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Green Energize: Unleashing a Samba Biopolymer for Enhanced Oil Recovery

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Abstract

Background: Several challenges exist in obtaining an optimal biopolymer material that is cost-effective, resilient under harsh conditions, and environmentally friendly. Currently, only xanthan and Schizophyllan biomaterials have been applied at a field scale. This study introduces a novel Samba biopolymer material for Enhanced Oil Recovery (EOR), which is economically viable, readily available, and stable.

Methods: Experimental tests were conducted to evaluate the Samba biopolymer. Infrared (IR), X-ray diffraction (XRD), and X-ray fluorescence (XRF) analyses were performed on the seeds before extracting the biopolymer solution. Subsequently, rheology, compatibility, membrane tests, particle size distribution, and filtration tests were conducted. A core flooding test was also carried out to measure the biopolymer's oil displacement efficacy; however, this test was performed for only one concentration scenario to serve as a preliminary evaluation of the polymer's performance under reservoir-like conditions.

Significant Findings: The extraction of the biopolymer solution from organic seeds was successful, with thermal extraction being sensitive to heating time and temperature. The Samba biopolymer enhanced water viscosity, recommended for reservoir conditions. Different concentrations revealed variations in rheological and filtration properties due to distinct morphology and surface functionality. Under optimal conditions, a remarkable 74% maximum oil recovery was achieved, demonstrating the biopolymer's potential as a promising EOR agent.

Keywords:
Samba Biopolymer, Enhanced Oil Recovery, Biopolymer Rheology, Core Flooding

1. Introduction

Enhanced Oil Recovery (EOR) encompasses any reservoir process that introduces external fluids or energy from outside the reservoir to increase oil recovery. EOR comprises various methods, each with its specific considerations for implementation. One such method is Chemical EOR (CEOR), which can be categorized into several approaches: Surfactant, Alkaline, Nanoparticles, and Polymer EOR (**Samba et al., 2019; Elsharafi and Samba et al., 2018; Massoud et al., 2022; Green et al., 1998**).

Polymer EOR is widely recognized as one of the most significant contributors to enhanced oil recovery (EOR) due to its mechanism and cost-effectiveness in comparison to other methods (**Jabbar et al., 2017**). The primary aim of polymer injection is to increase the mobility ratio and delay breakthrough. Its efficacy in delaying breakthrough has been successfully demonstrated in various oil fields globally. A survey conducted in 2016 documented over 865 polymer flooding projects worldwide (**Guo 2017**). As per available paper reports, no polymer project has encountered complete failure. While certain papers have highlighted challenges during polymer injections, most of these issues have been resolved through rigorous problem-analysing. Nevertheless, ongoing studies face the challenge of identifying an optimal polymer material that is not only cost-effective, but also capable of withstanding harsh conditions while remaining environmentally friendly. Consequently, researchers have increasingly focused on biopolymer materials. Presently, biopolymers have gained prominence as a subject of interest in discovering materials that address the previously mentioned challenges. To date, only Xantant and Schizophyllan biomaterials have been applied at a field scale (**Schnepp 2013**). Some companies have introduced polymer materials with undisclosed compositions, potentially related to biopolymer materi-

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als. Moreover, certain materials have solely undergone laboratory-scale testing and are yet to be tested in the field. Simultaneously, several biopolymer materials require further examination and testing to ascertain their potential for enhancing oil recovery (**Hamidi et al., 2021**). While materials such as okra, cassava starch, Detarium microcarpum, exudate gum, and aqueous beans have undergone laboratory testing, they have not been applied in field applications. Nevertheless, not every material exhibiting highly viscous solutions qualifies as a viable EOR agent. Before field application, various mechanisms including solution rheology, viscosity changes over time and temperature, degradation, crystallization, adsorption, and plugging should be thoroughly assessed. A recent study in 2019 focused on biopolymer EOR using guar and arabic gum. The research involved core flooding, comparing chemical polymer, water, biopolymer, and a chemical biopolymer slug. The biopolymer injection displayed superior performance compared to other scenarios (**Hassan et al., 2019**).

Similarly, in 2020, another study explored the use of green grass jelly leaves and seaweed as biopolymer materials, comparing them with xanthan gum and other chemical polymers. The results indicated comparable performance between biopolymers and chemical polymers. Additionally, it was observed that polymer viscosity decreased with an increase in salinity. However, seaweed and grass jelly exhibited good compatibility across different shear rates (**Huljannah et al., 2020**).

During the same year, research on tragacanth gum was published, testing it as a biopolymer-surfactant solution alongside smart water. The findings showed that fluid viscosity in Carboxymethyl Cellulose (CMC) reached an optimal value, resulting in a 21.4% increase in Recovery Factor (RF) (**Nowrouzi et al., 2020**).

Another study in 2020, focused on hybrid nanoparticles (NPs) with biopolymers. It aimed to characterize xanthan, arabic, and guar gum combined with copper, silica, and alumina NPs. The study analyzed the rheology of these biopolymers under various conditions such as salinity, temperature, NPs weight, and biopolymer concentrations. The materials were recommended for potential use in the EOR section (**Sowunmi et al., 2020**).

Challenges in finding an ideal biopolymer material persisted into 2021. A study introduced a novel biopolymer-surfactant extracted from the Acanthophyllum plant. Extraction procedures were conducted and characterized using Thermo-Gravimetric-Analysis (TGA), Fourier-Transform-Infrared-spectroscopy (FTIR), and Proton-Nuclear-Magnetic-Resonance-spectroscopy (H-NMR). The study also calculated surface tension (ST) and interfacial tension (IFT) between water-air and water-oil, resulting in 92.6% and 61.6%, respectively. This biopolymer-surfactant showed the capability to alter carbonate rock wettability from oil-wet to water-wet. The adsorption test introduced the Freundlich isotherm model as the best-fitted scenario (**Mehrabianfar et al., 2021**).

Though many plants offer effective biopolymer materials, the challenge lies in the complexity of extraction and preparation for high quantity implementation. Concurrently in 2021, research on grafted and dispersed nanoparticles of graphene combined with arabic gum biopolymer solution in harsh conditions was published. The results compared functional groups of graphene nano-platelets synthesized through grafting and mixing. This method aimed to enhance NPS stability in high-pressure and high-temperature conditions, holding promise as CEOR agents (**Hamdi et al., 2022**).

In recent times, the mixing of biomaterials with chemical substances to enhance their performance has emerged as an intriguing topic. A study titled ‹Application of Biopolymer and NPs in EOR at Reservoir Conditions› explored three distinct biopolymers derived from local materials in Nigeria and Malaysia. Chemical additives were employed in synthesizing the biopolymer NPs, demonstrating their resilience under harsh conditions (**Maghzi et al., 2014**).

Building upon the aforementioned studies, various biopolymers have been assessed for their potential to augment oil recovery at a laboratory scale. Concurrently, numerous biomaterials warrant further examination to ascertain their suitability in enhancing oil recovery. Consequently, this study proposed a novel biomaterial (Samba Biopolymer) which is readily available in North Africa, known for its significant capability in augmenting water viscosity while maintaining long-term stability, has garnered attention. Surprisingly, this material has not yet been utilized in the EOR sector. Consequently, this material was selected for testing its efficacy in EOR processes.

2. Procedures

2.1. Biopolymer solution preparation

The Samba biomaterial was extracted from organic seeds utilizing the pyrolysis method in the presence of water. Initially, the seeds underwent a thorough washing with fresh water and were subsequently immersed in a synthesis formation water solution with varying concentrations, as outlined in **Table 1**. All prepared concentrations underwent the same procedural steps to ensure consistent extraction protocols. The solution was then heated for 2 hours at 80°C while maintaining the pres-

Table 1. Biopolymer solution concentrations that were prepared in this study

Concentration	Water salinity, ppm	Preparation time, hr	Temperature, °C
2%	2500	2	80
4%	2500	2	80
6%	2500	2	80
8%	2500	2	80

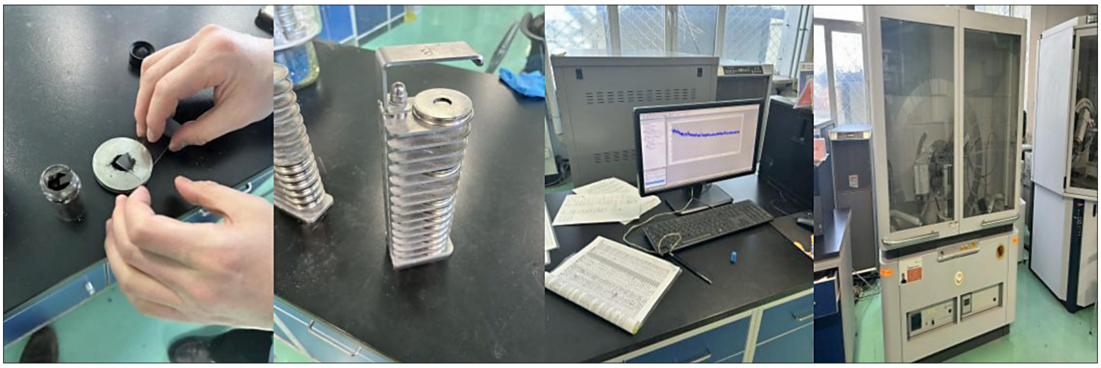

Figure 1. Devices and apparatus that have used to characterize the implemented materials

ence of 0.1% citric acid to prevent crystallization, a phenomenon that could occur with temperature variations. Additionally, 0.1% sodium benzoate was included during the extraction process to inhibit bacterial activation.

2.2. Biopolymer Solution Characterizations

To analyze and characterize the material utilized in this study, various tests were conducted. The Infrared (IR) test was performed to examine the functional groups present. Meanwhile, X-Ray Fluorescence (XRF) analysis was employed to ascertain the precise percentage composition. Lastly, X-Ray Diffraction (XRD) was utilized to investigate particle diameter and shape. **Figure 1** illustrates the devices utilized to conduct these tests.

2.3. Particle Size Measurements

The test was conducted using a Zetasizer device, depicted in **Figure 2**. A rectangular-shaped core holder was employed for this procedure. The objective was to calculate the particle size in the solution of the biopolymer, aiming to verify the success of the mixing procedures and assess the consistency of particle size obtained.

Figure 2. Zetasizer device that has been used to measure particle size

2.4. Solution Stability Test

Various samples of the biopolymer solution at concentrations of 2%, 4%, 6%, and 8% were assessed for compatibility at a reservoir temperature. These solutions were initially maintained under standard conditions (room temperature) for 3 days, followed by an additional 4 days at 70°C.

2.5. Rheology Investigations

The solution's rheology was assessed by measuring the viscosity of the biopolymer at various concentrations, temperatures, and shear stresses. Viscosity calculations were performed to determine whether the solution exhibited characteristics of a Newtonian or non-Newtonian fluid. The Viscometer Brookfield, illustrated in **Figure 3**, was utilized for viscosity measurements, functioning based on the fluid's resistance to shear stress during spinning. Viscosity measurements at different concentrations were conducted at 2500 ppm with shear rate. Furthermore, this study investigated the impact of temperature on viscosity, particularly focusing on the influence of temperature variations on this novel biopolymer material.

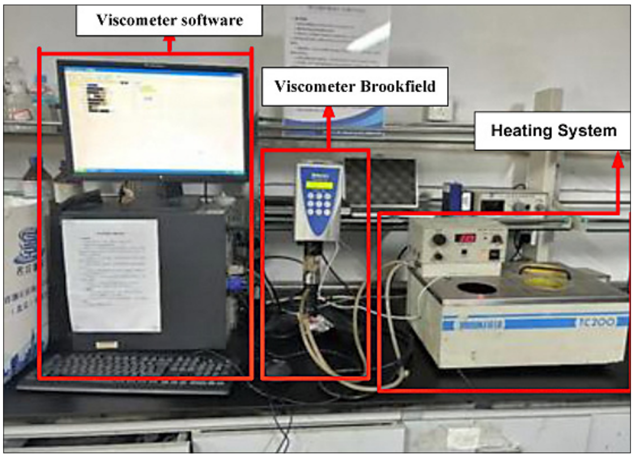

Figure 3. Viscometer Brookfield device that has been used to characterize the solutions

2.6. Membrane Test

A membrane test was conducted to assess the ability of the selected Biopolymer concentrations to penetrate pores without causing blockages. The filtration apparatus was prepared by placing a suitable pore size, which was a Millipore cellulose filter 5 µm in the filter holder. The suction valve was adjusted to 0.2 MPa. Subsequently, 500 ml of solution was filtered, and the cumulative time in seconds for each 100, 200, 400, and 500 ml was recorded. Visual monitoring of the filtered layer was conducted to detect any signs of potential plugging. Moreover, the assessment was based on the filtration ratio, calculated using the following equation:

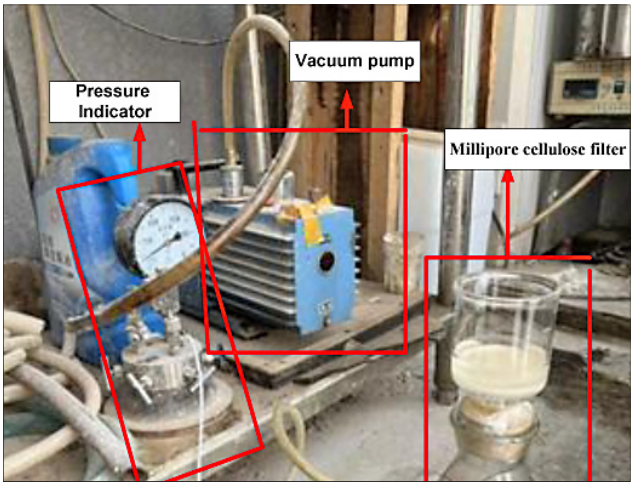

Figure 4. Membrane test apparatus

Figure 5. Experimental instruments

$$FR = \frac{(\mathbf{T}500 - \mathbf{T}400)}{(\mathbf{T}200 - \mathbf{T}100)} \tag{1}$$

Where: FR is the filter ratio. While, the T100, T200, T400, and T500 are the times when 100, 200, 400, and 500 ml.

2.7. Core Flooding

The objective of this experiment was to determine whether the Samba biopolymer could enhance oil recovery in sandstone reservoir rock. To achieve this, a core flooding test was designed for a single scenario, involving the sequential injection of formation water, followed by a 2% Samba biopolymer solution, and then a slug of formation water. This setup aimed to evaluate the polymer's effectiveness in improving displacement efficiency under controlled conditions. **Figures 5** and **6** illustrate the experimental setup and the core flooding system used to implement this scenario.

Initially, the dimensions and weight of the core were measured using a Vernier caliper and a sensitive balance. Subsequently, the core underwent vacuuming using a vacuum pump and was saturated with formation water.

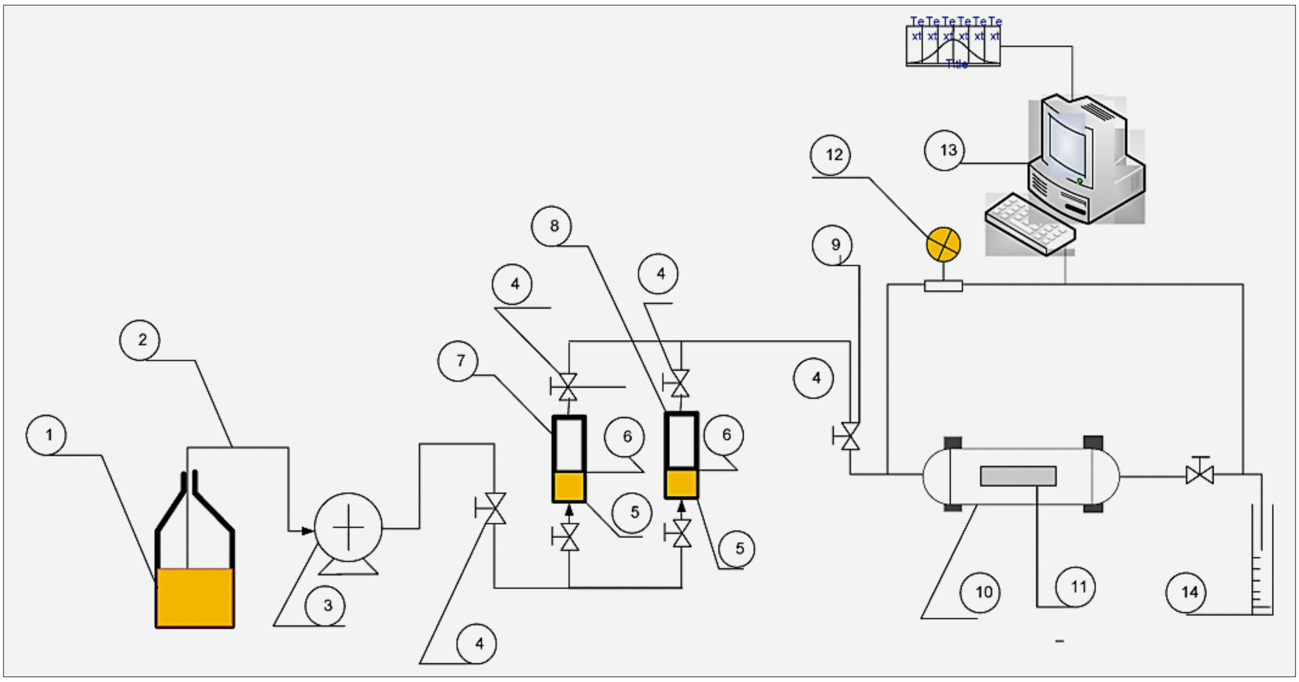

Figure 6. Core flooding apparatus: 1) Pump oil, 2) Injection pipeline, 3) Injection pump, 4) valve, 5) Pump oil in the fluid storage, 6) piston inside the fluid storage, 7) formation water, 8) Biopolymer solution, 9) Bypass valve, 10) Core holder, 11) Core plug inside the core holder, 12) pressure gauge and regulator, 13) computer, 14) accumulator.

After achieving saturation, crude oil was injected into the cores to establish the initial oil saturation in the presence of connate water saturation.

The saturated core was then placed within the core holder and subjected to an overburden pressure of 4 MPa, ensuring it exceeded the injection pressure to facilitate fluid passage through the core. The core flooding system operated through several stages. The injection pump introduced oil into the fluid storage to push the piston situated inside the storage. This piston, in turn, propelled the injected fluid stored within, acting as a separator between the two fluids. The storages were filled with formation water and biopolymer fluid and equipped with inlet and outlet valves to regulate fluid flow. The effluent from the core during the injection process was collected in the accumulator. It's crucial to ensure the absence of gas in the lines before initiating the injection process.

Figure 7. Samba biopolymer solution that has been prepared

3. Results

3.1. Biopolymer Solution

Samba biopolymer solution was successfully extracted from organic seeds, the successful extraction of the biopolymer solution revealed the sensitivity of the thermal extraction process to various parameters, including heating time, ambient temperature, and several other factors. Despite this sensitivity, all concentrations extracted exhibited a consistent pattern of viscosity progression based on concentration levels. This consistency indicated the successful extraction across all scenarios, affirming the efficacy of the established procedures.

3.2. IR Analysis

The infrared spectrum of the compound exhibits notable peaks, notably a broad peak at 3401 cm^{-1} attributed to the presence of the OH group. Additionally, the spectrum displays a peak at 2926 cm^{-1}, indicating the presence of the υ C-H Aliphatic group. Another distinctive peak at 1437 cm^{-1} is likely associated with the υ C=C Aromatic group. Furthermore, two additional peaks, as depicted in **Figure 8**, correspond to the υ C-OH and υ C-H (Aromatic) groups.

3.3. XRF Analysis

Table 2 presents the chemical composition of the Biomaterial, revealing that the sample is primarily composed of potassium (K) at 28.7%, followed by calcium (Ca) at 15.9%, phosphorus (P) at 9.30%, and aluminium (Al) at 8.41%. Additionally, trace amounts of other elements such as Si, Sn, Sb, Cl, etc., were detected in smaller quantities.

3.4. XRD Analysis

Figure 9 displays the X-ray diffraction (XRD) analysis of organic seeds. The XRD diagram notably depicts the

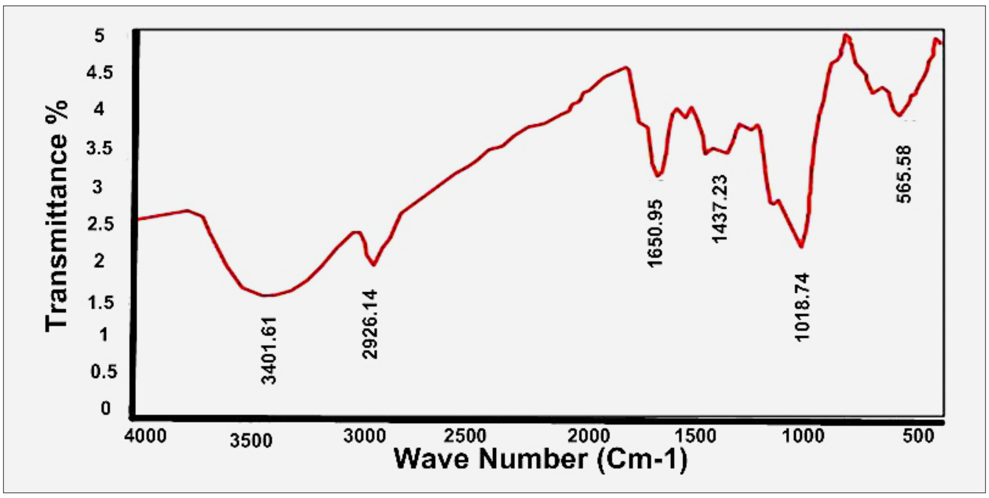

Figure 8. FTIR analysis for the Biopolymer material

Table 2. Chemical constituents for the Biopolymer material

Compounds	Concentration %
K	28.7
Ca	15.9
P	9.3
AL	8.41
Si	7.99
Sn	6.36
Sb	4.7
Cl	2.61
Fe	1.77
Ni	1.68
Zn	0.89
Mn	0.805
Cu	0.56
Co	0.282
Rb	0.0711
Ln	<0.0001

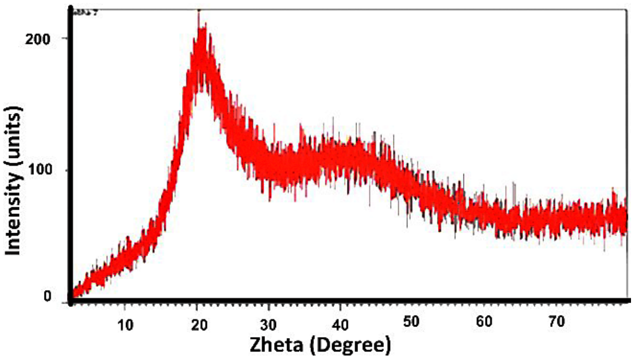

Figure 9. XRD Analysis for the Samba Biopolymer material

amorphous nature of the biopolymer seeds. Interestingly, no peaks were observed in the results, indicating the absence of distinct peaks in the diffraction pattern of organic seeds and thus revealing its amorphous character.

3.5. Compatibility Analysis

Four different concentrations of the biopolymer were evaluated for compatibility at a salinity level of 2500 ppm. The samples were stored at room temperature for 3 days and at 70°C for 4 days. Throughout this duration, no particles precipitation were observed. As depicted in **Figure 10**, the samples remained clear on the 7th day. These findings affirm the biopolymer's effectiveness in maintaining stability and compatibility with formation water, particularly at elevated reservoir temperatures.

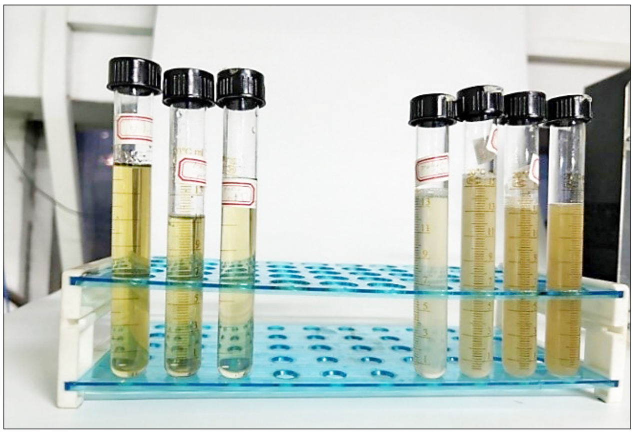
Figure 10. Compatibility tests for Samba biopolymer solutions after 7 days at (2, 4, 6 and 8%)

3.6. Biopolymer Rheology

Figure 11 illustrates the relationship between the biopolymer's behaviour concerning shear stress and shear rate at 25°C. All concentrations exhibited non-Newtonian behaviour attributed to polymer particles and biopolymer properties. The shear stress of various concentrations remained stable with increasing shear rate due to stable particle dispersion, wall slip motion in the polymer solution, and the movements of the shear plates.

In **Figure 12**, changes in shear rate were measured to determine Samba biopolymer viscosity, ranging from 2.45 s^{-1} to 244 s^{-1}. At 2% concentration, the viscosity showed minimal variation with increasing shear rate. However, at 4%, 6%, and 8% concentrations, higher shear rates led to decreased viscosity of the Samba biopolymer. Visual observations during extraction indicated that higher concentrations resulted in gel-like solutions. Consequently, there were notable differences in viscosity among concentrations, notably between 2% and higher concentrations. This phenomenon can be attributed to the relationship between biopolymer chain length, molecular weight, and viscosity. Higher molecular weight and polymer concentration led to increased viscosity through longer or stronger polymer chains. Conversely, concentrations at 4%, 6%, and 8% exhibited longer chains and higher molecular weight but were weaker resistance to shear rate degradation, resulting in higher viscosity that was susceptible to degradation by shear rate. The 2% concentration displayed lower viscosity but higher resistance to shear rate degradation.

In **Figure 13**, polymer viscosity measurements were taken at a shear rate of 7.34 s^{-1} under various temperatures. These conditions mimicked reservoir conditions. The viscosity reduction at 4%, 6%, and 8% concentrations was significant but still viable for facilitating oil flow within the reservoir, potentially improving the mobility ratio. However, the 2% concentration showed minimal viscosity reduction at reservoir temperatures, but the viscosity remained relatively low. This result still falls within acceptable safety margins for polymer injection, as it maintains a viscosity higher than that of water. A sharper viscosity decline was observed between 45°C and 50°C for the higher concentrations, which is typical

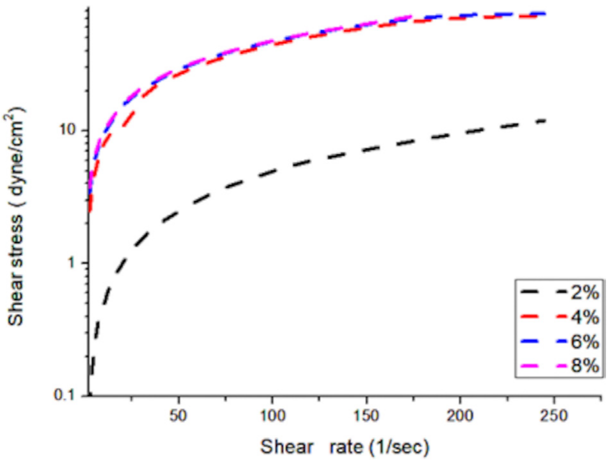

Figure 11. Shear stress as a function of shear rate Samba biopolymer solution samples at 25°C

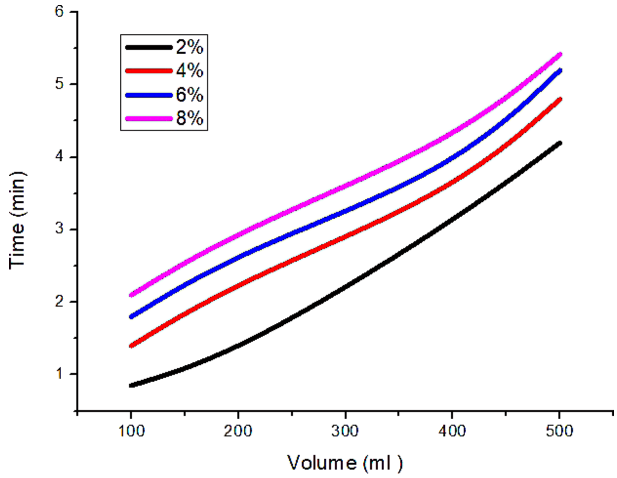

Figure 14. Filtration test results

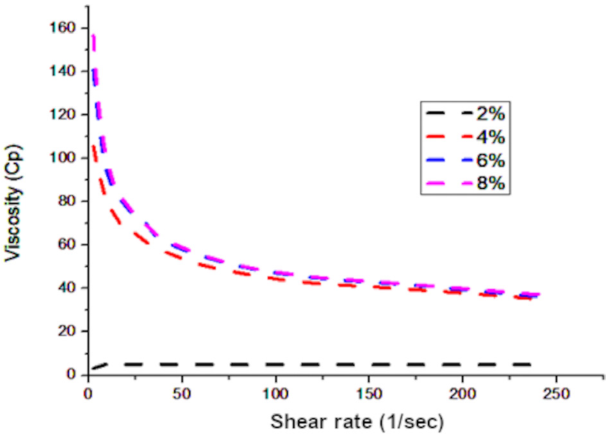

Figure 12. Samba biopolymer viscosity for variation Shear Rate

Figure 15. Gel formed at 8%

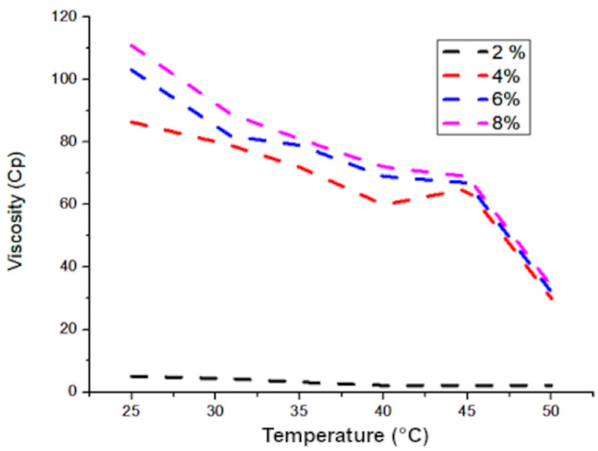

Figure 13. Samba biopolymer viscosity at variation temperature

Table 3. Summary of filtration ratio measurement

Concentration	Filtration ratio	Polymer remaining in the filter paper
2%	2.34	No
4%	1.08	No
6%	1.55	No
8%	1.35	Small amount of polymer gel remaining

for polymer solutions as thermal energy disrupts molecular interactions. Nonetheless, the viscosity levels remained sufficiently high to ensure effective performance.

3.7. Filtration Test Results

The filtration test was conducted to assess the potential for polymer plugging within the pores. The results demonstrate the passage of Samba biopolymer molecule particles through the pore throat size, accounting for interactions observed in real field conditions such as electrostatic forces, surface roughness, and rock wettability. As depicted in **Figure 14**, the relationship between passed polymer volume and time indicates that an increase in concentration correlates with extended passage time across all scenarios. Notably, it appears that the pore size accommodates all concentrations adequately. At an 8% concentration, as shown in **Figure 15**, a small amount of gel on the filter screen. This occurrence of unfiltered polymer is attributed to the high molecular weight characteristic of high concentrations.

3.8. Particle Sizes

Figure 16 displays the particle size distribution of the Samba biopolymer solution, measured using a Zetasizer

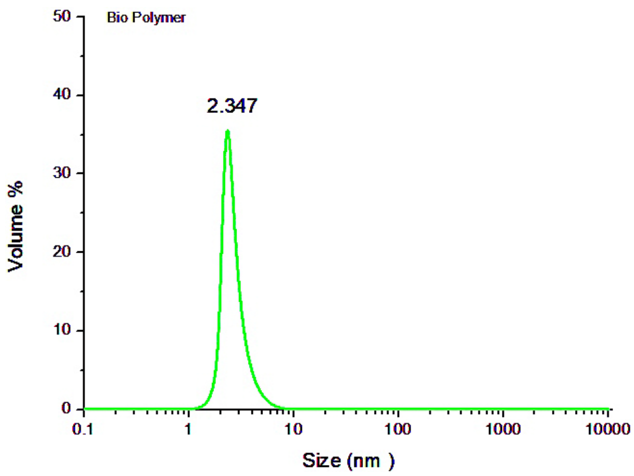

Figure 16. Particle size for Samba biopolymer solution

device employing a helium-neon laser light and integrated analysis software. The measurements were conducted at a temperature of 25°C, with the solution's salinity set at 2500 ppm. The analysis revealed a biopolymer size of 2.75 nm in the solution, demonstrating the success of the mixing procedures and the uniformity achieved in solution sizes. Furthermore, the results indicated that the particles did not exhibit swell-up when mixed with formation water.

3.9. Core Flooding

Figure 17 illustrates the recovery factor and pressure versus pore volume. The results indicate that breakthrough occurred after 0.92 pore volumes (Pv) of injected fluid. Following the breakthrough, oil continuously emerged until the recovery factor reached 60%, demonstrating a cessation in oil output. Subsequently, the introduction of Samba Biopolymer into the cores led to a substantial increase in pressure compared to the formation of water flooding.

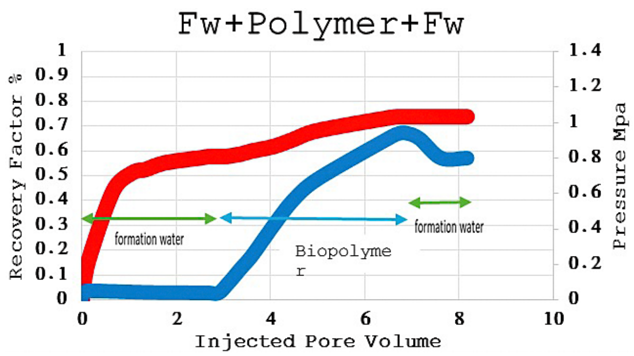

Figure 17. Oil recovery performance, pressure differential vs injected pore volume for formation/ biopolymer / formation water

Upon the Samba Biopolymer injection, oil extraction resumed, elevating the recovery factor to 74% before halting again. Subsequent formation water flooding post-biopolymer injection did not yield any additional oil production. These findings suggest that while the biopolymer material was effective in displacing the recovery factor, it did not alter the residual oil saturation.

Following the collection of produced oil, samples were extracted from the produced fluid and examined under a microscopic device to analyze the produced droplets for the presence of biopolymer. A comparison was made with samples from the water scenario.

In the formation water scenario (see **Figure 18a**), the oil droplets were observed to disperse naturally without the assistance of any external agent. Conversely, in the Biopolymer scenario (see **Figure 18b**), it was observed that the biopolymer material facilitated the dispersion of oil droplets into smaller ones, indicating the material's capability to displace oil droplets effectively.

4. Conclusions

1) The conducted tests confirm that the Samba biopolymer material is well-suited for elevating water viscosity under high temperatures and in saline conditions.
2) Results from core flooding tests demonstrate the effectiveness of the Samba biopolymer solution in increasing the recovery factor during extraction processes.
3) The high concentrations of the Samba biopolymer solution indicate its potential application as a gel material.
4) The ease of extraction in comparison to other published materials serves as a key motivation for the utilization of this material.

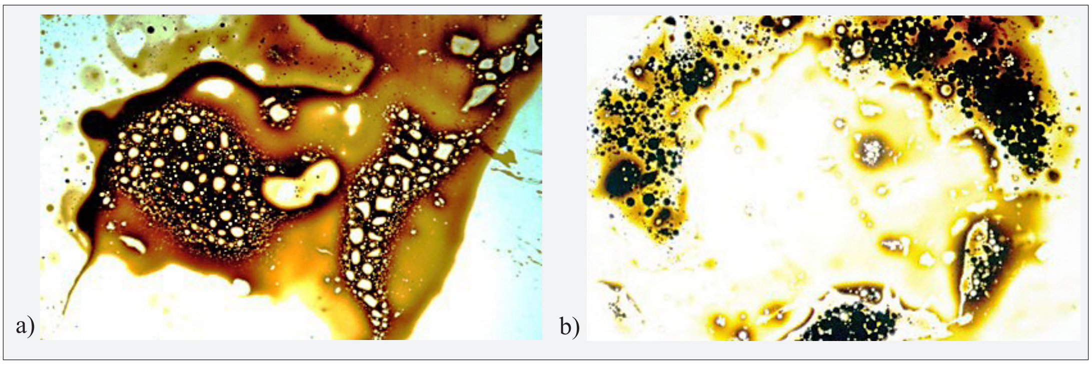

Figure 18. An optical microscope image for Microemulsion during a) formation water and b) Samba biopolymer

5) Initial investigations into the Samba biopolymer material have shown promise. Further development, including transforming the material into powder form and exploring additives to enhance particle cohesion, presents an opportunity for specialized companies in this field.

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SAŽETAK

Zelena energija: korištenje polimera samba za povećanje iscrpka nafte

Trenutačno postoji nekoliko izazova u dobivanju optimalnoga biopolimernog materijala koji je ekonomski isplativ, otporan na teške uvjete i ekološki prihvatljiv. Sada se u praksi primjenjuju samo biomaterijali ksantan i shizofilan. Ovim istraživanjem predstavlja se novi biopolimerni materijal za povećanje iscrpka nafte (engl. *enhanced Oil Recovery*, EOR) samba, koji je ekonomski isplativ, lako dostupan i stabilan.
Provedena su eksperimentalna ispitivanja biopolimera samba. Prije ekstrakcije otopine biopolimera napravljena je infracrvena spektroskopija (IR), rendgenska difrakcija (XRD) i rendgenska fluorescentna analiza (XRF) sjemenki. Nakon toga je napravljena analiza reoloških svojstava, raspodjele veličine čestica, kompatibilnosti te analiza filtracijskih svojstava. Također je proveden test utiskivanja u jezgru (engl. *core flooding*) kako bi se izmjerila učinkovitost istiskivanja nafte biopolimerom; međutim, ovaj test proveden je za samo jednu koncentraciju kako bi poslužio kao preliminarna procjena djelovanja polimera u ležišnim uvjetima.
Rezultati istraživanja pokazuju da je ekstrakcija otopine biopolimera iz organskoga sjemena bila uspješna, pri čemu je termička ekstrakcija bila osjetljiva na vrijeme i temperaturu zagrijavanja. Biopolimer samba povećao je viskoznost vode na vrijednost koja je potrebna za ležišne uvjete. Različite koncentracije otkrile su varijacije u reološkim i filtracijskim svojstvima zbog različite morfologije i funkcionalnosti površine. U optimalnim uvjetima postignut je iscrpak nafte od 74 %, što pokazuje potencijal biopolimera u EOR metodi.

Ključne riječi:
biopolimer samba, povećanje iscrpka nafte, reologija biopolimera, test utiskivanja u jezgru

Author's Contribution

Mohammed A. Samba, a member of the Petroleum Engineering staff at Sebha University and CEO of the Med Fezzan Research Center, was responsible for the material innovation, conceptualization, methodology design, original draft preparation, manuscript review and editing, as well as overall project administration. **Yiqiang Li**, Head of the Oil and Gas Laboratory at the China University of Petroleum, Beijing (CUPB), contributed through supervision, methodology development, manuscript review and editing, and data curation. **Zheyu Liu**, Assistant to the Head of the Oil and Gas Laboratory at CUPB, was involved in the provision of materials, laboratory facilities, and the execution of formal analysis. **Mahdi Almakki**, a member of the Chemical Sciences staff at Sebha University, supervised the preparation and characterization of biopolymers, carried out investigations, provided resources, and contributed to data curation. **Agus Arsad**, a staff member at the Institute for Oil and Gas (IFOG), Universiti Teknologi Malaysia, contributed to result visualization, data curation, and formal analysis.
All authors have read and agreed to the published version of the manuscript.