

SAMPLE 2 (0.3g cement & 6g CSAC)	68	70	74.3	70.77
SAMPLE 3 (0.3g cement & 9g CSAC)	94.60	91.9	103	96.5
SAMPLE 4 (0.3g cement & 12g CSAC)	101.5	153	132	128.833

Table 5.1.4 Values obtained for Density

DENSITY (g/cm3)				
	TRIAL 1	TRIAL 2	TRIAL 3	Average Density
RAW DRILLING MUD	1.185	1.185	1.180	1.1833
SAMPLE 1 (0.3g cement)	1.19	1.19	1.19	1.19
SAMPLE 2 (0.3g cement & 6g CSAC)	1.21	1.20	1.21	1.2067
SAMPLE 3 (0.3g cement & 9g CSAC)	1.22	1.22	1.22	1.22
SAMPLE 4 (0.3g cement & 12g CSAC)	1.23	1.23	1.23	1.23

Data Analysis of Cake Thickness, Filtrate Volume and Calcium Ion Content after Filtration

Table 5.1.5 Values obtained for Cake thickness, Filtrate Volume and Calcium Ion Content

	Raw Drilling Mud	Sample 1 (0.3g cement)	Sample 2 (0.3g cement & 6g CSAC)	Sample 3 (0.3g cement & 9g CSAC)	Sample 4 (0.3g cement & 12g CSAC)
Cake Thickness (mm)	7	14	12	12	10
Filtrate Volume (mL)	128	176	157	155	144
Calcium Ion Content (mg/L)	0	54.425	45.426	43.950	32.754

Figure 5.1.1 Graph of Viscosity at 30 rpm

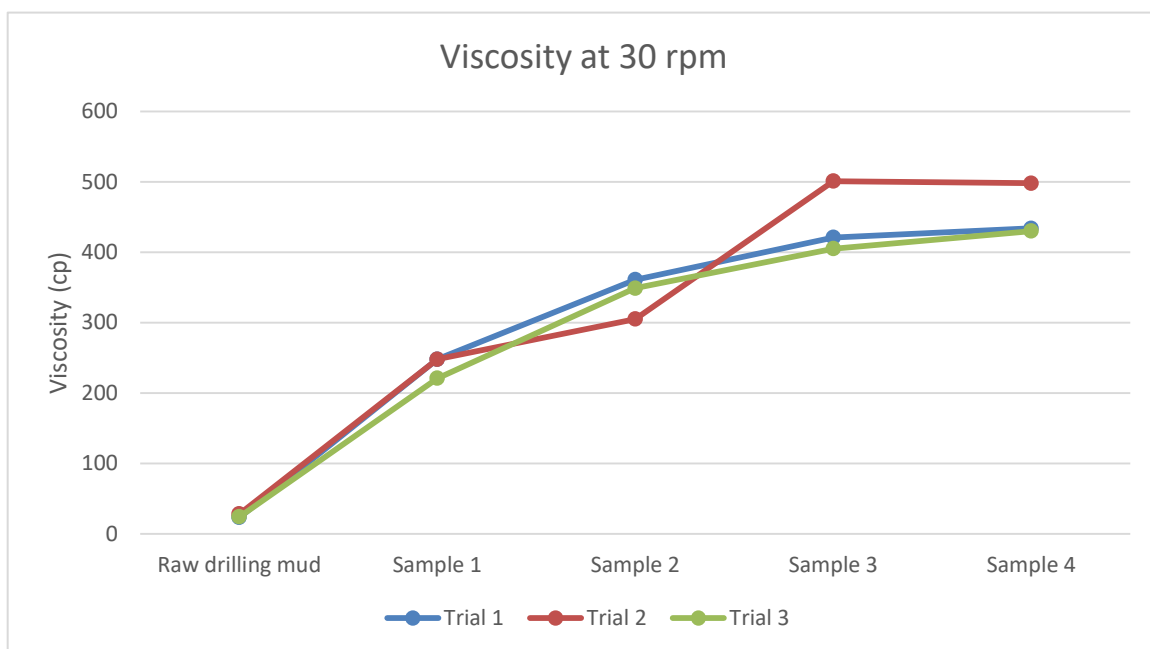


Figure 5.1.2 Graph of Viscosity at 60 rpm

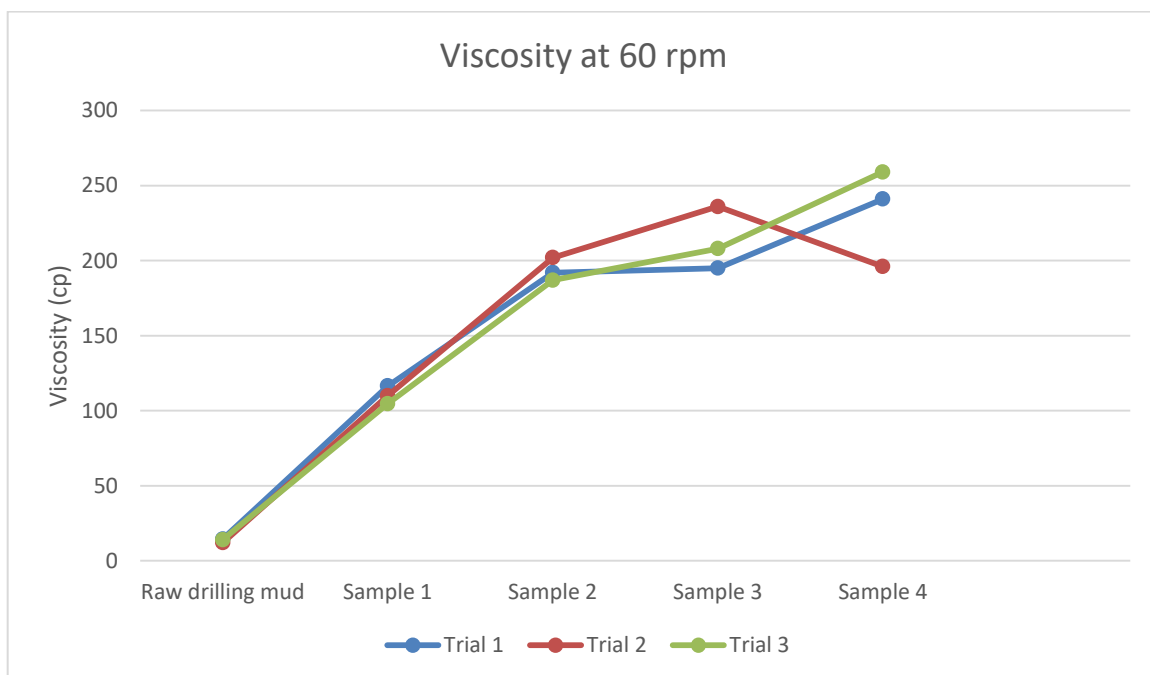


Figure 5.1.3 Graph of Viscosity at 150 rpm

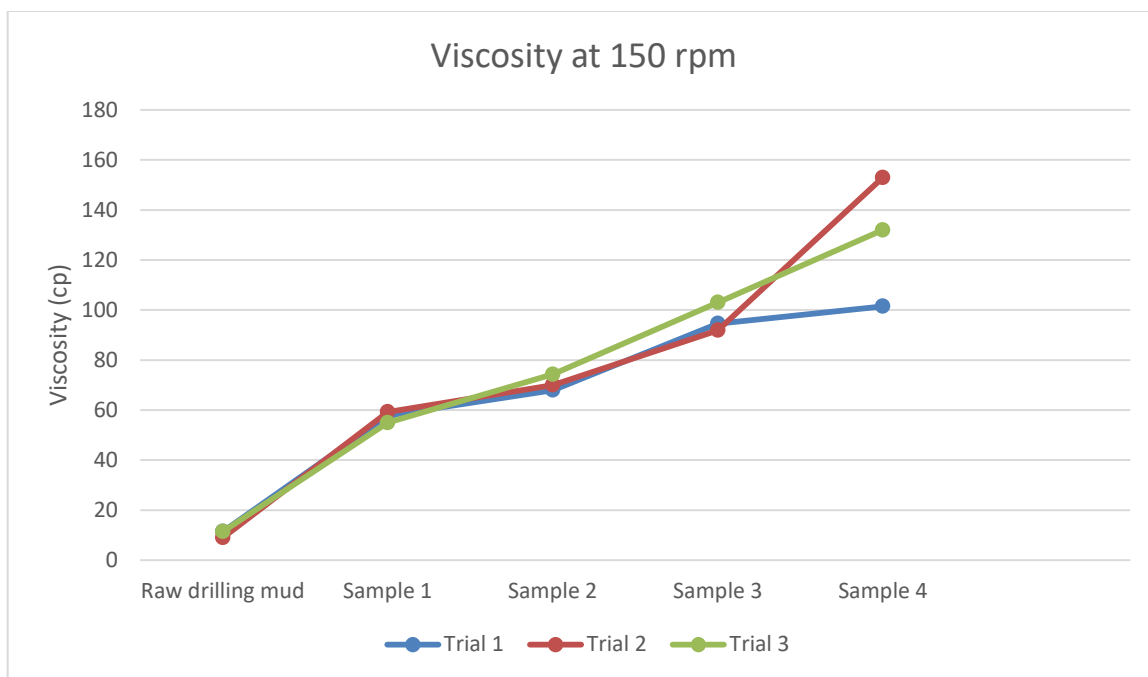


Figure 5.1.4 Graph of Values of Density

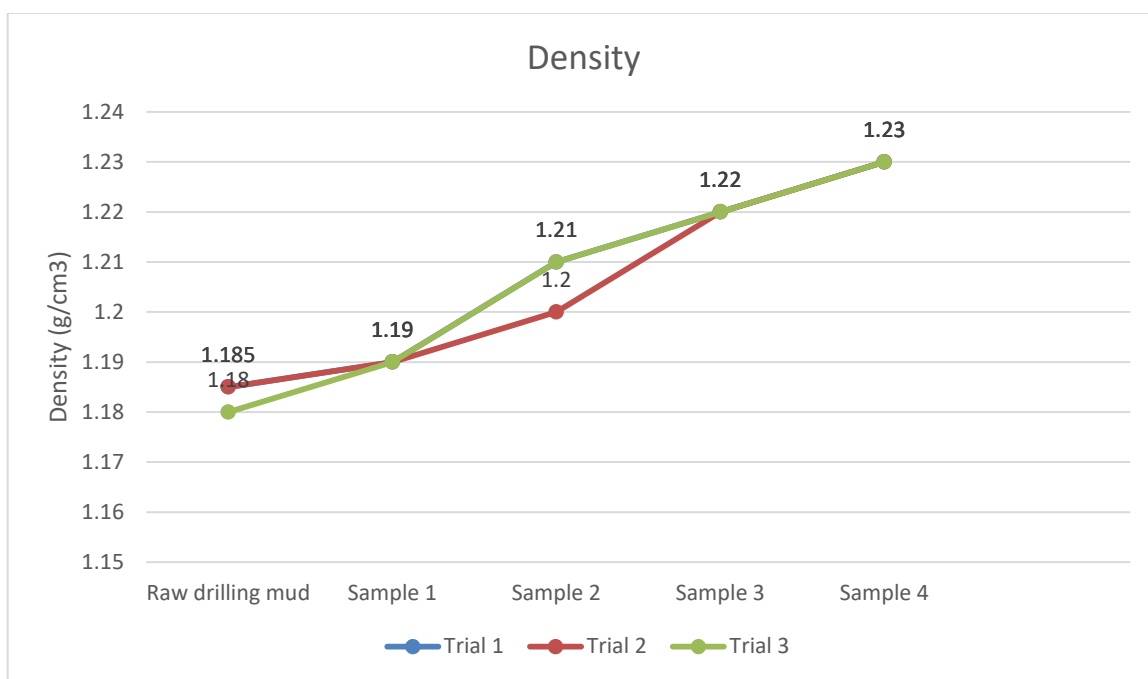
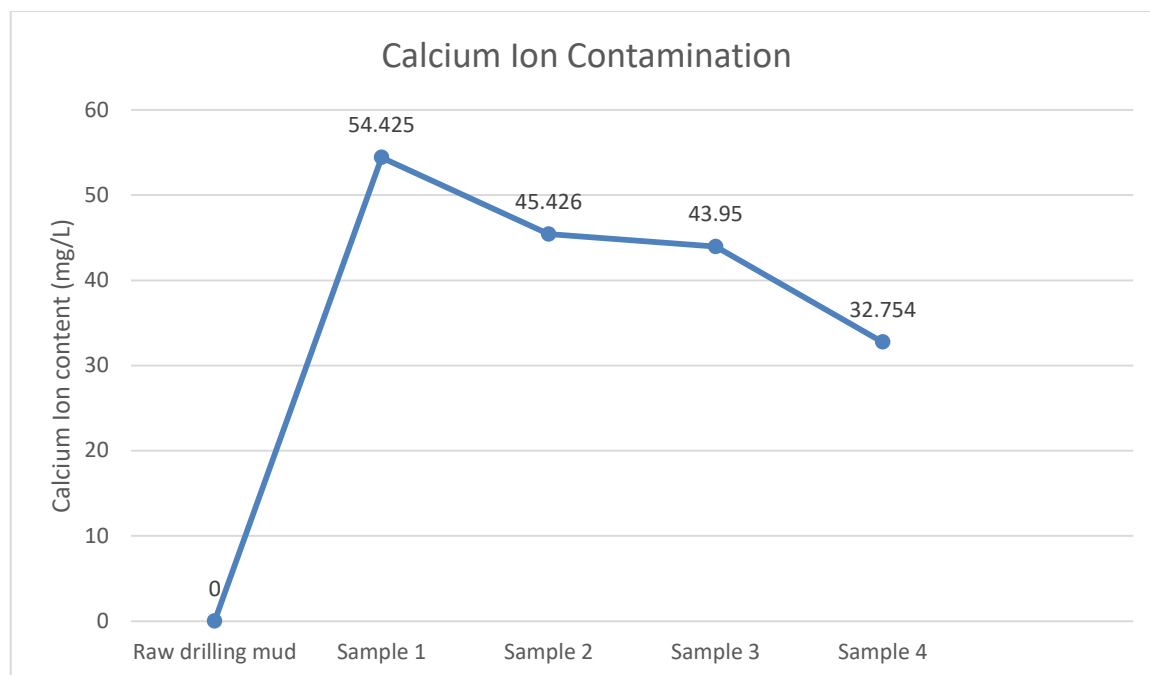


Figure 5.1.5 Graph of Calcium Ion Contamination



5.2 EVALUATION OF THE RHEOLOGICAL PROPERTIES OF RESEARCH TREATMENTS

For this study, Viscosity and Density formed part of the rheological properties of the different sample sets prepared for experimentation.

Table 5.2.1 Viscosity of the Different Research Treatments at different measurement speeds.

Treatments	Average Viscosity (cp)		
	30 rpm	60 rpm	150 rpm
Raw Drilling Mud	25.00	13.50	10.60
Sample 1	239.00	110.37	57.37

Sample 2	338.33	193.66	70.77
Sample 3	442.33	213.00	96.50
Sample 4	454.00	229.00	128.83

Table 5.2.2 Summary of ANOVA Result on Viscosity

Speed	F computed value	F critical	p-value
30 rpm	89.636	3.478	0.000
60 rpm	88.314	3.478	0.000
90 rpm	40.626	3.478	0.000

Result of the ANOVA test on the research treatments' viscosities reveal all computed F values are greater than the constant F critical equal to 3.478. This implies that the differences in the viscosities among the different treatments are statistically significant. This further means that there are enough observed data to affirm that the different sample sets have varying viscosities at different speeds of data collection. As observed in the computed average viscosities, the application of CSAC at different amounts produce different viscosities as well.

Having the p-value lower than 5% validates this result, which means that there is enough evidence to say that the results are not merely products of chance or coincidence.

To know specifically which among the different sets vary in viscosity, a post-hoc paired t-test presents the results summarized in Table 5.2.2.

Table 5.2.3 Summary of Paired T-test Results for the Viscosity of different experimental preparations

p-value	Raw	Sample 1	Sample 2	Sample 3	Sample 4
Raw	NA				
Sample 1	0.071	NA			
Sample 2	0.072	0.079	NA		
Sample 3	0.071	0.070	0.106	NA	
Sample 4	0.060	0.052	0.023	0.106	NA

As revealed in Table 5.2.3, all experimental Samples have statistically higher viscosities than the controlled (i.e. Raw). All probabilities of chance from the said result are lower than 10%, which is considered the acceptable percentage for committing coincidental results.

Moreover, the same table shows that all Samples with applied CSAC result to higher viscosities compared to Sample 1, which do not contain CSAC. All resulting p-values here are less than 10%.

On the other hand, the viscosities of Sample 2 and Sample 3 are considered not significantly different. The same goes with the viscosities of Sample 3 and Sample 4. However, the viscosities of Sample 2 and Sample 4 are considered statistically different, based on the acceptance level of 10%

Table 5.2.4. Summary of Regression Analysis between the amount of Coconut Shell Activated Carbon (CSAC) and the resulting Viscosity

Speed	Correlation r	r squared	p-value
30 rpm	0.925	0.855	0.000
60 rpm	0.926	0.857	0.000
90 rpm	0.868	0.755	0.000

As revealed in Table 5.2.4, the relationship between the amounts of CSAC applied to the samples have strong positive correlation with the resulting viscosities. This means that there is significant direct proportionality between the two variables: the amount CSAC and Viscosity. Furthermore, this implies that the more CSAC is applied to the contaminated drilling mud; a higher viscosity could be expected as a result.

Results also show that at least 75% of the resulting viscosities are influenced by the amount of CSAC. Other variables take the remaining percentage as they are considered neutral in this study.

Figure 5.2.1 Scatterplot of the amount of CSAC and Viscosity

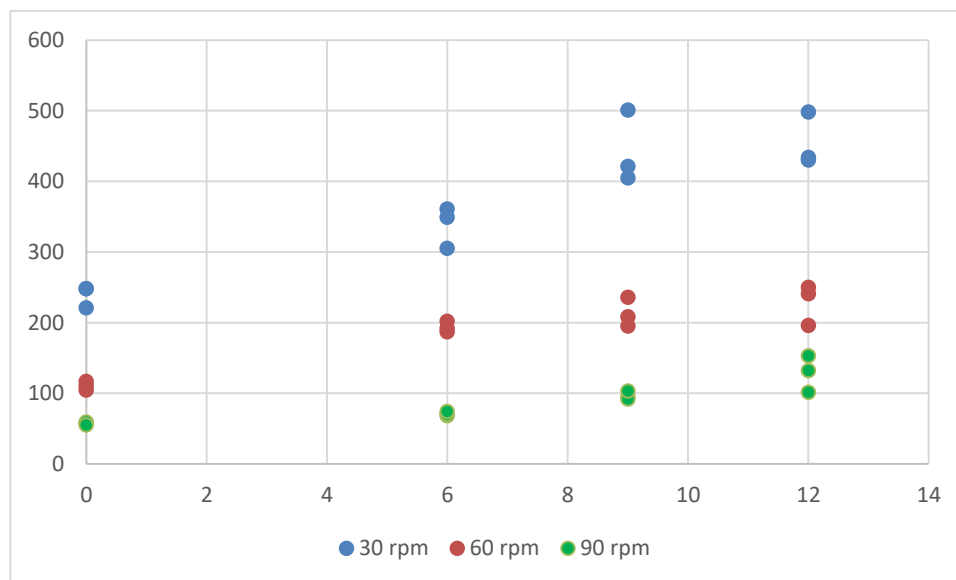


Table 5.2.5. Density of the Different Research Treatments.

Treatments	Average Density (Kg/L)
Raw Drilling Mud	1.18
Sample 1	1.19
Sample 2	1.20
Sample 3	1.22
Sample 4	1.23

Table 5.2.6 Summary of ANOVA Result on Density and Regression Analysis between the amount of CSAC and Density

F computed value	F critical	p-value
138.8	3.478	0.000
Correlation r	r squared	p-value
0.983	0.966	0.000

For Densities, both ANOVA and regression analysis report that different amounts of CSAC produce a different density. The correlation of these two variables is similar to that with viscosity, which presented strong positive correlations or direct proportions. Similarly, the higher the amount of CSAC applied, the higher the resulting density becomes.

Figure 5.2.2 Scatterplot of the amount of CSAC and Density

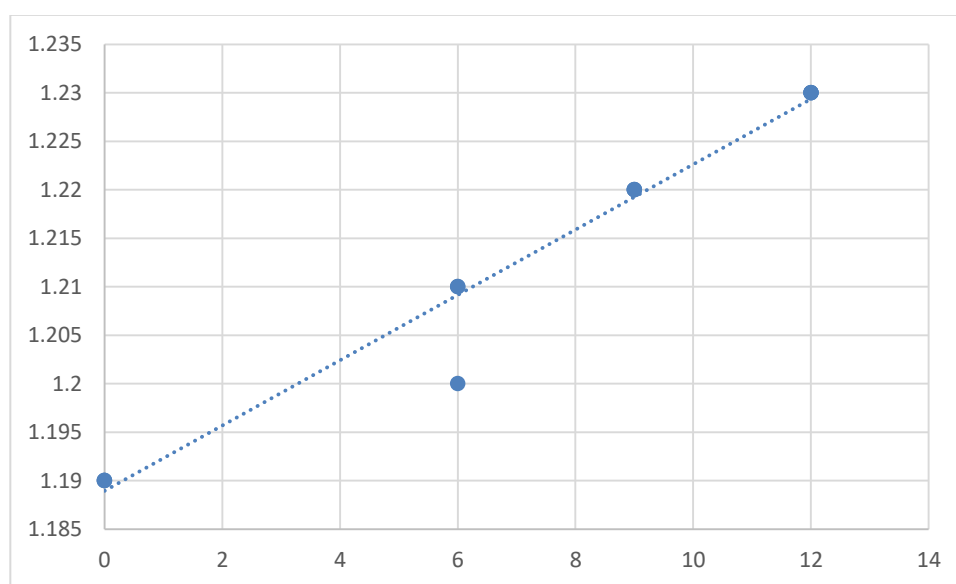


Table 5.2.6 Cake Thickness of the Different Research Treatments.

Treatments	Cake Thickness (mm)
Raw Drilling Mud	7
Sample 1	14
Sample 2	12
Sample 3	12
Sample 4	10

5.3 EVALUATION OF THE CAPACITY OF THE COCONUT SHELL ACTIVATED CARBON (CSAC) AS A POTENTIAL TREATMENT TO CALCIUM-ION CONTAMINATED DRILLING MUD

Treatments	Calcium Ion Contamination (Mg/L)
Raw Drilling Mud	0
Sample 1	54.425
Sample 2	45.426
Sample 3	43.950
Sample 4	32.754

For the purpose of fair analysis, a regression test was administered to the different research Samples with varying amounts of CSAC and the gathered calcium ion contamination. Data on the raw treatment was excluded in the analysis.

Summary of Regression Analysis between Amount of CSAC and Calcium
Contamination

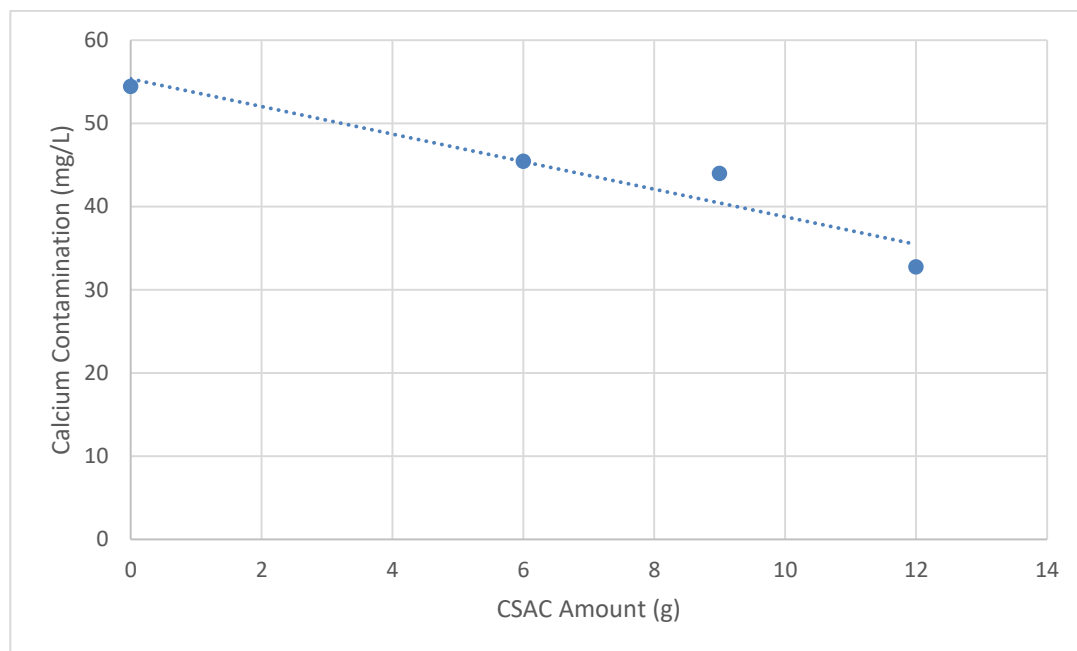
Correlation r	r squared	p-value
-0.955	0.913	0.044

Result of the regression analysis reveal a very strong negative correlation between the amount of CSAC and the gathered calcium ion contamination. This means that the two variables have indirect proportionality, which means that a higher amount of CSAC corresponds to a lower calcium ion contamination or that a lower amount of CSAC can be associated to a higher level of calcium ion contamination. To simply put, the higher the amount of CSAC applied to a contaminated drilling mud, the lower its contamination gets.

In addition, the computed r-squared reveals that about 91.3% of the variations in the levels of calcium ion contamination are influenced by the amount of amount CSAC used in the treatment. The other 8.7% may have been influences of other variables, which were considered neutral in this study.

All the said Regression Analysis results are considered statistically significant having a p-value lower than 10%. This implies that there is enough evidence to claim that the observation on the indirect proportionality of the amount of CSAC and the level of calcium ion contamination is not by chance alone, and thus could be generalized.

Figure 5.3.1 Scatterplot of the amount of CSAC and calcium ion contamination



The results of the regression analysis are further affirmed by the scatterplot shown above with a goodness-of-fit line sloping downward to the right, which resembles indirect proportionality.

Chapter 6

SUMMARY, CONCLUSION, AND RECOMMENDATION

This chapter presents the summary of the study and the findings presented in Chapter 5, conclusions based from the objectives of the study, and the recommendations indicating possible improvements for future studies involving Coconut Shell Activated Carbon as potential treatment for Calcium Ion Contamination.

6.1 SUMMARY OF THE STUDY

The study aimed to evaluate the capability of Coconut Shell Activated Carbon (CSAC) as a potential treatment to calcium ion contamination in water-based drilling mud at standard laboratory conditions and to evaluate the density, viscosity, and cake thickness with different amount of Coconut Shell Activated Carbon (CSAC) and with the same amount of Calcium-Ion Content. To achieve the goal, the proponents prepared the water-based drilling mud by gradually adding the bentonite to the distilled water. Coconut Shell Activated Carbon (CSAC) with varying concentrations: 6 gram, 9 gram, 12 gram and a fixed 0.3 gram of cement were added to the mud and were tested using digital viscometer, mud balance, API Filter Press, Atomic Absorption Spectroscopy (direct air-acetylene flame). The data from the experiment were analyzed with One-way ANOVA and Regression test to statistically prove any significant effects.

6.2 SUMMARY OF FINDINGS

The major findings of the study are the following:

- 1) From the four (4) samples which is contaminated by calcium, Sample 4 exhibits the best among the trials. The higher amount of the Coconut Shell Activated Carbon (CSAC) corresponds to a lower calcium ion contamination.
 Sample 1 (.03 g Ca^{++} , 0 g CSAC) – 54.425 mg/L
 Sample 2 (.03g Ca^{++} , 6 g CSAC) – 45.426 mg/L
 Sample 3 (.03g Ca^{++} , 9 g CSAC) – 43.950 mg/L
 Sample 4 (.03g Ca^{++} , 12 g CSAC) – 32.754 mg/L
- 2) It was statistically analyzed that the effects of activated carbon in a mixture of calcium contaminated water-based drilling mud is significant when it comes to improving rheological properties and adsorbing calcium ions but not efficient enough to fight off the contamination to be industrially accepted as a treatment.
- 3) The researchers observed that lumps of bentonite clay were flocculated in the waste mud after samples were subjected to the API filter press. The researchers consider that due to this contributing factor, the assumed amount of calcium contamination from Sample 1 was not met.

6.3 CONCLUSIONS

Based on the results of regression Analysis, calcium contamination and CSAC are indirectly proportional. This means that the higher the amount of CSAC applied to a contaminated water-based drilling mud, the lower its contamination gets. But when it

comes to treating a Calcium Ion Contaminated water-based drilling mud industrially speaking, it is not effective.

From the Evaluation of the Rheological Properties, such as viscosities at three rotational speeds: 30 rpm, 60rpm, and 150rpm and density readings implies that the differences in the viscosity and density of water-based drilling mud of 6% bentonite among the different samples are statistically significant. Thus, the addition of varying amounts of Coconut Shell Activated Carbon (CSAC) has a significant effect on the rheological properties of the water-based drilling mud. As the amount of CSAC added increased, the viscosities and densities of the water-based drilling mud also increased. The cake thickness decreased as the amount of CSAC increased which is an advantage.

6.4 RECOMMENDATIONS

The results of the study show that as the amount of CSAC increases, the amount of Calcium-ion adsorbed also increases. This study used Powdered Activated Carbon, for a potentially better result, Granular Activated Carbon is highly recommended as it has a higher surface area. Since the results of the study showed an increasing effect, more amount of activated carbon can also be used for better for a treatment-evaluation of calcium-ion contamination. Additives, and more rheological modifiers to water-based drilling mud can also be used in regards to setting more parameters in the future studies related to ours. Hence, other kind of drilling fluids can also be a subject of prospect.

GLOSSARY OF TECHNICAL TERMS

AAS - Atomic absorption spectroscopy is a spectro-analytical procedure for the quantitative determination of chemical elements using the absorption of optical radiation (light) by free atoms in the gaseous state.

Activated carbon - is a form of carbon that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption and chemical reactions.

Adsorption - is the adhesion of atoms, ions or molecules from a gas, liquid or dissolved solid to a surface.

Amorphous - lacking a crystalline structure, something without solid form.

API- American Petroleum Institute, trade association sponsored by the oil and gas industry and is recognized worldwide

Bulk density- is the weight of soil in a given volume.

Carbonate minerals- those minerals containing the *carbonate* ion

Coagulation- the action or process of a liquid, especially blood, changing to a solid or semi-solid state.

Density- mass per unit volume

Drilling fluids- fluids used in operations to drill boreholes

Filter cake - substances that are retained on a *filter*

Filtrate - liquid or solution that has passed through a filter

Flocculation - refers to the process by which fine particulates are caused to clump together into a floc.

Fluid loss - leakage of the liquid phase of drilling fluid, slurry or treatment fluid containing solid particles into the formation matrix

Fluid-loss control - means of controlling (usually lowering) the volume of filtrate that passes through a filter medium

Gel strength - A measure of the ability of a colloidal dispersion to develop and retain a gel form, based on its resistance to shear.

PH - Hydrogen ion potential

Plastic viscosity - resistance to flow of a fluid

Pore Volume - defined as the ratio of a porous material's air volume to a porous materials total volume

Rheology - scientific field that encompasses the flow phenomena of matter (solids, liquids, and gases) and notably involves time-dependent behavior under the influence of stresses.

Specific Gravity - dimensionless ratio of the density of a material to that of the same volume of water

Surface Area -The total *area* of the *surface* of a three-dimensional object.

Viscometer – measures viscosity of fluid

Viscosity - fluids' resistance to flow

Water hardness - the amount of dissolved calcium and magnesium in the water.

Yield point- stress required to start fluid movement

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APPENDICES

Appendix A - Documentation

	The bentonite (105 g) was weighed using a digital scale with high sensitivity. To do this, a plastic cup is first placed on the scale and is tared to zero before placing the powder and obtaining the reading. This process was repeated with 3 trials per sample.
	The cement (0.3 g) was weighed using a digital scale with high sensitivity. To do this, a plastic cup is first placed on the scale and is tared to zero before placing the powder and obtaining the reading. This process was repeated with 3 trials per sample.
	The Coconut Shell Activated Carbon (6 g, 9 g, 12 g) was weighed using a digital scale with high sensitivity. To do this, a plastic cup is first placed on the scale and is tared to zero before placing the powder and obtaining the reading. This process was repeated with 3

	trials per sample.
	350 mL of Distilled Water is measured by pouring the water in a beaker to get an accurate volume reading.
	The bentonite was added to the water while mixing to avoid formation of lumps.

	
	The Coconut Shell Activated Carbon (CSAC) was added to the bentonite-water mixture.
	The cement was carefully added to the mixture.
	Specific Gravity was measured using Mud Balance.

	
	The Viscometer (Brookfield DV2T model) was immersed in the indicated line in the bentonite-water suspension. After, the mixture is subjected to a speed of 30, 60 and 150 rpm using Needle #2. Their corresponding viscosity readings were recorded.
	The API Filter Press (Ofite model) was used to filter out the Mud to obtain Cake Thickness and Filtrate Volume.

Appendix B – Statistical Analysis

ANOVA – Test of Significant Differences in Viscosity at 30 rpm

<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	374369.6	4	93592.4	89.63644	8.63E-08	3.47805
Within Groups	10441.33	10	1044.133			
Total	384810.9	14				

ANOVA – Test of Significant Differences in Viscosity at 60 rpm

<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	96904.96	4	24226.24	88.31465	9.27E-08	3.47805
Within Groups	2743.173	10	274.3173			
Total	99648.13	14				

ANOVA – Test of Significant Differences in Viscosity at 60 rpm

<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	23437.76	4	5859.439	40.62623	3.74E-06	3.47805
Within Groups	1442.28	10	144.228			
Total	24880.04	14				

Post-hoc Paired T-test Results between different experimental preparations

Statistical Measures	Pair 1		Pair 2		Pair 3		Pair 4	
	Raw	Sample 1	Raw	Sample 2	Raw	Sample 3	Raw	Sample 4
Mean	16.3	140.7667	16.3	207	16.3	236.8667	16.3	258.8333
Variance	36.07	9485.663	36.07	21631	36.07	27948.85	36.07	27877.58
Observations	3	3	3	3	3	3	3	3
Pearson Correlation	0.999		0.985		0.999		0.986	
Hypothesized Mean Difference	0		0		0		0	
df	2		2		2		2	
t Stat	-2.359		-2.340		-2.370		-2.608	
P(T<=t) one-tail	0.071		0.072		0.071		0.060	
t Critical one-tail	2.920		2.920		2.920		2.920	
P(T<=t) two-tail	0.142		0.144		0.141		0.121	
t Critical two-tail	4.303		4.303		4.303		4.303	

Statistical Measures	Pair 5		Pair 6		Pair 7	
	Sample 1	Sample 2	Sample 1	Sample 3	Sample 1	Sample 4
Mean	140.7667	207	140.7667	236.8667	140.7667	258.8333
Variance	9485.663	21631	9485.663	27948.85	9485.663	27877.58
Observations	3	3	3	3	3	3
Pearson Correlation	0.992		1.000		0.992	
Hypothesized Mean Difference	0		0		0	
df	2		2		2	
t Stat	-2.205		-2.385		-2.865	
P(T<=t) one-tail	0.079		0.070		0.052	
t Critical one-tail	2.920		2.920		2.920	
P(T<=t) two-tail	0.158		0.140		0.103	

t Critical two-tail	4.303	4.303	4.303
---------------------	-------	-------	-------

Statistical Measures	Pair 8		Pair 9		Pair 10	
	Sample 2	Sample 3	Sample 2	Sample 4	Sample 3	Sample 4
Mean	207	236.8667	207	258.8333	236.8667	258.8333
Variance	21631	27948.85	21631	27877.58	27948.85	27877.58
Observations	3	3	3	3	3	3
Pearson Correlation	0.992		1.000		0.992	
Hypothesized Mean Difference	0		0		0	
df	2		2		2	
t Stat	-1.806		-4.511		-1.809	
P(T<=t) one-tail	0.106		0.023		0.106	
t Critical one-tail	2.920		2.920		2.920	
P(T<=t) two-tail	0.213		0.046		0.212	
t Critical two-tail	4.303		4.303		4.303	

Regression Statistics – Amount of CSAC and Viscosity at 30 rpm

Multiple R	0.925
R Square	0.855626
Adjusted R Square	0.841188
Standard Error	38.32821
Observations	12

ANOVA

	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	87062.4	87062.4	59.26437	1.65E-05
Residual	10	14690.51	1469.051		

Total	11	101752.9
-------	----	----------

Regression Statistics – Amount of CSAC and Viscosity at 60 rpm

Multiple R	0.926141
R Square	0.857737
Adjusted R Square	0.843511
Standard Error	19.88986
Observations	12

ANOVA

	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	23852.04	23852.04	60.29232	1.53E-05
Residual	10	3956.066	395.6066		
Total	11	27808.11			

Regression Statistics – Amount of CSAC and Viscosity at 90 rpm

Multiple R	0.868975
R Square	0.755117
Adjusted R Square	0.730629
Standard Error	15.92933
Observations	12

ANOVA

	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	7824.39	7824.39	30.83581	0.000243
Residual	10	2537.437	253.7437		
Total	11	10361.83			

ANOVA – Test of Significant Differences in Density

<i>Source of Variation</i>	<i>SS</i>	<i>df</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F crit</i>
Between Groups	0.004627	4	0.001157	138.8	1.02E-08	3.47805
Within Groups	8.33E-05	10	8.33E-06			
Total	0.00471	14				

Regression Statistics – Amount of CSAC and Density

Multiple R	0.983338
R Square	0.966954
Adjusted R Square	0.963649
Standard Error	0.003024
Observations	12

ANOVA

	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	0.002675	0.002675	292.6042	9.84E-09
Residual	10	9.14E-05	9.14E-06		
Total	11	0.002767			

Regression Analysis for Amount of CSAC and Calcium Contamination

Regression Statistics

Multiple R	0.9556874
R Square	0.9133385
Adjusted R Square	0.8700077
Standard Error	3.2053286
Observations	4

ANOVA

	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	216.5611	216.5611	21.07829	0.044313
Residual	2	20.54826	10.27413		
Total	3	237.1094			

Appendix C – Computation of the Concentration from Optimal Laboratory

Calcium Ion Content Result

COMPUTATION

The following computation were based and proportioned from the result given by the Optimal Laboratories.

SAMPLE NUMBER	Ca ²⁺⁺ Contamination	Original Amount of Filtrate
SAMPLE 1	27.368 mg/L	176 mL
SAMPLE 2	20.377 mg/L	157 mL
SAMPLE 3	19.457 mg/L	155 mL
SAMPLE 4	13.476 mg/L	144 mL

SAMPLE 1

$$\frac{27.368 \text{ mg}}{1\text{L}} = \frac{x}{0.350\text{L}}$$

$$1\text{L} \quad 0.350\text{L}$$

$$x = 9.5788 \text{ mg Ca}^{2++}$$

$$\text{Original Concentration: } 9.5788 \text{ mg} / 0.176 \text{ L} = \mathbf{54.425 \text{ mg Ca}^{2++} / \text{L}}$$

SAMPLE 2

$$\frac{20.377 \text{ mg}}{1\text{L}} = \frac{x}{0.350\text{L}}$$

$$x = 7.13195 \text{ Ca}^{2++}$$

$$\text{Original Concentration: } 7.13195 \text{ mg} / 0.157 \text{ L} = \mathbf{45.4264 \text{ mg Ca}^{2++} / \text{L}}$$

SAMPLE 3

$$\frac{19.457 \text{ mg}}{1\text{L}} = \frac{x}{0.350\text{L}}$$

$$x = 6.80995 \text{ mg Ca}^{2++}$$

$$\text{Original Concentration: } 6.80995 \text{ mg} / 0.155 \text{ L} = \mathbf{43.935 \text{ mg Ca}^{2++} / \text{L}}$$

SAMPLE 4

$$\frac{13.476 \text{ mg}}{1\text{L}} = \frac{x}{0.350\text{L}}$$

$$x = 4.7166 \text{ mg Ca}^{2++}$$

$$\text{Original Concentration: } 4.7166 \text{ mg} / 0.144 \text{ L} = \mathbf{32.754166 \text{ mg Ca}^{2++} / \text{L}}$$

Appendix D – Optimal Laboratory Calcium Ion Content Result

F-MR-04-13163
Rev. 04



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Department of Environment and Natural Resources
DA - Bureau of Animal Industry
Department of Health
Food and Drug Administration

CERTIFICATE OF ANALYSIS

Page 1 of 1

Customer: GABRIEL DOMINIC B. TRESPACES

Address: 431 Abad Santos St., Koronadal City, South Cotabato

Job Order No: 18-09-88491

Date Sampled: September 14, 2018

Date Received: September 20, 2018

Date Analyzed: September 20 - 27, 2018

Date Reported: September 27, 2018

Time Sampled: 4:00 PM

Sampled by: Customer

RESULTS OF ANALYSIS:

Type of Sample: Water - Trial 1 18-2383		Sample ID: 18-192134
PARAMETER	METHOD	RESULT
Calcium	Direct Air-Acetylene Flame	27.3680 mg / L

Sample Description/Condition: The sample is clear and received in plastic bottle transported without ice.

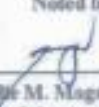
Reference/s: Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2012

Certified True and Correct by:



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Laboratory Analyst II
PRC Lic. # 001-2904

Noted by:



Enelle M. Maglino, R. Ch.
Laboratory Head
PRC Lic. # 000-7584

Approved by:



Jennifer R. Maralit, R. Ch.
General Manager
PRC Lic. # 990-7374

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Page 1 of 1

Customer: **GABRIEL DOMINIC B. TRESPACES**
Address: **431 Abad Santos St., Koronadal City, South Cotabato**

Job Order No: **18-08-89491**
Date Sampled: **September 14, 2018**
Date Received: **September 20, 2018**
Date Analyzed: **September 20 - 27, 2018**
Date Reported: **September 27, 2018**
Time Sampled: **8:00 PM**
Sampled by: **Customer**

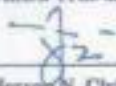
RESULTS OF ANALYSIS:

Type of Sample: Water - Trial 2 18-2364	Sample ID: 18-192135	
PARAMETER	METHOD	RESULT
Calcium	Direct Air-Acetylene Flame	20.3770 mg / L

Sample Description/Condition: The sample is clear and received in plastic bottle transported without ice.

Reference/s: Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2012

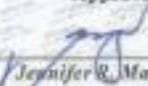
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Approved by:


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F-MR-04-13/83
Rev. 04

Recognition:

Department of Environment and Natural Resources
DA - Bureau of Animal Industry
Department of Health
Food and Drug Administration

CERTIFICATE OF ANALYSIS

Page 1 of 1

Customer: **GABRIEL DOMINIC B. TRESPACES**
Address: **431 Abad Santos St., Koronadal City, South Cotabato**

Job Order No: **18-09-89491**
Date Sampled: **September 14, 2018**
Date Received: **September 20, 2018**
Date Analyzed: **September 20 - 27, 2018**
Date Reported: **September 27, 2018**
Time Sampled: **6:00 PM**
Sampled by: **Customer**

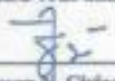
RESULTS OF ANALYSIS:

Type of Sample: Water - Trial 3 18-2365	Sample ID: 18-182136	
PARAMETER	METHOD	RESULT
Calcium	Direct Air-Acetylene Flame	19.4570 mg / L

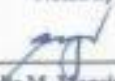
Sample Description/Condition: The sample is clear and received in plastic bottle transported without ice.

Reference/s: Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2012

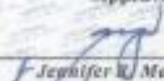
Certified True and Correct by:


Jerson N. Chiraco, R. Ch.
Laboratory Analyst II
PRC Lic. # 001-2304

Noted by:


Enelle M. Magallon, R. Ch.
Laboratory Head
PRC Lic. # 000-7384

Approved by:


Jennifer R. Maralit, R. Ch.
General Manager
PRC Lic. # 006-7174

Results of analysis refer only to the sample of material submitted by client. This report/certificate cannot be reproduced without written authorization from Optimal Laboratories, Incorporated and will be on file for 5 years from date of issue.

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Physico-chemical Analysis for Foods, Feeds & Pharmaceutical Products
Microbiological Testing / Stability Testing
Water and Wastewater Analysis & Monitoring
Ambient Air / Stationary Source Emission Sampling & Analysis
Workplace Environment Measurement (WEM)

LTR No **179804**

F-MR-04-13183
Rev. 04

Recognition:

Department of Environment and Natural Resources
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Food and Drug Administration

CERTIFICATE OF ANALYSIS

Page 1 of 1

Customer: GABRIEL DOMINIC B. TRESPACES
Address: 431 Abad Santos St., Koronadal City, South Cotabato

Job Order No: 18-09-89491
Date Sampled: September 14, 2018
Date Received: September 20, 2018
Date Analyzed: September 20 - 27, 2018
Date Reported: September 27, 2018
Time Sampled: 7:00 PM
Sampled by: Customer

RESULTS OF ANALYSIS:

Type of Sample: Water - Trial 4 18-2386		Sample ID: 18-192137
PARAMETER	METHOD	RESULT
Calcium	Direct Air-Acetylene Flame	13.4760 mg / L

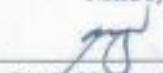
Sample Description/Condition: The sample is clear and received in plastic bottle transported without ice.

Reference/s: Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 2012

Certified True and Correct by:


Jerson M. Cirasco, R. Ch.
Laboratory Analyst II
PRC Lic. # 001-7304

Noted by:


Eniela M. Magano, R. Ch.
Laboratory Head
PRC Lic. # 000-7384

Approved by:


Jennifer K. Maralit, R. Ch.
General Manager
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Results of analysis refer only to the sample of material submitted by client. This report/certificate cannot be reproduced without written authorization from Optimal Laboratories, Incorporated and will be on file for 5 years from date of issue.

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Water and Wastewater Analysis & Monitoring
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Workplace Environment Measurement (WEM)

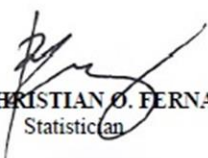
LTR No: 179805

Appendix E – Statistical Analysis Certification

CERTIFICATION

This certifies that the statistical treatment and analysis implemented in the study entitled, "Coconut Shell Activated Carbon (CSAC) as a Potential Treatment to Calcium-Ion Contaminated Drilling Mud", prepared and submitted by De Los Santos, K., et. al., have been reviewed and validated by the undersigned.

Issued this 3rd day of October 2018.


RUSSELL CHRISTIAN O. FERNANDEZ
Statistician

Appendix F – English Language Certification**C E R T I F I C A T I O N**

This is to certify that the manuscript entitled, “Coconut Shell Activated Carbon (CSAC) as a Potential Treatment for Calcium-Ion Contaminated Drilling Mud”, by De Los Santos, K.M.E., Ong, R.M.C. and Trespeces, G.D.B., has been edited and proofread. Neither the research content nor the proponents’ intentions were altered in any way during the editing process.

Issued this 16th day of October 2018.



Dr. Marie Vic Cabrera-Velasco
English Critique