APPLICATION OF NATURAL POLYMERS AND NANOPARTICLES IN ENHANCING OIL RECOVERY AT RESERVOIR CONDITION

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DEDICATION

Primarily to my beloved friend and brother Engr Godspower Precious John who died a few days before I arrive in Malaysia.

Secondarily to my dear cousin Owokonu Onyeoma Agi who was born a few days after I arrived in Malaysia

Tertiarily in cherish memory of my cousins Brig. Gen William Ogar and Bose Alaba who passed away a year and few months respectively before I completed my studies.,

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ABSTRACT

Nanofluid flooding has been proven to be very effective in enhanced oil recovery (EOR). The performance depends on the material and formulation process. Previous studies have focused only on the use of inorganic, metal and metal oxides nanoparticles which have limited full-scale field application due to cost and environmental concerns. The use of nanofluid from natural sources has not been investigated to a reasonable extent for possible application in EOR. In this research, three natural polymers were formulated from local materials from Malaysia and Nigeria. Ascorbic acid was used to synthesize the natural polymers to nanoparticles. The physical properties of the nanoparticles including their size distribution, crystalline structures were investigated. The rheological properties of the nanofluids were compared with natural polymers and commercial polymer xanthan. The interfacial properties of the nanofluids at the interface of oil and water were investigated at different concentrations and temperatures. The effect of interaction between electrolyte and ultrasonic was determined. The wettability alteration efficiency of the nanofluids on oil-wet sandstone surface was investigated using the sessile drop method. Core flooding experiment was conducted at high temperature high pressure (HTHP) reservoir conditions to justify the effect of wettability alteration and dispersion of the nanofluids on additional oil recovery. The performance of the nanofluids were compared with conventional EOR chemical. The methods were effective in producing spherical and polygonal nanoparticles with a mean diameter of 100 nm and increased in crystallinity of 7%. The viscosity increased with increase in surface area and temperature of the crystalline starch nanofluid (CSNF), *Cissus populnea* nanofluid (CPNF), *Cissus populnea* (CP) and cassava starch (CS) compared to a decrease in viscosity as the temperature increases for okra, okra lignocellulose nanofluid (OLCNF) and xanthan. The interfacial tension decreased with increase in concentration of the nanofluids, electrolyte and temperature. The results show that the nanofluids can change the wettability of sandstone at low concentration, high salinity and elevated temperature. Oil recovery after waterflooding was 48% of original oil in place (OOIP), the oil recovery of okra, CS and CP increased by 13%, 15% and 17% respectively, compared to 11% OOIP obtained with xanthan. The pressure drop data shows stability of OLCNF, CPNF and CSNF at 120 ºC and the formation of oil bank was enough to increase the oil recovery by 20%, 23% and 26% respectively. The nanofluids were found to be very effective in mobilizing residual oil at HTHP reservoir condition.

ABSTRAK

Banjiran bendalir nano telah terbukti sangat berkesan dalam perolehan minyak tertingkat (EOR). Prestasinya adalah bergantung pada bahan dan proses perumusan. Kajian terdahulu hanya memberikan tumpuan terhadap penggunaan partikel nano bukan organik, logam dan logam oksida yang mempunyai aplikasi lapangan berskala besar yang terhad berikutan kebimbangan tentang kos dan alam sekitar. Penggunaan bendalir nano daripada sumber semula jadi belum lagi dikaji secara meluas tentang kemungkinan penggunaannya dalam EOR. Dalam kajian ini, tiga jenis polimer semula jadi telah dirumuskan daripada bahan tempatan di Malaysia dan Nigeria. Asid askorbik telah digunakan untuk mensintesis polimer semula jadi menjadi partikel nano. Sifat-sifat fizikal partikel nano yang dikaji termasuk taburan saiz dan struktur hablurannya. Sifat-sifat reologi bendalir nano telah dibandingkan dengan polimer semula jadi dan polimer komersial xantan. Sifat antara muka bendalir nano pada antara muka minyak dan air telah dikaji pada kepekatan dan suhu yang berbeza. Kesan interaksi antara elekrolit dengan ultrasonik turut dinilai. Kecekapan perubahan keterbasahan bendalir nano pada permukaan batu pasir basah minyak telah dikaji memerusi kaedah titis sesil. Eksperimen banjiran teras telah dilaksana pada keadaan reservoir bersuhu tinggi dan bertekenan tinggi (HTHP) bagi menunjukkan kesan pengubahan keterbasahan dan penyebaran bendalir nano terhadap tambahan perolehan minyak. Prestasi bendalir nano telah dibandingkan dengan EOR kimia konvensional. Kaedah ini didapati berkesan dalam menghasilkan partikel nano sfera dan poligon yang bec diameter purata 100 nm dengan penghabluran tertingkat sebanyak 7%. Kelikatan didapati meningkat seiring dengan peningkatan dalam luas permukaan dan suhu bagi bendalir nano kanji habluran (CSNF), bendalir nano *Cissus populnea* (CPNF), *Cissus populnea* (CP), dan kanji ubi kayu (CS), berbanding penurunan kelikatan apabila meningkadnya suhu untuk bendi, bendalir nano bendi lignoselulos (OLCNF) dan xantan. Ketegangan antara muka didapati menurun apabila meningkadnya kepekatan bendalir nano, elektrolit dan suhu. Keputusan menunjukkan bahawa bendalir nano boleh menukar keterbasahan batu pasir pada kepekatan yang rendah, kemasinan tinggi dan suhu tinggi. Perolehan minyak selepas banjiran air telah 48% daripada minyak asal di tempat (OOIP), manakala perolehan minyak menerusi bendi, CS dan CP masing-masing meningkat sebanyak 13%, 15% dan 17% berbanding dengan 11% OOIP yang diperoleh menerusi xantan. Data kejatuhan tekanan menunjukkan kestabilan OLCNF, CPNF dan CSNF pada 120 ºC, dengan pembentukan batas minyak sudah cukup untuk meningkatkan perolehan minyak yang masing-masing sebanyak 20%, 23% dan 26%. Kesimpulannya didapati bendalir nano sangat berkesan dalam menggerakkan minyak baki pada keadaan reservoir HTHP.

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LIST OF ABBREVIATIONS

AFR	-	Advanced Flow Reactor
AHPE	-	Aromatic Hydrocarbon with ethylene
AIE	-	Aggregated Induced Emission
AM	-	Acrylamide Methyl
AOT	-	Aerosol-OT
API	-	American Petroleum Institute
ASP	-	Alkaline Surfactant and Polymer
ATO	-	Antimony-Doped Tin
ATRP	-	Atomic Transfer Radical Polymerization
BC	-	Before Christ
BSA	-	Bolvin Serum Albumin
CEOR	-	Chemical Enhanced Oil Recovery
CNC	-	Cellulose Nanocrystals
CP	-	*Cissus populnea*
CPNF	-	*Cissus populnea* Nanofluid
CPNP	-	*Cissus populnea* Nanoparticles
CS	-	Cassava Starch
CSNF	-	Crystalline Starch Nanofluid
CSNP	-	Crystalline Starch Nanoparticles
DA	-	Degree of Acetylation
DEC	-	Diethylene Glycol
DeTAB	-	Decyltrimethylammonium Bromide
DIW	-	Deionized Water
DLS	-	Dynamic Light Scattering
DLVO	-	Derjaguin-Landau-Vervey-Overbeek
DMAc	-	Dimethyl Acetamide
DMF	-	Dimethylformamide
DMSO	-	Dimethyl Sulfoxide
DSC	-	Differential Scanning Calorimetry
DW	-	Distilled Water

EDL	-	Electric Double Layer
EDTA	-	Ethylene Diamine Tetraacetic Acid
EDX	-	Energy Dispersive X-Ray
EG	-	Ethylene Glycol
EOR	-	Enhanced Oil Recovery
FTIR	-	Fourier Transform Infrared Spectroscopy
HCHO	-	Formaldehyde
HCl	-	Hydrochloric Acid
HD	-	Hexadecane
HLP	-	Hydrophobic Polysilicon
HLPN	-	Hydrophobic and Lipophobic Nanoparticles
HPAM	-	Hydrolysed Polyacrylamide
HTHP	-	High Temperature High Pressure
IFT	-	Interfacial Tension
IOR	-	Improved Oil Recovery
IOWA	-	Oil Wettability Control Agent
IWWCA	-	Water Wettability Control Agent
JHNPS	-	Janus Hybrid Nanoparticles
KYPAM	-	Salinity-Tolerant Polyacrylamide
LHPN	-	Lipophobic and Hydrophobic Nanoparticles
LSW	-	Lifshitz-Slyozov-Wager
MR	-	Micro Reactor
Na-AMPS	-	Sodium- Acrylamide Methyl Propane Sulfonate
NaCl	-	Sodium Chloride
NMP	-	Nitroxide-Mediated Polymerization
NMPy	-	N-methyl-2-pyrrolidone
NWP	-	Neutrally Wet Polysilicon
NWPN	-	Neutrally Wet
O/W	-	Oil-Water
OLCNF	-	Okra Lignocellulose Nanofluid
OLCNP	-	Okra Lignocellulose Nanoparticles
OOIP	-	Original Oil in Place
PAMCS	-	Poly (alkylene maleate citrates
PDI	-	Polydispersity Index

PES	-	Polyesthersulfonate
PFA	-	Performic Acid
PG	-	Propylene Glycol
PHEMA	-	Hydroxyl Ethyl Methacrylate
PLA	-	Poly (lactic acid)
PLGA	-	Poly-D-L-Lactide-co-Glycolide
PMMA	-	Methyl Methacrylate
ppm	-	Parts Per Million
PRSB	-	Petronas Research Sdn Bhd
PS	-	Polystyrene
PSDVB	-	Poly (styrene-divinyl benzene)
PTMC	-	Trimethylene Carbonate
PV	-	Pore Volume
PVA	-	Poly Vinyl Alcohol
RAFT	-	Reversible Additional and Fragmentation Transfer Chain
RC	-	Relative Crystallinity
RI	-	Refractive Index
rpm	-	Revolution Per Minute
SABS-n	-	Sodium 4-(ω-acryloyloxyalkyl)oxy Benzene Sulfonate
SDS	-	Sodium Dodecyl Sulfate
SEM	-	Scanning Electron Microscopy
SERS	-	Surface-Enhanced Raman Scattering
SI-PISA	-	Surface-Initiated Polymerization-Induced Self-Assembly
SLS	-	Sodium Lauryl Sulfate
TEM	-	Transmission Electron Microscopy
TEOS	-	Tetraethylortho-Silicate
TGA	-	Thermogravimetric Analysis
TPE	-	Tetraphenyl Ethylene
USA	-	United States of America
UTM	-	Universiti Teknologi Malaysia
UV	-	Ultraviolet
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

H_2SO_4	-	Sulphuric Acid
G	-	Gram
ºC	-	Degree Celsius
S^{-1}	-	Per Second
wt%	-	Weight Percent
mL/min	-	Milli-Litre Per Minute
CO_2	-	Carbon Dioxide
%	-	Percentage
Ca^{2+}	-	Calcium Ion
Mg^{2+}	-	Magnesium Ion
pH	-	Hydrogen Power
Kg/y	-	Kilogram Per Year
H	-	Hydrogen
C1	-	One Carbon Molecule
C12	-	Carbon-12
A	-	Ionic Functional Group
F	-	Force
τ	-	Stress
m	-	Metre
cp	-	Centipoise
g/L	-	Gram Per Litre
$CaCl_2$	-	Calcium Chloride
$MgCl_2$	-	Magnesium Chloride
$CONH_2$	-	Amide Groups
COO^-	-	Carboxyl Groups
mg/L	-	Milligram Per Litre
mPa.s	-	Millipascals Second
CO	-	Carbon Monoxide
H_2O	-	Water
M	-	Mobility Ratio

So	-	Oil Saturation
Krw	-	Relative Permeability of Water
Kro	-	Relative Permeability of Oil
S	-	Spreading Coefficient
ZrO_2	-	Zirconium Dioxide
NiO	-	Nickel (II) Oxide
TiO_2	-	Titanium Dioxide
MgO	-	Magnesium Oxide
Al_2O_3	-	Aluminium Oxide
CeO_2	-	Cerium Oxide
CNT	-	Carbon Nano Tube
h	-	Height
θ	-	Contact Angle
r	-	Radius of the Interface Area
b	-	Interface area
ψ	-	Electric Potential
z	-	Axis Perpendicular to the Surface
K_B	-	Boltzmann Constant
s	-	Cross Section
η_o	-	Particle Concentration
T	-	Temperature
e	-	Charge of Electron
mN/m	-	Metre Newton Per Metre
SiO_2	-	Silicon Dioxide
bbl	-	Barrels
γ	-	Shear Rate
μ	-	Viscosity
nm	-	Nano Metre
η	-	Intrinsic Viscosity
Fe_2O_3	-	Iron (III) Oxide
$CaCO_3$	-	Calcium Carbonate
ZnO	-	Zinc Oxide
SnO_2	-	Tin (IV) Oxide

ml/s	-	Millilitre Per Seconds
$AgNO_3$	-	Silver Nitrate
ms	-	Millisecond
mL	-	Millilitre
µm	-	Micrometre
NaOH	-	Sodium Hydroxide
mV	-	Millivolt
mol/L	-	Mole Per Litre
mg/ml	-	Milligram Per Millilitre
H_2O_2	-	Hydrogen Peroxide
H_2	-	Hydrogen Atom
Pd	-	Palladium
Pt	-	Platinum
PP-3	-	Polypropylene
$PSiO_2$	-	Triphenylphosphine
Si-O	-	Silicon
P-N	-	Phosphazenes
S-S	-	Polymer Sulphur
B_2O_3	-	Boric Oxide
g/mol	-	Gram Per Mole
g/mL	-	Gram Per Millilitre
Kg	-	Kilogram
mL	-	Millilitre
g	-	Acceleration Due to Gravity
Ge4+	-	Germanium Ion
Ge2+	-	Germanium (II) Ion
v/v	-	Volume Per Volume
W	-	Width
cm	-	Centimetre
L	-	Length
H	-	Height
kHz	-	Kilohertz
W	-	Watt

w_a	-	Weight of Dry Sample after Synthesis
w	-	Weight of sample before synthesis
kV	-	Kilovolt
mA	-	Milliampere
Cu_K	-	Copper Potassium
°/min	-	Degree Per Minute
Ac	-	Crystalline Area
Aa	-	Amorphous Area
°C/min	-	Degree Celsius Per Minute
T_o	-	Onset Temperature
T_m	-	Melting Temperature
K_{Br}	-	Potassium Bromide
σ	-	Interfacial Tension
$\Delta\rho$	-	Density Difference
Cos θ	-	Surface Wettability
R, r	-	Outer and Inner Radii Respectively
Psi	-	Pound Per Square Inch
KeV	-	Kiloelectron Volt
C	-	Carbon
O	-	Oxygen
Mg	-	Magnesium
P	-	Phosphorus
Cl	-	Chlorine
K	-	Potassium
Ca	-	Calcium
$-OSO3^-$	-	Sulphate Ester Group
T_c	-	Endset Temperature
T_p	-	Peak Temperature
T_g	-	Glass Transition Temperature
m	-	Consistency Index
n	-	Flow Behaviour Index
R^2	-	Stability Index
w/v	-	Weight Per Volume

η	-	Apparent Viscosity
ΔG	-	Gibb’s Free Energy
ΔH	-	Change in Enthalpy
ΔS	-	Change in Entropy
P	-	Pressure
γ	-	Free Energy Per Unit Surface Area
N_{ca}	-	Capillary Number
μ	-	Fluid Viscosity
v	-	Fluid Velocity
$\emptyset$	-	Packing Volume Fraction

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Recently, the oil and gas industry are grappling with the reality of scarcity of new sizeable or commercial discoveries and low production from existing reservoirs. Significant amount of oil remains in the reservoir after the primary and secondary recovery methods due to capillary trapping and heterogeneity of reservoirs. To recover the remaining oil, Enhanced Oil Recovery (EOR) methods are the only viable option which can reduce the gap between demand and supply. Since the exploration of new field requires huge amount of capital cost. EOR processes are used to improve the productivity of the fields which aims to recover the oil left in the reservoirs after the primary and secondary oil recovery methods.

To enhance the overall oil displacement efficiency, numerous EOR methods, such as gas, chemical, and thermal, have been devised and utilized. Amongst all the EOR techniques, chemical EOR (CEOR) method has been adjudged as the most promising because of its higher efficiency, reasonable capital cost, technical and economic feasibilities. CEOR methods increase oil recovery by increasing the effectiveness of water injected into the reservoir to displace the oil. Depending on the type of CEOR process, chemicals injected with the water slug alter the fluid-fluid and/or fluid-rock interaction in the reservoir. This includes lowering of the interfacial tension (IFT) between the imbibing fluid and oil or an increment in the viscosity of the injectant for improving mobility and conformance control. Besides, the injected chemicals result in wettability alteration of the rock to increase oil permeability.

The well-known traditional CEOR methods were the polymer flooding, surfactant and/or alkaline flooding. Recently, different modes of chemical flood injections have been studied and applied for EOR processes. Recently, the use of foam enhanced by surfactants and polymers, for mobility control have been studied and found to improve oil recovery. Field practices have shown that polymer flooding can increase oil recovery up to 5-30% original oil initially in-place (OOIP) (Pope, 2011). Hydrolysed polyacrylamide (HPAM) are widely used in polymer flooding because of inexpensive handling cost, relatively resistant to bacterial attack, high solubility in water and high ability to reduce permeability of water. However, with changes in reservoir conditions (high temperature, pressure and salinity) and crude oil properties, existing chemical flooding materials used in CEOR, such as polymers and surfactants do not function desirably. These conditions have detrimental effect on the performance of EOR chemicals, like degradation and precipitation. Therefore, various studies are being carried out to improve the limitation of polymer such as HPAM against high temperature and high salinity reservoir conditions.

More recently, nanofluid flooding has been evaluated and explored as a CEOR process. Nanofluid which is the synergy of base fluid with nanoparticles has the advantages of being more tolerant to high salinity, high temperature, and longer stability and less plugging and retention in highly permeable reservoir. Nanotechnology is the use of nanoparticles ranging from 1 nm to 100 nm size in the study of combination science, medical, engineering and technology. At the moment, nanotechnology has been used in medicine, electronics, electrical, space, science, and engineering (Kaul and Chauhan, 2014). It has enjoyed wide range of use because of its large surface area which makes it easier to interact with solvent molecules when added to make suspension, optical transparency (copper nanoparticle), electrical conductivity (silicon nanoparticle), chemical catalyst (platinum nanoparticle), color change (gold nanoparticle), thermal properties like heat transfer, cooling, insulation, and property of mechanical strength like ultra-high strength of material (Bera and Bel Haaj, 2016).

Nanotechnology has also found its way to petroleum engineering; it is a well-accepted path in the oil and gas industry to recovery more oil trapped in the reservoir. It has recorded success in reservoir characterization, drilling and well-completion jobs (Mahmoud *et al.*, 2016). In EOR, nanoparticle is still in the laboratory stage where its efficiency is being studied and few field trials have been reported (Zabala *et al.*, 2016).

Different laboratory studies (Onyekonwu and Ogolo, 2010; Ogolo *et al*., 2012) and pilot field application have been reported (Zabala *et al*., 2016) that nanofluids can recover oil trapped in the reservoir. Therefore, nanoparticle can change the wettability of the rock surface, reduce IFT between oil and water interface, and lower the chemical adsorption into the reservoir rock surface (Bayat and Junin, 2015).

The methods for formulating nanoparticles can be grouped into two: the 'top down' synthesis or the 'bottom up' synthesis. In the top-down synthesis, nanoparticles are produced by size reduction from a suitable starting material while in bottom-up synthesis, the nanoparticles are built from smaller entities, by joining atoms, molecules, and smaller particles. In top-down synthesis size reduction is achieved by physical and chemical treatment but its limitation is the imperfect nature of the surface structure, which tells us about the surface chemistry and physical properties of the nanoparticles, an example is a physical method. In the bottom-up synthesis, the building block of the nanostructure are first formed and then assembled to form the final particle example is the chemical and biological methods (Thakkar *et al*., 2010). The biological method is most preferable and will be the method for this study.

The main aim of this study is to formulate natural polymers and nanoparticles from agricultural produce and forest products of Malaysia for EOR. The challenges encountered in formulating cheap, readily available and environmentally friendly products that have opened new frontier for research are focussed in this study. The polymer will be produced from cassava starch (CS), okra fruit and *Cissus populnea* (CP) stem/bark. Whereas the nanoparticle will be produced from the synthesis of natural polymers using ascorbic acid extracted from pineapple, citrus and lemon grass assisted with ultrasonic and nanoprecipitation. Starches and cellulose are the most abundant natural polymers in the world. The advantages of selecting them as a starting raw material to produce nanoparticles for EOR is because they are in abundance, readily available, environmentally friendly, and biodegradable. Also, easy to handle, their rigid structure and long polysaccharides chains make them suitable to withstand the harsh reservoir conditions.

1.2 Problem Statement

The main property which is of interest in polymer flooding is the viscosity of the polymer, the main aim is to increase the viscosity of the brine, which will in turn improve the mobility ratio of the oil and brine. In the oil and gas industries, the synthetic HPAM and the biopolymer xanthan are mainly used, but they have their limitations, HPAM is susceptible to high temperature and high salinity, while, xanthan is degradable. Also, they are usually imported from other countries, as such, it takes a lot of time and money before these polymers arrive at their destination of use, and need to be used before the expiring date, these polymers are very expensive. Therefore, there is need to formulate low-cost, readily available polymers that can combat these problems.

Nanoparticles can localize at oil-water (O/W) interface, reduce residual oil saturation, increase the viscosity of brine and decrease the viscosity of crude oil emulsion. Previous studies have focused only on inorganic, metal and metal oxide nanoparticles and have reported excellent results. But cost and environmental concerns have limited full-scale field application of these nanoparticles. With increased demand for energy and inaccessibility to oil deposits, it is important that effective economic and environmentally friendly alternative such nanoparticles derived from natural source are considered.

Nanofluid flooding has been proven to be very effective in EOR. The performance depends on the material and formulation process. Hitherto, most of the published works have focused on the use of classic acid (HCl and H_2SO_4). Acid hydrolysis can produce nanoparticles 5-7 nm in size, but the drawback is the long duration and low yield of the nanoparticles. Also, the use of chemical is a source of concern as nanoparticles obtained by classic acid are limited for practical industrial utilization because the nanoparticles have strong tendency to aggregate especially in dry powder form. Physical treatment such as ultrasonic is a very effective method for the physical disruption of cellular structures. The exposure of natural polymers solution to high intensity ultrasonic can reduce the molar mass. The preparation time becomes shortened and the ultrasonic can effectively prevent aggregation. However, previous studies, reported disruption of the nanoparticle's crystallinity by ultrasonic. They did not consider the use of weak-acid hydrolysis and the intensification of the process parameters. The modification and intensification of the process parameters and homogenization can also enhance crystallinity by ultrasonic. If the crystallinity of nanoparticles is preserved after treatment, the powder products could be readily obtained and their accessibility to industrial items such as composites, nano-fillers, emulsifiers, viscocifiers and stabilizers could be improved.

In this research, a new polymer will be produced from agricultural produce and forest plant. The formulated polymer will be synthesized with ascorbic acid assisted with ultrasonic and nanoprecipitation to improve its properties. The polymers were selected as starting raw material because of their availability, low-cost and their environmental friendliness. They also contain long-chain polysaccharides that can withstand harsh reservoir conditions of high temperature, high pressure (HTHP) and high salinity.

Unlike the detailed studies on inorganic, metal and metal oxide nanoparticles, the use of nanoparticles from a natural source has not been investigated to any significant level in the oil and gas industry for possible application in EOR thus, this research will solve the following problems/research questions:

i. How to synthesize natural polymers with weak-acid hydrolysis assisted with ultrasonic and nanoprecipitation to form nanoparticles?
ii. What is the influence of concentration, electrolyte and temperature on the disperse phases of crystalline starch nanoparticles (CSNP), okra-lignocellulose nanoparticles (OLCNP) and *Cissus populnea* nanoparticles (CPNP) at fluid-fluid interfaces?
iii. What is the effect of concentration, electrolyte and temperature on the dynamic spreading and wetting of CSNP, OLCNP and CPNP in a three-phase contact region (oil-solid-aqueous)?
iv. How is the properties of the natural polymers and nanoparticles in solution and their flow behaviour at reservoir condition?

v. How is the oil recovery performance, if the natural polymers and nanoparticles are applied in EOR process at reservoir condition?

1.3 Research Objectives

The objectives of the research are:

i. To formulate, synthesize and characterize natural polymers and its polymeric nanoparticles.
ii. To evaluate the flow behaviours of nanoparticles and natural polymers in solution.
iii. To determine the IFT and wettability of the nanofluids at reservoir condition.
iv. To evaluate the oil recovery performance of the nanoparticles and natural polymers at reservoir condition and compare with conventional EOR chemical.

1.4 Scope of Study

Based on the objectives listed above, the scope of this research is as follows:

i. Extracting starch from grated cassava pulp and dried in powdered form, whereas, the dried fruits of the okra and dried stem/bark of the CP were blended to powdered form.
ii. Extracting juice from the pulpy fruit of pineapple and citrus, and storage for synthesis, while 5 g of lemon grass was collected, dried and boiled for 20 minutes, the extract was filtered and refrigerated at 4 ºC.
iii. Synthesizing the natural polymers to nanoparticles using weak-acid hydrolysis assisted with ultrasonic and nanoprecipitation.

iv. Isolating and determining the nanoparticles yield after synthesis

v. Characterising CS, okra, CP, CSNP, OLCNP and CPNP by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM) and differential scanning calorimetry (DSC) analyses to determine their chemical structure and surface properties, crystalline structural variation, morphology and thermal stability respectively.

vi. Determining the natural polymers and nanoparticles aggregation sizes and surface charge in distilled water by zeta potential analysis and dynamic light scattering (DLS) to interpret the stability of the polymers and nanoparticles in solutions.

vii. Determining the rheological properties of the natural polymers and nanoparticles at high and low shear rate using a rheometer (1- 1000 s^{-1}).

viii. Determining the effect of brine concentrations (0.9 wt% - 2.2 wt%) and temperature (26-80 ºC) on the natural polymers and nanoparticles viscosity and compare with commercial polymer xanthan.

ix. Determining O/W IFT modification induced by introducing different concentration of nanoparticles (0.05 wt% - 2 wt%), different brine concentration (0.9 wt% - 2.2 wt%) at different temperatures (26-80 ºC).

x. Measuring the contact angle in a 3-phase system (oil-solid-aqueous).

xi. Determining the effect of salinity (0.9 wt% - 2.2 wt%) and temperature (26-80 ºC) on the wettability alteration of the nanoparticles.

xii. Determining the properties of the crude oil and sandstone core samples.

xiii. Investigating the oil recovery performance of the polymers and nanoparticles in comparison to commercial polymer xanthan.

xiv. Performing all the displacement experiment at reservoir condition (temperature of 120 ºC, pressure of 3000 psi and salinity of 2.2 wt%). Restricting a constant flow rate (0.5 mL/min) to represent a laminar flow.

1.5 Significances and Original Contributions of this Study

The novelties of this research can be listed as follows:

i. The use of weak-acid hydrolysis assisted with ultrasonic and nanoprecipitation to produce nanoparticles.
ii. The use of CS, okra, CP as a displacing fluid at reservoir condition.
iii. The use of CSNP, OLCNP, CPNP for EOR and evaluating oil recovery mechanism by measuring the nanofluid viscosities, IFT between the crude oil and nanofluid, wettability alteration through contact angle in a three-phase contact region (oil, nanofluid, sandstone).

Whereas, the contribution to knowledge of this research work are:

i. This research will provide cost effective and readily available polymer and nanoparticles for EOR applications.
ii. This research adds new insight and open new frontiers to the synthesis of natural polymers using weak acid from plants for EOR. And its efficiency in altering wettability and reducing IFT of O/W system for EOR.
iii. The natural polymers and nanoparticles formulated can be used in other areas of oil and gas such as; drilling engineering as drilling fluid additives, well completion and workover jobs and cementing jobs.
iv. This research provides a basis for controlling the rheology, IFT and wetting of CSNF, OLCNF and CPNF which can be useful in a lot of practical applications such as confectionary, pharmaceuticals, coating products, freeze-casting, drilling fluids and EOR.

1.6 Thesis Structure and Organization

In chapter one, an introduction to this research is discussed, the objective of the research, problem statement, scopes of research and significance of study are clearly stated. Chapter two discusses the fundamental, methods and polymers used in EOR. The properties of natural polymers in solution, their flow behaviours and the review of existing literatures using natural polymers in EOR were also discussed. It also discussed the introduction to nanotechnology and mechanism of nanoparticles in EOR. Oil displacement using nanoparticles in EOR were reviewed. The chapter also discussed the synthesis of nanoparticles and natural polymers. The gap in the previous research are identified and the importance of the current study is highlighted. The sample preparation procedures, the experimental set-up and procedures, apparatus and theory behind the calculations, and finally the methodology used in achieving the desired objectives is discussed in chapter three. Chapter four presents the characterisation results of the polymer and nanoparticles. The results of the physical properties of the nanoparticles and their flow behaviour in comparison to that of natural polymer and xanthan was discussed. The results from the displacement test and the mechanism of oil recovery were also discussed in this chapter. The conclusions and main findings from this research with future recommendations are highlighted in chapter five.

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