UNIVERSITI PUTRA MALAYSIA

USE OF CASSAVA DERIVATIVE IN WATER BASED DRILLING MUD

RAHELEH SAMAVATI

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By

RAHELEH SAMAVATI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

May2016

DEDICATIONS

I wish to dedicate this work to my father, Dr. Heshmatollah Samavati, who has been a constant source of support and encouragement during the challenges of graduate school and life. Who have always loved me unconditionally and whose good examples have taught me to work hard for the things that I aspire to achieve.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia, in fulfillment of the requirements for the degree of Doctor of Philosophy

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Chairman: Associate Professor Norhafizah Abdullah, PhD
Faculty: Engineering

Drilling mud is a mixture of clays, chemicals and water applied in the drilling operation. The mud is pumped down the drill hole to achieve various functions such as cooling and lubricating the drill bits, flushing out the cuttings and strengthening the hole stability. The use of drilling mud in oilfield is often challenged by a number of factors, which require mud to withstand extreme temperatures and pressure without losing its functional integrity. Due to their excessive polluting characteristic, they are subjected to increasing number of waste management regulation and reinforcement. Biopolymers such as starch from agriculture based are reported to be a good replacement, but hampered by their thermal instability and low shear tolerability. To date, none of these polymers can be used as fluid loss and temperature reducing agent in mud formulation. Besides, they also reportedly failed to function after one pass of mud circulation system. The use of biodegradable polymer is desirable due to their environmental friendly, non-toxic, cheap and easily available for industrial application.

The objective of this study is to investigate and optimize starch from cassava as alternative option to a more expensive commercially applied starch counterpart, such as corn, potato and wheat. Various derivatives from cassava was used, namely *ubi kayu*, *elubo garri* (yellow garri), *ijebu garri* (white garri) and *fufu*. All the experiments were carried out according to the National Iranian South Oil Company and American Petroleum Institute Standards set for the actual oil-field drilling condition. The influence of starch modification and usage of carbon black and gilsonite as additives for thermal stability enhancement and fluid loss reduction of water based muds (WBM), in extreme drilling temperatures was also investigated.

The WBM was prepared by suspending different starch in saturated salt water, followed by addition of weighting materials. The WBM samples were formulated in different mud weights (light, average and heavy) intended for various borehole sizes of an actual drilling operation set-up. The WBM samples were subjected to drilling environment by placing them in a hot rolling oven for 8hrs. Four sets of temperatures were used; 200, 250, 275 and 300 □F, representing the actual drilling hole temperature. After the hot roll, fluid loss and rheological (plastic viscosity, yield point, apparent viscosity, gel strength (10s and 10min) properties of each WBM samples was analyzed.

Results revealed that at temperatures of 200 and 250 °F, cassava derivatives used in light, average and heavy mud weights of WBM formulations were acceptable as a fluid loss agent. However, the light weighted WBM formulation failed at higher temperature of 275 °F, unlike the average and heavy WBMs. At 300 °F, all of them were rejected in all WBM weights formulations. Therefore, to improve the thermal stability and fluid loss control of cassava WBM at this temperature, starch was subjected to acid modification step prior to mud preparation. Results from the WBM containing modified starch showed insignificant improvement of the fluid loss property.

Another option to enhance thermal stability of WBM at 300 °F was by adding thermal enhancer agent. In this study, carbon black (CB) and CB+ gilsonite blends were used as a thermal enhancer agents. Results showed, the addition of 1% CB and CB+ gilsonite blends into the WBM formulation successfully reduce the fluid loss at 300 °F, at 98% and 99%, respectively. The average WBM weight formulation with CB addition was qualified as a fluid loss agent, while in the heavy WBM weight samples, only *fufu* and *ubi kayu* were qualified. When the mud was added with CB+ gilsonite, similar results were observed for light and average WBM weight formulations, whereas only *ubi kayu* and yellow *garri* were qualified in the heavy WBM weight formulations. Cassava derivatives showed its potential to be industrially applied as a fluid loss additive in WBM intended for the drilling well temperature of 250 °F. For extreme drilling temperature of 300 °F, the addition of CB+ gilsonite to the WBM formulation successfully improved the fluid loss control to an acceptable range. The overall assessment among all the starches investigated showed that the *ubi kayu* presented a superior functionality as fluid loss agent in WBM formulations.

In summary, all starches used in this study failed API Standard as fluid loss control agent in WBM. Temperature of water use in preparing mud base does not affect rheological properties of WBM. The rheological properties of cassava-WBM samples in various formulation differs when subjected to different drilling temperatures. The acid modification of cassava derivatives improved WBM performance at HTHP circumstances. The addition of carbon black and gilsonite to the WBM formulation, further enhanced the thermal stability and fluid loss control of WBM. In short, this study found that cassava derivatives exhibited commercial potential as fluid loss agent in WBM.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGGUNAN CASSAVA TERBITAN DALAM LUMPUR GERUDI BERASASKAN AIR

Oleh

RAHELEH SAMAVATI

Mei 2016

Pengerusi: Profesor Madya Norhafizah Abdullah, PhD
Fakulti: Kejuruteraan

Lumpur gerudi adalah campuran tanah liat, bahan kimia dan air yang digunakan dalam operasi penggerudian. Lumpur dipam ke dalam lubang gerudi, dengan tujuan untuk mencapai pelbagai fungsi seperti penyejukan dan pelinciran mata gerudi, curahan keluar keratan dan mengukuhkan kestabilan lubang. Penggunaan lumpur gerudi di medan explorasi minyak sering dicabar oleh beberapa faktor, yang memerlukan ketahanan lumpur pada suhu lampau dan tekanan yang lampau tanpa kehilangan integriti fungsi yang dinyatakan di atas. Oleh kerana ciri-ciri pencemaran yang berlebihan, ianya tertakluk kepada peningkatan jumlah peraturan pengurusan sisa dan penguatkuasaan. Biopolimer seperti kanji dari sumber pertanian dilaporkan berpotensi menjadi pengganti alternatif yang baik, tetapi dihalang oleh ketidakstabilan terma dan toleransi ricih yang rendah. Setakat ini, tiada satu pun biopolimer boleh digunakan sebagai agen kehilangan bendalir dan penurunan suhu dalam pembentukan lumpur gerudi. Selain itu, ianya juga dilaporkan gagal berfungsi selepas satu pas sistem peredaran lumpur gerudi. Polimer yang boleh biorosot adalah wajar digunakan kerana ianya mesra alam, tidak beracun, murah dan mudah didapati untuk aplikasi perindustrian.

Tujuan penyelidikan ini adalah untuk menyelidik dan mengoptimakan penggunaan kanji dari ubi kayu sebagai pilihan alternatif daripada kanji komersial yang lebih mahal, seperti jagung, kentang dan gandum. Berbagai jenis ubi kayu digunakan seperti *ubi kayu*, *elubo garri* (garri kuning), *ijebu garri* (garri putih) dan *fufu*. Semua eksperimen telah dijalankan mengikut permatuhan *National Iranian South Oil Company* dan *American Petroleum Institute* yang ditetapkan untuk keadaan penggerudian medan minyak sebenar. Pengaruh pengubahsuaian kanji dan penggunaan karbon hitam dan gilsonit sebagai bahan tambahan untuk meningkatkan kestabilan terma dan pengurangan kehilangan bendalir dari lumpur dasar air (WBM) dalam suhu ekstrem penggerudian juga disiasat.
WBM telah disediakan dengan merampaikan kanji yang berbeza di dalam air garam tepu diikuti dengan penambahan bahan pemberat. Sampel WBM telah diformulasi pada berat lumpur yang berbeza (ringan, sederhana dan berat) bagi pelbagai saiz lubang gerudi dari persediaan operasi penggerudian sebenar. Sampel WBM telah diujilari

kepada persekitaran penggerudian dengan meletakkannya di dalam ketuhar guling panas selama 8 jam. Empat set suhu telah digunakan; 200, 250, 275 dan 300 °F, yang mewakili suhu lubang penggerudian sebenar. Selepas penggulian panas, kehilangan bendalir dan reologi (sifat kelikatan plastik, sifat takat alah, sifat kelikatan ketara, sifat kekuatan gel (10 s dan 10 min) bagi setiap sampel WBM dianalisis.

Keputusan menunjukkan bahawa pada suhu 200 dan 250 °F, derivatif ubi kayu dalam formulasi WBM yang ringan, sederhana dan berat, boleh diterima sebagai ejen kehilangan cecair. Walau bagaimanapun, formulasi WBM ringan gagal pada suhu 275 °F, sementara WBM sederhana dan berat boleh bertahan pada suhu ini. Walaubagaimanapun, pada suhu 300 °F, semuanya telah gagal berfungsi. Oleh itu, untuk meningkatkan kestabilan terma dan kawalan kehilangan bendalir WBM ubi kayu pada suhu ini, adalah disyorkan supaya langkah modifikasi asid terhadap kanji dilakukan sebelum penyediaan lumpur. Keputusan daripada WBM yang mengandungi kanji yang diubahsuai telah menunjukkan peningkatan pada kandungan kehilangan cecair yang ketara.

Pilihan lain bagi meningkatkan kestabilan terma WBM pada 300 °F adalah dengan menambah agen penambahbaik terma. Dalam kajian ini, karbon hitam (CB) dan sebatian CB-gilsonit digunakan sebagai agen penambahbaik terma. Hasil kajian menunjukkan, penambahan 1% pada CB dan sebatian CB-gilsonit ke dalam formulasi WBM telah berjaya mengurangkan kehilangan bendalir pada 300 F, pada 98 % dan 99 %, masing-masing. WBM formulasi sederhana yang mengandungi CB didapati layak sebagai ejen kehilangan bendalir, manakala sampel WBM formulasi berat, hanya *ubi kayu* dan *fufu* sahaja yang layak. Apabila lumpur ditambah dengan sebatian CB-gilsonit, keputusan yang sama diperhatikan bagi formulasi WBM yang ringan dan sederhana, manakala hanya *ubi kayu* dan *elubo garri* telah berkelayakan dalam WBM formulasi berat. Derivatif ubi kayu menunjukkan potensi untuk digunakan dalam proses perindustrian penggerudian sebagai bahan agen kehilangan bendalir dalam formulasi WBM yang digunakan untuk suhu penggerudian 250 °F. Bagi suhu penggerudian 300 °F, penambahan CB dan gilsonit kepada formulasi WBM telah berjaya meningkatkan kehilangan kawalan cecair kepada julat yang boleh diterima. Penilaian keseluruhan antara semua kanji yang disiasat menunjukkan bahawa *ubi kayu* berfungsi unggul sebagai ejen kehilangan bendalir dalam formulasi WBM.

Sebagai ringkasan, semua kanji didapati gagal berfungsi sebagai agen kawalan cecair di bawah piawaian *API*. Suhu air yang digunakan semasa penyediaan lumpur asas didapati tidak menjejaskan sifat reologi WBM. Sifat reologi sampel ubi kayu -WBM dalam pelbagai formulasi didapati berbeza apabila dikenakan suhu penggerudian yang berbeza. Pengubahsuaian asid terhadap derivatif ubi kayu telah meningkatkan prestasi WBM pada keadaan HTHP. Penambahan karbon hitam dan gilsonite turut meningkatkan lagi kestabilan haba dan kawalan kehilangan bendalir WBM. Secara amnya, derivatif ubi kayu berpotensi komersil sebagai ejen kehilangan cecair dalam WBM.

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Raheleh Samavati
May 2016

I certify that a Thesis Examination Committee has met on 04 May 2016 to conduct the final examination of Raheleh Samavati on her thesis entitled "Use of Cassava Derivative in Water-Based Drilling Mud" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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This is to confirm that:

- The research conducted and the writing of this thesis was under our supervision;
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°C	Degree Celsius
°F	Degree Fahrenheit
%	Percent
AHR	After Hot-Roll (Drilling)
$Al_2Si_2O_5(OH)_4$	Kaolinite
API	American Petroleum Institute
AV	Apparent Viscosity
Ba_2SO_4	Barite
BHR	before Hot-Roll (Drilling)
$CaCO_3$	Calcium Carbonate
$CaMg\ (CO_3)_2$	Dolomite
CaO	Limestone
CB	Carbon black
CMC	Carboxymethyl Cellulose
cP	Centipoise
eq	Equation
Fe_2O_3	Hematite
FLC	Filtrate Loss Control
GBM	Gas Based Mud
GS	Gel Strength
H_3O^+	Hydroxonium Ion
HCl	Hydrochloric Acid
HEC	Hydroxyethyl Cellulose
HTHP	High Temperature-High Pressure
IO	Isomerized Olefins
LAO	Linear Alpha Olefins
LP	Linear Paraffins
LTLP	Low Temperature-Low Pressure
$MgCO_3$	Magnesite
min	Minute
N	Normality
Na	Sodium
NaCl	Sodium Chloride
NaCMC	Carboxymethyl Cellulose
NaOH	Sodium Hydroxide
NISOC	National Iranian South Oil Co.
OBM	Oil Based Mud
PAC	Polyanionic Cellulose
PAO	Polyalphaolefin
PAM	Polyacrylamide
PDF	Pars Drilling Fluid Co.
PHPA	Partially-Hydrolyzed Polyacrylamide
POAM	Polyoxyalkyleneamine
PPG	Polypropylene Glycol
PV	Plastic Viscosity
rpm	Round Per Minute
s	Second
SBM	Synthatic Based Mud

WBM	Water Based Mud
WG	White Garri (*Ijebu garri*)
XG	Xanthan Gum
YG	Yellow Garri (*Elubo garri*)
YP	Yield Point
ϕ	Torque Dial Readings
v/v	Volume Per Volume
w/v	Weight Per Volume
g/cm^3	Gram Per Cubic Centimeter
g	Gram
µg	Microgram
µL	Microliter
µg/mL	Microgram Per Milliliter
µM	Micromolar
τ	Shear Stress
□	Shear Rate
□$_0$	Yield Point
□$_p$	Plastic Viscosity
V_s	Volume of Solid
ρ_s	Density of Solid
psi	Pounds Per Square Inch
sp.gr g/m^3	Specific Gravity
ppg	Pound Per Gallon
lb/gal	Pound Per Ggallon
pcf	Pound Per Cubic Foot
lb/ft^3	Pound Per Cubic Foot
$lb/100\ ft^2$	Pound Per 100 Square Foot
cm^3	Cubic Centimeter
cm^2	Square Centimeter
cc	Cubic Centimeter
m^3	Cubic Meter
ml	Milliliter
lb	Pound
ft^2	Square Foot
ft^3	Cubic Foot

CHAPTER 1

INTRODUCTION

1.1 Background

In recent years, there has been an immense mass of petroleum explorations accomplished by applying water based drilling mud (WBM) as favored drilling fluid. The main motive for this inclination is expenditure and environmental affinity. Conventional oil based drilling muds (OBM) manufactured from oils or diesel, besides being considerably more costly compared to WBM, are environmentally irreconcilable. Consequently, the application of OBMs have been restricted only to obligatory drilling situations. The efficiency of a drilling mud, particularly the additives are valued by standard measurement of specific characteristics of the formation. The viscosity (plastic and apparent), yield point, gel strength (10s and 10min), pH and filtrate loss characteristics of the mud systems are unerringly attributable to the functionality of the drilling mud (Darley and Gray, 1998).

Through time, the operational circumstances of oil/gas drilling and manufacturing have become progressively more excessive. For example, operational depth alterations, disposition of subterranean geohazards with mounting depth, intricacy of drilling process, structure of wellbore, and so on, impose a direct effect on drilling procedures. Furthermore, major alterations in the thermal, chemical and physical circumstances of deeper wells, limit the application of numerous conservative drilling muds above a definite operational set-point. Therefore, to accomplish the intentions and requirements of the drilling procedure, optimization of drilling fluids characteristics by specific ingredient customization, is obligatory (Benayada et al., 2003).

Fluid loss is an assess of the inclination of aqueous phase of a WBM to traverse the filtrate cake into the mud formation. The adequately low filtrate value and the declaration of a dilute filter cake with minute permeability are common vital aspects for the drilling mud fine performance (Caenn and Chillinger, 1996; Civan, 2007). The virtual assessments of these filtration individualities are reliant on the penetrated mud. Additionally, drilling muds can moreover contain preservatives that are designated to facilitate in achieving well-bore intensification by adjusting the fracture technicalities of the mud. During rotary drilling, drilling muds filtrate could be enforced into the contiguous ulterior formation, causing detrimental effects on drilling muds efficiency.

1.2 Problem Statement

Principally, various material and additives such as starches, cellulose, and polymers have been employed to manage the fluid loss requirements. However, these category of materials own certain limitations. For instance, starches and cellulose derivatives are thermally degraded when subjected to high temperatures (Darley and Gray, 1988). Whereas, polymers have precincts in high concentration of salts and valence cation contagions in addition to their high market price and unavailability. Accordingly, the necessity for a thermally stable WBM additive with high temperature-high pressure (HTHP) stability, and considerably unaltered by contamination of salt/solids particles has been clearly defined (Khodja, 2008; Sanchez *et al*., 2003).

The rheological appearance of drilling muds are intricate in their conjectural modeling and composition under various conditions. Assembling accurate information on the down-hole performance of drilling mud cannot be misrepresented. Drilling impostors are only acceptable if their provided data are processed attentively (Seymour and MacAndrew, 1994). The hydraulics preparation and composition process of the mud dependants on inaugurating fluids rheology when wellbore experiences various pressure and temperature settings. Any miss-calculation of these facts would laterally lead to flawed results, course alteration and associated expenses throughout advanced phases of drilling. Therefore, drilling fluids require to be formulated with the prime objective of preserving its properties in the wellbore (RP13D, 2006; Rommetveit and Bjorkevoll, 1997).

The necessity to enumerate rheological alterations in drilling muds along well-bore cannot be totally understood. An efficacious formation and how systematically it has been verified when subjected to all possible settings is often the pivotal factor in whether the HTHP-drilling venture is successful. The degree of variation in WBMs is fairly radical, while the fluid at the bottom-hole may not endure the rheological resemblance experienced at the surface.

Separately from conservative rheological evaluation, a complete testing database should also include the succeeding parameters which are vital inputs to HTHP drilling process: Muds chemical features in down-hole settings (such as pH); thermo-physical assets of mud based on its composition (Bland *et al*., 2006; Caenn and Chillingar, 1995). Many of researches and studies on WBM fluid loss control additives have been focused on commercial starches, in laboratory measurements and applicable for typical drilling trials (Chesser and Enright, 1980; Rayborn and Dickerson, 1992; Issham and Ahmad Kamal, 1997; Rayborn Sr.and Rayborn, 2000; Ademiluyi *et al*. 2001; Amanullah and Long, 2004; Baba Hamed and Belhadri,2009; Agarwal *et al*, 2011; Egun and Achadu, 2013; Dias *et al*, 2015). But, the use of novel starches in WBM formation with actual industrial application, which can also tolerate various aggressive circumstances such as HTHP drilling are yet to be discussed.

1.3 Research Objectives

The objectives of this research are:

1) To investigate the utilization of cassava starch derivatives as fluid loss control agent in WBM
2) To evaluate rheological properties of cassava-WBM in various formulation subjected to different drilling temperatures
3) To investigate acid modification of cassava derivatives and its impact on WBM performance in HTHP circumstances
4) To improve formulation of cassava-WBM via enhancement of thermal stability and fluid loss control by addition of carbon black and gilsonite

1.4 Scope of Work

The use of polymeric products as WBM additives has globally spread throughout petroleum industries. Potato, corn and wheat starches are the most common additives applied for WBM fluid loss control, as well as other pricey polymers such as xanthan gum and polynomic cellulose (American Institute of Petroleum, 1998; Barnes, 2000; Azar and Samuel, 2007).

For this study, 4 types of cassava derivatives (*ubi kayu*, *ijebu garri* (WG), *elubo garri* (YG) and *fufu*) were used as novel and commercially compatible fluid loss control additives for WBM formation. The designed formulations are all based on American Petroleum Institute (API) and National Iranian South Oil Co. (NISOC) standards.

Recognizing what and when to expect is an influential preparation tool in designing an efficient drilling fluid. Having defined the complications related with HTHP water based mud design and experiments, and the motive for the emphasis on cassava-WBM. This research will focus exclusively on eminence areas being:

1) Introducing various cassava derivatives as fluid loss control additive in WBM formation
2) Evaluation of temperature-based effective inceptions of WBM samples throughout experimentation.
3) Understanding the rheological behavior of cassava-WBMs in various drilling circumstances
4) Developing cassava-WBM formulation with improved thermal stability, compatible for HTHP Drilling
5) Providing generic guiding through experiments on fine treatment of the muds for restored performance, and avoiding instability which appears to plague drilling muds at extended temperatures.

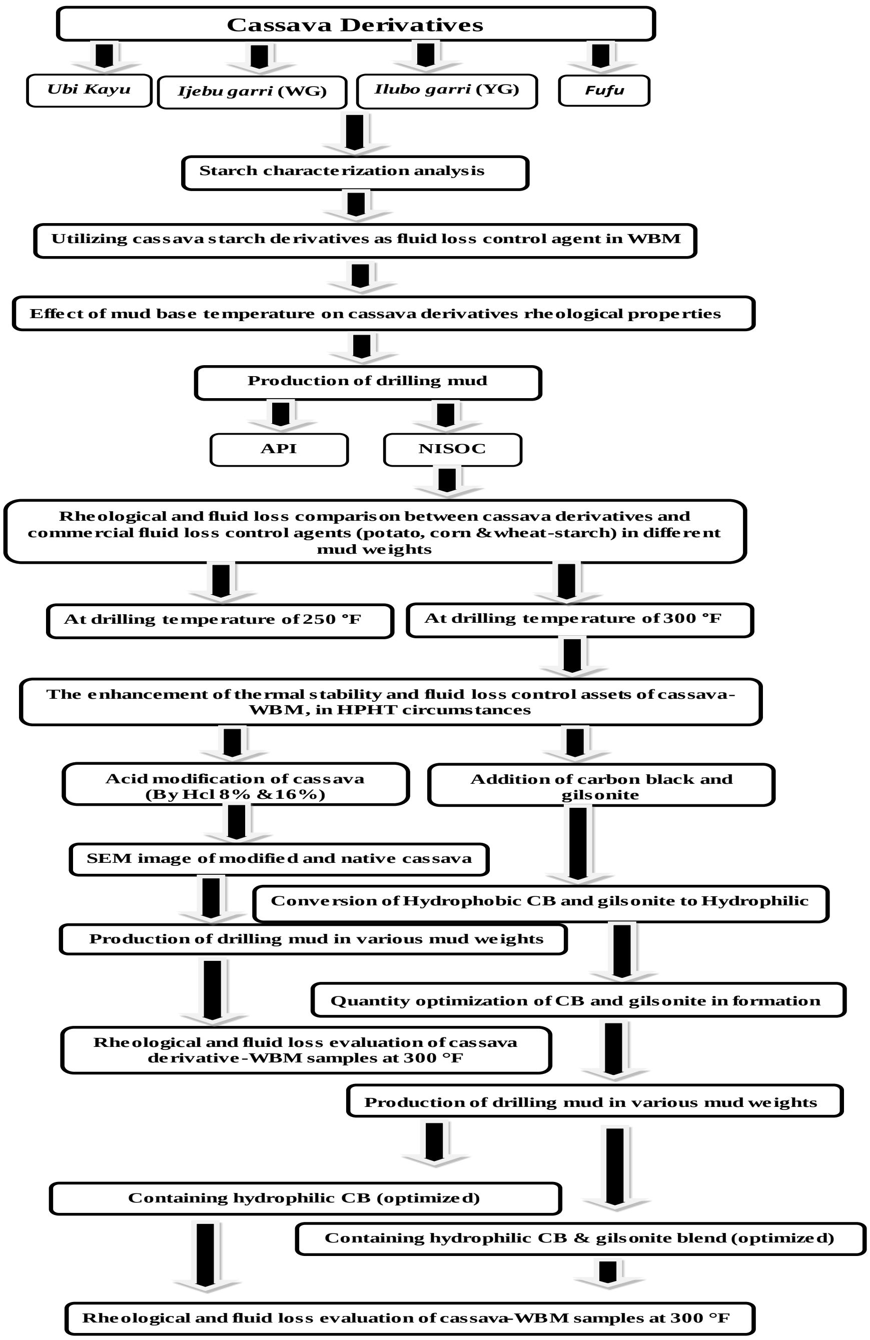

Figure 1.1. Schematic diagram illustrating the flowchart of this thesis.

1.5 Thesis Layout

In the first chapter, the importance and benefits of water based drilling fluids were discussed. It also illustrates the thesis objectives, problem statement and scope of work.

Chapter two covers the literature review on drilling fluid relevant subjects including background study, classifications, practical additives, rheological characterization and models.

Chapter three covers the experimental design/formulations and measures surveyed in the study. It elucidates the applied equipment, mud preparation procedure and the materials employed.

In chapter four, cassava derivatives (*ubi kayu*, *ijebu garri* (WG), *ilubo garri* (YG) and *fufu)* were introduced as a fluid loss control agent in WBM formation and their characteristics analyzed. Prior to mud preparation, samples were examined with various water (mud base) temperatures, in order to define the effect of the mud base on starch/mud rheology and function. Mud samples were prepared and examined based on terms of API and NISOC standards for starch. The basis behind using the incessant procedures for examining drilling muds was clearly defined.

In chapter five, the WBM samples were prepared in three different mud weights (75pcf, 100pcf and 150pcf) according to NISOC standard requirement and standards as described previously. Rheological properties (plastic viscosity, apparent viscosity, yield point and gel strength, pH) and filtration loss appraisal of the samples were evaluated in various drilling temperatures (200, 250, 275 and 300 °F) in comparison to commercial starches (potato, corn and wheat-starch). In an attempt to study the influence of starch quantity on WBMs functionality, a reduced amount (10g) of each cassava sample were studied in optimized temperature found (250 °F).

In chapter six, the main focus is on acid modification of cassava derivatives, with the visualization of improving the fluid loss control and thermal stability of cassava-WBM formations under HTHP circumstances (300 °F). Starches were modified by the acid-alcohol treatment based on two acid concentrations (HCl 8 and 16%). Prior to cassava-WBM preparation and its rheological and fluid loss evaluation in 3 mud weights (light, average and heavy). The effect of acid modification on cassava granules was investigated using SEM Imaging.

In chapter seven, in order to further advance cassava-WBM thermal stability of cassava-WBM in HTHP drilling, carbon black N326 and gilsonite CH 110 were added to the formation as thermal stabilizer and fluid loss reducer. Primarily, hydrophobic CB and gilsonite were converted to Hydrophilic state and the influence of dispersant in conversion was studied. Then, mud samples were prepared and evaluated by using optimized quantity of CB and gilsonite in formation in various mud weights.

The last chapter presents the conclusions attained from the study, outlining the recommendations for future exertions on drilling fluid utilization and rheological advancement.

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