

# Application of Aluminium and Iron Oxides Nanoparticles and Tardigrade-Based Microbial Enhanced Oil Recovery (MEOR)

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**Abstract:-** Enhanced oil recovery is a different approach to enhance oil production from existing reservoir. The enhanced oil recovery (EOR) process uses microorganisms, chemicals and produced water to extract the residual oil trapped in the capillaries of reservoir rocks. This research focuses on the use of nanoparticles and Tardigrade in solving problems of recovering entrapped oil in the formation. Enhance Oil Recovery using Tardigrade (Micro-Animal) compared with Nanoparticle (Aluminium oxide & Iron oxide) was used. The Effects of Tardigrade and Nanoparticles on API Gravity, Flash point, kinematic Viscosity, Cloud Point, Density and contact time were investigated. The Nano Particle were characterized on the basis of moisture content (2.75%), bulk density (8.2 m/v), ash content (2.2%) and methylene blue test (0.63 g/kg). Crude oil was obtained from Otakikpo marginal oil field and its rheological and physiochemical property was determined. Five samples (A, B, C, D and E) were formulated as follows: Sample A 10 liters of crude oil alone. Sample B 5grams of Tardigrade and 10 liters of crude oil, Sample C contains 5grams of aluminium nanoparticle and 10 liters of crude oil, Sample D 5grams of iron oxide nanoparticle and 10 liters of crude oil, Sample E is the mixture of 5grams of tardigrade, 5grams of aluminium oxide and 5grams of iron oxide, 10 liters of crude oil were mixed and tested to determine the volume of oil recovered. The result shows that there was a rapid increase of oil recovery in sample B, which yielded 15L, Sample A with a recovery of 7.8L, Sample C recovering 9L, Sample D yielded 11L, Sample E yielded 19L, the mixtures of sample (E) gave the maximum yield 19L flow within 10mins. The result reveals that the addition of Tardigrade gave quick increase in the recovery of oil. The alteration in the viscosity and density of the crude oil indicates the possibility of achieving higher recovery with the application of Tardigrade. It gave an increase in the recovery rate of oil production and will reduce cost on EOR. Therefore, based on the results obtained, Tardigrade (Micro-Animal) have the potential of being an efficient tool for Enhance Oil Recovery of Crude oil in attempting to resolve the age long challenge in recovering significant amount of residual oil left behind in the reservoir after primary and secondary oil recovery in Oil and Gas industries.

**Keywords:** Aluminium Oxide, Iron Oxide, Tardigrade and Microbial Enhanced Oil Recovery (MEOR)

## I. INTRODUCTION

Auxiliary recuperation procedures have shown a normal of 35-45% of original oil in place (OOIP) [1]. With the utilization of essential and auxiliary recuperation techniques, around 60% of OOIP stays in the repository. This has given explanations behind upgraded oil recuperation, which is a tertiary recuperation technique. Microbial Enhanced oil recuperation

is an alternate way to deal with upgrade oil creation from existing repository. The Microbial improved oil recuperation (MEOR) process utilizes microorganisms to separate the lingering oil caught in the vessels of repository rocks. It is generally utilized when the warm and synthetic EOR strategies have been executed and a lot of unrefined petroleum actually still needs to be recuperated from the supply rocks. Chosen miniature creatures are equipped for processing hydrocarbon delivering natural solvents. The utilization of bio-surfactants for improved oil recuperation applications. Bio-surfactants can bring down interfacial pressure, fundamentally and change the supply rock wettability toward good condition in microbial improved oil recuperation (MEOR). Nano technology has shown great potential in extracting about 60% of original oil in place (OOIP) that is left in the reservoir after primary and secondary recovery stages, increase oil production rates from existing fields, and fill the gap between energy supply and demand worldwide Nano particles can alter the reservoir wettability by absorbing on the reservoir rock to develop a new surface roughness. They can also absorb into oil/water interface and decrease the interfacial tension between the two phases. Nano particles also have the ability to increase the viscosity of EOR fluid and reduce the viscosity of heavy crude. Because of their ability to alter crude oil or reservoir properties. Research have shown an increase recovery of oil by Nanoparticles and biosurfactants. But comparing the effect of the combination of different nanoparticles and Tardigrade is not known.

This strategy is extensively more conservative and harmless to the ecosystem than other EOR techniques. Energy that is utilized in microbial cycles to upgrade oil recuperation doesn't rely upon the cost of raw petroleum. Organisms can develop freely under many circumstances and produce a lot of helpful items quickly from modest, sustainable materials that are accessible in enormous amounts. As an organic specialist, microbial bio-items are frequently biodegradable, which brings about lower levels of contamination and a low poisonousness [2], [3].

[4], directed a review utilizing three native kinds of microbes: *Bacillus subtilis*, *Pseudomonas aeruginosa*, and *Bacillus cereus*. Every one of the bacterial strains delivered biosurfactants fit for emulsifying raw petroleum. Besides, the biosurfactants exhibited great steadiness under unforgiving ecological circumstances (pH 4, 25 g/L saltness, and 120°C) like those found in oil supplies. In a research center scale oil dislodging explore different avenues regarding lamp oil, the

biosurfactant from *Bacillus subtilis* recuperated 25% of the extra oil.

The biosurfactant was fermentatively produced from *Bacillus* strains, and the viability of rough biosurfactant union went from 30.22 to 34.19 percent of the water flood remaining oil immersion in a sand pack section [5]. There have been different reports on the capacity of *Bacillus* strains to make biosurfactants for MEOR [6]. Different examinations which detailed the effectiveness of biosurfactants in upgraded oil recuperation incorporates, rhamnolipids created by *Pseudomonas aeruginosa* recuperated 27% of the first oil after water flooding in sand stuffed section [7] biosurfactant delivered by *Rhodococcus* sp. strain TA6 recuperated 70% of leftover oil from oil immersed sand packs [8].

## II. LITERATURE REVIEW

### A. Mechanisms of Oil Recovery by Microorganisms

A sound understanding of the basic mechanisms governing enhanced oil recovery is important in order to get the best results of increased recovery. Oil recovery by microorganisms is achieved through a number of mechanisms which include; Degradation of Crude Oil serves as food source for microorganisms as they use carbon in crude oil as energy source for their growth. Bacteria support its life processes by degrading crude oil. Degradation of oil by microorganisms could be aerobic or anaerobic. Microbial activities have resulted in significant biodegradation of crude oils worldwide for millions of years and changes in oil quality [8] "The degradation of hydrocarbons has a significant effect on the composition of crude oil. Under anaerobic conditions, *Bacillus* strains have been known to degrade large alkyl chains thereby reducing the viscosity of hydrocarbon mixtures [9]. The degradation of crude oil by microorganisms" leads to break down of heavy fractions into lighter fractions which are less viscous and flow easily to the surface.

The delay communication of metabolites with the oil in the repository, replaces the oil properties so that fixed unrecoverable oil is changed over into mobile oil that can stream to the creation wells expanding oil yield appropriately. Microbial corruption of oil, which is a perplexing synthetic compound, is the consequence of digestion of microorganisms where the digestion's results of the hydrocarbon-oxidizing microscopic organisms act as the substrate for other physiological gatherings in the repository [10].

The advantages of Microbial Enhanced oil recovery are:

- i. The injected bacteria and nutrients are cheap and simple to procure and use in the field.
- ii. MEOR procedures are economically appealing for marginally producing oil fields and are viable alternatives to abandoning marginal wells.
- iii. Microbial cell factories use minimal energy to create MEOR agents.
- iv. Compared to other EOR technologies, MEOR technologies require less change of current field features to conduct the recovery process, making them more cost-effective to install and apply.

[12], conducted research on the isolation and characterization of biosurfactant generating bacteria for use in enhanced oil recovery. A biosurfactant-producing bacterial strain was

isolated, tested, and identified in the study. The isolated strain produced biosurfactants and was identified as Genus-*Pseudomonas* species *aeruginosa*. The isolate grew best at a pH of 7.0 and a temperature of 37°C. The biosurfactant produced by the isolate was detected as rhamnolipid and having significant reduced interfacial tension with good stability at harsh condition of the reservoir. The results showed that the produced biosurfactant was highly efficient for MEOR. Several studies suggest that biosurfactants improve oil recovery through a variety of methods, including lowering interfacial tension and surface tension between oil and water, forming oil-water emulsions, and altering rock wettability from oil wet to water wet [13].

### B. Biosurfactants Production

Biosurfactants are amphiphilic molecules that are produced by microorganisms. Biosurfactants are also known as surface active substances. One of the mechanisms for improved oil recovery through the use of biosurfactants is emulsification which results from the combination of water and oil phase to form a middle phase. A mixture of the oil and water phase results in the swelling of the oil volumes leading to increase in oil phase, saturation and permeability. The high level of toxicity of chemical surfactants as well as their large costs has necessitated the need for the use of biosurfactants which are cheaper, less toxic, environmentally friendly, safe and easy to handle. The production of biosurfactants for MEOR has shown a lot of potentials for recovering substantial quantities of residual oil from mature oil fields [10].

### C. Production of Biomass

Biomass produced by microorganisms as a by-product of their metabolism accumulates between the oil and the rock surface of the well, increasing its displacement efficiency thereby increasing oil recovery. Biomass improves oil recovery by plugging of high permeable zones due to rapid growth of microbial cells at the large pore channels thereby displacing residual oil towards producing wells [14].

### D. Production of Gases

Microorganisms in oil reservoirs produce gases such as CO<sub>2</sub>, CH<sub>4</sub>, H<sub>2</sub>, N<sub>2</sub> which increase the pressure of the reservoir dissolve in to oil causing it to swell, thereby reducing its viscosity and subsequently increasing the ease of flow. Microorganisms that produces gases includes; *Clostridium*, *Enterobacter*, *Methano bacterium*, *Desulfovibrio*, *Pseudomonas* and certain methanogens. These gases also find application in pressure depleted reservoirs where they contribute to pressure buildup [14].

### E. Biosurfactant

Biosurfactant is applied both ex-situ and in-situ. In ex-situ Biosurfactants are created external the oil-wells and either can be infused as slug(s) trailed by water flood or straightforwardly broke up in infusion water and applied with next to no hermit period and recuperation rate is checked and dissected. In-situ, potential biosurfactant creating microorganisms are infused in the oil wells alongside or without extra supplements; essentially adding the supplements to the oil wells and increment development and action of native biosurfactant delivering organisms and upgrading the oil recuperation. By

and large the microorganisms or the supplements are infused to the oil-wells as slug(s) trailed by water-flood and closing the well for 1-6 months, trailed by water-flood and the recuperation rate is investigated [2].

#### F. Application of MEOR

Before the utilization of MEOR advances, the tasks are evaluated to decide the similarity of the raw petroleum and repository properties with MEOR considering the physicochemical properties of the raw petroleum, supply creation execution, and repository properties (i.e., temperature). At the fundamental stage, repository liquid examples are gathered and tried for similarity with the MEOR frameworks. The principal stage is the distinguishing proof of the native hydrocarbon-consuming microorganisms, which is as of now adjusted to the in-situ supply conditions; after which the best activity technique for each task is planned and created. MEOR can be applied on individual wells as follows:

- i. From the well being treated.
- ii. From the target well and adjacent wells of the same reservoir.

The MEOR arrangement is infused into neighbouring wells similarly as water is infused into the supply. The volume of the MEOR biomaterial to be infused is determined in light of the pore volume of the objective supply. The arrangement is blended and siphoned through the infusion very much followed by the infusion of water to drive the organic arrangement into the oil soaked zones. Then, the treated well is closed in for the necessary timeframe (regularly 24 h to 7 days) after which oil creation is continued. This technique is reshaped each 3-6 months to empower microorganisms to move further into the store to oil immersed zones [11].

#### G. Nano-Technology (Nanofluid/Nano-Particles)

Nanofluid flooding is the most recent chemical enhanced oil recovery method, it focuses on the use of nanoparticles that are metal oxides such as Tin oxide, Silicon oxide ( $\text{SiO}_2$ ), Zinc oxide ( $\text{ZnO}$ ), Iron oxide ( $\text{Fe}_2\text{O}_2$ ), Magnesium oxide ( $\text{MgO}$ ), Aluminium oxide ( $\text{Al}_2\text{O}_3$ ), Nickel oxide ( $\text{Ni}_2\text{O}_3$ ), and Zirconium oxide ( $\text{ZrO}_2$ ). Nanoparticles are particles with diameters ranging from 10 to 500nm; this particle aids in the alteration of reservoir factors and the alteration of oil characteristics, resulting in improved recovery. Some of the mechanisms are wettability alternation, fluid property changes, improved trapped oil mobility, sand consolidation, and decreased interfacial tension (IFT), and formation wettability alternation, i.e., Water-wet formations are said to produce more than oil-wet formations. Although intermediately wet formations produce better than water wet formations nanoparticles affect the wettability of the rock for successful oil recovery [15]. A nanoparticle such as aluminium oxide ( $\text{AL}_2\text{O}_2$ ) alters the viscosity of oil at the surface. It has been proposed that iron oxide nanoparticles have the power to enhance the viscosity of displacing fluids [16]. Nanoparticles are often distributed in a fluid, such as ethanol, distilled water, brine, or, in certain situations, diesel. [17], conducted a study on the usage of nanoparticles suspended in various dispersing fluids for injection into sand and sand soaking in various nano9-fluids. The results showed that the kind of fluid used for

dispersing nanoparticles, as well as the kind of nanoparticle employed, are critical factors in improving oil recovery.

#### H. Tardigrade

Tardigrades have a place with this gathering of numerous semi-sea-going (or semi-earthly) types of this taxon and the flowing variety Echiniscoides can endure all out drying up in a condition of totally captured digestion called cryptobiosis [18] Cryptobiosis actuated by drying up is called anhydrobiosis [19] however the cryptobiotic state may likewise be incited by freezing (cryobiosis), osmotic tension (osmobiosis) and anoxia (anoxybiosis). When presented to parching, tardigrades contract into a supposed tun, and the penetrability of the fingernail skin declines [20] this decreases the pace of happening and gives the creature time for blend of tissue protectants, for example trehalose. As per the primary speculation, these protectants supplant the bound water in layers and permit underlying cell trustworthiness to be kept up with in totally dry creatures [21]. Anhydrobiosis permits tardigrades and a few other miniature metazoans (for example nematodes, rotifers and scavengers), protozoans and plants, to continue in conditions prohibited to different living beings. Aside from a security against parching and freezing under normal circumstances, anhydrobiosis likewise permits an opposition against unnatural abiotic limits.

Tardigrade (miniature creature) in a real sense signifies "slow steppers." But they're generally regularly known as water bears because of their strolling walk looking like that of a bear. They have eight legs, three sets for strolling and one sets for getting a handle on, each closure in hooks. A Tardigrade's normal life expectancy, a few carry on with a couple of months, while others can live for as long as 2 years. In any case, they can likewise go into a lethargic state which extends their life expectancy considerably further. In this state, they can go over 30 years with next to no food or water. Tardigrades are truly small and are best seen through a magnifying lens. Most species are in the scope of 0.3 to 0.5 mm (or 0.012 to 0.02 inches). Having said that, the biggest types of water bear, in any case, you don't have to take to such drastic courses of action to see one. They are most usually tracked down on greeneries and lichens. As a matter of fact, one normal epithet for Tardigrades is "greenery piglets." A Tardigrade's got dried out structure is known as a tun and it can live in this state for a considerable length of time prior to being rehydrated. It does this by delivering a unique protein, called TDP, which replaces water. Tardigrades can be carried on the breeze or on the feet of different creatures to another climate. It really is something else that far this can take them. When they arrive at a more neighbourly climate, they can return to their regular structure and begin another state and can be lowered in unsafe synthetic substances causes little to damage. Liquor is extremely harming to cells and can totally disturb their construction in under a moment. Tardigrades can endure being drenched in liquor for a whole day. As though that wasn't adequately great, there are instances of Tardigrades enduring carbolic corrosive openness, as well as openness to hydrogen sulphide, which is both exceptionally combustible and profoundly destructive.

This implies that Tardigrades don't simply get their hereditary material in the typical way from parent to posterity, however by different means too. Their Genes Could Be Used to Preserve Food in The Future. Researchers have previously



affirmed that that the Tardigrade qualities for creating TDPs can be utilized to shield yeast and microscopic organisms from drying out. They currently imagine that a similar thought could be applied to harvests to ensure that they can endure dry seasons. This strategy actually might assist with creating drug that needn't bother with to be refrigerated. As you can envision, this would be a truly helpful turn of events, particularly in more unfortunate nations where emergency clinics don't necessarily have the gear that they need. There Are Possibly Tardigrades On the Moon.

The Israeli lunar lander, Beresheet, was sent off in April 2019. They had ready, in addition to other things, a whole province of Tardigrades. Tragically, the lander crashed onto the Moon's surface, which carried the mission to a startling end. However, given their close to indestructible nature, all things considered, the Tardigrades endure the effect and are still there in a dried out state. Tardigrade can so broad and ready to endure such countless various conditions, including the vacuum of room, some figure that this might be where they started. In this note, we center around quite possibly of the most successive story about tardigrades, that of very long term endurance in cryptobiosis, and show an extensive error between current realities from the first record and resulting reports. following twelve days from the outset of the hosing, in the mostly expanded example (scarcely recognizable to species yet absolutely to credit to the request Eutardigrada and liable to the variety Macrobiotus) trembles in a few zones of its body were noted. Specifically, in the front legs and it was seen to broaden development followed by withdrawal. For this situation, it was not the typical interaction by which the tun, after hydration, changed into a close to ordinary state of the creature however without indication of something going on under the surface. All things considered, there was a development, which drove us to presume that an action of life showed up, regardless of whether extremely slight.

Creatures and plants that can restore following quite a long while in cryptobiosis need to have an exceptionally effective innovation to safeguard their cells against ecological obliteration like oxidation and radiation. Information about the organic chemistry, physiology and hereditary qualities of this normal innovation will be of impressive significance for creating techniques for long haul conservation of natural material, for example in the food and clinical enterprises. Likewise, regardless of whether the capacity of long haul cryptobiotic endurance can't be viewed as a transformation, and has no endurance esteem today, it actually addresses a pre-variation to conditions that might be acknowledged from now on. Consequently, the chance of cryptobiotic endurance over long stretches of time ought not be excused as a natural interest. Rather, we really want more researcher concentrates on this peculiarity. Tardigrades are interesting creatures, and cryptobiosis is to be sure one of the most momentous transformations among living organic entities. It merits impressive consideration, as we actually see moderately bit of its overall science and organic chemistry, and know hardly anything about its natural and developmental ramifications. Thusly, in spite of the fact that tardigrades will be unable to endure hundred years in cryptobiosis, they will unquestionably.

### I. Mechanism of Different Types of Nanoparticles

A few sorts of nanoparticles and a large portion of which are silicate and metal oxide nanoparticles have been applied to EOR. Silicate mixtures, for example, SiO<sub>2</sub> have been generally utilized in the oil field because of their wide accessibility, minimal expense, and low poisonousness. Accordingly, reasonable particles can be chosen to develop Nano liquids in view of these exceptional properties.

Contrasting the EOR systems of various nanoparticles, it is not difficult to track down that the non-metal particles addressed by silicate basically further develop the oil recuperation by changing the wettability and lessening the water-oil interfacial strain. The outer layer of these particles has certain hydrophilic/lipophilic gatherings, which favor their adsorption at the oil-water point of interaction and assume a comparable part as surfactants (Liu *et al.*, 2020).

#### i. Oil Displacement Mechanism of Nanofluids

To more readily comprehend the impact of different variables on the development of nanofluids, the conceivable oil relocation systems of nanofluids are summed up in this part. Nanofluids in the field of petrol allude to uniform and stable liquids made by adding at least one nanoparticles to the base arrangement with a specific goal in mind and legitimate extent (the size is by and large under 100 nm). Numerous exploratory investigations of miniature model flooding have demonstrated the way that nanofluid infusion can lessen the lingering oil content. A few different investigations demonstrated the way that the oil recuperation could be expanded by 25% while utilizing nanofluids rather than deionized water. The consequences of other nanofluid dislodging tests additionally showed that the oil recuperation rate could be expanded by 4.48-10.33%. [11]

#### ii. Effects of Nanoparticles on Oil Recovery

A mix of surfactants and NPs causes the arrival of oil drops, which are caught in flimsy throats and miniature channels of the supply rock. These peculiarities are connected with a specialist that lead to an expansion in oil recuperation; these incorporate boundaries, for example, the wettability modification of repository rocks, unconstrained emulsion development, changing IFT between supply liquids and stream properties of permeable media. They additionally concentrated on the interfacial properties of water-oil, dilatational viscoelasticity, IFT and zeta possibilities. The outcomes showed that rising NP focuses cause a diminishing in the IFT and zeta capability of oil drops and expands the dilatational viscoelasticity. With expanding NP focuses, dilatational viscoelasticity and IFT were expanded, while zeta potential became steady after 250mg/l fixation. In reality, in the weighty oil repository, polymer-covered nanoparticles can change the pH states of the supply and cause recuperation of weighty oil, which is displayed in Figure 2.4

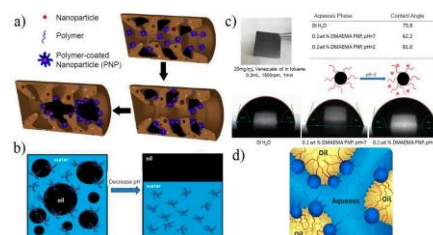


Fig 1: Schematic Representation of (Source: Liu *et al.*, 2020).

- i. The use of polymer-coated NP additives to recover heavy oil trapped in porous media
- ii. The emulsification of oil-in-water emulsions with changes in solution pH,
- iii. Wettability alteration by polymer-coated NPs and
- iv. Schematic of oil–water micro emulsion phases with polymer-coated NP additives.

### III. MATERIALS

The following materials and reagents were used for the experiment;

Table 1: Materials and Reagents

Materials	Reagents
Pycnometer	Ethanol
Beaker	hydrogen peroxide
Capillary viscometer	Kovac reagent
Measuring Cylinder	crystal violet
Thermometer	phenol red
CO <sub>2</sub> gas cylinder	Tetramethyle-phenylendiamine,
Weighing balance	dihydrochloride
Spatula	biritt reagents
Distilled water	
Autoclave	
Hot-air	
Oven	
Incubator	
Bunsen burner	
Microscope	
Stop watch	
Capillary viscometer	
Hydrometer	

### IV. METHODS

The set up utilizes a carbon dioxide chamber (CO<sub>2</sub>) that serves as the supply pressure, this chamber is associated with a 12liter metal tank which stands as the repository, a line is associated with this tank which along its line includes a tap handles (remains at the well head valve) and a tension check used to peruse the tank outlet pressure (repository outlet pressure), a condenser to consolidate any gas on the off chance that present, a subsequent tap handle which remains as a valve which prompts the assortment holder (capacity tank).” The procedure is as follows; 10 litres of crude and is measured and poured into the tank. Make sure the carbon dioxide cylinder is closed, make sure the tank is empty and the flow line is free from any liquid. Make sure the tap handles are in closed positions. Fill the tank with the measured quantity of the crude that will to be recovered and close the tank properly to avoid spill of the content. Once the cylinder is opened, the tap handles should be opened and the stop watch set for 10 minutes. Record the inlet pressure and outlet pressure as the fluid flows. As the 10 minutes elapses the carbon dioxide cylinder should be closed, the tap handles should also be returned to a closed position. Record the volume of the crude and water recovered, transfer the recovered mixture into a 1000ml cylinder and allow to settle. Empty the tank totally. After the mixture settle, record the water cut and calculate the amount of water and crude recovered. Take a sample of the recovered crude and test for its properties again and label it sample (A). Repeat the process for samples (B, C, D and E). Presented in Fig 2.



Fig 2: Enhanced Oil Recovery Set-up

#### A. Crude Oil Density Determination

The weight of empty dry pycnometer was determined with a weighing balance after which the pycnometer was filled with crude oil and re-weighed and ensure that there is no bubble inside the pycnometer containing the crude oil, the weight was recorded. The procedures were repeated for concentrations. This procedure was repeated; for the crude oil alone after recovery, for crude recovered using Tardigrade and for crude oil recovered using bacteria and nanoparticle (aluminium oxide & iron oxide). The density of the crude was calculated by subtracting the weight of the filled pycnometer from weight of the empty pycnometer and dividing the result by the volume of the pycnometer.

$$\text{Density } (\rho) = \frac{M_2 - M_1}{V} \quad (1)$$

$$\text{API}^o = \frac{141.5}{S.G} - 131.5 \quad (2)$$



Fig 3: Weighing Balance and Pycnometer

#### B. Crude Oil Flash Point Determination

The Pensky-Martens flash point tester consists of a closed-cup tests arrangement that contains any vapour produced and essentially simulate the situation in which a potential source of ignition is introduced into a container. For this test, a test portion is introduced into and a close-fitting lid is fitted to the top of the cup, the cup and test portion are heated and stirred, then opened in the lid to allow air into the cup and also the ignition source to be dipped into the vapors to test for a flash.” Before the crude was poured into the Pensky-Martens, the brass test cup was cleaned and dried, the brass test cup was filled with the sample (crude oil) to the specified mark. The cup was fitted into the pensky – Marten and covered properly, the pensky-marten was connected to an electric source to heat and stir the liquid in the brass cup. At regular interval, the ignition source was directed to the ignition point to check if the

flash had occurred, if it does not continue to heat, stir and check till it ignites. After the ignition had occurred, the temperature was recorded and the procedure was repeated for enhanced oil recovery using Tardigrade and the nanoparticles.



Fig 4: Pensky – Marten Flash Point Tester

### C. Crude Oil Viscosity Determination

The redwood viscometer comprises of vertical barrel shaped oil cup with a hole in the focal point of its base. The hole can be shut by a ball. A snare facing up fills in as an aide mark for filling the oil. The barrel shaped cup is encircled by the water shower. The water shower keeps up with the temperature of the oil to be tried at consistent temperature. The oil is warmed by warming the water shower through a drenched electric warmer in the water shower. The arrangement is made for blending the water, to keep up with the uniform temperature in the water shower and to put the thermometer to record the temperature of oil and water shower. The chamber is 47.625mm in width and 88.90mm profound. The hole is 1.70mm in width and 12mm long.

The redwood viscometer cylindrical oil cup was cleaned, and the orifice tube is kept free from dirt. The orifice was closed with the orifice bulb and a 50 ml measuring cylinder was set below the opening of the orifice. The cylindrical oil cup was filled with crude oil to the mark in the cup. Thermometer was inserted in the crude oil to measure the crude oil's initial temperature, the orifice bulb was lifted up and the oil flowed through the orifice into the measuring cylinder and the time was taken in seconds using a stop watch. The procedure was repeated for enhanced oil recovery using Tardigrade and nanoparticles.

### D. Synthesis of Nanoparticles by Co-Precipitation Method

The starting materials were aluminium trichloride ( $AlCl_3$ ), ethanol and 25% ammonia solution as precipitant agent. At first,  $AlCl_3$  was dissolved in 150 mL ethanol and a little amount of distilled water was added to get a transparent solution. 60 mL  $NH_3$  was added to the stirred  $AlCl_3 \cdot 6H_2O$  solution drop by drop at the rate of 2.5 mL/min until precipitation became white as gelation of  $Al^{3+}$  cations in the form of hydroxides  $Al(OH)_3$ . After filtering in vacuum system, drying at  $200^\circ C$  for 2 h in an oven, and annealing at 750, 1,000 and 1,250 $^\circ C$  for 1 h, a white fine alumina nano-powder was obtained to study the effect of precipitant concentration on the final structure of nanoparticles. Other samples were prepared with 10 and 30 mL  $NH_3$  under the same conditions.

### E. Characterization of Samples of Nano Particle

The Nanoparticle was characterized based on Bulk density, Methylene Blue number, moisture content and Ash content.

The morphology, the micro-structure, homogeneity and the particle size of alumina powders were examined with the transmission electron microscope.

#### i. Bulk Density

A centrifuge bottle is filled with Nano particle up to 10ml by volume and tapped for ten (10) times. The Nano particle was weighed with a weighing balance to obtain the mass of the Nano particle. The bulk density was calculated by dividing the mass of the Nano particle used.

$$\rho_b = \frac{\text{mass of test tube plus sample} - \text{mass of empty test tube}}{10} \quad (3)$$

#### ii. Adsorption of Methylene Blue Dye

0.2g of each sample of the Nano particle was poured into 20ml of the methylene blue (MB) dye solution and transferred into labelled bottles and shaken thoroughly using a mechanical shaker for 20 mins to attain equilibrium condition for adsorption. Therefore, the carbons were filtered and the adsorptions were determined. The concentrations of methylene blue before and after adsorption were determined using a Jenway 6405UV/spectrophotometer at 500nm wavelength. The amount of adsorption at equilibrium, was calculated using the formula

$$MB = (C_o - C_e) V/M \quad (4)$$

where

$C_o$  = Liquid-phase concentration of the dye at initial concentration. Concentration (mg/L)

$C_e$  = Concentration of the dye at equilibrium state

V = Volume of the solution (l)

M = Mass of dry adsorbent used (g)

#### iii. Moisture Content

Some Nano molecule when put away under damp circumstances will adsorb significant dampness over a time of 1-3 months adsorb as much as 25 to 30 % dampness nevertheless seem dry. For some reasons, this dampness content doesn't influence the adsorptive power until the overall stickiness surpasses 75 %, however clearly it weakens the Nano molecule (Tierney et al, 2006).

The moisture content was obtained using; % Moisture

$$\text{content} = \left( \frac{(b-c)g}{a(g)} \right) \quad (5)$$

where

Mass of crucibles + original Sample = (b) gram

Mass of crucible + sample after drying = (c) gram

Original mass of sample = a (g)

#### iv. Ash Content

The ash content of a Nano particle is the residue that remains when the carbonaceous material is burnt off. As Nano particle contain inorganic constituents derived from the source materials and from activating agents added during manufacture, the total amount of inorganic constituents will vary from one grade of carbon to another. The inorganic constituents in a carbon are usually reported as being in the form in which they appear when the Nano particle is ashed. Ash content can lead to increased hydrophilicity (ability to dissolve in water) and can have catalytic effects, causing restructuring process during regeneration of used Nano



particle.” The % ash content (dry basis) was obtained from the equation below;

$$\% \text{ Ash} = \frac{F - B}{G} \times 100 \quad (6)$$

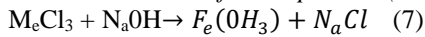
Where,

F= Mass of crucible + ashed sample (g)

B= Mass of crucible + dried sample (g)

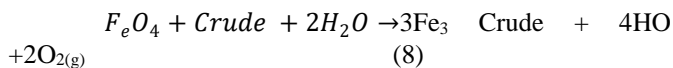
G= Mass of empty crucible (g)

v. *Production of Nano particle (Fenton)*



6g of iron chloride (fecl<sub>3</sub>) is dissolved in 25ml of distilled water 14g of sodium hydroxide little by little to give a dark colour precipitation. Both the nanoparticle (fention) reagent where dried into powder form produced 69.48g (fention)

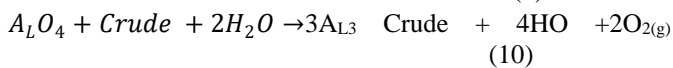
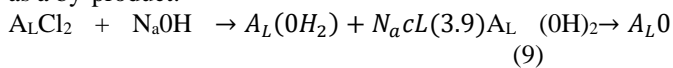
Fsention reagent is a soluble iron (II) salts reacting with Crude and Water.



Ferric iron reacted with crude oil in the presence of two molecules of water to produce 3 molecule of ferric iron crude and 4 molecules of water giving out oxygen as a by-product.

vi. *Production of Nano Particle (Aluminium Oxide)*

40g aluminium hydroxide dissolve in 250ml distilled water (exothermic reaction, co-precipitate method). 26g of aluminium (ii) chloride is dissolve in 160ml of distilled water endothermic reaction ‘wet chemical method’ both were mixed together to give a light yellowish substance and 74.86 grams of aluminium oxide was produced, Filtered, dried at 80<sup>o</sup>c and aluminium (II) reacting with Crude oil and Water in the presence of two molecules of water to produce 3 molecule of ferric iron crude and 4 molecules of water giving out oxygen as a by-product.



V. RESULTS

i. *Characterization of the Crude*

The crude oil was characterized on the basis of cloud point, density, viscosity, flash point and API gravity. The physiochemical properties of crude oil before and after being recovered are shown in Table 4.1, the change in the physiochemical properties of the crude oil shows that nanoparticle and tardigrade has significant impact on the crude oil. From the analysis, it was noticed that cloud point, density, viscosity, flash point and API gravity increased after the application of nanoparticle and tardigrade. From the result, analysis of the crude oil sample before the application, it was revealed that the crude oil has lower values of physiochemical properties. The analysis showed that sample B and E recovered more crude oil and it is represented in Table 2.

Table 2: Characterization of initial Crude Oil

Parameters	Value
Density @ 30°C	0.9088g/ml
API Gravity	25.20°C
Viscosity	0.0787cp
Flash point	104 °C
Cloud point	2.8°C

ii. *Characterization of Nano Particle*

Table 3 shows the characterization of Nano Particle. The parameters considered for the characterization of Nano Particle include particle size, bulk density and ash content.

Table 3: Shows the Characterization of Nano Particle

S/N	PARAMETERS	NANO-PARTICLE	UNIT
1	BULK DENSITY	8.2	g/cm <sup>3</sup>
2	ASH CONTENT	2.2	%
3	pH	5.4	
4	PARTICLE SIZE	140	nm

iii. *Pressure Yield during Recovery*

Fig 5 is the plot of pressure against sample. The inlet pressure for samples A, B, C, D and E was maintained at 3.5 bar. The outlet pressure for sample A and E is 1.7 bar. The outlet pressure for sample B is 1.4 bar. Sample C and D is 1.5 bar.

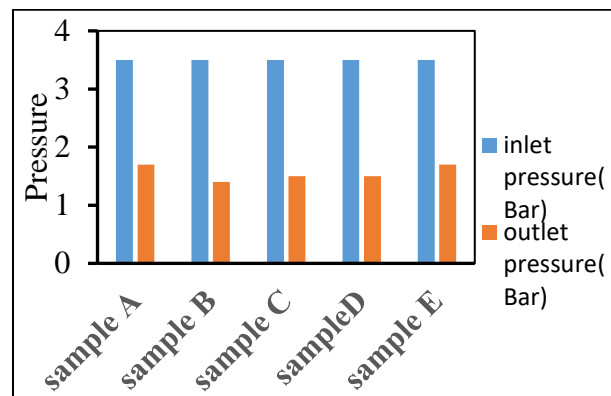


Fig5: Pressure Differential of the EOR

iv. *Relationship between Total Volume Recovered and Oil Recovered*

The Total volume of the recovery for the different samples were obtained under the same temperature and pressure conditions containing samples of the same quantity and properties. The samples total recovery volume varies and so also does the oil recovered for each sample vary. From the graph below, the samples with the largest volume recovered are sample E, sample B and sample D; sample C and sample A had the lowest volume recovered. The samples with the highest volume of oil recovered are sample E and sample B, followed by sample D and sample C, sample A had the lowest volume of oil recovered presented in Fig 6. The water cut calculation from the experiment is shown in Appendix A2. Table 4.4 presents the summary of the results gotten.

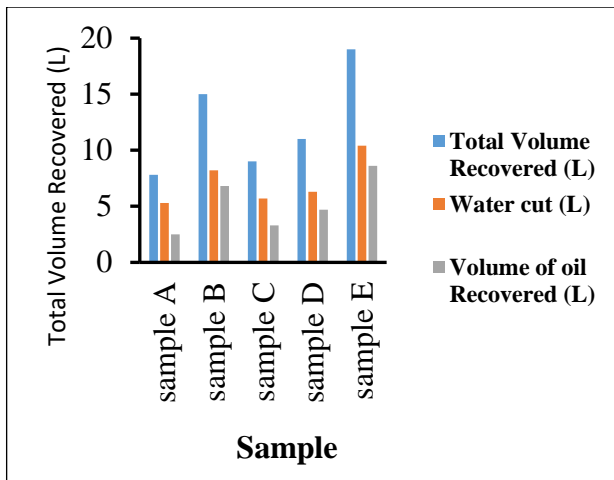


Fig 6: The Total Recovery, Oil Recovered and Water Cut

v. *Recovery between Nanoparticle and Tardigrade*

Sample B containing tardigrade recovered large volume of crude oil above the control sample A. sample C containing Aluminium Oxide nanoparticle recovered more crude oil than the control sample. Sample D is the sample containing iron oxide nano particle. It also had a good recovery above the control sample. Sample E, containing tardigrade, iron and aluminium oxide nano particles had a very good recovery of crude oil. Although, it was noticed that the nano particles when added to tardigrade caused the tardigrade to degrade. It was able to be recover more crude oil than sample B containing only tardigrade presented in Fig 7.

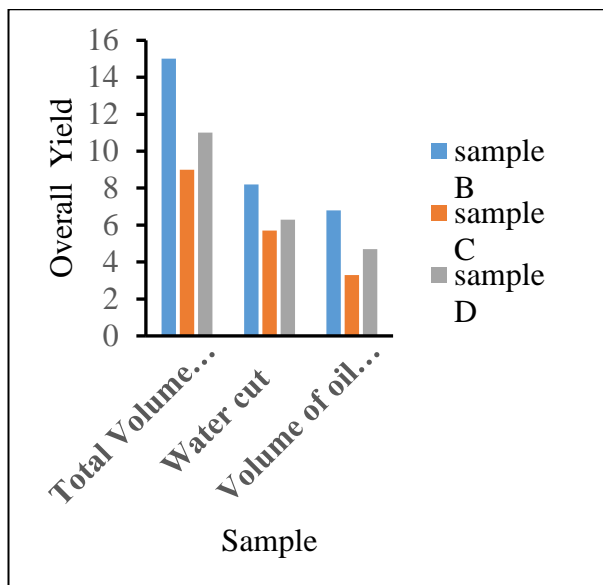


Fig 7: The Recovery between Tardigrade and Nano Particle Used

vi. *Physiochemical Properties of Crude before and after EOR*

After crude oil was recovered for the following samples, crude oil properties test was carried out for each of them to know which of the properties the Tardigrade and the nanoparticles changed that helped us to recover more crude. Fig 8 below, the samples with the lowest specific gravity are Sample C and Sample D, they have higher API gravity which makes them the

lighter crude samples. Samples with the lowest API gravities are Sample B, Sample E and Sample A which has the highest specific gravity which means they are the heaviest crude samples.

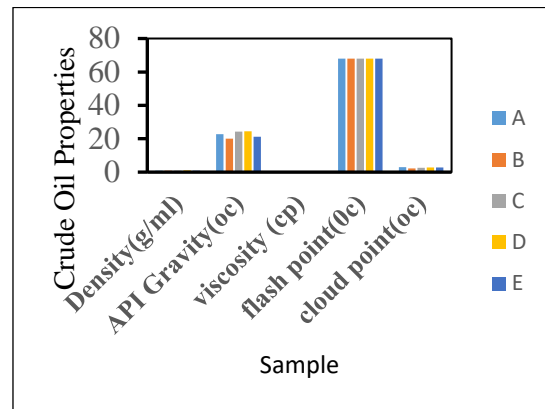


Fig 8: Physiochemical Parameter of Crude before and after EOR.

VI. CONCLUSION

The effect of nanoparticles on Tardigrade during EOR is a broad course and issue to look into in the petroleum industry, institution and laboratory. Tardigrade generally increases the recovery from a reservoir because of its tendency to change the crude oil properties like viscosity and API gravity, when nanoparticles were added to the Tardigrade the oil recovery also increased which means nanoparticles and Tardigrade when used together for enhanced oil recovery can help to recover more from an already depleted reservoir. From the Tardigrade enhanced oil recovery carried out with a laboratory set up, the result is concluded as follows;

- i. Sample A which was crude oil and water, had a high volume of recovery but a lower volume of crude oil recovered.
- ii. Sample B is sample containing crude oil and the Tardigrade, also had a high volume of total recovery and a high volume of crude oil recovery.
- iii. Sample C contains the crude oil and Aluminum oxide, has lower recovery compared to other samples. It recovered more crude oil than Sample A which was just Crude oil and water alone.
- iv. Sample D consist of Iron oxide (nanoparticles), recovered more crude, this implies that the addition of nanoparticles helped to reduce the viscosity of the oil.

Sample E had the highest total recovery and the highest oil recovered which means that the combination of Tardigrade, Aluminium and iron oxide Nano particles was very active in the recovery of crude oil.

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