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An Experimental Assessment of the Impact of TiO_2 and Al_2O_3 Nanoparticles and Tragacanth on the Augmentation of Oil Recovery in Limestone Reservoirs

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Abstract

Chemical Enhanced Oil Recovery (CEOR) methods are garnering significant global attention in light of the substantial decline in available oil resources. These techniques are employed to enhance oil recovery from various types of reservoirs by influencing critical parameters, such as the mobility of trapped oil within the reservoir rock's pore network, interfacial tension (IFT) between water and oil, wettability and the spreading behaviour of chemical solutions on rock surfaces. Nanoparticles (NPs) represent novel agents in CEOR processes, offering promising contributions owing to their enhanced surface activity at the crude oil-brine-rock interface and their impact on disjoining pressure. Incorporating polymers into nanofluids improves the dispersion stability of NPs, extending their capability for wettability alteration and achieving faster equilibrium states compared to polymer-free nanofluids. Experimental investigations involved the use of various combinations of tragacanth, TiO_2, and Al_2O_3 with water at different temperatures for oil recovery. The results revealed that the tragacanth + Al_2O_3 combination at 50°C exhibited the highest recovery rate of 16.57%. The most significant decrease in interfacial tension was observed at 25°C (16.75%), while the most favourable characteristics for reducing interfacial tension (9.38%) were observed at 50°C compared to 75°C (18.3%). Tragacanth demonstrated the highest viscosity among the tested substances at 25°C, with a measured value of 4.27 centipoise (cP), while reductions of 2.87 cP and 1.45 cP were observed at 50°C and 75°C, respectively. According to the obtained results, it is appropriate to say that the solution of tragacanth + Al_2O_3 has shown the best performance in increasing EOR and decreasing the interfacial tension in sand-pack flooding.

Keywords:
nanoparticles; polymers; EOR; limestone; reservoirs

1. Introduction

The International Energy Agency (IEA) predicts that the global demand for fossil fuels will increase by over 33% by 2035 (**Medina et al., 2019, Mortaghi et al., 2023**). Therefore, countries that strongly depend on petroleum should contemplate intensifying their oil extraction endeavours (**Sorbie, 1991, Ahmadi et al., 2022**). Nevertheless, the decrease in production rates from current reservoirs and the limited availability of financially feasible new reservoirs have compelled oil-producing corporations to utilize diverse enhanced oil recovery (EOR) methods to extend the productive lifespan of wells (**Cheraghian, 2015, Ali et al., 2018**).

Enhanced oil recovery (EOR) techniques are essential in the petroleum business as they can significantly enhance the recovery rate of oil reservoirs by up to 60% (**Tan et al., 2022; Ghosh and Mohanty, 2019**). Thermal recovery, gas injection, and chemical injection are some of the techniques employed for EOR (**Abadshapoori et al., 2018**). Thermal recovery is a process where steam is injected into the reservoir to decrease the thickness of the oil, making it easier to extract (**Patel et al., 2015; Yusuf et al., 2024**). Gas injection involves the injection of gases, such as natural gas or carbon dioxide to move oil towards producing wells (**Alvarado and Manrique, 2010**). Chemical injection involves the introduction of chemicals into the reservoir in order to modify the properties of the oil. This method has been proven to be effective, as demonstrated by studies conducted by **Scott and Romero-Zero´n (2020)**, as well as **Shafiei et al. (2023)**.

EOR techniques, such as alkaline, surfactant, and polymer fluid injections, have demonstrated exceptional efficacy and cost-efficiency (**Gbadamosi et al., 2018; Ahmadi et al., 2022**). These substances can be utilized either separately or in conjunction, for example, in the form of surfactant-polymer (SP) and alkaline-surfactant polymer (ASP) blends. Accurate evaluation of the rheological characteristics of these substances is essential in order to decrease interfacial tension, decrease capillary pressure, and improve sweep efficiency for the purpose

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of displacing remaining and trapped oil towards producing wells. Polymer flooding enhances the effectiveness of fluid displacement by minimizing the unevenness in permeability and regulating the ratios of fluid mobility. On the other hand, surfactant flooding modifies the wetting properties of the rock and reduces the tension between different fluid interfaces (**Huh and Pope, 2008**).

Currently, there is an increasing focus on the development of more effective technology for extracting oil from already existing reservoirs. This requires substantial progress in fundamental scientific and engineering principles within the oil and gas sector. Nanotechnologies have great opportunities to transcend existing energy supply alternatives with technologically advanced, dependable, and eco-friendly solutions. Nanotechnology's interdisciplinary nature guarantees accuracy in its applications, making it a more desirable option compared to traditional methods. Nanotechnology is an advanced industrial breakthrough that involves the manipulation of atoms and molecules to build materials, structures, and components at the nanoscale (**Hornyak et al., 2009**). Nanoparticles (NPs) within the size range of 1 to 100 nm (**Ali et al., 2018**) have significant importance in Enhanced Oil Recovery (EOR) as a result of their distinct chemical and physical characteristics (**Nazarahari et al., 2021, Mortaghi et al., 2023**). In addition, nanotechnology is used in various ways in the field of oil production and hydrocarbon-fuel technologies (**Huh et al., 2019; Panchal et al., 2021; Thakar et al., 2018, Davoodi et al., 2022**). The research conducted by Al-Shargabi et al. and Alsaba et al. suggests that nano-EOR has the potential to revolutionize oil production and recovery (**Al-Shargabi et al. 2022a, b; Alsaba et al., 2020; Davoodi et al., 2022**).

Nanomaterials can manifest in diverse forms and arrangements, such as spherical, tubular, or irregular geometries. They can exist as individual entities, fused structures, aggregates, or agglomerates (**Medhi et al., 2020**). They have the ability to appear as solid composites, complex liquids, or fluid components (**Ahmadlouydarab et al., 2021**). These materials, including nanoparticles (NPs), nanoclays, and nanoemulsions, need to undergo thorough testing in actual reservoir settings to demonstrate their effectiveness in subsurface environments (**Peng et al., 2018, Davoodi et al. 2022; Yusuf et al., 2024**).

Adsorption, precipitation, and phase transfer are the primary mechanisms employed in the flooding process to effectively retain surfactants within porous media. Surfactants that are impervious to salt and temperature can effectively inhibit the formation of precipitates and the entrapment of different phases. However, the attachment of a surfactant to rock can only be reduced. The presence of surfactants in the injected solution is diminished due to adsorption, leading to a decrease in the interfacial tension between oil and water. Consequently, the effectiveness of surfactant-based enhanced oil recovery (EOR) in releasing trapped oil is impacted (**Belhaj et al., 2020**).

Nanoparticles, which usually have sizes between 1 and 100 nm, are used in water flooding, gas flooding, and chemical EOR processes to enhance oil recovery (**Krishnamoorti, 2006**). The use of silica, alumina, and titanium oxide nanoparticles is common in chemically enhanced oil recovery to tackle issues like pore plugging and permeability reduction. These nanoparticles are preferred due to their small size and ability to penetrate hard-to-reach areas, resulting in improved microscopic efficiency (**Ahmadi et al., 2011; Alomair et al., 2014, Ahmadi et al., 2022**).

Nanoparticles have unique characteristics that are beneficial for improving oil recovery, including their exceptional surface area-to-volume ratio and their ability to interact with oil and water in diverse ways (**Rezk and Allam, 2019**). The use of nanoparticles (NPs) in enhanced oil recovery (EOR) can increase the pace at which oil is extracted from reservoirs by altering the characteristics of oil and water, therefore improving the movement of oil (**Kazemzadeh et al., 2015a, b; Sun et al., 2017; Rezvani et al., 2018**). In addition, customized nanoparticles (NPs) have the ability to specifically target certain types of oil, hence enhancing selectivity in the process of oil recovery (**Agista et al., 2018; Kazemzadeh et al., 2018; Yusuf et al., 2024**). Chemical approaches have demonstrated superior efficiency when compared to all nonthermal treatments. This is likely a result of reducing the interfacial tension (IFT), modifying the capacity of a liquid to spread on a solid surface (wettability), and enhancing viscosity by the best possible method (**Gbadamosi, et al., 2018**).

The utilization of nanoparticles (NPs) in enhanced oil recovery (EOR) has several advantages. NPs have the ability to reduce the interfacial tension (IFT) between the fluid and oil, hence enhancing oil recovery through their surfactant-like characteristics (**Ali et al., 2018; 2020; Yusuf et al., 2024**). In addition, nanoparticles (NPs) that possess polymer-like characteristics might improve the movement of fluids in porous materials, hence assisting in the recovery of oil by creating gels or substances that increase viscosity (**Kazemzadeh et al., 2019**). Nanoparticles (NPs) can also reduce the thickness of oil, making it easier to extract, and enhance the movement of oil inside the reservoir (**Hou et al., 2022**). Moreover, NPs that possess catalytic qualities have the ability to modify the chemical features of oil, hence enhancing the efficiency of the recovery process (**Rezaei et al., 2016; Tavakkoli et al., 2022; Ali, 2023, Yusuf et al., 2024**). Nanoparticles (NPs) can sometimes block the channels via which fluids and oil move in porous materials, which might be beneficial in some Enhanced Oil Recovery (EOR) techniques (**Taborda et al., 2016**).

Several categories of nanoparticles (NPs) have a role in enhanced oil recovery (EOR) methods, such as synthetic or natural NP polymers, ceramic NPs, metal NPs, carbon-based NPs, and Janus nanoparticles (**Crucho and Barros, 2017; Medhi et al., 2020; Deng et al., 2021; Astefanei et al., 2015, Davoodi et al., 2022**). The study conducted by Rahimi and Adibifard provides evidence of the beneficial effects of NPs and surfactants on oil recovery in sandstone reservoirs. The injection of NPs resulted in an 11% increase in ultimate oil recovery by modifying wettability and reducing interfacial tension (**Rahimi and Adibifard, 2015**).

Nanoparticles exhibit fascinating properties, specifically their notable surface area-to-volume ratio, which allows them to facilitate diffusion efficiently, particularly at elevated temperatures (**Chegenizadeh et al., 2016; Al-Anssari et al., 2018**). Due to their diminutive size, they may easily navigate through narrow openings in porous materials (**Rodriguez et al., 2009**). Several studies have demonstrated the positive effects of using nanoparticles in enhanced oil recovery (EOR). These effects include reducing interfacial tension (IFT), changing wettability, lowering oil viscosity, and improving the viscosity of injection fluids. Multiple studies emphasize the crucial significance of nanoparticles in chemical enhanced oil recovery (CEOR) procedures (**Ali et al., 2018; Emadi et al., 2017; Roustaei et al., 2012; Karimi et al., 2012; Taborda et al., 2016; Mohammadi et al., 2017; Ehtesabi et al., 2015; De Castro Dantas et al., 2017; Motraghi et al., 2023**). The benefits of using nanoparticles in enhanced oil recovery (EOR) applications include reducing interfacial tension (IFT), altering wettability, swelling heavy oil, stabilizing asphaltene, modifying viscosity, plugging pore channels, and altering disjoining pressure (**Chegenizadeh et al., 2016; Ju and Fan, 2006; Zaid et al., 2013; Torsater et al., 2012; Saien and Gorji, 2017; Al-Anssari, 2016; Kazemzadeh, 2015b; El-Diasty and Ragab, 2013; Bobbo et al., 2012; Anganaei et al., 2014; Mcelfresh et al., 2012a**). In addition, the distribution of nanoparticles in reservoir fluids can have several beneficial impacts, such as modifying wettability and improving sweep efficiency (**Zamani et al., 2012; Mcelfresh et al., 2012b**).

The effective transport of nanoparticles in porous medium is vital and has been thoroughly examined. According to **Rodriguez et al. (2009)**, nanoparticles may effectively pass through narrow openings in pores because of their small dimensions and can stay evenly distributed in solutions due to their reactive surface and strong stability. Aurand et al. claimed that nanofluids containing fumed silica nanoparticles have superior adsorption efficiency and recovery performance compared to those containing colloidal silica nanoparticles (**Aurand et al., 2014**). **Li and Torsaeter (2014)** conducted a comparison of the transportation and adsorption characteristics of different silica nanoparticles (NPs). They found that hydrophilic silica non-structure particles (NSP) exhibited superior adsorption ability compared to hydrophilic silica colloidal nanoparticles (CNP).

Several recent studies have highlighted the potential of several types of nanoparticles, namely those made of silica, in improving the process of oil recovery. These nanoparticles have been shown to change the ability of a surface to be wetted, decrease the tension between two interfaces, and improve the ratio of mobility. This has been demonstrated by **Suleimanov et al. (2011)**, **De Castro et al. (2017)**, and **Moradi et al. (2015)**. Onyekonwu and Ogolo conducted a study to examine how different polysilicon nanoparticles (PSNP) can change the wettability of reservoir rocks. They found that various PSNPs successfully transformed the rocks into strongly water-wet systems (**Onyekonwu and Ogolo, 2010**). Hendraningrat et al. conducted a study that confirmed the substantial influence of silica-based nanoparticles on enhancing oil recovery. They achieved this by altering surface forces, taking into account factors such as nanoparticle size, initial rock wettability, nanoparticle concentration, and injection rate. This research was conducted in multiple studies by **Hendraningrat et al. (2012, 2013a, and 2013b)**. **Shahrabadi et al. (2012)** conducted a study on the impact of hydrophobic and lipophilic polysilicon (HLP) nanofluids on oil recovery. They observed a notable change in wettability and a decrease in interfacial tension (IFT). In their study, **Mohammadi et al. (2014)** discovered that aluminum oxide (Al_2O_3) nanoparticles are efficient agents for enhanced oil recovery (EOR) in sandstone reservoirs. Tarek introduced an innovative enhanced oil recovery (EOR) technique that employs nanofluids including different types of nanoparticles such as Al_2O_3, Fe_2O_3, and SiO_2. This method has been shown to provide better oil recovery results compared to nanofluids containing only a single type of nanoparticle (**Tarek, 2015; Ali et al., 2018**).

Aluminium nanoparticles have the ability to stick to the walls of reservoir pores, which changes the way the surface interacts with liquids and helps to extract more oil. However, they can clump together and cause difficulties in manufacturing and potential harm to the environment under high-pressure/high-temperature conditions. Titanium nanoparticles demonstrate exceptional performance in oxidation, photocatalysis, and adsorption. However, their effectiveness is impeded by the presence of a restricted number of heterogeneous binding sites for polymerization. This information is supported by studies conducted by **Lashari et al. (2022), Elakkiya et al. (2020)., Mathew Simon et al. (2021), Jalilian et al. (2019), Surya Mohanty et al. (2021), Mariyate and Bera (2021), Adil et al. (2020), Barkhordari and Jafari (2018), Tavakkoli et al. (2022), El-hoshoudy (2022), Tran and Nguyen (2014), Sun et al. (2018), Norhasri et al. (2017), Pitroda and Dave (2016), Davoodi et al. (2022)**. Due to the above-mentioned features of nanoparticles and surfactants, their potential for resolving and preventing some of the existing challenges that are facing the oil industry is high. Hence, nanoparticle injection alone and combined with other chemicals offers great opportunities to address the challenges caused by traditional EOR methods (**Almahfood and Bai, 2018**).

Nanofluids, new types of liquids that consist of a base liquid with suspended particles at the nanoscale, present a significant opportunity for CEOR. Nanofluid flooding is the process of pumping fluids containing nanomaterials or nanocomposites into reservoirs in order to improve the displacement of oil. This is achieved by modifying the wettability, reducing interfacial tension, and altering the disjoining pressure (**Mohammadi et al., 2019; El-Diasty and Aly, 2015**). The process of creating "smart fluids" involves the addition of nanoparticles to specific fluids. Inorganic nanoparticles are preferable since they have a lower tendency to accumulate and clump together (**Manshad et al., 2022; Jia et al., 2021**).

In addition, nanofluids have shown the capacity to reduce interfacial tension (IFT) between oil and water phases, enhancing the capillary number and enabling the migration of nanoparticles towards oil-water interfaces (**Li and Torsaeter, 2014**). Polymer flooding, a chemical process that utilizes polymers, surfactants, and alkalines, has shown improved sweep efficiency. However, it is important to note that this method has disadvantages, such as high cost, potential formation damage, and chemical loss (**Wei, 2016; Ahmadi et al., 2022**).

Injecting polymers into water alters the flow characteristics, leading to the improved extraction of oil by decreasing the ratio of fluid mobility, raising the effectiveness of sweeping, stabilizing the mixture of oil and water, and minimizing the formation of water channels. The efficacy of polymer flooding is impacted by reservoir conditions, crude oil characteristics, temperature, and salinity. While fines may include residual oil, the process of fines migration can lead to a reduction in permeability due to the partial blockage of pore space. Polymer flooding mechanisms involve the reduction of permeability by the adsorption of polymers on pore surfaces and an increase in flow resistance due to polymer elasticity. This technique is commonly applied in sandstone reservoirs (**Jiang and Zhu, 2014**).

Silicon dioxide (SiO_2) is the most often used type of nanoparticles (NPs) for enhanced oil recovery (EOR) applications, as mentioned by **Hendraningrat et al. (2013)** and **Ju and Fan (2006)**. In addition, iron oxide (Fe_2O_3), nickel oxide (Ni_2O_3), and aluminium oxide (Al_2O_3) have been found to act as catalysts and support catalysts, affecting the flow properties of oil, especially at high temperatures in reactors. These findings have been reported by **Li et al. (2007), Nares et al. (2015), and Hashemi et al. (2013)**. The findings have shown that aquathermolysis reactions, aided by metal particles, can decrease the viscosity of generated oils by converting hydrogen atoms to free radicals. This process hinders the occurrence of polymerization, condensation reactions, and coke production (**Hashemi et al., 2013**). Furthermore, research conducted by **Hamedi Shokrlu and Babadagli (2010), Bayat et al. (2014)** demonstrates that metal nanoparticles are effective in decreasing the thickness of oil, both at elevated temperatures and at lower temperatures. In general, the combination of nanoscale metals and metal oxides shows potential for energetics since it results in a substantial release of energy during oxidation (**Darlington et al., 2009**). It is important to emphasize that all the described research used metal nanoparticles in a context that was separate from porous materials.

Ehtesabi et al. (2014), Ogolo et al. (2012), Zaid et al. (2013), and Hendraningrat and Torsaeter (2014) examined the use of metal oxide nanoparticles (NPs), such as Al_2O_3, titanium dioxide (TiO_2), and zinc oxide (ZnO), for enhanced oil recovery (EOR) purposes. Nevertheless, their research could not document any discernible alterations in the calibre of the generated oils. It is worth mentioning that the majority of these tests were carried out at normal temperatures. This could be the reason why there is no mention of the rheological qualities of the oils produced. Hence, it is imperative to conduct a thorough investigation to evaluate the impact of temperature on EOR while utilizing metal nanoparticles (NPs).

Multiple research investigations have examined the efficacy of various types of nanoparticles in improving the process of extracting oil. **Roustaei et al. (2012)** found that hydrophobic and lipophilic polysilicon nanoparticles had a positive influence on oil recovery, with hydrophobic polysilicon showing better results. Furthermore, researchers have investigated the effectiveness of several metal nanoparticles, including Fe_2O_3, Al_2O_3, and SiO_2, in improving the performance of enhanced oil recovery (EOR). It has been found that Al_2O_3 and SiO_2 are more successful in recovering oil compared to Fe_2O_3. In addition, the surface modification of nanoparticles has been proven to boost their efficacy in enhancing oil recovery through different methods, as evidenced by **Ju and Fan (2009)** and **Qi et al. (2018)**.

Recent developments in water flooding techniques, including low salinity and smart water injection, as well as hybrid approaches, have prioritized features such as wettability change, interfacial tension reduction, and viscosity augmentation to maximize efficiency. In addition, polymers have been used to regulate water viscosity and reduce fingering effects in flooding processes. Nevertheless, the amalgamation of polymers and nanoparticles necessitates the resolution of obstacles such as exorbitant expenses, compatibility discrepancies, and diminished permeability.

In this study used tragacanth as a polymeric surfactant in injected water effect on interfacial tension and effectiveness at optimum salinity. **Nowrouzi et al. (2019)** examined the effect of tragacanth gum as a natural polymeric surfactant and soluble ions on chemical smart water injection in oil reservoir. Even at a low concentration of tragacanth, interfacial tension between water and oil reduced and reaches CMC at low concentrations. Therefore, it can be considered as a useful additive for injected water. Tragacanth increases the viscosity of water, and at high concentrations, has non-Newtonian be-

Table 1: Specifications of the polymer solution prepared

Deionized water	pH of water	NaOH 1mol	NaCl	pH of Solution	Temperature	Tragacanth	Mixer speed
999 ml	4.5	1 ml	1 g	9	25°C	1 g	900 rpm

haviour. Increasing the viscosity of the injectable phase can regulate the ratio of water and oil mobility in the porous medium and cause the piston movement to prevent the phenomenon of fingering and the early production of the injectable phase.

The objectives of this study were to examine the effectiveness of polymer water flooding in enhancing oil recovery, assess the impact of low concentrations of silica and aluminium nanoparticles on oil recovery, and evaluate the performance of polymer nanoparticles combined with polymer in enhancing oil recovery in calcareous reservoirs.

One of the primary aims of the experiments conducted in this study is to transition polymer and nanoparticle improved oil recovery trials from the research phase to the practical phase. This implies that the economic feasibility of incorporating nanoparticles and polymers in the enhanced oil recovery process can be determined by optimizing the concentration of nanoparticle oxides and the temperature of the polymers. This optimization allows for the practical implementation of nanoparticle oxides and polymers in large-scale operational settings. Nevertheless, the focus of these investigations is to utilize minimal concentrations of nanoparticles in conjunction with a polymer. Another aim of these investigations is to do trials at temperatures higher than the surrounding environment. In contrast, the use of crushed limestone as a permeable medium enables us to approximate the conditions seen in limestone reservoirs, hence enhancing the fidelity of experimental outcomes to real-world scenarios.

2. Experimental work

2.1. Materials

2.1.1 Polymer fabrication & solution preparation

The polymer utilized in this study was derived from tragacanth, a naturally occurring polymer sourced from the Astragalus plant. The polymer of interest is obtained in the form of dehydrated leaves and subsequently transformed into a powdered state through the process of milling. The tragacanth employed should possess a pristine, white and clear appearance, devoid of any impurities manifesting as yellow streaks. To facilitate the dissolution of the tragacanth and initiate the polymerization process, a sodium chloride solution (NaCl) with a concentration of 0.1% by weight was first prepared. To dissolve the tragacanth and prepare its polymer from a solution (0.1wt%, NaCl): 1 gramme of NaCl was weighed with an electronic balance and then 999 ml of deionised water (DIW) was added. Following this, to make 0.1wt% of tragacanth (1 gram of tragacanth) was introduced into the solution and subjected to mechanical mixing for a duration of 3 hours, ensuring the formation of a homogeneous solution. It should also be mentioned, the density of nano particles and polymers are equal to 50 mg/l.

It is crucial to acknowledge that adjusting the pH of deionized water is necessary for enhancing the stability of tragacanth and preventing sedimentation. In order to alter the pH of the solution, a 1 molar solution of sodium hydroxide (NaOH) is introduced into deionized water. When considering the use of nanoparticles in reservoir engineering, several limitations and challenges come to light, which can affect their practical applicability and long-term effectiveness: nanoparticles may aggregate or disperse unevenly, leading to ineffective distribution and reduced efficiency in altering reservoir properties. While nanoparticles may initially enhance oil recovery or alter reservoir permeability, their long-term effects are not well understood. There could be unintended consequences, such as reservoir clogging, alteration of rock properties, or environmental impacts that only become apparent over time. Nanoparticles may face challenges in transport through porous media. Factors such as pore size distribution, fluid composition, and surface interactions can influence their mobility within the reservoir. The cost of synthesizing, deploying, and monitoring nanoparticles in reservoirs may be prohibitive, especially for large-scale applications. Specifications of the prepared polymer solution are presented in **Table 1**.

2.1.2 Nanoparticle suspension & solution

In this study, two nanoparticle oxides, TiO_2 and Al_2O_3 were used and their properties are respectively as follows:

20 nm, specific surface area = 160 m^2/g, and purity percent= 99.5%,

40 nm, specific surface area = 60 m^2/g, and purity percent= 99%.

The oxide of nanoparticles was prepared from Sky Spring Nano-Materials (Houston, TX, US). The nanoparticle suspension before each test is made as follows:

In order to dissolve the nanoparticle and make a suspension with the previously prepared solution (1% tragacanth and 0.1% NaCl), after weighing the nanoparticles and taking the nanoparticles required amount, and adding it to the fabricated solutions, the suspension is mixed for one hour by a Mechanical Mixer at a speed of 900 rpm. Characteristics of nanoparticle solutions are presented in **Table 2**.

Table 2: Characteristics of nanoparticle solutions

Sample name	Base solution	Nanoparticles mg/l	Experiment temperature °C	Mixer speed rpm
Water + TiO_2	deionized water (0.1 w% NaCl)	50	25	900
Water + Al_2O_3	deionized water (0.1 w% NaCl)	50	25	900
Tragacanth solution + TiO_2	Tragacanth solution	50	25	900
Tragacanth solution+ Al_2O_3	Tragacanth solution	50	25	900

Table 3: Characteristics of solutions

Sample	Experiment temperature (°C)	Density (g/cm^3)	Viscosity (cP)	pH
Deionized water solution (0.1w% NaCl)	25	0.9997	0.97	4.9
Tragacanth solution		0.998	4.27	5.2
Water + TiO_2		0.999	0.98	5.2
Water + Al_2O_3		0.9984	1.01	5.1
Tragacanth solution + TiO_2		0.9997	3.19	5.3
Tragacanth solution + Al_2O_3		0.9989	1.96	5.4
Deionized water solution (0.1w% NaCl)	50	1.005	0.62	5.1
Tragacanth solution		0.9877	2.87	5.3
Water + TiO_2		0.9902	0.63	5.6
Water + Al_2O_3		0.987	0.63	5.4
Tragacanth solution + TiO_2		0.99	1.94	5.5
Tragacanth solution + Al_2O_3		0.9882	1.24	5.1
Deionized water solution (0.1w% NaCl)	75	0.9817	0.44	5.6
Tragacanth solution		0.9334	1.45	5.9
Water + TiO_2		0.9753	0.50	5.2
Water + Al_2O_3		0.9671	0.49	5.1
Tragacanth solution + TiO_2		0.9997	1.25	5.6
Tragacanth solution + Al_2O_3		0.9731	0.71	5.4

The results of the density and viscosity tests of the suspensions made with a viscometer (Model – SVM 3000) and the pH was measured with a pH meter.

2.2. Characteristics of solutions

The results of the density, viscosity, and pH tests of different prepared solution presented in **Table 3**.

2.3 Core & Porous media

In order to conduct laboratory scale experiments on increased oil recovery, it is necessary to utilize a core sample of porous media that possesses distinct physical characteristics, such as permeability and porosity. Sand pack was utilized in the conducted experiments due to the ongoing development of the polymer nanoparticle uptake technique. The objective of employing these packs was to assess various parameters including permeability, porosity, and the influence of nano and polymer materials on wetness and interfacial tension (IFT).

Limestone particles are utilized in the production of porous media. In order to get calcareous grains, the initial step involves procuring limestone from limestone mines. Initially, it is recommended to employ a hammer to divide these rocks into smaller fragments, thereby achieving an appropriate size for subsequent utilization as feed material for the jaw crusher machine. Subsequently, the stones should be subjected to crushing using the jaw crusher, ensuring a reduction in size to approximately 0.5 cm. This crushing process should be carried out in two distinct stages. The material produced by the jaw crusher machine is further processed in a roller crusher machine in a two-step process to achieve a particle size of up to 1 mm. The resulting product derived from the utilization of a roller crusher machine will be propelled by a mill machine. It is important to acknowledge that in the operation of jaw crusher machines, roller crushers, and ball mills, the initial step involves feeding the machine with glass material. Subsequently, an air compressor is employed to thoroughly clean the machine, hence enhancing the level of certainty in its performance. The powder acquired from the ball mill was subjected to sieving, resulting in particle sizes ranging from 125 to 212 microns. The sample was subjected to rinsing using a 125-micron filter, followed by drying at a temperature of 110°C in an oven.

2.4. Procedure of sand-pack making

A cylindrical tube, measuring 2.18 mm in diameter and 36 cm in length, composed of stainless steel, has been selected for the purpose of constructing a sand pack. The valves for opening and closing were attached to the two ends of the pipe via connectors. It is important to acknowledge that the pipes lack any visible seams, and just let the flow of fluids through the valves for entry and exit. Following the closure of one end of the pipe, the lime particles and distilled water were gradually introduced into the tube, ensuring uniform compression. Through this process, we were able to obtain a uniform core, which was next subjected to drying at a temperature of 105°C for a duration of 12 hours. In addition, we implemented the installation of 50-micron filters at both extremities of the pipe in order to effectively mitigate the potential discharge of grain particles. Following the drying process, the cores underwent vacuuming and subsequent weighing.

2.5. Porosity of sand-pack

The core sample was subjected to a flow of deionized water at a rate of 1 ml/min. It is important to acknowledge that in all instances of injection, the cores were oriented vertically and injected in a bottom-to-top manner. The injection was sustained until an approximate volume of 150 ml of water was generated. Following the thorough saturation of the cores with deionized water, we proceeded to measure their weights and determine the pore volumes using the prescribed formula (for each sand-pack made, there will be a new porosity which has been presented in **Table 2**):

Vp = [(Weight of core saturated with deionized water) - (Dry and vacuum core weight of deionized water)] /1 g/cm^3

$$V_b = \pi r^2 L \tag{1}$$

Where:

V_b – bulk volume (cm^3),
r – core radius (cm),
L – core length (cm).

Using the porosity formula, we obtain it in percent:

$$Ø = \frac{V_p}{V_b} \times 100 \tag{2}$$

Where:

Ø – porosity,
V_b – bulk volume (cm^3),
V_p – pore volume (cm^3).

2.6. Absolute permeability of sand-pack

It is widely accepted among petroleum engineers that the Darcy formula is among the most commonly used and, one might even say, the basic formula for understanding fluid flow in reservoirs.

Darcy's formula (1856):

$$q = \frac{KA}{\mu}\frac{\Delta P}{L} \tag{3}$$

Where:

q – flow rate (m^3/sec),
K – absolute permeability (m^2),
μ – fluid injection viscosity (Pas),
A – cross-sectional area of core (m^2),
ΔP – the pressure difference between two ends of core (Pa),
L – core length (m).

The Darcy formula can be rearranged for absolute permeability:

$$K = \frac{q\mu L}{A\Delta P} \tag{4}$$

Sand pack and water parameters: A = 0.000369 m^2, L = 0.365 m, μ = 0.001 Pas.

To obtain absolute permeability, we choose three different flow rates: $1.7*10^{-8}$, $3.3*10^{-8}$and $8.3*10^{-8}$ m^3/sec for measurement, and the absolute permeability were calculated by simple arithmetic averaging (for each sand-pack made, there will be a new absolute permeability which has been presented in **Table 2**):

$$K = \frac{K1 + K2 + K3}{3} \tag{5}$$

2.7. Connate water and oil saturation of sand-pack

The inherent water present in reservoirs is characterized by its salinity, which varies in a unique manner for each reservoir. In the course of the experimental procedure, a solution consisting of sodium chloride (NaCl) and distilled water with a weight percentage of 3% was employed. The core was subjected to vertical injection of saline water at a rate of 1ml/min, which corresponded to 2 times the pore volume (2Vp). Subsequently, the oil was introduced into the core, which had been saturated with salty water, at a rate of 1 ml/min, equivalent to two times the pore volume (2Vp). The oil and saline water were measured with a great degree of precision. Through the utilization of basic mathematical operations:

$$S_0 = \frac{(Amount\ of\ oil\ injected\ (2V_p)) - (Amount\ of\ oil\ produced(ml))}{V_p} \tag{6}$$

Where So is the amount of saturated oil inside the sand pack.

$$S_{wc} = \frac{(Amount\ of\ sailine\ water \in the\ core) - (amount\ of\ water\ produced\ during\ oil\ injection)}{V_p} \tag{7}$$

Where S_{wc} is the amount of saturated connate water inside the sand pack.

The oil used during experiment was from one of the oil fields of Iran. Its API gravity was 36 and its dynamic viscosity was 6.35 cP.

2.8. Waterflooding

After saturating the core with oil, water flooding experiments were performed. In these experiments, saline water (NaCl 1 wt%) was injected into the core at 1ml/min flow rate, twice the pore volume (2Vp).

2.9. Enhanced oil recovery using polymer and nanoparticle suspension

The abovementioned steps were required to prepare the cores for the EOR experiments and research. During the EOR process, 2Vp amount of the desired solution was injected to the cores at a flow rate of 1 ml/min.

2.10. Calculation of interfacial tension

The contact angle method was employed in the analysis of porous medium to ascertain its hydrophilic or oleophilic nature, following established conventions. Furthermore, we investigated whether the suspension angles of oil and fluid wettability were altered subsequent to the injection of the tragacanth suspension and the nanoparticles.

A limestone slab measuring 5 cm in length and 2 cm in width is horizontally immersed in solutions of varying temperatures, given that the porous media consists of limestone. Utilizing an insulin syringe, a volume of 0.1 cm^3 of oil was spread over the surface of the limestone specimen, subsequently allowing it to remain undisturbed for a duration of 48 hours to attain equilibrium. Subsequently, a digital camera equipped with a lens of microscopic scale was modified to capture photographs. By employing analytical tools, we performed calculations to determine angles and assess wettability.

Typically, within hydrocarbon reservoirs, the three phases of water, oil, and gas exist in a state of equilibrium, with a shared interface that has a thickness to the order of a few molecules. The concept of interfacial tension (IFT) is employed in the analysis of the forces acting between two surfaces that are heterogeneous in nature and composed of liquids. There are various methodologies for measuring interfacial tension between surfaces and oil, one of which involves utilizing the Tensometer instrument (specifically the Data Physics/DCAT9 model). The Du NoFuy Ring technique is employed in our study. The machine is capable of autonomously entering the loop within the fluid and detecting the contact surface between the two fluids by analysing variations in weight.

2.11. Challenges of nanoparticle application

When considering the use of nanoparticles in reservoir engineering, several limitations and challenges come to light, which can affect their practical applicability and long-term effectiveness. Nanoparticles may aggregate or disperse unevenly, leading to ineffective distribution and reduced efficiency in altering reservoir properties. While nanoparticles may initially enhance oil recovery or alter reservoir permeability, their long-term effects are not well understood. There could be unintended consequences, such as reservoir clogging, alteration of rock properties, or environmental impacts that only become apparent over time. Nanoparticles may face challenges in transport through porous media. Factors such as pore size distribution, fluid composition, and surface interactions can influence their mobility within the reservoir. The cost of synthesizing, deploying, and monitoring nanoparticles in reservoirs may be prohibitive, especially for large-scale applications.

3. Results

3.1. The results of nanoparticles and polymers application on EOR

The results of the use of nanoparticles and polymers for EOR tests are summarized in **Table 4**. In order to enhance the assessment and examination of oil recovery by saline water flooding and nanopolymeric fluids, the quantification of oil recovery was carried out with regards to the volume of fluid injected.

3.2. The Comparison result of EOR by using polymeric and nanofluids and tertiary at different temperatures

This study examines the comparative analysis of oil recovery using polymeric and nanofluid agents, alongside the tertiary recovery process applying polymeric and nanofluid agents, across different levels of temperature (25, 50 & 75°C**)** as shown in **Figure 1, Figure 2** and **Figure 3** and **Table 4**.

Figure 1, **Figure 2** and **Figure 3** show a comparison of oil recovery by polymeric and nanofluids at different temperatures.

3.3. Movement mechanism

Different processes are involved during oil displacement by Nano-polymer water flooding. To evaluate these processes, the capillary number is a widely used parameter to evaluate the effect of the nanoparticle and polymer nanoparticles in the EOR process. The capillary number is calculated by dividing viscous force by capillary force.

The capillary number (Nc) is mathematically defined as follows (**Jeong, 2005**):

$$N_c = \frac{Viscose\ force}{capillary\ force} = \frac{\mu \times V}{\times \cos} \tag{8}$$

Table 4: Enhanced Oil Recovery (EOR)

Test. No	Solution	Temp °C	ϴ %	K (Darcy)	S_O (ml)	S_{WC} (ml)	Secondary recovery (%)	Additional oil recovery via NPs+polymer (%)	Ultimate oil recovery (%)
1	NaCl	25	40	2.33	47.26	3.23	58.22	3.28	61.5
2	Tragacanth	25	42.7	2.39	54.73	3.67	58.01	8.57	66.58
3	Water + TiO_2	25	39.10	2.37	49.40	4.06	58.7	6.98	65.68
4	Water + Al_2O_3	25	36.8	2.38	46.05	4.27	59	7.01	66.01
5	Tragacanth + TiO_2	25	44.13	2.35	55.03	5.31	59.7	9.08	68.78
6	Tragacanth + Al_2O_3	25	41.18	2.30	52.78	3.54	59.46	10.68	70.14
7	NaCl	50	41	2.7	51.35	5.04	63.1	6.02	69.12
8	Tragacanth	50	40.7	2.1	51.15	4.5	63.26	11.86	75.12
9	Water + TiO_2	50	38	2.23	48.17	3.79	63.9	10.3	74.2
10	Water + Al_2O_3	50	39.6	2.36	49.55	4.6	63.33	11.45	74.78
11	Tragacanth + TiO_2	50	42.1	2.8	52.51	5.06	64.1	13.85	77.95
12	Tragacanth + Al_2O_3	50	40.15	2.4	49.42	4.49	63.54	16.57	80.11
13	NaCl	75	39	2.2	50.13	3.2	66.5	8	74.5
14	Tragacanth	75	39.5	2.31	49.69	4.32	66.59	16.41	83
15	Water + TiO_2	75	42.6	2.45	53.30	4.95	65.8	13.5	79.01
16	Water + Al_2O_3	75	40.5	2.3	50.12	5.26	65.51	15.3	81.1
17	Tragacanth + TiO_2	75	38.1	2.47	48.34	3.75	67.3	18.2	86
18	Tragacanth + Al_2O_3	75	43.7	2.15	54.46	5.31	67.8	22.6	89.9

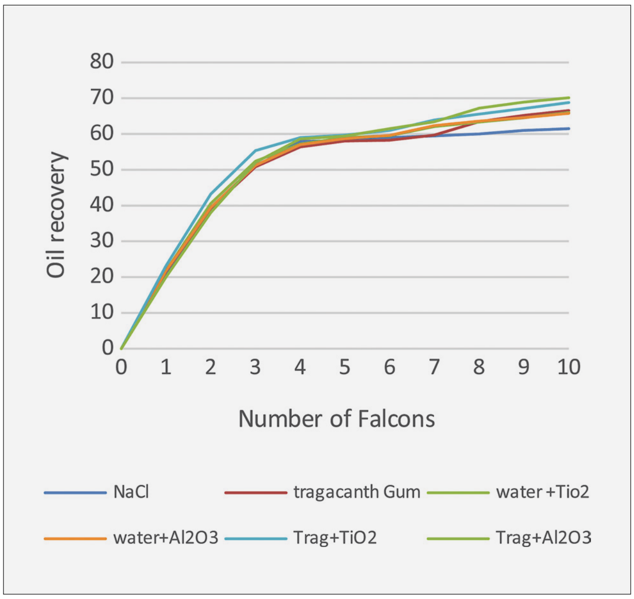

Figure 1: Comparison of oil recovery by polymeric and nanofluids at 25°C

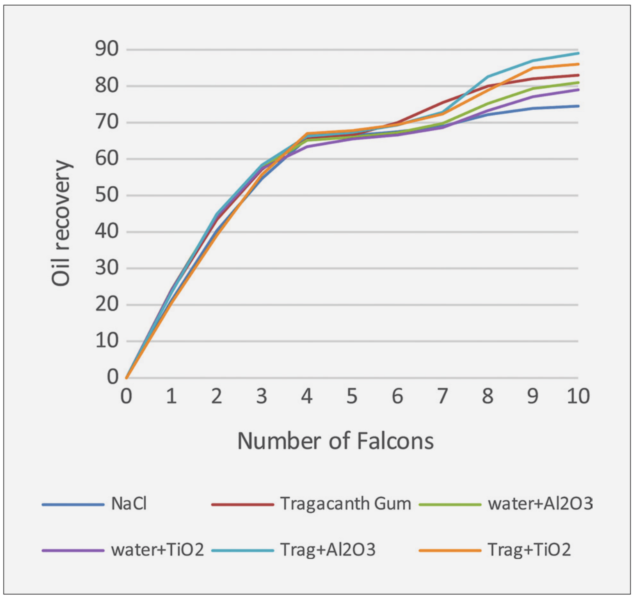

Figure 2: Comparison of oil recovery by polymeric and nanofluids at 50°C

Where:

ν – injection velocity (m/s),
σ – interfacial tension (IFT) (N/m),
θ – contact angle (degree),
μ – dynamic viscosity of the injection fluid (Pa·s).

To evaluate the capillary number improvement by nanofluids, all its parameters must be analysed, better recovery will be achieved if the capillary number is greater. To reach this goal based on the equation above, we need to increase the viscous force by adding a polymer, and decrease the capillary force by adding nanoparticles to our solution.

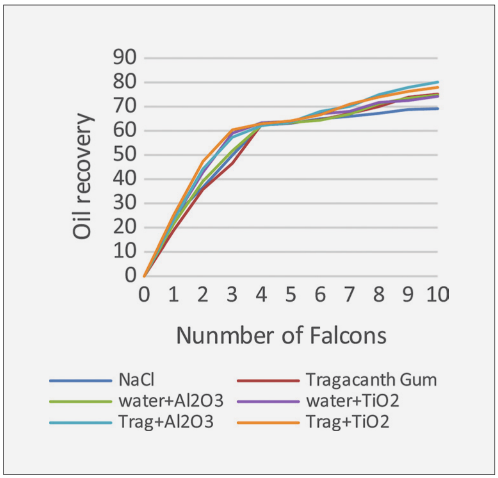

Figure 3: Comparison of oil recovery by polymeric and nanofluids at 75°C

Table 4: Comparison of tertiary recovery by polymeric and nanofluids at different temperatures

Test. No	Solution	Temperature °C	Tertiary Recovery (%)
1	Tragacanth	25	8.57
2	Water + TiO_2		7.01
3	Water + Al_2O_3		6.98
4	Tragacanth + TiO_2		10.68
5	Tragacanth + Al_2O_3		9.08
6	Tragacanth	50	11.86
7	Water + TiO_2		11.45
8	Water + Al_2O_3		10.3
9	Tragacanth + TiO_2		16.57
10	Tragacanth + Al_2O_3		13.58
11	Tragacanth	75	16.1
12	Water + TiO_2		15.3
13	Water + Al_2O_3		13.5
14	Tragacanth + TiO_2		22.6
15	Tragacanth + Al_2O_3		18.2

3.4. Viscous force

The above equation specifies that the viscous force is influenced by two parameters, denoted as μ and v. As demonstrated in **Figure 4, Figure 5** and **Figure 6**, the viscosity of each fluid was determined at temperatures of 25, 50, and 75°C. Fluids displayed Newtonian behaviour at certain temperatures. It can be suggested that the efficiency of oil production by fluids with viscosities exceeding those of saline water would be comparable to or greater than that of saline water. The present investigation places an assumption that the enhanced oil recovery (EOR) process is not significantly impacted by the viscosity of the nanofluid free of polymers at a temperature of 25°C. However, it is seen that the viscosity of polymer-based nanofluids has a substantial impact on both

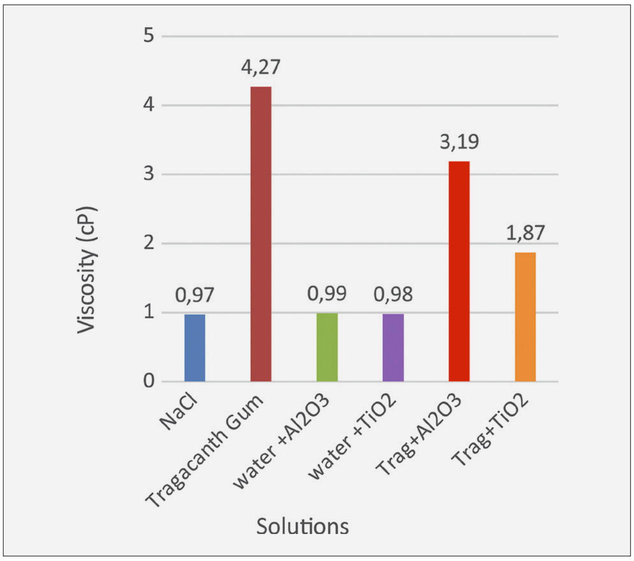

Figure 4: Fluid viscosity at 25°C

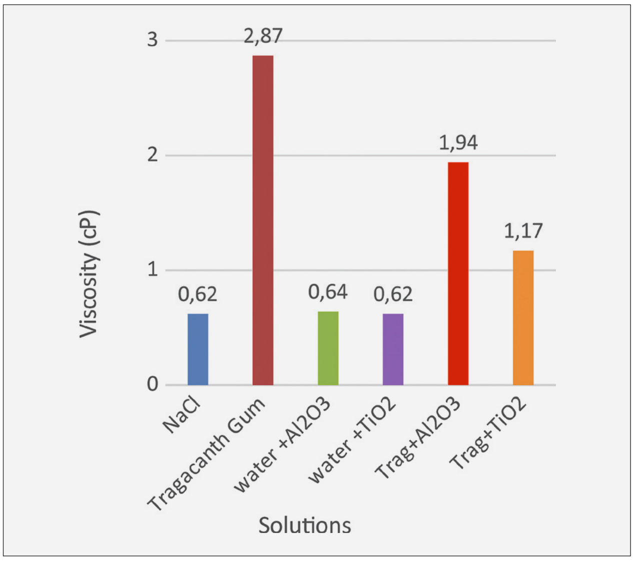

Figure 5: Fluid viscosity at 50°C

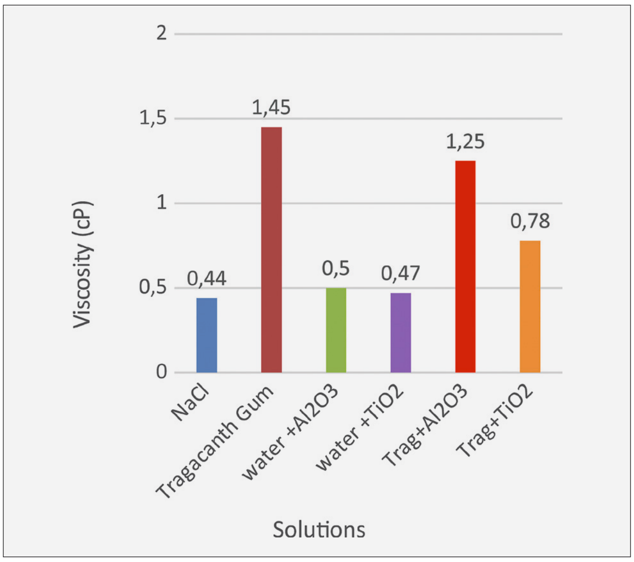

Figure 6: Fluid viscosity at 75°C

the EOR and capillary number, hence yielding a favourable outcome. As the temperature rises to 50°C and 75°C, there is a corresponding decrease in viscosity. However, it should be noted that the viscosity of polymer-free nanofluids is insufficient to significantly impact the enhanced oil recovery (EOR) process. On the other hand, the presence of polymer and polymer nanofluids shows high viscosity, which improves both the EOR and the capillary number.

3.5. Capillary force

The measurement of the contact angle θ may indicate the type of wetness displayed by the stone. Ordinary stones tend to have one of three types of wettability: hydrophilic (θ< 70°), moderate wettability (70°< θ <110°), or oleophilic (θ> 110°) (**Onyekonwo and Ogolo, 2010**). The contact angles of the oil droplet at various temperatures are given in **Table 5**.

Table 5: Comparison contact angle of oil droplet in the presence of polymeric and nanofluids at different temperatures

Test. No	Solution	Temperature (°C)	Contact angle (°)	Tertiary Recovery (%)
1	NaCl	25	60.51	3.28
2	Tragacanth		47.02	8.57
3	Water + Al_2O_3		30.99	7.01
4	Water + TiO_2		44.61	6.98
5	Tragacanth + Al_2O_3		30.31	10.68
6	Tragacanth + TiO_2		46.79	9.08
7	NaCl	50	60.01	6.02
8	Tragacanth		58.28	11.86
9	Water + Al_2O_3		76.18	11.45
10	Water + TiO_2		59.21	10.3
11	Tragacanth + Al_2O_3		62.31	16.57
12	Tragacanth + TiO_2		46.76	13.85
13	NaCl	75	46.21	8
14	Tragacanth		60.47	16.41
15	Water + Al_2O_3		44.21	15.3
16	Water + TiO_2		58.35	13.5
17	Tragacanth + Al_2O_3		43.47	22.6
18	Tragacanth + TiO_2		45.29	18.2

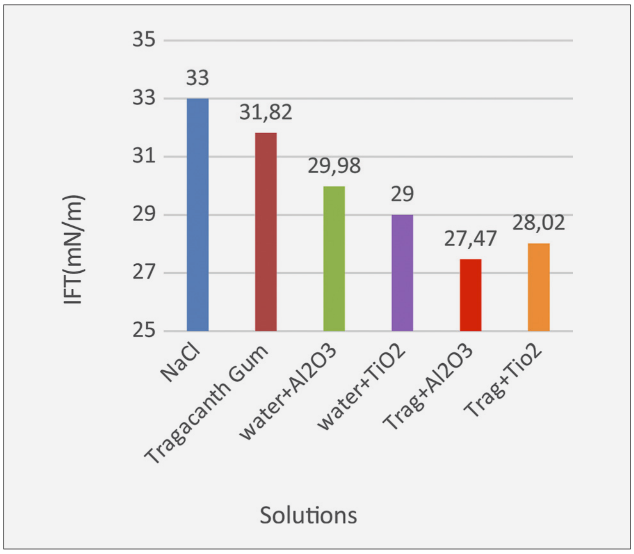

Figure 7: Quantitative amounts of interfacial tension at 25°C

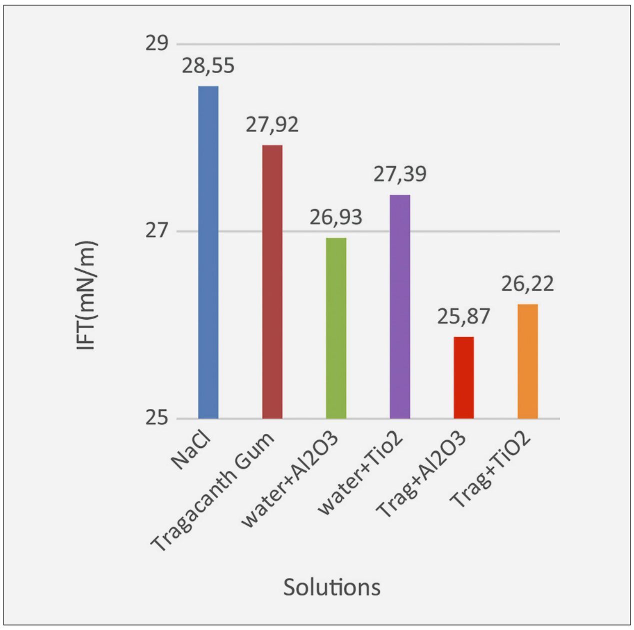

Figure 8: Quantitative amounts of interfacial tension at 50°C

3.6. Influence of nanofluids and polymers on interfacial tension

Interfacial tension performs a key part in the enhanced oil recovery (EOR) process when utilizing nanoparticles and polymers. Minimizing it is crucial in oil recovery, and an increasing amount of research suggests the use of nanoparticles to decrease interfacial tension would enhance the effectiveness of enhanced oil recovery (EOR) techniques. The cited references include **Onyekonwu and Ogolo (2010), Ogolo et al. (2012), Hendraningrat et al. (2014), Hendraningrat and Torsæter (2014), Joonaki and Ghanaatian (2014), Anganaei et al. (2014), Bayat et al. (2014), Mohajeri et al. (2015), Jafarnezhad (2016), Nowrouzi et al. (2019), and Zargar et al. (2020)**. The deformation of confined oil droplets is facilitated by the reduction of interfacial tension, so enabling them to pass through the pores with less difficulty. The present investigation determined that interfacial tension appeared as the most influential compo-

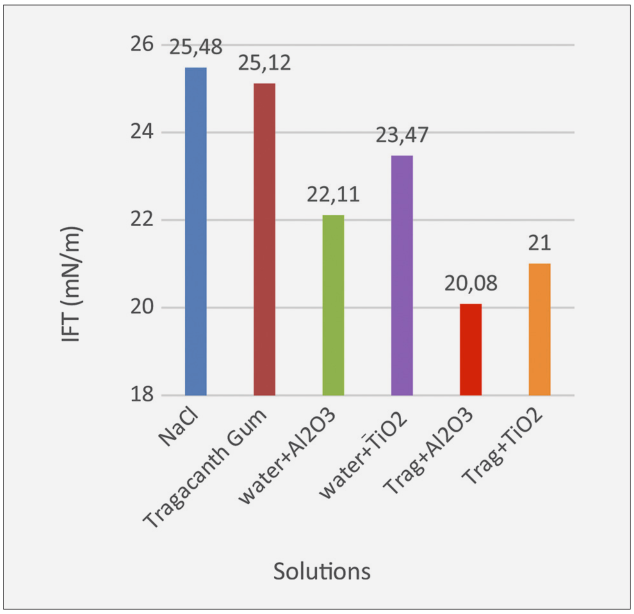

Figure 9: Quantitative amounts of interfacial tension at 50°C

nent for enhancing recovery. **Figure 7, Figure 8** and **Figure 9** illustrate the observed impacts of various additives on interfacial tension.

4. Conclusions

This study examined the impact of polymer and nanoparticles on calcareous grain porous media at temperatures of 25, 50, and 75°C. The concentration used was 50mg/l ml. The outcomes are as follows:

- The experimental investigation involved the utilization of various combinations of tragacanth, TiO_2, and Al_2O_3 in conjunction with water for oil recovery purposes. The experiments were conducted at a controlled temperature of 25°C. Based on the obtained results, it was determined that the combination of tragacanth + Al_2O_3 exhibited the highest recovery rate, specifically 10.68%. In comparison, the recovery rates for the combinations of tragacanth, water + TiO_2, water + Al_2O_3, and tragacanth + TiO_2 were found to be 8.57%, 6.98%, 7.01%, and 9.08% respectively, indicating their respective recycling efficiencies.
- The experimental results indicate that the addition of tragacanth + Al_2O_3 at a temperature of 50°C resulted in the highest recovery rate of 16.57%. In comparison, tragacanth, water + TiO_2, water + Al_2O_3, and tragacanth + TiO_2 exhibited recycling rates of 11.86%, 10.3%, 11.45%, and 13.85% respectively.
- By subjecting the samples to a temperature of 75°C, it was shown that tragacanth + Al_2O_3 with a recycling rate of 22.6% and tragacanth, water + TiO_2, water + Al_2O_3, and tragacanth + TiO_2 showed recovery rates of 16.41%, 13.5%, 15.3%, and 18.2% correspondingly.
- At a temperature of 25°C, the combination of tragacanth and Al_2O_3 exhibits the highest potential as a means to decrease interfacial tension. A drop of 16.75% in interfacial tension was observed.
- At a temperature of 50°C, the combination of tragacanth and Al_2O_3 has the most favourable characteristics for reducing interfacial tension. A decrease of 9.38% in interfacial tension was observed.
- Tragacanth + Al_2O_3 exhibits the most favourable characteristics for mitigating interfacial tension when subjected to a temperature of 75°C. A drop of 18.3% in interfacial tension was observed.
- Tragacanth has the highest viscosity among substances tested at a temperature of 25°C, with a measured value of 4.27 centipoise (cP).
- Tragacanth has the maximum viscosity among substances tested at a temperature of 50°C, with a recorded value of 2.87cP.
- Tragacanth exhibits the highest viscosity at a temperature of 75°C, with a measured value of 1.45 centipoise (cP).

According to the obtained results, it is appropriate to say that the solution of tragacanth + Al_2O_3 has shown the best performance in increasing EOR and decreasing the interfacial tension in sand-pack flooding.

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SAŽETAK

Eksperimentalna procjena utjecaja nanočestica TiO_2 i Al_2O_3 i gume tragakant na povećanje iscrpka nafte u vapnenačkim ležištima

Kemijske metode povećanja iscrpka nafte (engl. *Chemical Enhanced Oil Recovery*, CEOR) privlače veliku globalnu pozornost u svjetlu znatnoga smanjenja raspoloživih naftnih rezervi. Navedene metode koriste se za povećanje iscrpka nafte iz različitih vrsta ležišta, pri čemu se njima utječe na kritične parametre kao što su pokretljivost zarobljene nafte unutar pornoga prostora ležišne stijene, međufazna napetost (engl. *interfacial tension*, IFT) između vode i nafte, močivost i ponašanje širenja kemijskih otopina na stijeni površine. Nanočestice (eng. *nanoparticles*, NP) predstavljaju nove agense u CEOR procesima nudeći obećavajuće rezultate zahvaljujući poboljšanoj površinskoj aktivnosti na granici faza sirova nafta – slojna voda – stijena i njihovu utjecaju na sile razdvajanja različitih faza. Uvođenje polimera u nanofluide poboljšava disperzijsku stabilnost NP-ova, proširujući njihovu sposobnost za promjenu močivosti i postizanje bržih ravnotežnih stanja u usporedbi s nanofluidima bez polimera. U ovome su radu opisana eksperimentalna istraživanja povećanja iscrpka nafte koja su uključivala primjenu različitih kombinacija gume tragakant, TiO_2 i Al_2O_3 s vodom na različitim temperaturama. Dobiveni rezultati pokazuju da se najveći iscrpak nafte od 16,57 % dobiva primjenom gume tragakant u kombinaciji s Al_2O_3 pri temperaturi od 50 °C. Najveće smanjenje međufazne napetosti zabilježeno je na 25 °C (16,75 %), dok je veće smanjenje međufazne napetosti (9,38 %) uočeno na 50 °C u odnosu na 75 °C (18,3 %). Među ispitivanim tvarima na 25 °C guma tragakant pokazala je najveću viskoznost, s izmjerenom vrijednošću od 4,27 cP, dok su smanjenja od 2,87 cP i 1,45 cP opažena na 50 °C odnosno 75 °C. Prema dobivenim rezultatima moguće je zaključiti da je otopina gume tragakant i Al_2O_3 pokazala najbolju učinkovitost u povećanju EOR-a i smanjenju međufazne napetosti prilikom ispitivanja na uzorku nabijenoga pijeska (engl. *sand-pack flooding*).

Ključne riječi:
nanočestice, polimeri, povećanje iscrpka nafte (EOR), vapnenac, ležišta

Author’s contribution

Sobhan Anvari (1) (PhD Student, Petroleum Engineer) performed the laboratory test and presented the results. **Zoltan Turzo (2)** (Assoc. Prof. Dr., Petroleum Engineer) provided the analysis of the results.