UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF GRAPHENE-ENHANCED ESTER-BASED DRILLING FLUID

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By

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

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DEVELOPMENT OF GRAPHENE-ENHANCED ESTER-BASED DRILLING FLUID

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SITI ZULAIKA BINTI RAZALI

August 2019

Chairperson: Professor Robiah binti Yunus, PhD
Institute: Institute of Advanced Technology

Synthetic base fluids have been introduced to substitute non-environmentally friendly diesel at strict discharged regulation area, particularly in sensitive marine zone. Issues such as high viscosity and pour point, poor thermal and hydrolytic stabilities limit the manipulation of ester for various drilling fluid's density and oil-water ratio (OWR) at high pressure-high temperature (HPHT) although it is superior in biodegradability, lubricity and zero aromatic content. In addition to that, nanomaterials have been proven in improving drilling fluid's properties, especially in water based. Nevertheless, there is very limited knowledge of nanomaterial's role in the improvement of ester-based drilling fluid (EBDF).

The main objectives of this work are to evaluate the performance of EBDF from 2-Ethylhexyl caprylate/caprate and to investigate the effects of carbon-based nanomaterials on EBDF for properties improvement. The ester was synthesized through a pulsed loop reactor and optimized by response surface methodology. The maximization of conversion (97.96%) and yield (89.43%) were achieved under optimum conditions of 78 °C of reaction temperature, 10 mbar of vacuum pressure, 20 minutes of reaction time and 155 rpm of oscillation speed. Important properties of 2-Ethylhexyl caprylate/caprate such as viscosity (3.26 cSt), density (862 kgm^{-3}), pour point (-69 °C) and flash point (158 °C) have been analyzed according to ASTM standard and met drilling fluid's base oil requirements.

Ester then has been formulated as drilling fluid at different OWR (70:30, 80:20, 85:15) and drilling fluid density (9.5 ppg, 12.5 ppg, 16.5 ppg) to suit with a wide range temperature (275 °F - 360 °F). The optimized formulation for condition of 70:30 OWR and 9.5 ppg (ester=180.30 lb/bbl; water=89.54 lb/bbl; calcium chloride=34.44 lb/bbl; primary emulsifier=8 lb/bbl; secondary emulsifier=6 lb/bbl; organophilic clay=3 lb/bbl; fluid loss control=8 lb/bbl; lime=2 lb/bbl and

barite=67.89 lb/bbl) remained stable after 16 hours of hot rolling at 275 °F with satisfactory rheology (plastic viscosity = 22 cp and yield point =19 lb/100 ft^2), electrical stability (601 V) and HPHT filtration properties (filtrate volume= 2.8 ml and cake thickness= 0.79 mm). The optimized formulation for HPHT conditions with 80:20 OWR and 12.5 ppg (ester= 177.16 - 182.72 lb/bbl; water= 51.32 - 52.93 lb/bbl; calcium chloride= 19.74 - 20.36 lb/bbl; primary emulsifier= 10 - 12 lb/bbl; secondary emulsifier= 2-6 lb/bbl, organophilic clay= 3 lb/bbl; fluid loss control=8 lb/bbl, lime 4=lb/bbl and barite=245.20 - 246.07 lb/bbl) remained stable after 16 hours of hot rolling at 300, 330 and 350 °F with satisfactory plastic viscosity (32-38 cp), yield point (17-20 lb/100 ft^2), electrical stability (820-910 V) and HPHT filtration properties (filtrate volume= 2.8 - 3.8 ml and cake thickness= 0.79 - 1.59 mm). The ester also demonstrated the ability to tolerate high solid content with optimized formulation for 16.5 ppg (ester= 154.23 lb/bbl; water= 31.54 lb/bbl; calcium chloride= 12.13 lb/bbl; primary emulsifier= 12 lb/bbl; secondary emulsifier= 8 lb/bbl; organophilic clay= 3 lb/bbl; fluid loss control= 6 lb/bbl; lime= 2 lb/bbl and barite= 464.38 lb/bbl) which was also remained stable after 16 hours of hot rolling at 360 °F with satisfactory rheology (plastic viscosity= 69 cP and yield point= 36 lb/100 ft^2), electrical stability (1416 V) and HPHT filtration properties (filtrate volume= 4.0 ml and cake thickness= 1.59 mm).

The 16.5 ppg of EBDFs demonstrated different properties in the presence of commercial graphene nanoplatelets (GNPs), in-house GNPs, graphene nanopowder, graphene oxide (GO) and carbon nanotube cotton (CNT cotton). Plastic viscosity increased to 10.14% (GO-EBDF), 4.35% (commercial GNPs-EBDF) and 2.90% (in-house GNPs-EBDF) while decreased to by 5.80% (graphene nano-powder) and 2.90% (CNT cotton) relative to Control. The GO augmented EBDF's gel strengths by 27.27% (10 sec) and 12.5% (10 min) while the others gave a slight reduction of gel-strengths. The emulsion stability of EBDF improved with graphene nanopowder (1480 V) while decreased with other materials. HPHT filtration properties of EBDF was improved with graphene nanopowder (20% and 24.53% reduction of filtrates amount and cake thickness respectively). In addition to that, 0.007wt% is the optimum concentration of graphene nanopowder in 16.5 ppg EBDF. The improvement of properties of EBDF by graphene nanopowder may due to the action of graphene nanopowder as an inhibitor for micelle formation, a co-secondary emulsifier, a bridging agent and increases oil-wettability of filter cake.

As a conclusion, 2-EH caprylate/caprate has been successfully synthesized and optimized via a vacuum assisted-pulsed loop reactor. The performances of ester as green drilling fluid at HPHT conditions also has been demonstrated according to API 13B-2 standards. Graphene nanopowder enhanced the EBDF's performance, particularly in the filtration properties. Finally, knowledge beyond the enhancement of EBDF through the addition of graphene nanopowder has been elucidated.

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

PEMBANGUNAN BENDALIR PENGGERUDIAN BERASASKAN ESTER YANG DITAMBAH BAIK DENGAN GRAFIN

Oleh

SITI ZULAIKA BINTI RAZALI

Ogos 2019

Pengerusi: Professor Robiah binti Yunus, PhD
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Bendalir asas sintetik telah diperkenalkan untuk menggantikan diesel yang tidak mesra persekitaran di kawasan peraturan pelepasan ketat terutama di zon marin sensitif. Ester diketahui sebagai pilihan terbaik minyak asas dari segi pelinciran, biodegradasi, rendah toksik dan kandungan sifar aromatik untuk bendalir penggerudian. Isu-isu seperti kelikatan, titik tuang, ketidakstabilan haba dan hidrolitik menghadkan manupulasi ester untuk ketumpatan bendalir penggerudian dan nisbah minyak/air yang pelbagai pada tekanan tinggi-suhu tinggi (HPHT). Penambahan bahan nano telah dilaporkan dapat memperbaiki sifat-sifat bendalir penggerudian, terutamanya bendalir penggerudian berasaskan air. Walau bagaimanapun, terdapat pengetahuan yang terhad terhadap kelakuan bahan nano dalam penambahbaikan bendalir penggerudian.

Objektif utama kerja ini adalah untuk menilai prestasi bendalir penggerudian berasaskan ester (EBDF) dari ester 2-etilheksil kaprilat/kaprat dan untuk mengkaji kesan-kesan bahan nano berasaskan karbon ke atas EBDF. Ester tersebut disintesis menggunakan rektor gelung denyut dan dioptimasi dengan kaedah permukaan gerak balas. Pemaksimuman penukaran (97.96%) dan hasil (89.43%) ester dicapai pada kondisi optimum iaitu suhu 78 °C, tekanan vakum 10 mbar, masa tindakbalas 20 minit dan kelajuan ayunan 155 rpm. Sifat-sifat penting ester 2-etilheksil kaprilat/kaprat seperti kelikatan (3.26 cSt), ketumpatan (862 km-3), titik tuang dan titik kilat (158 °C) telah dianalisis mengikut piawaian ASTM and memenuhi keperluan bendalir penggerudian.

Ester seterusnya diformulasi sebagai bendalir penggerudian pada nisbah minyak:air (70:30, 80:20, 85:15) dan ketumpatan bendalir penggerudian (9.5ppg, 12.5 ppg, 16.5ppg) yang berbeza untuk menyesuaikan dengan suhu yang pelbagai 275 °F - 360 °F. Formulasi optimum untuk keadaan 70:30 OWR dan 9.5 ppg (ester= 180.30 lb/bbl; air= 89.54 lb/bbl; kalsium klorida= 34.44 lb/bbl;

pengemulsi utama= 8 lb/bbl, pengemulsi kedua= 6 lb/bbl, tanah liat organofilik=3 lb/bbl, pengawal kehilangan bendalir = 8 lb/bbl, kapur= 2 lb/bbl dan barit= 67.89 lb/bbl) kekal stabil selepas 16 jam di dalam ketuhar pada suhu 275 °F dengan reologi (kelikatan plastic= 22 cP dan titik hasil= 19 lb/100 ft^2), kestabilan eletrik (601 V) dan sifat penurasan HPHT (isipadu turasan= 2.8 ml dan ketebalan kek= 0.79mm) yang memuaskan. Formulasi optimum untuk keadaan HPHT dengan 80:20 OWR and 12.5 ppg (ester= 177.16-182.72 lb/bbl; air= 51.32-52.93 lb/bbl; kalsium klorida= 19.74-20.36 lb/bbl; pengemulsi utama= 10-12 lb/bbl; pengemulsi kedua= 2-6 lb/bbl; tanah liat organofilik= 3 lb/bbl; pengawal kehilangan bendalir= 8 lb/bbl; kapur= 4 lb/bbl dan barit= 245.20-246.07 lb/bbl) kekal stabil selepas 16 jam di dalam ketuhar pada suhu 300 °F, 330 °F dan 350 °F dengan reologi (kelikatan plastic= 32-38 cP dan titik hasil= 17-20 lb/100 ft^2), kestabilan eletrik (820-910 V) dan sifat penurasan HPHT (isipadu turasan= 2.8-3.8 ml dan ketebalan kek= 0.79-1.59 mm) yang memuaskan. Ester tersebut turut menunjukkan kebolehan bertoleransi dengan kandungan pepejal yang tinggi dengan formulasi untuk 16.5 ppg (ester= 154.23 lb/bbl; air= 31.54 lb/bbl; kalsium klorida= 12.13 lb/bbl; pengemulsi utama= 12 lb/bbl; pengemulsi kedua= 8 lb/bbl, tanah liat organofilik=3 lb/bbl; pengawal kehilangan bendalir= 6 lb/bbl, kapur= 2 lb/bbl dan barit=464.38 lb/bbl) di mana bendalir penggerudi kekal stabil selepas 16 jam di dalam ketuhar pada suhu 360 °F dengan reologi (kelikatan plastic= 69 cP dan titik hasil=36 lb/100 ft^2), kestabilan eletrik (1416 V) dan sifat penurasan HPHT (isipadu turasan =4.0 ml dan ketebalan kek= 1.59 mm) yang memuaskan.

EBDF menunjukkan sifat berbeza dengan kehadiran platlet nano grafin komersial (GNPs), GNPs dalaman, serbuk nano grafin, grafin oksida dan kapas tiub nano karbon (CNT Cotton). Kelikatan plastik meningkat sehingga 10.14% % (GO-EBDF), 4.35% (komersial GNPs-EBDF) and 2.90% (dalaman GNPs-EBDF) manakala menurun kepada 5.80% (graphene nano-powder) and 2.90% (CNT cotton) masing-masing berbanding Kawalan. GO meningkatkan kekuatan gel EBDF sebanyak 27.27% (10 saat) dan 12.5% (10 min) manakala yang lain memberikan sedikit penurunan. Kestabilan emulsi EBDF bertambah dengan serbuk nano grafin (1480 V) manakala bahan lain mengalami penurunan. Sifat penurasan HPHT EBDF dipertingkatkan dengan serbuk nano grafin (20% and 24.53% bagi isipadu turasan dan ketebalan kek). Sebagai tambahan, 0.007wt% adalah kepekatan optimum serbuk nano grafin bagi 16.5 ppg EBDF. Penambahbaikan sifat EBDF oleh serbuk nano grafin mungkin disebabkan tindakan serbuk nano grafin sebagai penghalang pembentukan misel, pengemulsi kedua bersama, ejen penyambung dan mempertingkatkan kelembapan kek turasan dengan minyak.

Sebagai penutup, 2-EH kaprilat/kaprat berjaya disintesis dan dioptimisi menggunakan rektor gelung denyut. Prestasi ester sebagai bendalir penggerudian hijau di dalam keadaan HPHT juga telah dikaji berdasarkan piawaian API 13B-2. Serbuk nano grafin meningkatkan prestasi EBDF terutamanya sifat penurasan. Akhir sekali, pengetahuan disebalik penambahbaikan EBDF melalui serbuk nano grafin telah diperjelaskan.

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I would like to thank all the member of the Institute of Advanced Technology, my colleagues, my labmates and Biobased Research Group. Thank you all.

I certify that a Thesis Examination Committee has met on 27 August 2019 to conduct the final examination of Siti Zulaika binti Razali on her thesis entitled "Development of Graphene-Enhanced Ester-based Drilling Fluid" in accordance with the 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 thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

2-EH Caprylate/caprate ester	2-Ethylhexyl caprylate/caprate ester
ANOVA	Analysis of variance
API	American Petroleum Institute
CCI	Carrying capacity index
CNT cotton	Carbon nanotube cotton
DOBDF	diesel oil-based drilling fluid
ES	Emulsion stability
FCCD	Face-centered central composite design
FBDF	Foam-based Drilling Fluid
FESEM	Field-emission scanning electron microscopy
GNPs	Graphene nanoplatelets
GO	Graphene oxide
HBPA	Hyperbranched polyamine
HPHT	High-pressure high-temperature
HRTEM	High-resolution transmission electron Microscopy
IO	Internal olefin
LAO	Linear alpha olefin
LTMO	Low toxicity mineral oil
LTOBDF	Low toxic oil-based drilling fluid
MWCNT	Multiwall CNT
NPG	Neopentylglycol
OBDF	Oil-based drilling fluid
OBR	Oscillatory baffle reactor
OTMOS	Hydrophobic octadecyltrimethoxysilane
OWR	Oil-water ratio
PV	Plastic viscosity
PE	Pentaerythritol
POA	Poly-alpha olefin
POME	Palm oil methyl ester
SBDF	Synthetic-based drilling fluid
TMP	Trimethylolpropane
WBDF	Water-based drilling fluid
YP	Yield point

CHAPTER 1

INTRODUCTION

This chapter covers the background of the drilling operation and general information about drilling fluids, problem statement, objectives, contribution, and scope of this research, as well as the overall organization of the thesis.

1.1 Background

Drilling fluid is one of the important components in the drilling fluid system. It serves crucial functions in drilling system including the removal of cuttings; lubricating and cooling the drill bit; sealing permeable formations; suspending solids even after drilling (and consequently fluid flow) has halted; and controlling subsurface pressure to prevent formation damage (Fink, 2015; Lyons *et al*., 2016). Drilling fluid will be pumped from the surface pits over the drill pipe and will exit at the end of the bit then flow up through the annular space in the wellbore while carrying all the cuttings resulted from the spinning of bit at the bottom hole. The fluid will be transferred back to the drilling fluid shaker for cuttings removal and drilling fluid treatments. During the process of carrying the cuttings to the surface, part of the solids in drilling fluid will be deposited onto the wellbore wall and form a layer called filter cake through a mechanism, namely filtration. The filter cake will act as a temporary support for the wellbore and reduce invasion of filtrates into formation therefore enhance the wellbore stability before casing and cementing installation (Salehi *et al*., 2015, 2014; Salehi and Kiran, 2016; Vasheghani Farahani *et al*., 2014; Zhong *et al.*, 2019).

The types of drilling fluids used in drilling operation depend on well types and formation (Rabia, 1985; Wu and Massoudi, 2016). In general, drilling fluids are classified based on the physical properties; oil-based drilling fluid (OBDF), water-based fluid (WBDF) and pneumatic-based drilling fluid (PBDF). Synthetic based drilling fluid (SBDF), as well as ester-based drilling fluid (EBDF), fall under OBDF classification. OBDF is originally derived from petroleum diesel but has been discontinued due to negative impact on the environment. Alternatively, less toxic mineral oil and synthetic oils such as poly-alpha olefin (PAO), internal olefin (IO), linear alpha olefin (LAO), ester, ether and paraffin have been considered as base oils for drilling fluids. Although all SBDF derived from hydrocarbon derivatives are less toxic, they are only 15-60% biodegradable under anaerobic condition (IOGP, 2001). Synthetic base oils have been developed due to the stringent cuttings discharge requirements in the marine ecosystem whereby diesel fails to comply. Diesel has been strictly banned in the Gulf of Mexico, the North Sea and many other places in the world since 1984 due to its toxicology issue to marine life (Speight, 2015). WBDF although non-toxic, biodegradable and has lower cost of drilling fluid; it is unsuitable for drilling complicated wells, particularly in the shale formation. Shale formation complicates drilling process. It can be observed that SBDF may the only option available in shallow vertical onshore wells to deeper and deviated holes; higher temperature, higher pressure or deeper water (Fornasier *et al*., 2017; Sinha *et al*., 2017). About 75% of the footage drills worldwide contain shale formation and 90% of the wellbore stability problems originate from the shale since it contains a significant amount of clays such as smectite and montmorillonite (Chen *et al*., 2003; Claudio *et al*., 2005; Darley, 1969; Ewy and Cook, 1990; Helstrup *et al*., 2004; Oort, 2003). SBDF is preferred in shale formation drilling due to its ability to stabilize clay, high lubricity, less corrosion, fewer formation damage, minimal temperature effect and low-cost factor (Lyons *et al*., 2016).

SBDF is an invert emulsion drilling fluid which consists of three main phases (water-in-oil and solids) with several additives (emulsifier, alkali reserve, viscosifier, thinning agent, fluid loss control, and stabilizer) (Caenn *et al*., 2017). Invert emulsion system assists in optimizing the cost of drilling operation by reducing the amount of synthetic base oil without compromising the performance of an OBDF. In addition, small and well-dispersed water droplets help to increase fluid viscosity by acting as solids particles, thus reducing the amount of viscosifier in the system. Reduced viscosifier helps in cold temperature drilling operation by allowing rheological and fluid loss properties to be controlled through the fluid-emulsion characteristics (Said and El-Sayed, 2018).

1.2 Problem Statements

Oil retained on the cuttings has been a major issue in the increasingly stringent limitations for cuttings discharge offshore. The cuttings disposal policy is different from one country to another. Currently, various technologies have been developed as alternatives to sea bed disposal that includes solids control, thermal desorption, solvent extraction, incineration, solidification/ stabilization, bioremediation and cuttings dryer (Ball *et al*., 2012; Huang *et al*., 2018). Those technologies are effective in removing the retained oil; however, they are not exactly flexible, costly, requires high temperature and high pressure, limited feasibility, and safety hazards.

It has been reported that SBDF is less toxic. SBDF degrades faster than diesel and mineral oils, without compromising the performance of OBDF (Friedheim, 1997; Neff *et al*., 2000). SBDF is also designed to fulfil specific requirements guided by International Petroleum Industry Environmental Conservation Association (2009) such as it should be less volatile than conventional OBDF (higher flash point) and its vapors must be free of aromatic compound. Such base oil properties are very important for health and safety aspects. However, SBDFs derived from hydrocarbon-based show issues on biodegradability which raise environmental concerns (Munro *et al*., 1998; Steber *et al*., 1995). The degradation of hydrocarbon derived SBDF much faster in aerobic condition (Candler *et al*., 1999; Neff *et al*., 2000) than in anaerobic *(Candler et al*., 1999; Herman and Roberts, 2005; Van Slyke and Miller, 2016).

On the other hand, EBDF derived from plant oils have been recognized in providing the best environmental performance of any SBDF (Peresich *et al*., 1991). Apart from that, it is also fully biodegradable; aerobically and anaerobically (Blanchet *et al*., 1998; Imevbore *et al*., 2000; Lake *et al*., 2007; Neff *et al*., 2000; Steber *et al.*, 1995). Ester emits the lowest organic vapor compared to LAO and IO (Candler *et al*., 1996; Neff *et al*., 2000). EBDF has also been proven as superior in the aspects of biodegradation, no bioaccumulation, very low eco-toxicity, high lubricity and zero aromatic content, in comparison to other synthetic base fluids. However, it possesses a high kinematic viscosity, has poor thermal stability, high pour point, and prone to hydrolysis either in acidic or base condition (Caenn and Chillingar, 1996). Enhancement of the EBDF performance in order to fulfil the requirements of complex holes and deepwater operations are crucial especially in sensitive marine areas (Cordes *et al*., 2016; Tornero and Hanke, 2016). In spite of the rules that limit the discharge of SBDFs into the sea, EBDF remains as the best option, when environmental aspects are concerned.

In general, ester-based systems exhibit high kinematic viscosity, a condition that is magnified in the cold temperatures encountered in deep-water risers. Issues such as viscosity, pour point, thermal and hydrolytic stabilities limit the manipulation of ester for different drilling fluid's density and oil-water ratio (OWR) at high operating temperature. Various types of esters derived from vegetable oils and fatty acids have been explored as invert emulsion drilling fluid including peanut oil, soybean oil, methyl ester, ethyl ester and others (Dosunmu and Joshua, 2010; Habib *et al*., 2014; Jassim *et al*., 2016; Mueller *et al*., 1993c; Peresich *et al*., 1991). Most of the published works related to EBDF reported poor rheological and fluid loss properties after aging for 16 hours at 300 °F (149 °C) or above. Burrows *et al*. (2001, 2004) developed a low-viscosity ester for drilling fluid which was synthesized from a short-chain length (C8) fatty acid. However, the operating temperature was also limited to 300 °F. In other words, in any drilling operation that involves temperature higher than 300 °F only hydrocarbon derived base oil such as internal olefin can be used.

At present, basic formulation of a drilling fluid (base oil, brine, emulsifier, lime, viscosifier, fluid loss control and weighing agent) is unable to produce fluid that will perform satisfactorily in complicated drilling operations especially related to high pressure high temperature (HPHT) well, shale formation in deepwater well, narrow window of equivalent circulating density (ECD) and etc. (Burrows *et al*., 2001; Growcock *et al*., 2011; Lyons *et al*., 2016; Sinha *et al*., 2017; Zhao *et al*., 2017). Additives such as thinning agent, bridging agent, rheology modifier, anti-hydrolysis, antioxidant and etc. have been utilized to enhance the properties of drilling fluids such as in rheology, fluid loss reduction, filter cake formation, thermal stability and salty resistance in the harsh applications (Bastidas *et al.*, 2017; Cadix *et al*., 2015; Deville, 2010; Goncalves *et al.*, 2007; Mueller *et al*., 1993a; Pereira *et al*., 2017; Song and Yang, 2016). Most of the additives are produced from polymers. Adding nanomaterials to the formulation has been reported to improve properties of drilling fluid, especially for WBDF. Among the reported carbon-based nanomaterials, graphene oxide (GO) demonstrated as an excellent fluid loss control in WBDF (Kosynkin *et al.*, 2012). Other carbon-based nanomaterials such as CNT and graphene nanoplatelets also demonstrated the improvement of fluid loss of WBDF although they are not as good as GO (A. Aftab *et al*., 2017; Fazelabdolabadi *et al*., 2015). Unfortunately, there is very limited knowledge of carbon-based nanomaterials in the improvement of drilling fluid, particularly EBDF.

1.3 Contribution of Research

The main purposes of this project are to evaluate the performance of ester-based drilling fluid (EBDF) from 2-Ethylhexyl caprylate/caprate (2-EH caprylate/caprate) and to investigate the effects of carbon-based nanomaterials in enhancing the performance of EBDF. To achieve this, ester with low kinematic viscosity, low pour point and high flash point has been synthesized using a vacuum-assisted pulsed loop reactor. The three mentioned properties are the main criteria for a good base oil for drilling fluid. The new EBDFs have been formulated at different aging temperature and drilling fluid's density. The rheological and fluid loss properties of newly formulated EBDF have proven the positive attributes of the ester base oil. Carbon-based nanomaterials such as graphene nanoplatelets (GNPs), graphene nanopowder, graphene oxide (GO) and carbon nanotube cotton (CNT cotton) affect the properties of EBDF differently at similar concentration. Graphene nanopowder is a promising additive in enhancing the rheological and fluid loss properties of newly formulated EBDF. Several tests such as rheology, electrical stability and HPHT filtration test have elucidated the roles of graphene nanopowder in EBDF.

1.4 Research Objectives

The followings are the research objectives of the study.

1. To evaluate the optimum operating conditions of the vacuum-assisted pulsed loop reactor on the conversion and yield of 2-EH caprylate/caprate ester.
2. To investigate the effects of additives on the rheology and thermal stability of 2-EH Caprylate/caprate EBDF.
3. To examine the effects of adding carbon-based nanomaterials on the rheological performance of 2-EH caprylate/caprate EBDF.
4. To elucidate the roles of graphene nanopowder in improving the rheology and filtrates losses of 2-EH caprylate/caprate EBDF.

1.5 Scope of Research

Methyl caprylate/caprate and 2-Ethylhexanol are selected as raw materials to produce a new base oil for drilling fluid. The reactions were conducted in the presence of sodium methoxide as a catalyst to accelerate the transesterification process. The reactions took place in a vacuum-assisted pulsed loop reactor. The ratio of the reactants and catalyst concentration were fixed at 1:1.5 (methyl caprylate/caprate: 2-Ethylhexanol) and 0.4% respectively. Four factors have been considered in the optimization study, which are temperature (70-90 °C), time (20-60 minutes), vacuum pressure (10-30 mbar) and oscillatory speed (120-240 rpm).

The performance of 2-EH caprylate/caprate ester as base oil was evaluated at temperatures starting from 275 °F to 360 °F. The performance of 2-EH caprylate/caprate EBDF was also assessed based on its rheological and fluid loss properties. The drilling fluids were tested according to American Petroleum Institute (API) procedures; Practice 13B-2 (2014).

Commercial GNPs, in-house GNPs, graphene nanopowder, GO and CNT cotton were used to examine the effects of carbon-based nanomaterials on 2-EH caprylate/caprate EBDF. The governance of carbon-based nanomaterial on EBDF has been investigated based on the emulsion stability, rheology and HPHT filtration properties.

1.6 Organization of Thesis

The thesis consists of five chapters. Chapter One is an introduction to the background and significance of the study and comprises the objectives and scope of the research. Chapter Two provides an extensive review and analysis of previous research relevant to the present study including i) transesterification, synthesis 2-Ethylhexyl esters and factors that are affecting the yield of ester in an oscillatory flow reactor, ii) various aspects of EBDFs, and iii) addition of nanomaterials in drilling fluids. Chapter Three presents the general materials, methods and testing used in the study, including synthesis and optimization of transesterification reaction via vacuum-assisted pulsed loop reactor, testing and analysis of synthesized base oil, development and testing of 2-EH caprylate/caprate EBDFs (various temperatures, different density and OWR) and development and testing of nano-2-EH caprylate/caprate EBDFs. Chapter Four covers the discussion of each findings in relation to the research objectives, which include of 2-EH caprylate/caprate ester synthesis and optimization by using pulsed loop reactor, effects of basic additives on 2-EH caprylate/caprate EBDF, performance of 2-EH caprylate/caprate EBDF at various temperatures, performance of 2-EH caprylate/caprate EBDF after adding carbon-based nanomaterials and mechanism of nanomaterials in enhancing the performance of 2-EH caprylate/caprate EBDF. Finally, the conclusions of this research and recommendations for future works are presented in Chapter Five.

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