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**Faculty of Science**

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**Experimental Investigation of the  
Impact of Green Zinc Nanorods on the  
Performance of the Biodegradable  
Drilling Fluid**

**Bachelor Thesis**

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## *Experimental investigation of the impact of green zinc nanorod on the performance of the biodegradable drilling fluid*

### **Anotace:**

Jedním z nevyhnutelných problémů, který vzniká při vrtání, je ztráta kapaliny, při které je část vrtné kapaliny filtrována do porézních a propustných útvarů. Ztrátu tekutin lze účinně léčit kombinací nanorůtek, které mají speciální vlastnosti, s cenově dostupnými biologicky odbouratelnými materiály. Tato studie si klade za cíl vyvinout nano-biologicky odbouratelné vrtné výplachy z dubové kůry, semen Gundelia a prášku ze slupky rambutanu. Za tímto účelem byly zinkové nanorody (NR) syntetizovány zeleně z extraktu rostliny Cydonia Oblonga a charakterizovány pomocí několika analytických technik, jako je skenovací elektronický mikroskop (SEM), rentgenová difrakce (XRD) a infračervená spektroskopie s Fourierovou transformací (FTIR). ) metody.

Biologicky odbouratelné vrtné výplachy byly připraveny přidáním prášku z použitých odpadních materiálů (dubová kůra, semena Gundelia a slupka rambutanu) v různých koncentracích a velikostech částic. Poté byl vyhodnocen dopad syntetizované nanotyče přípravou nano-biodegradabilních vrtných kapalin. Všechna požadovaná měření reologických a filtračních vlastností vyvinutého vrtného výplachu, biodegradabilních vrtných výplachů a nanobiodegradabilních vrtných výplachů byla provedena za normálních a HPHT podmínek. Získané výsledky ukázaly, že přidání 1 % hmotn. prášku z dubové kůry do referenční (základní) vrtné kapaliny snížilo objem ztráty filtru na 10,7 ml ve srovnání s referenční kapalinou (14,8 ml). Protože optimální koncentrace byla 0,5 % hmotn. prášku z kůry rambutanu poté přidaného k referenční tekutině, objem ztráty filtru se snížil na 8,6 ml.

**Klíčová slova:** Odpadní materiály; nanorod; vrtné kapaliny; reologické vlastnosti; filtrační vlastnosti.

**Annotation:**

One of the unavoidable issues that arises while drilling is the fluid loss, in which some of the drilling fluid is filtrated into the porous and permeable formations. Fluid loss can be effectively treated by combining nanorods, which have special properties, with affordable biodegradable materials. This study aims to develop nano-biodegradable drilling fluids from the oak peel, Gundelia seeds, and rambutan peel powders. For this purpose, zinc nanorods (NRs) was synthesized greenly from the extract of the *Cydonia Oblonga* plant and characterized using several analytical techniques, such as scanning electronic microscope (SEM), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) methods.

Biodegradable drilling fluids were prepared from adding the prepared powders of the used waste materials (oak peel, Gundelia seed peel, and rambutan peel) at different concentrations and particle sizes. Then, the impact of the synthesized nanorods was evaluated by preparing nano-biodegradable drilling fluids. All the required measurements for the rheological and filtration properties of the developed drilling fluid, biodegradable drilling fluids and nano-biodegradable drilling fluids were performed. The obtaining results have shown that adding 1 wt.% of the oak peel powder to the reference (base) drilling fluid reduced the filter loss volume to 10.7 mL compared with the reference fluid (14.8 mL). As the optimal concentration was 0.5 wt.% of rambutan peel powder then added to the reference fluid, the filter loss volume was reduced to 8.6 mL.

**Keywords:** Waste materials; nanorod; drilling fluids; rheological properties; filtration properties.

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### **Declaration Statement**

I declare that I have prepared the bachelor's thesis myself and that I have stated all the used information resources in the thesis.

In Olomouc, May 27, 2022

.....

Rayan Gailani Hasan



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## List of abbreviations

- (CMC) (Sodium Carboxyl-Methyl Cellulose)
- (API) American petroleum Institute
- (HTHP) high temperature high pressure
- Zn (CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O, Zinc acetate dehydrate
- (NaOH) Sodium hydroxide
- (RPP) Rambutan powder peel
- (CCPP & GSM) Gudelia seeds powder
- (OPP) Oak Powder Peel
- (FTIR) Fourier Transform Infrared Spectroscopy
- (DLS) Dynamic Light Scattering
- (OBFs) Oil-based fluids
- (SBFs) synthetic-based fluids
- (BHA) bottomhole assembly

# **Chapter 1: Introduction, Objectives and Outlines**

## **1.1 Introduction**

The demand for oil and gas has expanded dramatically, as has the necessity for cost-effective methods of obtaining them. Nonetheless, the drilling process should be safe, ecologically benign, and cost-effective Nmegbue,Bari-Agara (2014). A huge number of difficulties arise throughout the drilling and production procedures in the petroleum sector. For example, when the fluids in the borehole are flowing, certain fluids, such as drilling fluid or completion fluid, might be lost underground to the subsurface formation Wagle,Kalgaonkar (2018). As a result, fluid loss is regarded as a critical issue that occurs during the majority of drilling processes Nutskova,Rudyaeva (2018). As the differential pressure and circulation rate between the circulating fluid and the wellbore rise, fluid loss becomes more critical Iscan et al (2007,2018). Nanotechnology may be utilized in drilling technology in the oil and gas sector, and it has the potential to bring many significant breakthroughs, particularly in overcoming numerous challenges in the area. The use of nanomaterials, such as nanoparticles, enhances the rheological and filtration properties of drilling fluids Al-Yasiri,Al-Sallami (2015). As environmental awareness increased worldwide, particularly in the oil and gas industry, and environmental protection agencies established stringent and firm regulations, rules, guidelines, and standards pertaining to drilling waste management, obeying the rules and regulations, as well as using, developing, and practicing environmentally friendly drilling fluids to reduce environmental impact of the produced drilling waste, became necessary. As a result, various initiatives and experiments have been carried out in order to reduce the waste generated during the drilling and completion processes Al-Saba et al (2018). As a result, pro-environmental research has become an appealing issue for promoting the use of biodegradable and eco-friendly waste products as drilling fluid additives. One of the goals in this study was to search for an appropriate use of agro-waste in the formulation of drilling fluids because most of the waste elements produced in the agriculture business are harmless to humans and the environment Irawan et al (2009). Several past studies and explorations on the impact of nanoparticles and naturally biodegradable materials on the rheological and filtration characteristics of drilling fluids are examined. Ragab and Noah (2014) observed, for example, that utilizing micro silicane

concentrations of 20-30% (by weight) reduced fluid loss by 56% when compared to the reference drilling fluid. Similarly, Ismail et al (2014) discovered that adding 0.01 g of nano silica to drilling fluid at 200°F results in a fluid loss volume of just 6.5 mL during their experiment analyzing the impacts of nano silica. Furthermore, Ismail and his colleagues (2014) investigated and discovered that when TiO<sub>2</sub> is added to a water-based drilling fluid, it reduces fluid loss by 50% and filter cake thickness by 30%. Nmegbu and Bari-Agara (2014) also investigated the effects of corncob cellulose on water-based drilling fluids. Because the drilling fluid manufactured from corncobs cellulose reduced fluid loss to between 5.2 and 5.8 mL, the results revealed that corncobs cellulose is a promising choice for decreasing fluid loss. Okon and colleagues (2014) investigated the use of rice husk as a fluid loss control additive through a series of studies. The results showed that adding 20 g of rice husk to 350 mL of drilling fluid decreased fluid loss by 65%. Furthermore, Hossain and Wajheuddin (2016) investigated the effect of powdered grass addition with different particle size distributions to the water-based drilling fluid on the rheological and filtration properties. The results revealed that the optimal concentration of grass particles in the drilling fluid at 300 mL was 0.75 ppb, which decreased fluid loss by 25%. Al-Saba and colleagues (2018) recently investigated the impact of 20 biodegradable waste materials on rheological qualities and filtering features, including pomegranate peels, soybean peels, henna, and tamarind gum.

## **1.2 Thesis objectives**

The main goal of this report is to improve quality of the drilling fluid using the green and environmentally friendly materials by conducting the following objectives:

- Preparation of the waste materials from oak peel, Gundelia seeds and rambutan peel at different sizes.
- Synthesize and characterize the green zinc nanorods.
- Evaluate the role of the prepared waste materials on the rheological and filtration properties of the drilling fluid.
- Study the impact of the synthesized nanorod on the role of the biodegradable drilling fluid.

### **1.3 Thesis outline**

Chapter one covers the introduction to the project including research objectives and the outline. Chapter two describes the background and literature review regarding the drilling fluid and applications of nanotechnology and biodegradable materials in the drilling fluids. Chapter three covers the materials and detailed explanation of methods that are successfully done in lab experiments and the results. Chapter 4 covers the results and discussions that were made during all this time. Chapter 5 covers the conclusions and recommendations. Finally, references of cited books, journals and articles are presented.

## **Chapter 2: Background and Literature Review**

### **2.1 Oil and gas industry**

Oil and gas have long been considered to be crucial resources for the benefit of humanity, and they are now essential parts of the global energy system (Vezirolu, T.N., and Sahi, 2008). The oil and gas sector is regarded as one of the largest, most diverse, and important global industries (Inkpen, et al., 2011). According to the American Petroleum Institute, using fossil fuels helps civilization advance and secures sustainable development for the future. The production of oil and gas also has a significant impact on global demand; as the world's population rises, so does the need for these fuels (Holditch, S.A. and Chianelli, R.R., 2008). The prediction tends to raise the demand for energy globally by 40% by 2030, as (Inkpen, et al., 2011) noted. Additionally, the demand is rising by roughly 60% in 2030 compared to 2000. This suggests that the oil and gas sector play a significant role and has a significant impact on the global economy. One of the key areas of petroleum engineering is drilling, which is responsible for wells that generate hydrocarbons. Drilling is the process of penetrating a target reservoir that contains hydrocarbons while also involving good design, drilling regime, and completion (Jahn, et al., 2008). There are many different types of drilling wells, each used for a different purpose, such as exploration wells, appraisal wells, and development wells. Exploration wells are drilled to identify hydrocarbons in the subsurface. Since it has been established that a location is appropriate for producing hydrocarbons, production wells are dug for their extraction, and the wells are abandoned if no hydrocarbons are discovered underground. Casing activities are the next step after the initial well drilling, followed by completion.

### **2.2 Drilling Process**

Drilling engineering is one of the branches of petroleum engineering that is responsible behind the wells that produce hydrocarbons. It is the process of quarrying the soil and rocks onshore and offshore to reach the target reservoir that contains hydrocarbons and involves the well design, cost, and drilling regime, well testing and completion. (Hossain and Al-Majed, 2015).

There are various types of drilling wells for different purposes, including:



1. *Wildcat or exploratory wells* are drilled for the intention of finding hydrocarbons subsurface.
2. *Appraisal wells*, often used to gather information of the reservoir for future planning.
3. *Development or production wells* are drilled for the extraction of hydrocarbons.
4. In the case, hydrocarbons were not found underground or the well reached its peak stage of its life production. Then, the well is named an *abandonment well*.

After the well is drilled initially, the second step is the casing operations and then the well completion. Figure 1 shows the well casings and its steps.

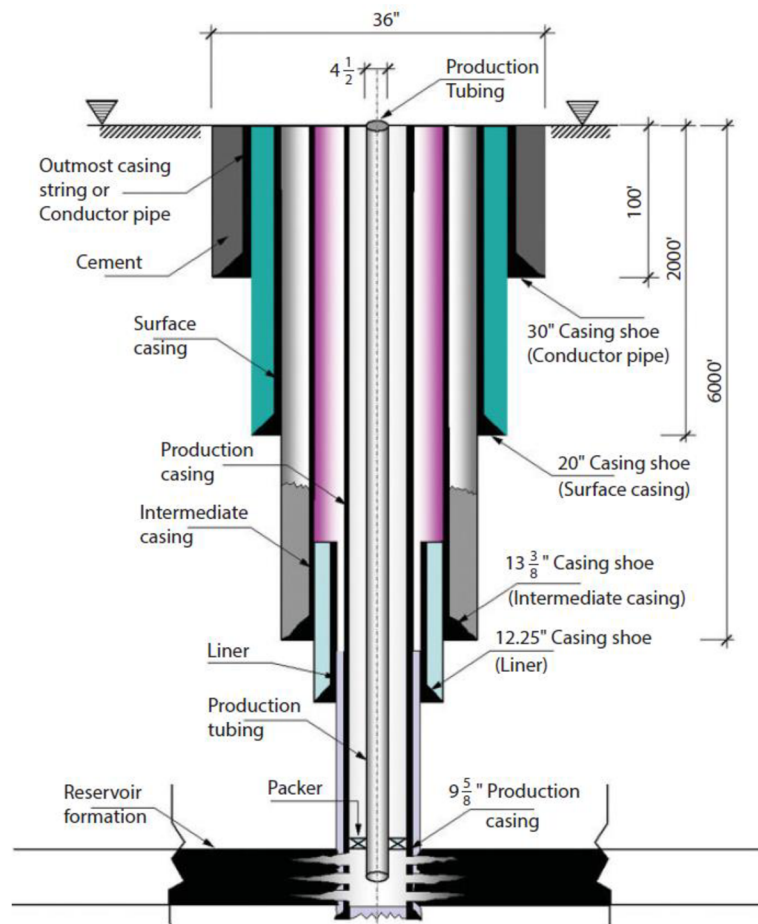


Figure 1 Typical casing program with different sizes and depths (Guilherme et al,2011)

### 2.3 Drilling fluid

Drilling fluid is an essential and crucial component of the drilling process. The term "mud" refers to a fluid that circulates continuously during the drilling operation in order to bring the cuttings back to the surface (Agung and Hamid, 2015). The simplest drilling fluid, according to Agung and Hamid (2015), is made up of a mixture of fluid (water) and clay. Drilling mud travels from steel tanks to mud pumps, then through the standpipe, rotary hose, Kelly, and drill string. The mud then moves to the bit at the bottom of the borehole, then moves up to the outside of the well through the annulus (Agung and Hamid, 2015).

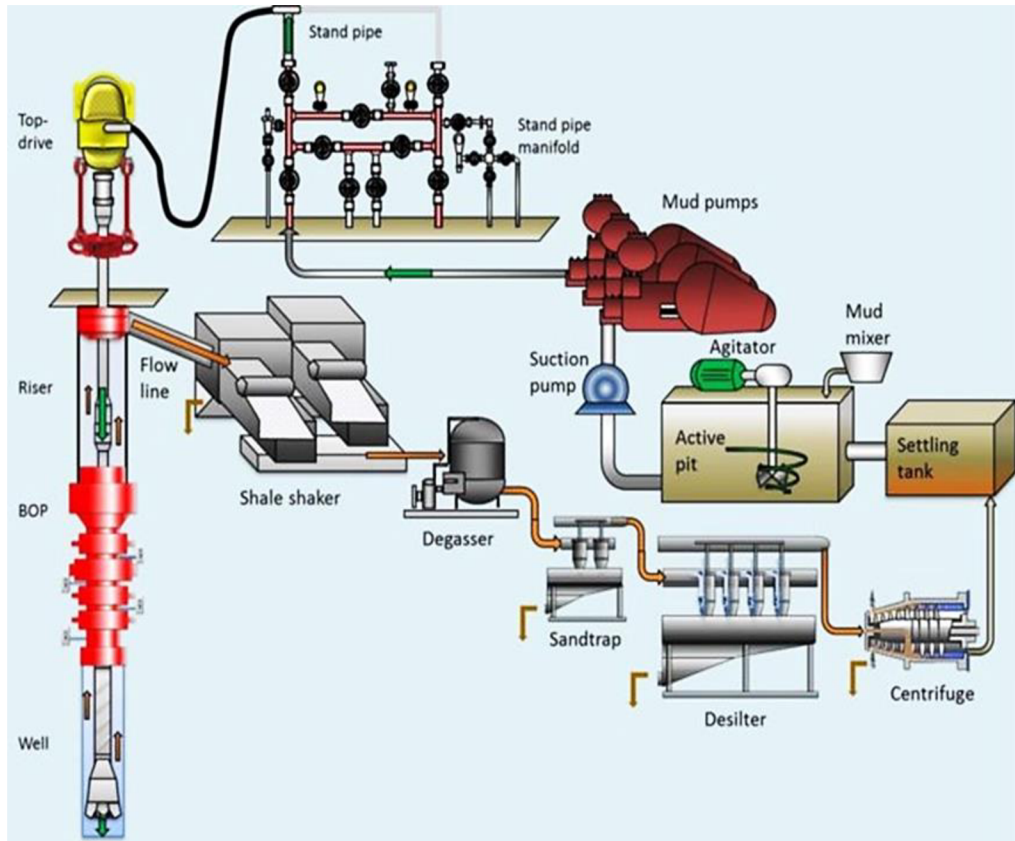


Figure 2 Drilling fluid system on a conventional drilling unit (Oyatomari et al,2002)

The returned mud and the transported cuttings are processed using degassers, desanders, shale shakers, desilters, and centrifuges near the surface to remove contaminants. The mud is then sent to the active pit once the cuttings have been removed (El Boubsi, 2017). Figure 2 shows the drilling mud circulation system. The major criteria

for choosing the drilling fluid types are the sorts of fluids that the formation exhibits, also the cost of the drilling fluid contributes significantly to the overall cost of drilling a well (El Boubsi, 2017). Therefore, Water-based drilling fluids are the most commonly utilized drilling fluid amongst the three major drilling fluid types in the drilling process. These types include water-based, oil-based, and air-based drilling fluids.

## **2.4 Classification of drilling fluids**

Drilling fluid is a fluid used in geotechnical engineering to drill boreholes into the earth. Drilling fluids aid in the exploration of oil and natural gas in drilling rigs. Drilling mud is another name for liquid drilling fluid (Mukherjee, 2013). Drilling fluids are broadly categorized into three types: water-based drilling fluids, oil-based drilling fluids, and synthetic-based drilling fluids. Figure 3 shows main three drilling fluid classifications.

Water-based drilling fluids account for 80 percent of all drilling operations since they are environmentally benign and cost-effective when compared to synthetic or oil-based drilling fluids. The elements influencing the drilling fluid selection are as follows: (Medhi et al., 2019). These parameters include (1) the location and kind of formation to be drilled, (2) the fluctuation in the pressure and temperature of the wellbore, (3) the nature of the formation fluids, i.e., strength, porosity, and permeability, and (4) other essential considerations considered while selecting drilling fluids. Water-based fluids are utilized in the drilling of nearly 80% of all wells. Fresh water, seawater, brine, saturated brine, or formate brine are all examples of fluids. The fluid type chosen is determined by predicted well conditions or the precise interval of the well being drilled. Commercial bentonite or attapulgite may also be applied to help in fluid loss management and hole cleaning efficacy. Water-based fluids are classified into two types: no dispersed and dispersed.

To assist with drilling challenges, oil-based drilling fluids were created and released in the 1960s. They're made from diesel, mineral oil, or low-toxicity linear olefins and paraffin's. Barite is utilized to improve system density, and the major viscosifier in most oil-based systems is carefully prepared organophilic bentonite. Fluid viscosity is also affected by the emulsified water phase. To assist regulate HP/HT (High pressure/High temperature), organophilic lignitic, asphaltic, and polymeric materials are applied.

Furthermore, surfactants used for oil-wetting can also be utilized as thinners. Lime is commonly used in oil-based systems to maintain a raised pH, resist the negative effects of hydrogen sulfide and carbon dioxide gases, and improve emulsion stability. One of the primary advantages of employing an oil-based technology is shale inhibition. The high-salinity water phase keeps shales from hydrating, expanding, or sloughing into the wellbore. Synthetic-based drilling fluids were developed in response to a growing need to lessen the environmental impact of offshore drilling operations while maintaining the cost effectiveness of oil-based systems. Field evidence collected since the early 1990s confirms that synthetic-based drilling fluids outperform diesel and mineral oil-based fluids in terms of drilling performance. Regulations prohibiting the disposal of cuttings drilled with oil-based drilling fluids in many offshore locations do not apply to some synthetic-based systems. The synthetic-based drilling fluids, which are created with linear alpha olefins and isomerized olefins, have lower kinematic viscosities, which are required in response to the growing relevance of viscosity difficulties as operators travel deeper into the sea.

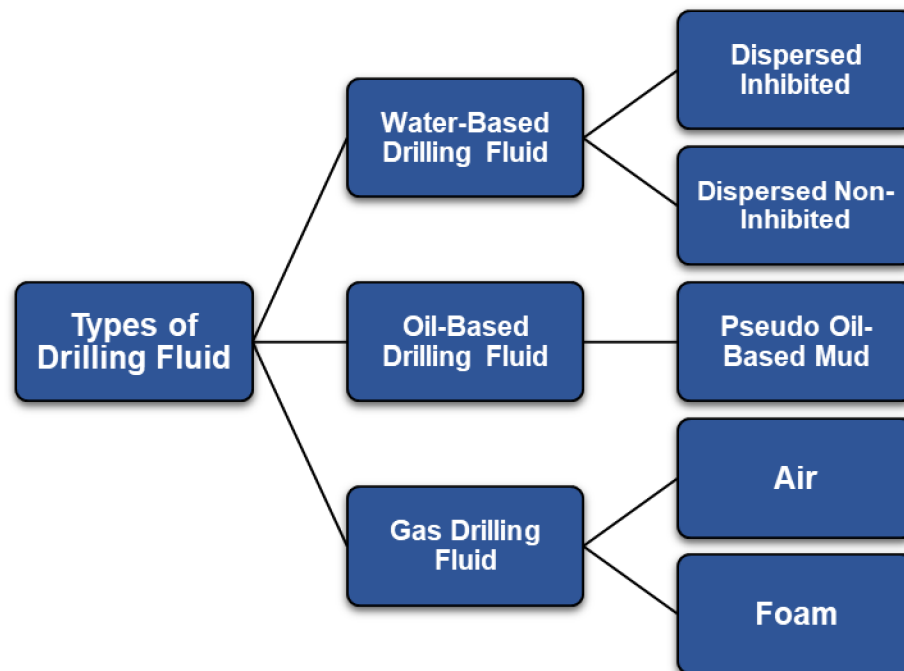


Figure 3 Drilling fluid classifications.

## **2.5 Functions of drilling fluid**

An effectively designed and maintained drilling fluid performs critical functions during well construction, such as transporting cuttings to the surface, preventing well-control issues and wellbore stability, minimizing formation damage, cooling, and lubricating the drill string, and providing wellbore information (Griffith, 1999).

- **Transport cuttings to surface**

The most fundamental function of drilling fluid is to transport drilled cuttings to the surface. To do this, the fluid must have enough suspension qualities to guarantee that cuttings and commercially added solids, such as barite weighing material, do not settle during static times. The fluid should have the necessary chemical qualities to assist avoid or reduce the dispersion of drilled materials, allowing them to be properly removed at the surface. Otherwise, these substances may break into ultrafine particles, causing harm to the producing zone and reducing drilling efficiency.

- **Prevent well-control issues**

The wellbore is subjected to hydrostatic pressure by the column of drilling fluid in the well. This pressure should balance or surpass the natural formation pressure under typical drilling circumstances to assist avoid an intrusion of gas or other formation fluids. As formation pressures rise, so does the density of the drilling fluid, which helps to maintain a safe margin and prevent "kicks" or "blowouts." However, if the fluid density grows too high, the formation may fail. If drilling fluid is lost in the resulting cracks, hydrostatic pressure falls. This pressure drop may also result in an inflow from a pressurized formation. As a result, maintaining the optimum fluid density for the wellbore pressure regime is crucial to wellbore safety and stability.

- **Preserve wellbore stability**

Maintaining proper drilling-fluid density not only helps keep formation pressures under check, but it also helps prevent hole collapse and shale instability. The wellbore should be clear of impediments and tight areas, allowing the drill string to flow smoothly in and out of the hole (tripping). After drilling a hole section to the desired depth, the wellbore should stay stable under static conditions while casing is run to the bottom and

cemented. The drilling-fluid program should specify the density and physicochemical qualities that are most likely to yield the best outcomes for a specific interval.

- **Minimize formation damage**

Drilling exposes the producing formation to the drilling fluid, as well as any particles and chemicals included in that fluid. It is unavoidable for fluid filtrate and/or fine particles to enter the formation. However, with proper fluid design based on testing with cored samples of the formation of interest, this invasion and the risk for formation damage may be reduced. Formation damage can also be reduced by skilled control of downhole hydraulics using precise modeling software, as well as the use of a specifically designed "drill-in" fluid, such as the systems used when drilling horizontal wells.

- **Cool and lubricate the drill string**

During typical drilling activities, the bit and drill string revolve at relatively high revolutions per minute (rev/min) all or portion of the time. Drilling fluid circulated through the drill string and up the wellbore annular space reduces friction and cools the drill string. The drilling fluid also offers lubricity to help the drill pipe and bottomhole assembly (BHA) go around angles made purposely by directional drilling and/or tight places caused by swelling shale. Oil-based fluids (OBFs) and synthetic-based fluids (SBFs) are the recommended fluid types for high-angle directional wells due to their high lubricity. Some water-based polymer systems produce lubricity comparable to oil- and synthetic-based systems.

- **Provide information about the wellbore**

Even though drilling fluid is constantly in contact with the wellbore, it reveals significant information about the formations being drilled and serves as a conduit for much data collected downhole by tools mounted on the drill string and wireline-logging operations performed when the drill string is removed from the hole. The capacity of the drilling fluid to maintain the cuttings as they go up the annulus has a direct impact on the quality of analysis that can be conducted on the cuttings. These cuttings are a major indication of the drilling fluid's physical and chemical state. An improved drilling-fluid

system that aids in the production of a stable, in-gauge wellbore can improve the quality of data supplied by downhole measurement and logging gear as well as wireline tools.

- **Minimize risk to personnel, the environment, and drilling equipment**

Drilling fluids need regular testing and monitoring by professionally qualified staff. The dangers of handling any sort of fluid are clearly noted in the fluid's paperwork. Drilling fluids are also rigorously inspected by international regulatory organizations to verify that the formulations in use conform with standards designed to preserve both natural and human populations where drilling occurs. The equipment used to pump or process fluid is continually monitored at the rig site for signs of wear from abrasion or chemical corrosion. To guarantee that safety is not jeopardized, elastomers used in blowout-prevention devices are evaluated for compatibility with the intended drilling-fluid system.

## **2.6 Biodegradable drilling fluid**

The data given in this paper were gathered during an experimental examination into the use of biodegradable, environmentally friendly drilling fluid additives derived from waste. Several dietary wastes were explored, and full-set measurements were taken when these wastes were added to the drilling fluid. Several researchers have investigated food wastes as drilling fluid additives, such as banana peels, potato peels, Arabic gum, olive pulp, corncob, corn starch, pomegranate powder, peach pulp, tamarind gum, soya bean, coconut coir, sugar cane, grass, henna powder, rice husk, cashew, and mango extracts (see Table 1). All of the researchers saw an improvement in the qualities of the drilling fluid. For more improvement in this project different and non-used materials is tested and added to the mud and the results are interesting. Okon et al. (2014) investigated the use of rice husk as a fluid loss control additive through a series of studies. The results showed that adding 20 g of rice husk to 350 mL of drilling fluid decreased fluid loss by 65 percent. Furthermore, Hossain and Wajheeuddin (2016) investigated the effect of powdered grass addition with different particle size distributions to the water-based drilling fluid on the rheological and filtration properties. The results revealed that the optimal concentration of grass particles in the drilling fluid at 300 mL was 0.75 ppb,

which decreased fluid loss by 25%. Al-Saba et al. (2018) recently investigated the impact of 20 biodegradable waste materials on rheological qualities and filtering features, including pomegranate peels, soy-bean peels, henna, and tamarind gum. The optimal concentration of soya bean peel powder was discovered to be 5 ppb for preparing a drilling fluid that may be utilized as a filter regulating agent and treat fluid loss of up to 60%.

Table 1 Summary table that shows the usages of the biodegradable materials in the drilling fluid.

Reference	Biodegradable materials and NPs	Optimum concentration	Optimum results
Okon et al. <sup>10</sup>	Rice husk	20 gm per 350 mL	Reduced the fluid loss 64.89% compared to PAC (59.57%) and CMC (62.77%), mud cake thickness of 3.2 mm
Hossain and Wajheuddin <sup>11</sup>	Grass	0.75 ppb in drilling fluid (300 µm)	25% filtration loss control.
Dagde and Nmegbu <sup>12</sup>	Cellulose processed from groundnut husk	4.0 gm	Fluid loss 6.5 m/s with maximum percentage deviation of -0.02% compared to PAC
Amanullah et al. <sup>13</sup>	Date seed powder (DSP)	6 gm	Spurt reduction by 40% compared to clay-free starch contain drilling mud and HPHT fluid loss reduction by 60%
Sharma and Vikas <sup>14</sup>	Tamarind gum and tragacanth	0.1% PAC and 0.2% Tamarind Gum	API filter press reduction of 12 mL and 21.40 mDarcy
Nmegbu et al. <sup>1</sup>	Corn cob	2 gm	5.2 m/s to 5.6 m/s lower fluid loss
Davoodi et al. <sup>15</sup>	Pistachio shell powder (PSP)	9 gm of PSP-1 in 350 mL	Efficiency with 44% fluid loss reduction and the highest value of plastic viscosity, yield point, and gel strength
Al-saba et al. <sup>6</sup>	Among 20 waste materials soya bean peel was most efficient	2 ppb	Reduced the fluid loss up to 60% and enhanced yield point 330% and gel strength 640%

## 2.7 Nano-drilling fluids

Drilling for unconventional reservoirs has increased dramatically in recent years, mostly for shales with Nano holes ranging in size from 10 to 30 nm and marked by exceptionally poor permeability (Sensoy et al., 2009). As a result, utilizing traditional mud additives to make a high-quality mud cake on such rocks is impossible. The particle sizes of conventional mud additions, such as bentonite and barite, range from 0.1 to 100



m. As a result, water enters the shale pores, where its ions react with the clay minerals, causing wellbore instability concerns (Egejuru et al, 2017). The petroleum sector utilizes oil-based muds that do not react with clay minerals in the shale to stabilize the borehole, wall during shale formation drilling, however because to their environmental effect, they are not always feasible to deploy (Sensoy et al., 2009). As a result, the use of nanoparticles is being studied, which, because to their small dimensions, may penetrate the nano pores of shale, fill this space, and reinforce the rock, resulting in higher fracture initiation pressure. Table 2 provides a summary of the types, sizes, and concentrations of nanoparticles utilized to minimize filtration and enhance wellbore strength. The majority of publications given demonstrate the possibility of nanoparticles plugging Nano holes, which decreases filtration and increases wellbore strength. Analyzing prior test results revealed that adding nanoparticles to some muds produced unanticipated outcomes. For example, Vryzas et al. (2015) discovered, that iron oxide nanoparticles dramatically decreased the filtration of bentonite suspensions. When iron nanoparticles at a concentration of 0.5 wt. percent were introduced to bentonite suspensions under high temperature high pressure (HTHP) conditions, the highest filtration decrease (reduction of 42.5 percent compared to bentonite solution without nanoparticles) was found. API filtration was also examined, and the findings revealed a reduction in filtration at higher concentrations (1.5 and 2.5 wt.%). Simultaneously, the inclusion of SiO<sub>2</sub> nanoparticles at all three concentrations greatly enhanced both API filtration and HTHP filtration (0.5, 1.5 and 2.5 wt.%). Taraghikhah et al. (2015) discovered that at concentrations of less than 1 wt.% SiO<sub>2</sub> nanoparticles, and filtration characteristics were unchanged when compared to mud without nanoparticles. Wahid et al. (2015) found that adding SiO<sub>2</sub> nanoparticles to a synthetic mud at a concentration of 0.32 to 0.71 wt. (%) reduced filtration and thickness while maintaining stable rheological parameters.

Table 2 Ttype, size and concentration of nanoparticles used in different laboratory research.

Reference	Type of nanoparticles	Size (nm)	NPs concentration (wt.%)	Mud type and formulation	Type of filtration test and conditions	Impact of adding nanoparticles on filtration	The highest measured reduction of filtration (%)
Contreras et al. (2014a)	Iron-based and calcium-based nanoparticles	N/A	<ul style="list-style-type: none"> <li>• 0.5</li> <li>• 1</li> <li>• 2.5</li> </ul>	Oil-based mud (oil/water ratio 90:10) emulsifier, CaCl <sub>2</sub> brine, hydrated lime, gilsonit, organophilic clay)	<ul style="list-style-type: none"> <li>• API filtration</li> <li>• PPT filtration (<math>\Delta p=34.5</math> bar, T=121 °C)</li> </ul>	Reduced filtration	90
Fakoya and Shah (2014)	SiO <sub>2</sub>	20	<ul style="list-style-type: none"> <li>• 0.058</li> <li>• 0.24</li> <li>• 0.4</li> </ul>	Surfactant-based, polymer-based and surfactant-polymer based mud (4% KCl solution, surfactant, guar, polyanionic cellulose (PAC))	API filtration	It reduced filtration highly	93.9
Vryzas et al. (2015)	Powder Fe <sub>2</sub> O <sub>3</sub> and powder SiO <sub>2</sub>	12 – 50	<ul style="list-style-type: none"> <li>• 0.5</li> <li>• 1.5</li> <li>• 2.5</li> </ul>	Water-based mud (7 % (w/w) bentonite suspension)	<ul style="list-style-type: none"> <li>• API filtration</li> <li>• HTHP filtration (<math>\Delta p=20.7</math> bar, T=121 °C)</li> </ul>	Fe <sub>2</sub> O <sub>3</sub> reduced filtration	42.5
Taragikhah et al. (2015)	SiO <sub>2</sub>	N/A	<ul style="list-style-type: none"> <li>• 0.5</li> <li>• 1</li> <li>• 2</li> </ul>	Water-based mud (salt, viscosifier, fluid loss controller)	• API filtration	Silica does not affect API filtration below 1 wt.%, at higher concentrations filtration reduced	115
Wahid et al. (2015)	SiO <sub>2</sub>	10-20	• 0.16-1.05	Synthetic-based mud (base oil, emulsifier, viscosifier, fluid loss control, lime, CaCl, barite, 2 drill solids)	• HTHP filtration (T=135 °C and 176.7 °C)	Reduced filtration	41.67
Vryzas et al., 2017	Fe <sub>3</sub> O <sub>4</sub>	N/A	• 0.5	Water-based mud (Na-bentonite 7 wt.%)	• HTHP filtration ( $\Delta p=34.5$ bar, T=121 °C)	Reduced filtration	47

## Chapter 3: Materials and methodology

### 3.1 Materials

For the synthesise of zinc nanorods and performing the experimental test, several chemical reagents were used, including zinc acetate dehydrate ( $Zn(CH_3COO)_2 \cdot 2H_2O$ ), sodium hydroxide (NaOH) and ethanol with the purity of 99% which were purchased from MERCK Germany. In addition, the soda ash and bentonite were ordered from Pulsar Petroleum Company. As local plants, the cydonia oblonga plant, gundelia seeds and oaks were collected from the Kurdistan Region and Rambutan was purchased from the international market.

Cydonia oblonga plant was used in the synthesis of zinc nanoroads, and it was collected from 20 kilometers outside of Soran city in the Iraqi Kurdistan Region's capital Erbil, in the autumn season, October 2021. The Cydonia oblonga is the common name for the plant that was used in this project, and the scientific name is Quince. Cydonia oblonga is a medicinal plant of family Rosaceae. The extract is aqueous extract of quince's leaf is rich in useful secondary metabolites such as phenolics, steroids, flavonoids, terpenoids, tannins, sugars, organic acids, and glycosides (Ashraf, 2016). Figure 4 shows the available phytochemicals of the leaf extract.

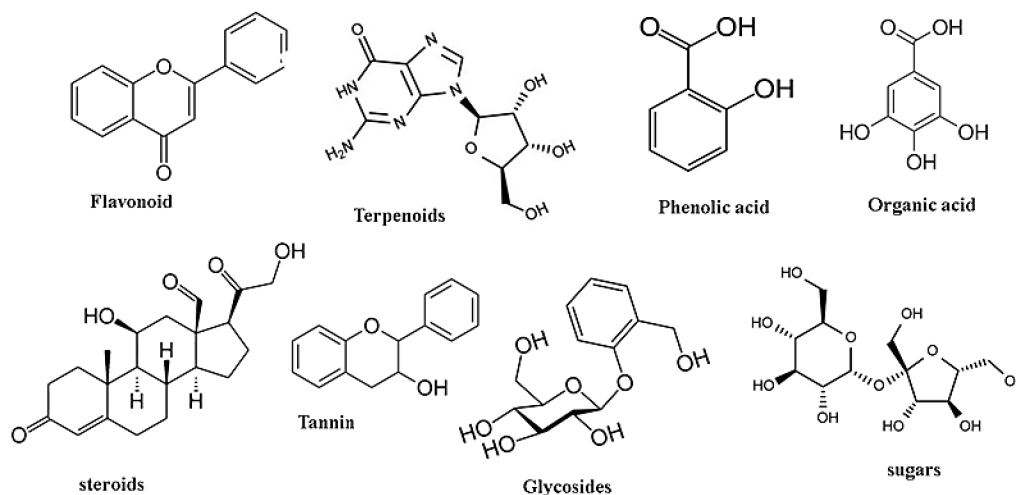


Figure 4 The available phytochemicals from Cydonia Oblonga plant extract.

### 3.2 Synthesise of zinc nanorods

The collected leaf part of the Cydonia oblonga plant was thoroughly rinsed with double distilled water to remove dust particles and were cut into small pieces and dried at room

temperature. In a flask containing 100 mL double distilled water, 10 gm of freshly cut quince's leaf were soaked for 40 minutes at 60 °C. The extract was allowed to cool down to room temperature and then filtered with a Whatman No. 1 filter paper to remove unwanted organic materials. Subsequently, the pure filtrate extract was stored in the refrigerator for further experimental work.

Zinc acetate, the molecular weight 219.51 gm/mol, was used in this study. The amount of 2 mg of zinc acetate was dissolved in 50 mL double distilled water and kept stirring for 20 min at 80 °C. Then, 50 mL of plant extract solution was drop-wise added to the dissolved zinc. The final mixture was put on the hotplate, heated, and stirred at 70 °C for 45 min till the color of the mixture reformed to a yellowish color. The new chromatic appearance of the mixture is considered a priority indicator for synthesizing ZnO nanorods after added 50 mL of NaOH. The obtained precipitates were separated from the mixture by centrifugation at 7000 rpm for 20 min and afterwards heated at 400 °C for 2 hrs using an oven to remove the impurities and organic materials around the ZnO nanorods (see Figure 5).

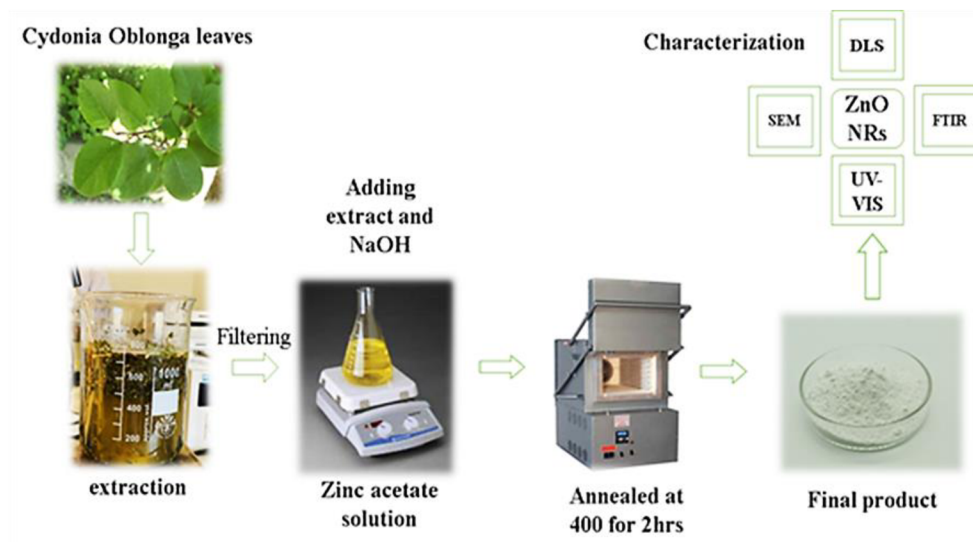


Figure 5 Schematic representation of ZnO NRs synthesis using the leaf extract of Cydonia Oblonga and zinc acetate dehydrate.

### 3.3 Preparation of base drilling fluid

The base drilling fluid sample was primarily made up of water (base fluid) and bentonite, with the addition of various additives such as caustic soda (NaOH) to manage alkalinity, soda ash ( $\text{Na}_2\text{CO}_3$ ) to address Ca ion pollution, and starch as a fluid loss agent. The drilling fluid base

samples are manufactured in line with the API-SPEC-13A-2010 standard, as described in a recent work by Ali et al. (2018).

The basic drilling fluid was mixed for 30 minutes with a Hamilton Beach 3-speed Type GM20 mixer. Table 2 shows the concentrations of the ingredients needed to make 400 mL of base drilling fluid. Drilling fluid samples were prepared using a 3-speed Type GM20 Hamilton Beach Commercial mixer, a Series 300 LPLT Filter Press was utilized to assess fluid loss, and the FANN 35 Viscometer was used to determine rheological parameters.

Table 3 The composition of the base drilling fluid sample.

Materials	Bentonite (gm)	Water (mL)	Soda ash (gm)	Polymer (gm)	NaOH (gm)
Concentration	20	350	0.50	0.50	0.10

### 3.4 Preparation of biodegradable and nano-biodegradable drilling fluids

With the exception of the base sample, twenty-eight additional drilling fluid samples were generated by varying the amounts of the synthesized zinc nanorods and biodegradable plant peel powders (rambutan, gundilia seed and oak) within the base drilling fluid as shown in Table 4. Biodegradable drilling fluids were prepared from mixing the powder of the selected waste materials within the base fluid at different concentrations of 0.5 to 1 wt.% and the particle sizes of 75, 150, 300 and 600  $\mu$ . In addition, the nano-biodegradable drilling fluids were formulated from mixing 1 wt.% zinc NRs within the base drilling fluid and the optimal biodegradable drilling fluids prepared from mixing the waste powders at 75  $\mu$  of size and 1 wt.% of concentration. All the drilling fluids were initially mixed using a 3-speed Type GM20 Hamilton Beach Commercial mixer and homogenized using UH-IID ultrasonic homogenizer for 2 hrs.

Table 4 The formulation of the nano-, biodegradable and nano-biodegradable drilling fluids used in this study for one size of waste powder.

Drilling fluid component	Drilling fluid	Biodegradable drilling fluid						Nano-drilling fluid			
		OPM		GSM		RPM		Zinc NRs			
		1	2	1	2	1	2	1	2	3	4
Bentonite, gm	20	20	20	20	20	20	20	20	20	20	20
Water, mL	350	350	350	350	350	350	350	350	350	350	350
Soda ash, gm	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
Polymer, gm	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
NaOH, gm	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
Oak peel, wt.%	-	0.5	1.0	-	-	-	-	-	1.0	-	-
Gunilia seed peel, wt.%	-	-	-	0.5	1.0	-	-	-	-	1.0	-
Rambutan peel, wt.%	-	-	-	-	-	0.5	1.0	-	-	-	1.0
Zinc NRs, wt.%	-	-	-	-	-	-	-	1.0	1.0	1.0	1.0

### 3.5 Rheological measurements of the developed drilling fluids

The FANN 35 viscometer was used to test the apparent viscosity, plastic viscosity, yield point, and gel strength of all the drilling fluid samples as shown in Figure 6. The FANN 35 viscometer's cup is filled with the reference drilling fluid, and the equipment is assembled. The gear switch is activated, and the rotor is rotated at various speeds. There are readings at 600 and 300 RPM. These measurements aid in the calculation of apparent viscosity, plastic viscosity, and yield point. The equipment is modified to measure gel strengths every 10 seconds and every 10 minutes by reading the highest, or maximum, deflection of the dial before the gel splits. Shear stresses and shear rates were also calculated using the rotating viscometer's recorded deflections at various speeds. The gel strength of drilling fluids was tested after 10 seconds and after 10 minutes.

For measuring the apparent viscosity, plastic viscosity and yield point the below equations were used:

$$\text{Plastic Viscosity } (\mu_p) \text{ (cP)} = 600 \text{ rpm reading} - 300 \text{ rpm reading} \quad (1)$$

$$\text{Apparent Viscosity } (\mu_a) \text{ (cP)} = 600 \text{ rpm reading}/2 \quad (2)$$

$$\text{Yield Point } (\tau_y) \text{ (lb/100 ft}^2\text{)} = 300 \text{ rpm reading} - \mu_p \quad (3)$$



Figure 6 FANN 35 Viscometer

### **3.5 Filtration measurements of the developed drilling fluids**

Using a Series 300 LPLT Filter Press at 100 pressure and room temperature shown in Figure 7. the filtering properties, including fluid loss and filter cake, were investigated for various concentrations of (RPP & CCPP & OPP) and combinations of biodegradable materials and Nano rod. The thickness of the filter cake after 30 minutes and the fluid loss at various time intervals from 0 to 30 minutes were determined for all samples. The filter paper is appropriately positioned in the cell of the Series 300 LPLT Filter Press, and the reference drilling fluid is pumped into the filter cell. The filter press is set up for the tests, and a graduated cylinder is positioned beneath the filtrate tube. When all of the equipment is in place, the timing is started, and the test begins. The test is permitted to run for another 30 minutes. Throughout the 30 minutes, the amount of filtrate in the graduated cylinder is read and recorded. The thickness of the filter cake is next measured by using a special roller from Fann instrument company as shown in Figure 7.

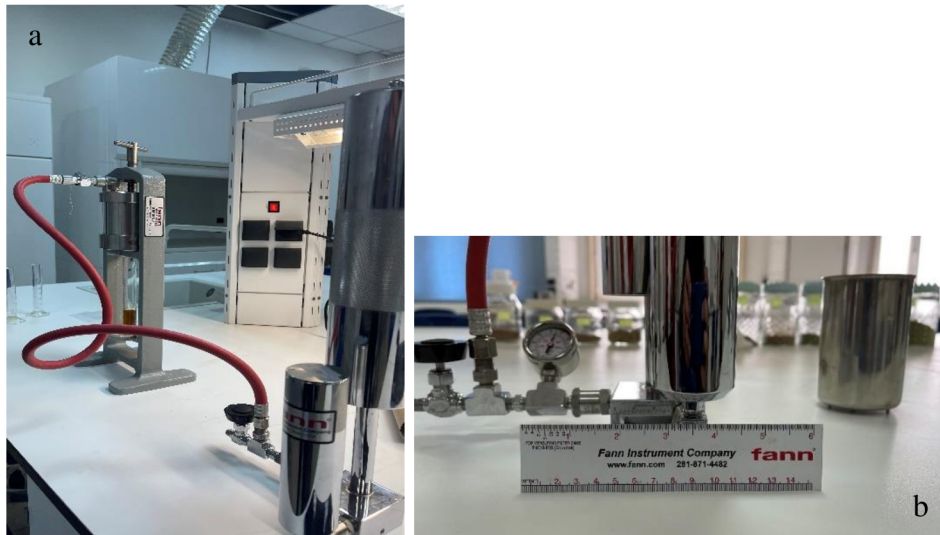


Figure 7 Filtration measurement apparatus; a) series 300 LPLT filter press, and b) roller for measuring filter cake thickness.



## Chapter 4: Results and discussions

### 4.1 Characterization of the synthesized NRs

Greenly synthesized zinc nanorods were characterized using UV-Visible spectroscopy, dynamic light scattering and transmission electron microscopy to ascertain their shape and size. Washed and dried zinc nano-powders was re-suspended in sterile deionized water to yield a dilute suspension and UV-Visible spectrum was generated using UV-Vis double-beam spectrophotometer (Super Aquarius spectrophotometer-1000) in the wavelength range of 200-700 nm with 1 nm resolution, was used to confirm the formation of zinc nanorods. Distilled water was used to set the reference baseline before performing spectral measurement studies and spectra for zinc acetate dihydrate, sodium hydroxide and plant extract were noted to serve application.

#### 4.1.1 UV-vis spectrum

The *Cydonia Oblonga* plant extract is mostly composed of flavonoids and phenolic chemicals. These phytochemicals enable to decrease zinc ions and stabilize them in nanoscale range. These phytochemicals are also having a direct impact on the shape and size of the nanorods that are being produced. Figure 8a displays the UV-Vis spectrum of *Cydonia oblonga* leaf extract. We believed that the dominant peaks, at 269.1 nm and 320.6 nm, are more likely related to the phenolic components, i.e., polyphenols and flavonoid available in *Cydonia oblonga* leaf extract. Further, the UV-Vis absorption spectrum (Figure 8b) indicator of the creation of zinc oxide nanorods is at the ultraviolet range of 341.79 nm.

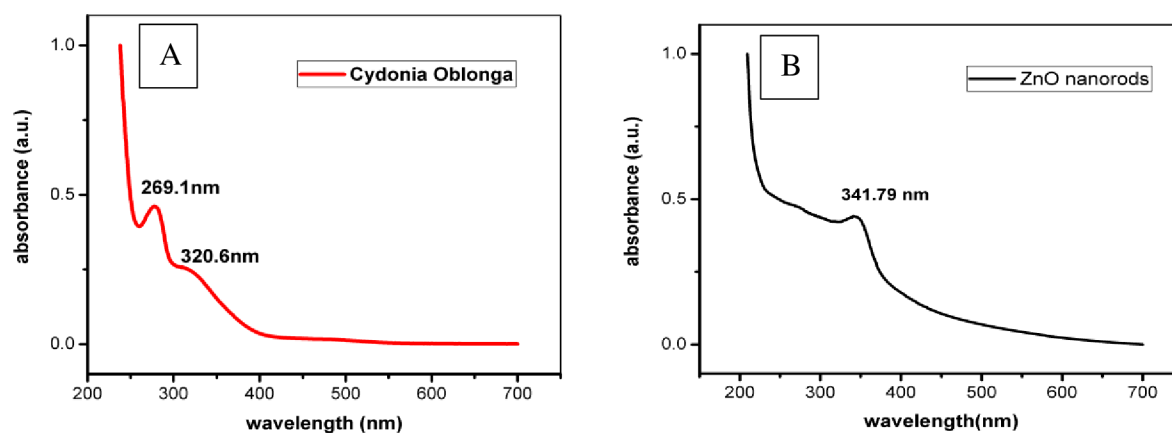


Figure 8 (a) UV-vis spectra of plant extract and (b) green nanorods

### 4.1.2 Field Emission Scanning Electron Microscope

The morphologies of the yellow layer were observed by the field emission scanning electron microscope (FE-SEM). FE-SEM image (see Figure 9) shows that the product consists of nanorods with diameters of about 47-50 nm and some of the nanorods are straight and hexagonal that SEM possess a uniform hexagonal shape as shown in fig 9 (a) and (b).

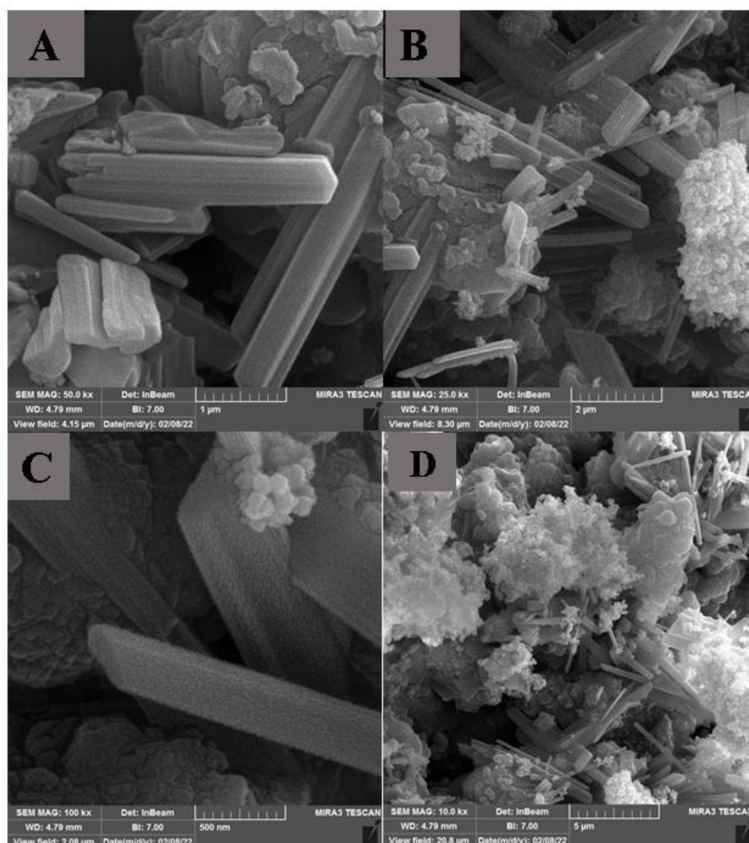


Figure 9 FE-SEM of ZnO nanorods at different scale (a). 1µm (b). 2µm (c). 500nm (d). 5µm.

### 4.1.3 FTIR Spectrum

The FTIR spectrum of the biosynthesized zinc nanorods is displayed in Figure 10, which demonstrates absorption peaks positioned between the region around  $4000\text{ cm}^{-1}$  and  $500\text{ cm}^{-1}$ . The FTIR spectra displayed absorption bands at  $3464.75\text{ cm}^{-1}$ ,  $2923\text{ cm}^{-1}$ ,  $1596\text{ cm}^{-1}$ ,  $1383\text{ cm}^{-1}$ ,  $1251\text{ cm}^{-1}$ , tention of studying the oxidation state of ZnO nanorods. The FTIR spectra displayed absorption bands at  $3441\text{ cm}^{-1}$ ,  $2923\text{ cm}^{-1}$ ,  $1596\text{ cm}^{-1}$ ,  $1383\text{ cm}^{-1}$ ,  $1251\text{ cm}^{-1}$ , and  $1076\text{ cm}^{-1}$ , representing the existence of reducing, capping, and stabilizing biomolecules with the ZnO nanorods. The band at  $3441\text{ cm}^{-1}$  in the spectra corresponds to the O-H

stretching vibration, indicating the presence of alcohol and phenol. The band at  $2923\text{ cm}^{-1}$  was related to the C-H stretching vibrations of the primary and secondary amines. The band at  $1596\text{ cm}^{-1}$  is more likely related to C-C/C-N stretching vibrations of Alkene or amines, while the band at  $1383\text{ cm}^{-1}$  is related to the N=O symmetry stretching typical of the nitro compound. Additionally, the bands at  $1251\text{ cm}^{-1}$  and  $1076\text{ cm}^{-1}$  correspond to C-N and C-C stretching, indicating the presence of proteins amines.

As stated before, these functional groups, in general, have a role in the stability/capping of ZnO nanorods as testified in various studies.

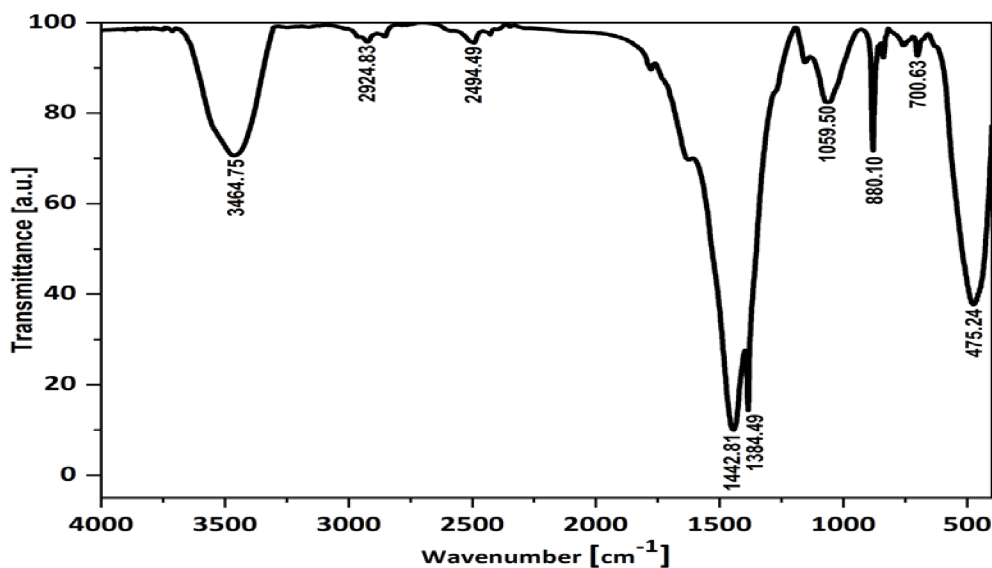


Figure 10 FTIR image of ZnO nanorods.

#### 4.1.4 DLS

DLS indicated that DLS is an extensively used technique for determining the hydrodynamic diameter of nanoparticles established on the brownian movement of particles in the suspension. The variation in the nanoparticle size could be associated with the polydispersity Index (PDI) values in turn related to the existence of nanoparticles as aggregates or agglomerates. Particle size was determined by DLS measurement. The average size of ZnO nano was determined 31nm (see Figure 11). The difference between the diameter values obtained from DLS and SEM measurement was due to the process involved in the sample preparation. By the laser light scattering (DLS) method the hydrodynamic diameter (dehydrate state) of nanorods was obtained. In summary, ZnO nanorods were synthesized UV-VIS, FE-SEM, DLS and FTIR images showed that the product was composed of nanorods and the nanorods were hexagonal.

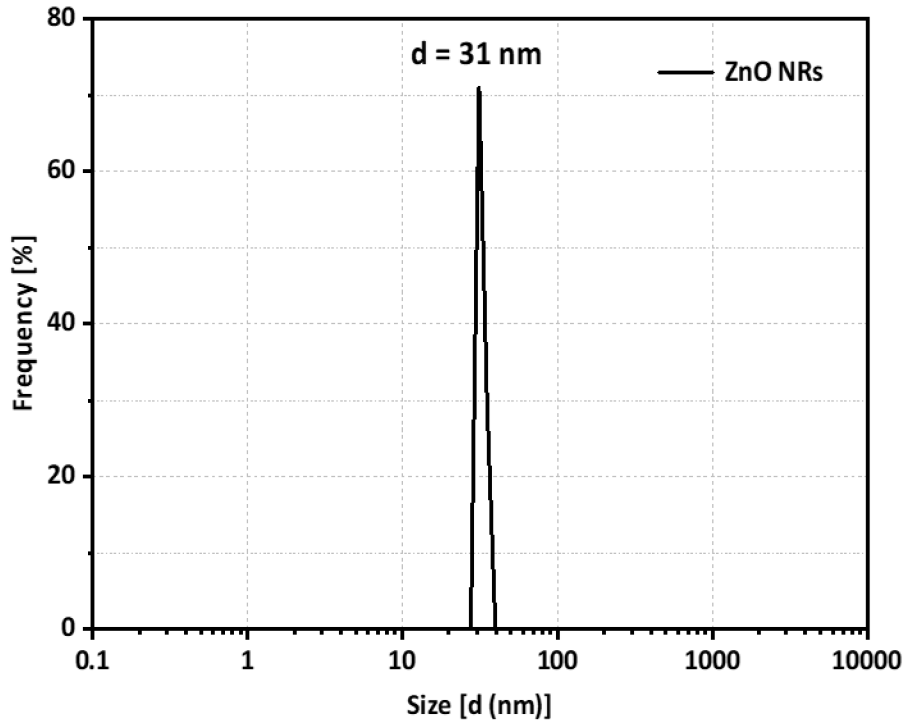


Figure 11 DLS analysis of ZnO nanorods from Cydonia Oblonga.

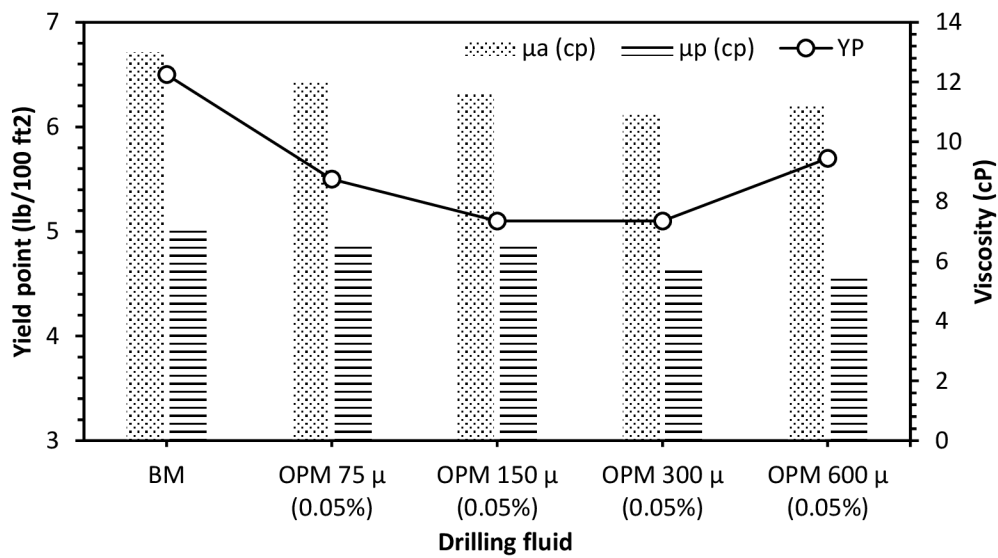
#### 4.2 Rheological Properties

This section looks into how the rheological properties of the produced drilling fluids are affected by zinc nanorods and biodegradable components like powdered oak peels, Gundelia seeds, and rambutan peels. Plastic viscosity, apparent viscosity, yield point, and gel strengths at 10 sec. and 10 min. were among the rheological characteristics of the base mud that were measured, and the plastic and apparent viscosities and yield point were 7 and 13 cP and 6.5 lb/100ft<sup>2</sup>, respectively. The 10 sec and 10 min gel strengths were 3 and 6 lb/100ft<sup>2</sup>, respectively.

The rheological properties of the drilling mud with 0.05 wt.% of oak peels powder at four different particle size as the additive were measured as shown in Table 5 and Figure 12. As can be see, it was found that the OPM at concentration 75  $\mu$  plastic viscosity, apparent viscosity, and yield point are 6.5 cP, 12 cP, and 5.5 lb/100 ft<sup>2</sup>, respectively, and the gel strengths at 10 sec and 10 min were also found to be 3.1 and 5.8 lb/100 ft<sup>2</sup>, respectively. As the Particle size of the OPM was increased, the plastic and apparent viscosities and gel strengths at 10 sec and 10 min were increased. However, the yield point slightly decreased in comparison to the mud with 0.05 wt.% of OPM at 75  $\mu$ .

Table 5 The rheological results of oak peels powder based drilling fluid.

Drilling fluid	Sample	Bio-size	Concentration (wt. %)	YP (lb/100ft <sup>2</sup> )	$\mu_p$ (cP)	$\mu_a$ (cP)	Gel strength, lb/100 ft <sup>2</sup>	
							Gel <sub>initial</sub>	Gel <sub>final</sub>
Base sample	BM	-	-	6.5	7	13	3	6
Biodegradable drilling fluids	OPM	75	0.5	5.5	6.5	12	3.1	5.8
	OPM		1	5	6	11	2.8	5.7
	OPM	150	0.5	5.1	6.5	11.6	3	5.8
	OPM		1	5.05	6.1	11.1	3.5	7
	OPM	300	0.5	5.1	5.8	10.9	4	7.4
	OPM		1	6	6.5	12.5	3.5	8.2
	OPM	600	0.5	5.7	5.5	11.2	5	9
	OPM		1	4	11	15	9	19



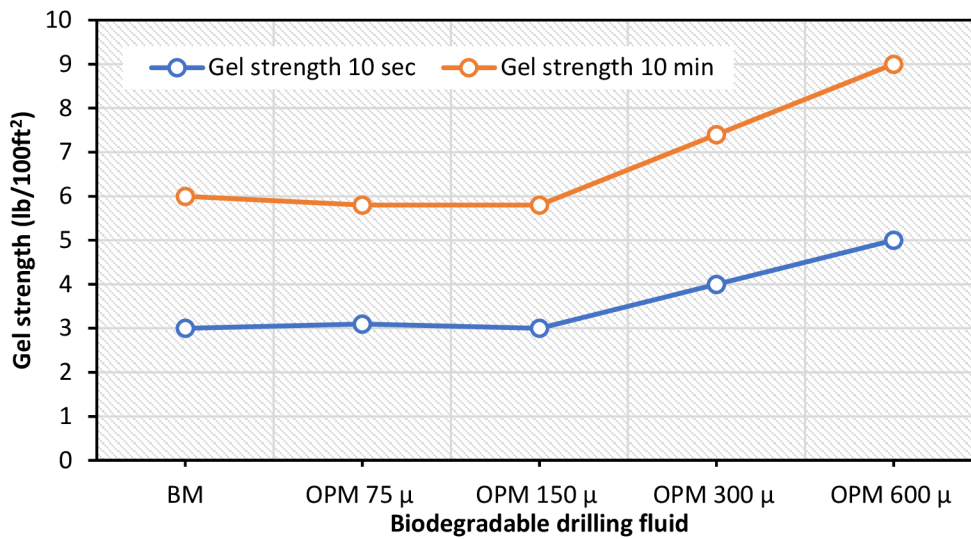


Figure 12 The rheological results of the biodegradable drilling fluids developed from mixing oak peels powder at different particle sizes and concentrations.

The results showed that using Gundelia seeds powder (GSM) in the drilling mud can considerably change the rheological properties of the drilling fluid. With the addition of 0.05 and 0.1 wt.% of GSM a particle sizes of 75 μ, 150 μ, 300 μ, and 600 μ as shown in Table 6 and Figure 13. With 75 μ particle sizes the rheological properties are reduced significantly. The plastic viscosity, apparent viscosity and yield point, were reduced to be 4 cP, 7.5 cP, and 3.5 lb/100 ft<sup>2</sup>, respectively, 10 sec and 10 min gel strength were decreased to 2 and 5 lb/100 ft<sup>2</sup>, respectively, as 0.05 wt.% GSM were added to the drilling mud.

Table 6 The rheological result of Gundelia seeds powder (GSM) based drilling fluid.

Drilling fluid	Sample	Bio-size	Concentration (wt.%)	YP (lb/100ft <sup>2</sup> )	μ <sub>p</sub> (cP)	μ <sub>a</sub> (cP)	Gel strength, lb/100 ft <sup>2</sup>	
							Gel <sub>initial</sub>	Gel <sub>final</sub>
Base sample Biodegradable drilling fluids	BM	-	-	6.5	7	13	3	6
	GSM	75	0.5	3.5	4	7.5	2	5
	GSM		1	4	4	8	3	6
	GSM	150	0.5	4.25	4.5	8.75	2	6
	GSM		1	5.75	6	11.7	4	9.5
	GSM	300	0.5	9.5	7	16.5	8	16
	GSM		1	6.5	5.5	12	6	11
	GSM	600	0.5	14.5	12	26.5	30	46
	GSM		1	16.5	12	28.5	32	48

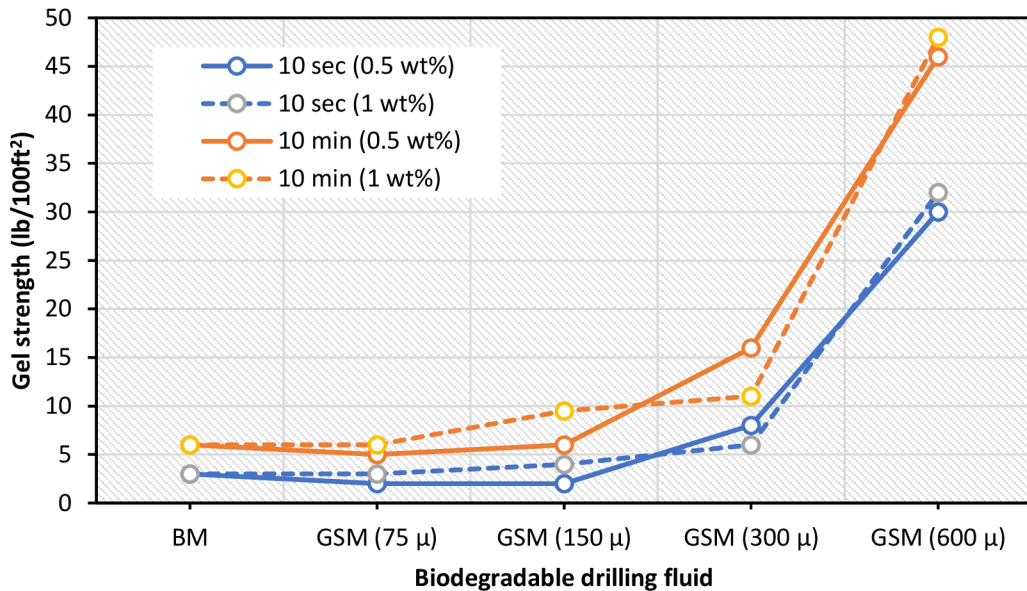
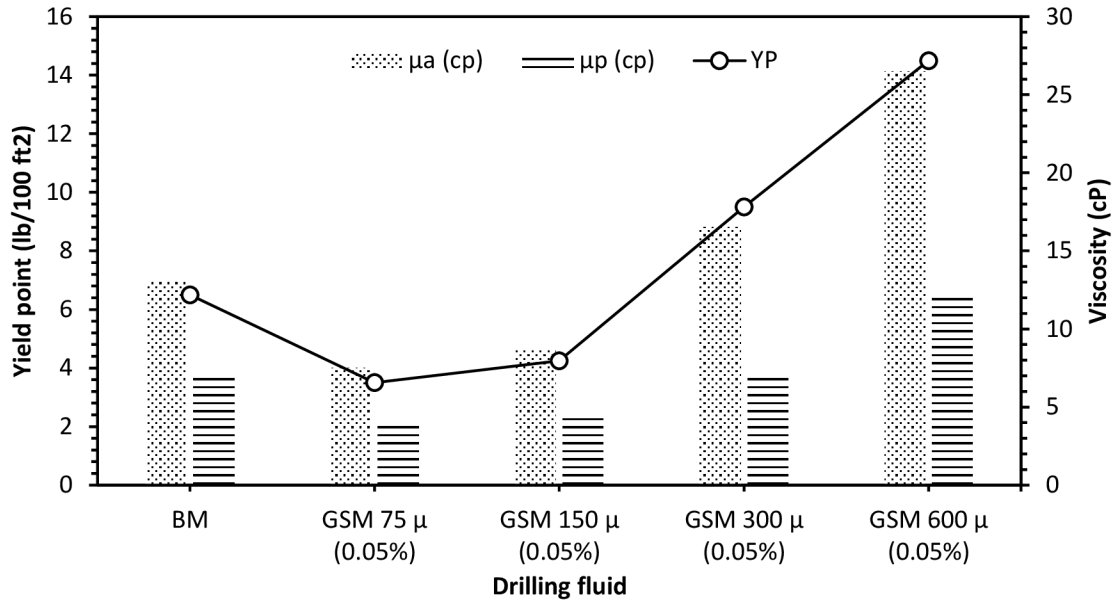


Figure 13 The rheological results of the biodegradable drilling fluids developed from mixing Gundelia seed peel powder at different particle sizes and concentrations.

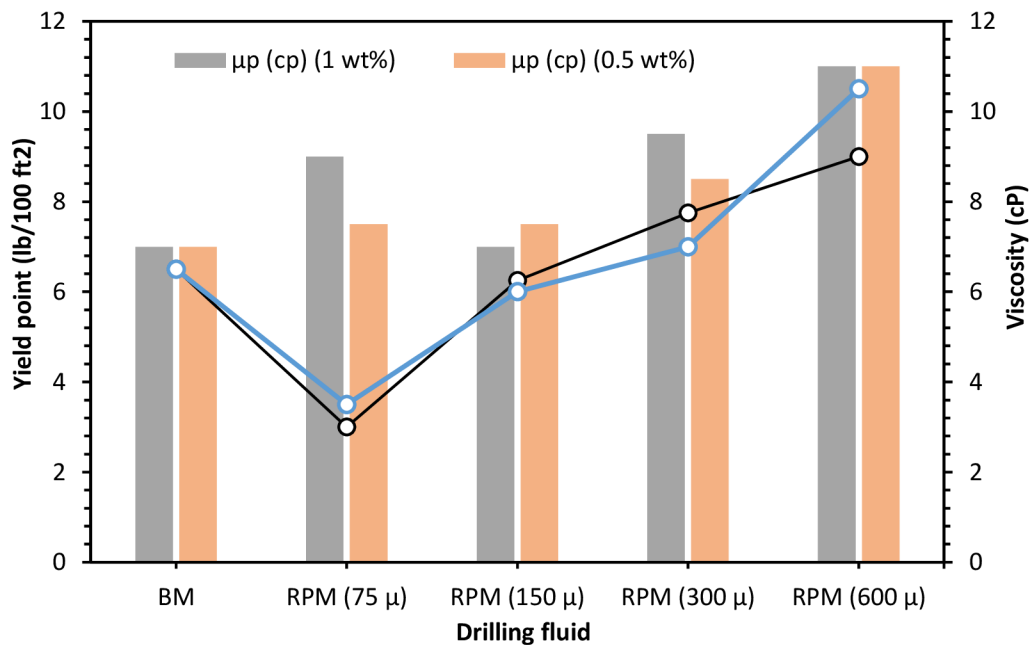
As the particle sizes increased, the rheological properties were increased significantly. It was noticed that at biggest particle size reached highest value of the plastic viscosity, apparent viscosity, and yield point, of 28.5 and 32 cP, 4 16.5 lb/100 ft<sup>2</sup>, respectively, and the gel strength at 10 sec and 10 min were increased to and 19 lb/100 ft<sup>2</sup>. The test results proved that at small particles sizes will not affect the rheological properties compared to the base sample. However, at big particle sizes all the rheological properties significantly changed. On

the other hand, as the two different concentrations of the rambutan peels powder were added to the mud, the yield point decreased at small particle sizes 3 lb/100 ft<sup>2</sup> and with bigger particle sizes the yield point started to increase reached highest value at biggest particle size 600  $\mu$  and increased to 10.5 lb/100 ft<sup>2</sup> (see Table 7 and Figure 14).

Table 7 Rheological results of rambutan peels powder-based drilling fluid.

Drilling fluid	Sample	Bio-size	Concentration (wt. %)	YP (lb/100ft <sup>2</sup> )	$\mu_p$ (cP)	$\mu_a$ (cP)	Gel strength, lb/100 ft <sup>2</sup>	
							Gel <sub>initial</sub>	Gel <sub>final</sub>
Base sample	BM	-	-	6.5	7	13	3	6
Biodegradable drilling fluids	RPM	75	0.5	3	7.5	10.5	4	5.5
	RPM		1	3.5	9	12.5	4	6
	RPM	150	0.5	6.25	7.5	13.7	10	10
	RPM		1	6	7	13	9.5	9.5
	RPM	300	0.5	7.75	8.5	16.2	11.5	13
	RPM		1	7	9.5	16.5	13	13
	RPM	600	0.5	9	11	20	19	29
	RPM		1	10.5	11	21.5	20	30

As RPM was used, the plastic viscosity, apparent viscosity, and gel strength at 10 sec and 10 min are increased with increasing particle sizes and at 600  $\mu$  was 11 and 21.5 cP, 20 and 30 lb/100 ft<sup>2</sup>.





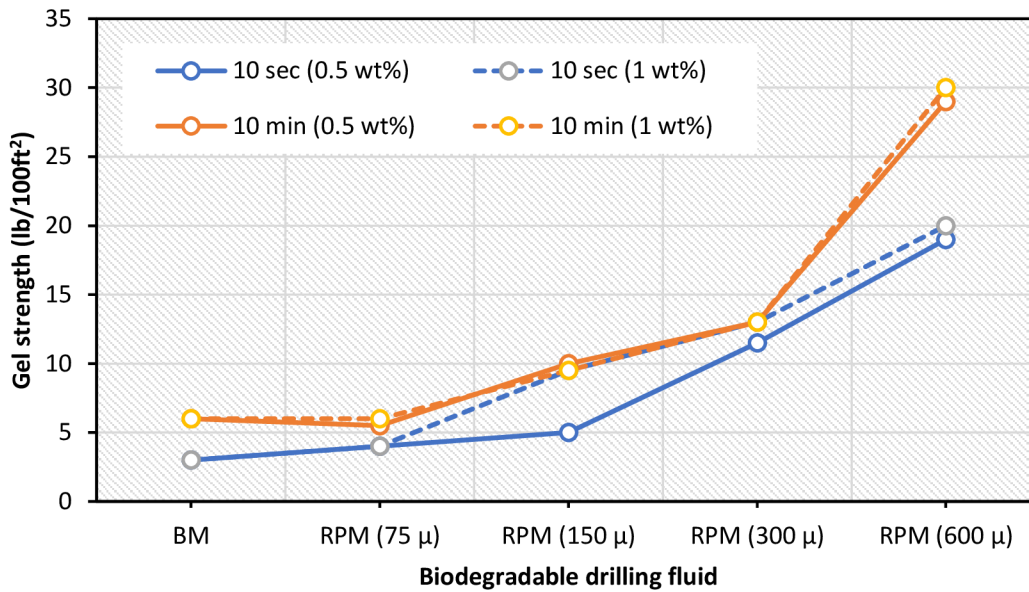


Figure 14 The rheological results of the biodegradable drilling fluids developed from mixing rambutan peels powder at different particle sizes and concentrations.

After that, the base sample was tested under the influence of zinc NRs with the concentration of 0.1 wt.% and all the rheological properties were enhanced and plastic viscosity, apparent viscosity, and yield point increased to 13 and 43 cP, 60 lb/100 ft<sup>2</sup> as shown in Table 8 and Figure 15. Then three more samples were formulated using the waste materials oak peels, Gundelia seeds, and rambutan peels with optimum concentration of 0.1 wt.% and smallest particle size of 75 μ to be added with zinc NRs. The plastic viscosity, apparent viscosity, and yield point values were discovered to decrease with addition of the oak peels powder and optimum concentration of zinc NRs. On the contrary, the test results showed that gel strength at 10 sec and 10 min possessed an increasing trend. This formulation had a different behavior when applied to Gundelia seeds. To be clearer, as the optimum nanorod concentration and 0.1% GSM (by weight) were used to formulate a drilling fluid sample, the plastic viscosity kept its value. However, the values of all measured rheological properties of the prepared samples of mud using optimum concentration of nanorod and GSM with concentrations of 0.1, wt.%, increased in comparison to the reference fluid except the apparent viscosity. During investigating the effects of addition of a combination of nanorod and rambutan peels powder to the reference mud, it was found that as the optimum concentration of nanorod is mixed with RPM and added to the base mud, plastic viscosity and apparent viscosity are reduced, whereas yield point gel strengths at 10 sec and 10 min were increased.

Table 8 The results of ZNO-nanorod and optimum of oak peels, gundelia seeds, and rambutan peels with zinc NRs

Drilling fluid	Sample	Bio-size	Concentration (wt. %)	YP (lb/100ft <sup>2</sup> )	$\mu_p$ (cP)	$\mu_a$ (cP)	Gel strength, lb/100 ft <sup>2</sup>	
							Gel <sub>initial</sub>	Gel <sub>final</sub>
Base sample	BM	-	-	6.5	7	13	3	6
Nano-drilling fluid	ZNO		1	60	13	43	63	70
ZnO-biodegradable drilling fluids	ZNO+OPM	75	1	4.5	6.5	8.75	5	7.5
	ZNO+GSM	75	1	9	7	11.5	10	10
	ZNO+RPM	75	1	10	6	11	5	7

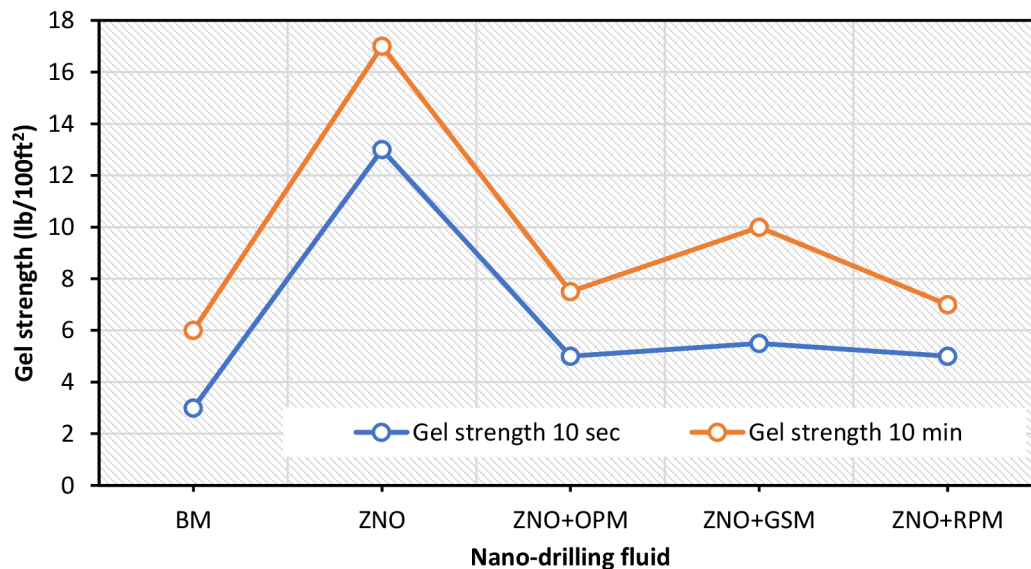
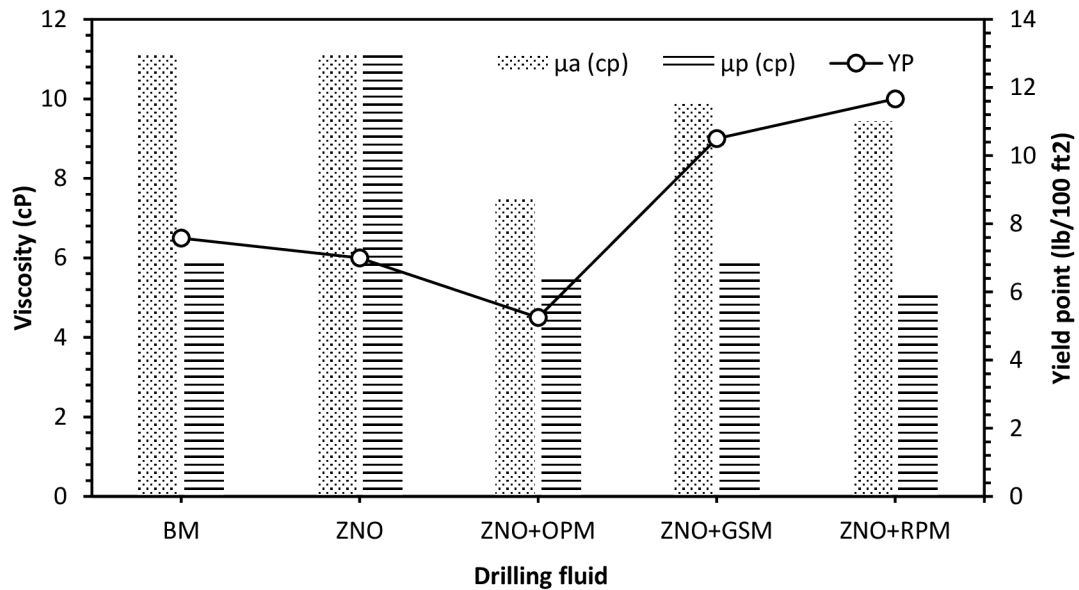


Figure 15 The rheological results of the nano-drilling fluids formulated from mixing zinc NRs within the base and biodegradable drilling fluids at 0.1 wt.%.

### 4.3 Filtration properties

The filtration characteristics shown, both fluid loss volume and filter cake thickness, of the water-based drilling fluids modified with nanorod and biodegradable materials were studied and measured in the laboratory. The base mud that was prepared first had a filtration rate of 14.8 cc and mud cake thickness of 3.7 mm.

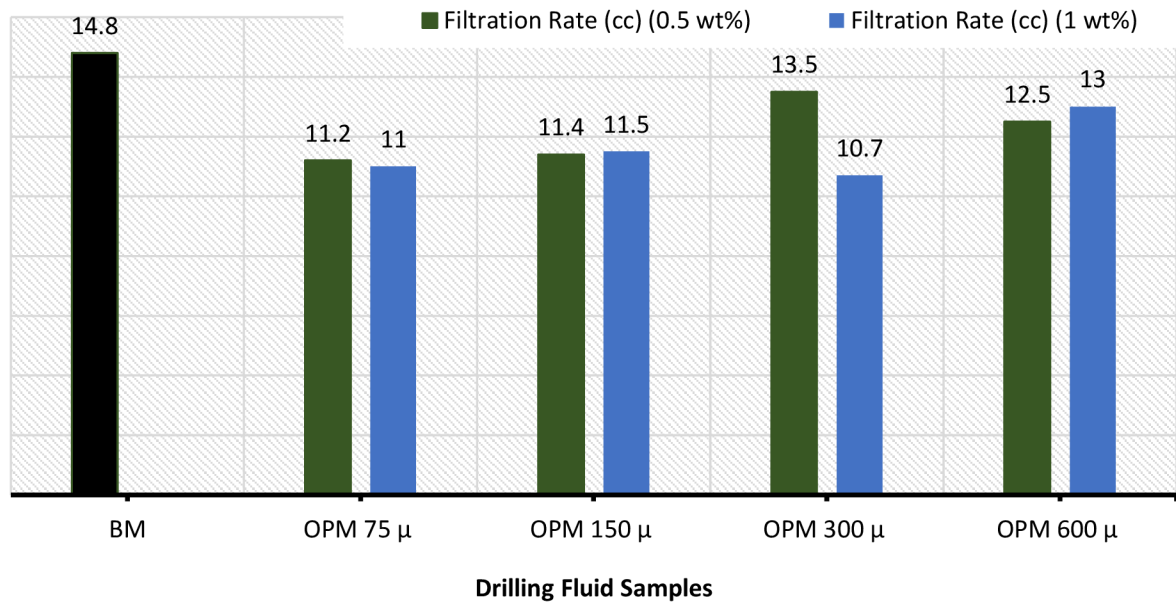


Figure 16 The results of filtration rate of oak peels at different concentration vs different particle sizes

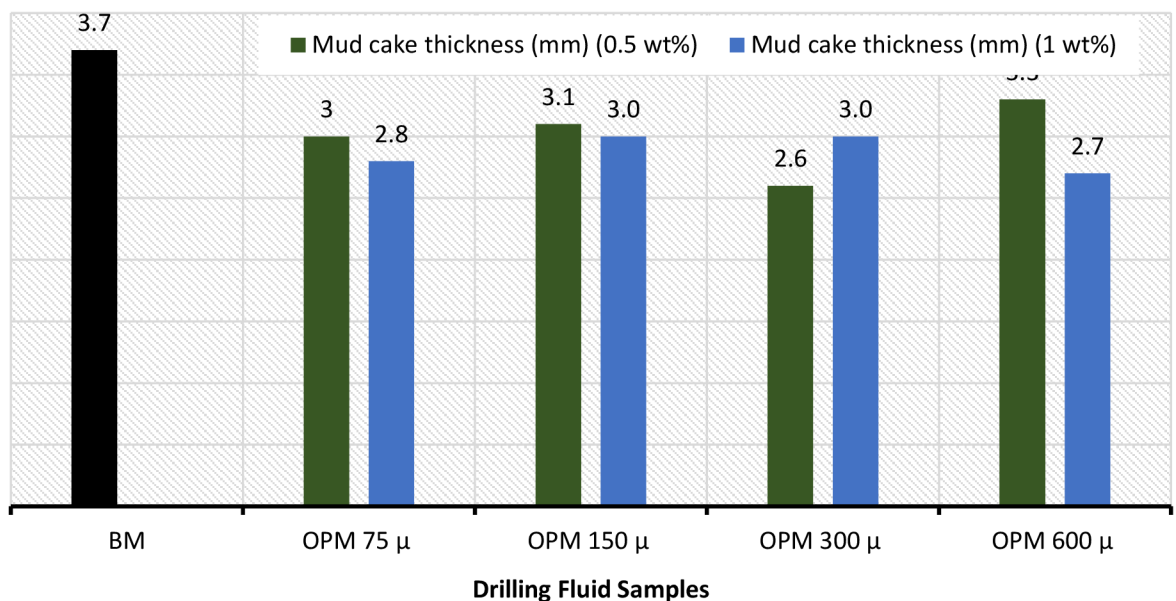


Figure 17 The results of mud cake thickness of oak peels different particle sizes and different concentrations

The synthesized nanorods and biodegradable materials as well as optimal concentrations of the combination of both of them were calculated and determined. Basically, the formulated drilling fluid with concentration (0.05 and 0.1 wt.%) of OPM recorded with different particle sizes starting from 75  $\mu$  to 600  $\mu$  and best result was recorded for 75  $\mu$  at 0.1 wt.% concentration of a fluid loss volume of 11 mL and a mud cake thickness of 2.8 mm. The result started increasing with bigger particle sizes and it reached highest value at 600  $\mu$  with fluid loss 13 mL and mud cake thickness 3.3 mm.

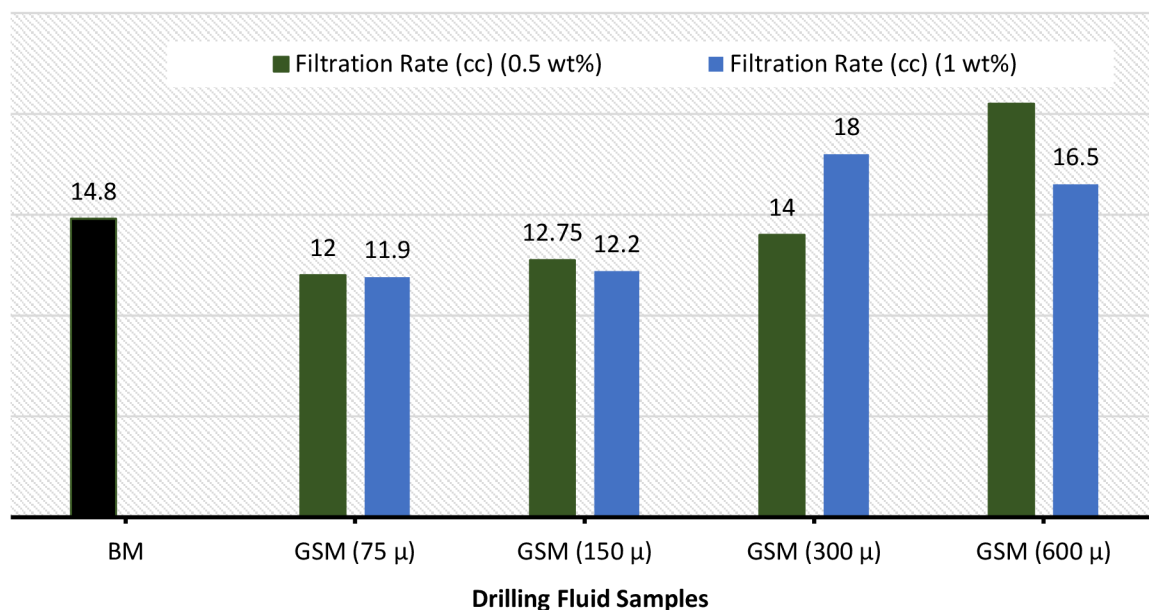


Figure 18 The results of filtration rate Gundelia seeds at different concentration vs different particle sizes

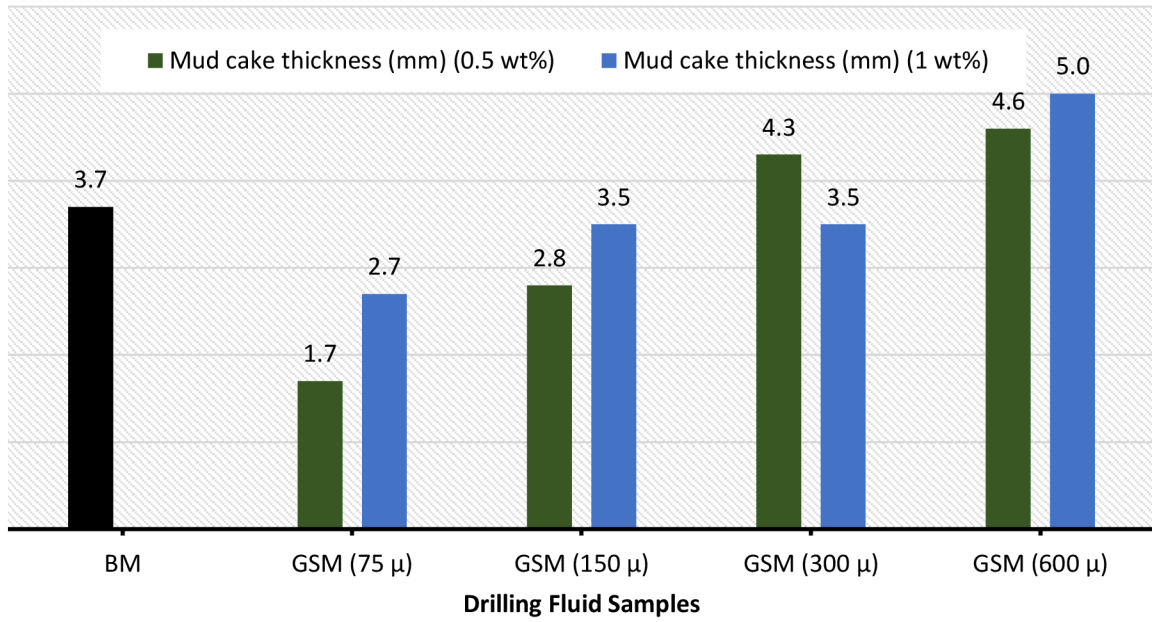


Figure 19 The results of mud cake thickness of Gundelia seeds at different particle sizes and different concentrations.

As Gundelia seeds powder were added to the base mud, at 75 μ a filtration rate of 11.9 mL was recorded, and the filter cake thickness was reduced by 2 mm compared to the base mud. After that, rambutan peel powder was added to the base sample and best values was receded at particle size 300 μ with fluid loss 9.75 mL and mud cake thickness 2.6 mm.

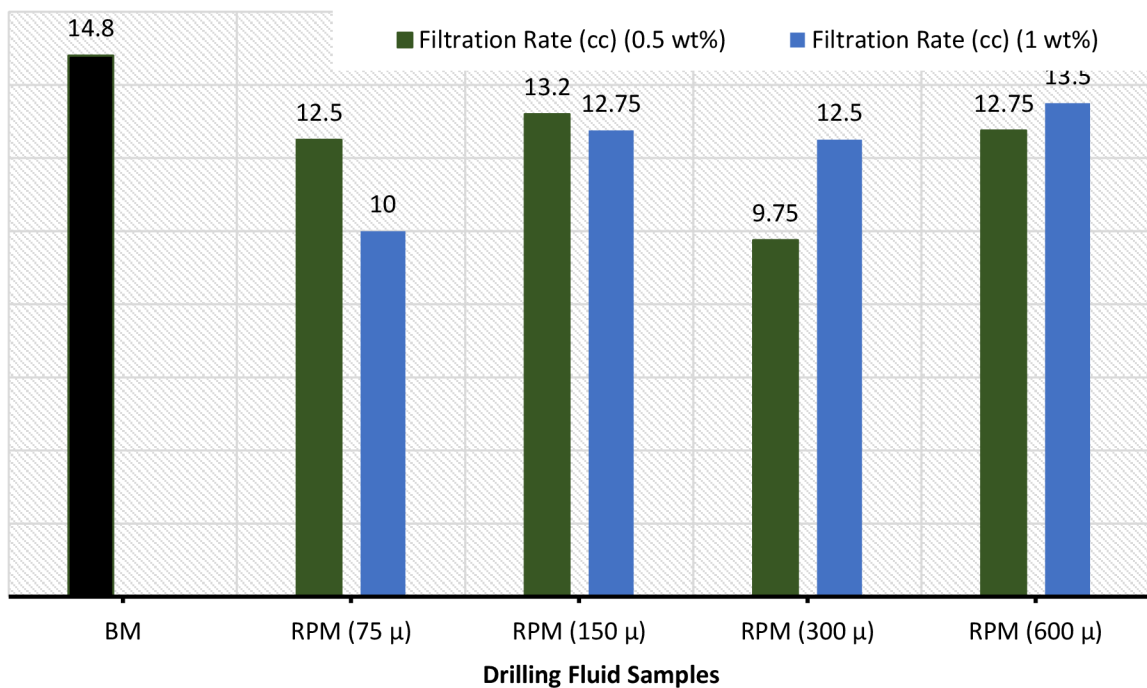


Figure 20 The results of filtration rate of rambutan peels at different concentration vs different particle sizes.

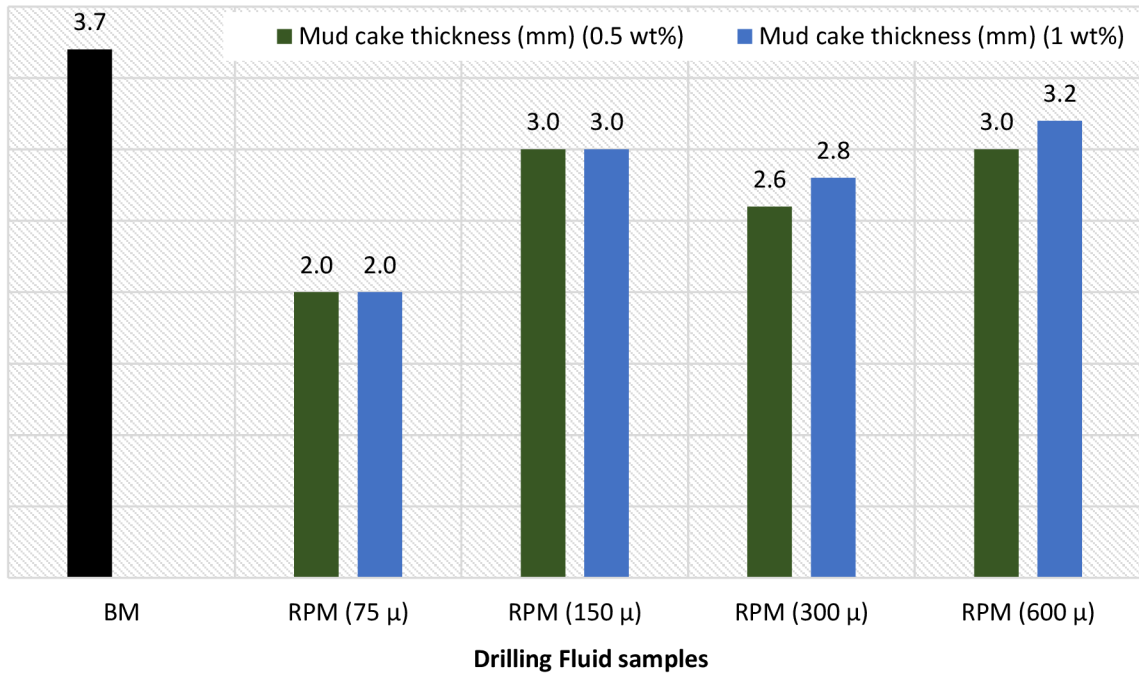


Figure 21 The results of mud cake thickness of rambutan peels powder different particle size vs different concentrations

Therefore, the result showing that with increasing particles size more fluid loss we will face and most losses was recorded was at particle size 600 μ with fluid loss 13.5 mL and mud cake thickness 3.2 mm. During the investigation of the mud with optimum concentration of nanorod and waste materials are added to study the optimum concertation in combination. The mud cakes thickness and filtration rates of the reference mud and combinations of biodegradable materials and zinc NRs modified drilling fluids. Figure 22 shows that the addition of 0.1 wt.% ZNO nanorod, reduced mud thickness and fluid loss to 3 mm and 10.2 mL in comparison with the base mud. After that, the addition of 0.1 wt.% of the zinc NRs to the OPM, reduced fluid loss and mud thickness to 1.7 mm and 10 mL compared with the base mud. Further on, the addition of 0.1 wt.% GSM to nanorod reduced the mud thickness to 3 mm. Meanwhile, the fluid loss is highly reduced to 9.3 mL in comparison with the base mud. However, the most efficient result was using 0.1 wt.% of RPM in addition with nanorod. Mud thickness is highly reduced to 1.5 mm and fluid loss is reduced to 9.5 mL.

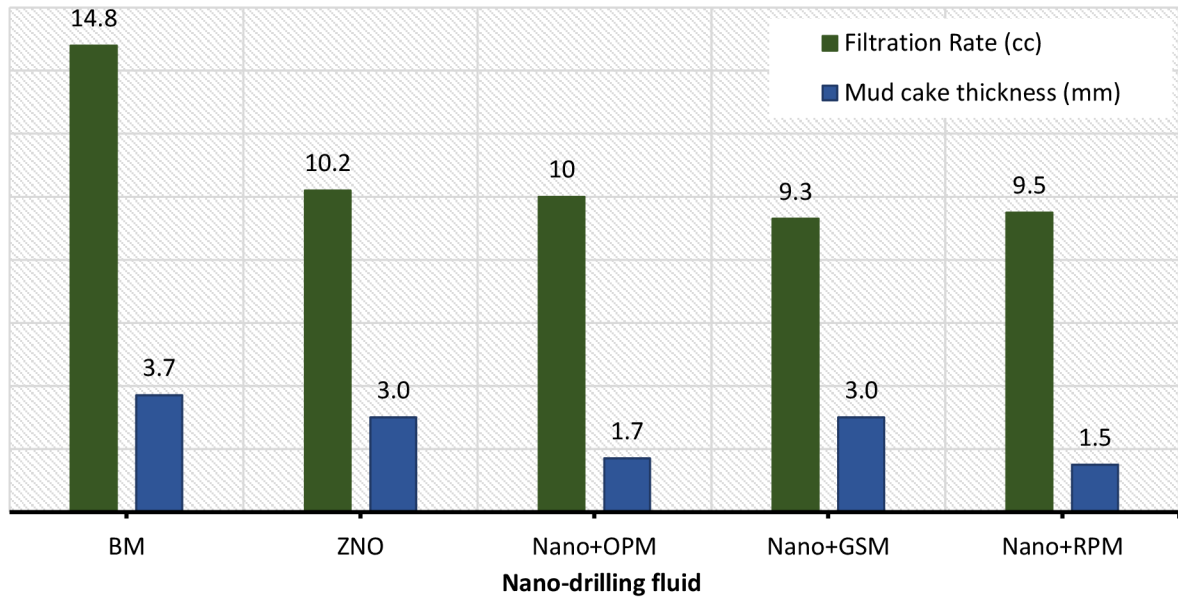


Figure 22 The results of filtration rate and mud cake thickness of waste materials (oak peels, gundelia seeds, and rambutan peels) and nanorod.

## Chapter 5: Conclusions and recommendations

### 5.1 Conclusions

The objective of the research study was to formulate a drilling fluid using either nanoparticles or biodegradable waste materials or a combination of both of them, as well as to analyze the effects of these materials on the filtration characteristics. During the investigation period, the optimum concentrations of nanorod were discovered to be 0.1 wt.%, respectively, based on the achieving the lowest filtration rate. Moreover, the different particle sizes were investigated to show the best particle size for OPM was 75  $\mu$ , GSM was 75  $\mu$ , and RPM was 300  $\mu$ . The results revealed that as the nanoparticles were mixed with various concentrations of the biodegradable

materials, optimum concentrations in weight percentage of the combination were 0.1 for OPM, GSM and RPM. Using the nanoparticles and biodegradable materials under study in any concentration resulted in reductions in the filtration rates and filter cake thickness. The results showed that the optimum results were acquired when GSM of 0.1% (by weight) was added to the base mud and fluid loss volume was reduced to 9.3 mL. Additionally, optimum filter cake thickness of 1.5 mm was achieved when 0.1 wt.% RPM was mixed with optimum concentration of nanorod.

## **5.2 Recommendations**

Throughout this study on the role of new biodegradable materials in combination with the synthesized green nanorods on the rheological and filtration properties of the drilling fluids, it can be recommended that:

- The effect of the temperature and pressure on the biodegradable drilling fluids prepared from the Gundelia, Oak peel and rambutan peel powders can be studied.
- The effect of the temperature and pressure on the nano-drilling fluids prepared from the synthesized zinc nanorods can be studied.
- The effect of the temperature and pressure on the nano-biodegradable drilling fluids prepared from the Gundelia, Oak peel, rambutan peel powders and zinc nanorods can be studied.
- The effect of the nano-biodegradable (nano-biopolymer) on the rheological and filtration properties of the drilling fluids can be studied.



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