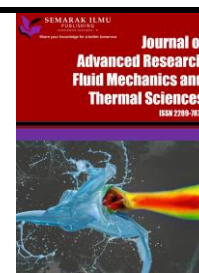




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Investigating the Effect of Okra Mucilage on Waxy Oil Flow in Pipeline

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ABSTRACT

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Wax deposition in the pipeline is one of the petroleum production system's most serious flow assurance issues. Wax deposition limits crude oil flow through the pipeline, resulting in unusual pressure conditions and an artificial barrier that reduces or stops production. This study discusses the effect of okra mucilage on preventing wax deposition in the waxy pipeline. Fourier Transform Infrared (FTIR) and X-ray diffraction analysis (XRD) analysis were done to characterize the okra mucilage as a wax inhibitor. An experimental flow loop system was fabricated to study the wax inhibition potential of okra mucilage. The wax deposition thickness was measured using the pressure drop method. After the experimental completion, the effectiveness was calculated. The FTIR result showed that sugar components (galactose, rhamnose, and galacturonic acid) found in the okra were the reason that made the okra slimy and slippery. This characteristic reduces the surface roughness, reducing wax deposition in the pipeline. Based on the result of the experiment, it was shown that using the diluted okra powder as wax inhibition is more effective than using okra mucilage. The correlated effectiveness of the result using diluted okra powder was approximately 10% more effective than using okra mucilage. Diluted okra powder gives 40.79% effectiveness; compared to okra mucilage, which was 30.75%. These results indicate that the diluted okra powder is a cost-efficient plant-based substitute inhibitor against wax inhibition in oil flow pipes and has good wax inhibition capabilities.

1. Introduction

One of the significant flow assurance problems in petroleum production systems is wax deposition in the pipeline. Wax accumulation can obstruct the passage of petroleum oil through a conduit, resulting in uncommon pressure conditions and a manufactured obstruction that reduces or stops production [1,2]. The problem comes when the temperature of the crude oil falls below the wax appearance temperature, causing crystallization in the crude oil [3]. This results in the paraffin molecules in petroleum oil precipitating and depositing on the cool pipe's wall, which affects the pipeline flow assurance problem in the pipeline system [4,5]. The thickness of the paraffinic wax can rapidly increase in the solid phase, clogging the pipeline. As a consequence, the flow-through

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diameter of the pipeline was lowered, resulting in inefficient production operations due to the decreased transportation capacity [6,7].

Numerous investigations have been done to determine the best methods for preventing or removing paraffin wax precipitation. The methods for preventing wax deposition can be divided into three categories. The categories are thermal, chemical, and mechanical. These methods can be combined in some instances [8-10]. These methods have disadvantages, and most are costly and toxic [11]. For example, Ragunathan, Wood [12] use palm oil additives as a wax inhibitor, while Akinyemi and Udonne [13] use plant seed oils to prevent wax deposition. Also, according to Gudala *et al.*, [14], minimizing viscosity via in situ operation or postproduction is critical to cut down on the expenses of surface operation. Triethanolamine (TEA), polyethylene-co-vinyl acetate (EVA), and similar polymers are currently used in products; however, they are both highly costly and unfriendly to the environment [15]. To solve this issue, scientists used vegetable oils and a triethanolamine-xylene mixture to prevent the wax formation and showed some improvement as a wax deposition inhibitor in waxy crude oil. However, plant seed oils need to be blended with another chemical for better efficiency of wax inhibitors [13,15]. But plant seed oils need to be blended with another chemical for better efficiency of wax inhibitors [13,16]. In this regard, there is a need to develop an environmentally friendly inhibitor with better inhibitor effectiveness.

Abelmoschus esculentus (synonym *hibiscus esculentus*), also known as okra. Okra is a member of the malvaceous family, and it has a slimy mucilage characteristic. Its mucilage is widely utilized in cooking and medicine [17,18]. Murtaza and Tariq [19], utilize the okra slurry as a fluid loss prevention component in drilling fluids, showing that it performs comparably to starch. This is because okra has less influence on rheological characteristics than starch. The filter cake of okra-based drilling fluid was thin. This was one of the examples of okra used in the petroleum industry. Besides that, Anwar *et al.*, [20] used okra seed oil for biodiesel production. The biodiesel was produced by methanol-induced transesterification using an alkali catalyst from okra seed oil. It was discovered that okra seed oil is a suitable feedstock for manufacturing biodiesel.

Therefore, Okra Mucilage was used as a wax inhibitor in this study. This is because its slimy characteristic can theoretically help enhance the pipeline's flow by minimizing the factor affecting the Wax deposition. The factor that was studied is surface roughness. The use of okra mucilage as a wax inhibitor has been studied using the Flow loop experiment. The wax deposition thickness was carefully observed and compared to get the effectiveness of using okra mucilage as a wax inhibitor in the pipeline. The experiment was repeated using diluted okra powder, and the results were compared to the study, which performs better as a wax inhibitor.

2. Methodology

2.1 Materials

Okra fruits and muslin cloth were purchased from a local store. Acetone with a purity of 89% was purchased from Merck and HCl (97%) was obtained from Fisher. In this study, Dulang light crude oil with an API value of 37.4° was used. All chemicals were used just as they were supplied, with no additional purification.

2.2 Extraction of Mucilage from Okra

The process of extracting the mucilage from okra was adapted from the method developed by de Alvarenga *et al.*, [21] and Zaharuddin [22]. The okra was sliced and blended with distilled water using a laboratory blender to extract mucilage from the okra. The okra was stored in a container at room

temperature for 12 hours to extract the mucilage. After soaking, the solid was separated from the okra mucilage with the muslin cloth. After extracting the okra mucilage, the okra powder needs to be prepared. The mucilage was precipitated using acetone with 3:1 of acetone and okra mucilage. Repeat this step until the precipitated okra mucilage turns white into color. Then, the precipitated mucilage was left inside the fume hood to slow the drying phase of the mucilage. After drying, the precipitate mucilage was powdered using a laboratory blender.

2.3 Characterization of Okra Mucilage

Fourier Transform Infrared (FTIR) and X-ray Diffraction Analysis (XRD) were used to characterize the okra mucilage for qualitative analysis. FTIR is an analytical method performed to determine the functional group of okra mucilage. The FTIR spectra of the okra mucilage were collected using a Frontier 01 Perkin Elmer spectrometer and calibrated between 500 and 4500 cm^{-1} . The infrared absorption bands in FTIR are used to determine the composition and structure of okra mucilage. Eight scans were used to validate the final structure.

XRD is a non-destructive method that offers comprehensive knowledge of a material's crystalline nature, chemical properties, and physical characteristics. The crystalline structure of the okra mucilage was identified by using an X-ray diffractometer (XRD, Xpert 3 Powder, Malvern Panalytical, UK) using a Ni-filtered $\text{Cu-K}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$) and collected at a scanning rate of 5° per min in the 2θ range of $10^\circ \sim 90^\circ$, the step size of 0.02° and the scan rate of 10 s per step. The INDEX software was indexing the crystal peaks of compounds. A Bruker Kappa APES II single-crystal X-ray diffractometer with MoK ($\lambda = 0.71073 \text{ \AA}$) radiation was used to analyze the single-crystal X-ray diffraction data and determine the molecular structure.

2.4 Flow Loop Experiment

The single-phase wax formation process was investigated using the flow loop experiment, and the pressure drop technique was used to determine wax thickness. The Flow loop setup that was used consists of a steel pipe, pump, crude oil reservoir tank, flow meter, temperature meter, testing section, and pressure gauge. The steel pipe was used to mimic the real petroleum industry for better studies on wax precipitation. To produce a favorable environment for deposition within the area under test and investigate the wax deposition process, waxy petroleum oil was pumped through the inner conduit at a temperature just a little higher than the wall cooling temperature.

The pressure decrease along the pipe length was measured at 30 min with its temperature. The fluid was circulated at a flow rate range of 2.5 L/min - 5 L/min with and without the inhibitors (okra mucilage). The temperature for the test section was in the range of 15°C to 40°C , and the pressure was around 600 Pa - 3000 Pa. Temperatures were reduced from 40°C , 33°C , and 24°C to 14°C . The procedure was performed while pouring the okra mucilage into the storage vessel before the start of the flow.

The Hagen-Poiseuille equation [23] was used to figure out the pressure drop caused by the smaller effective thickness of the tubing (Eq. (1)). The laminar motion of a substance through a circular pipe with a fixed interior diameter is represented by this expression.

$$\Delta P = \frac{128LQ\mu}{\pi D^4} \quad (1)$$

where,

ΔP = Pressure loss, pa Q = flowrate, m³/sec
 D = pipe diameter, m L = test section pipe length, m
 μ = viscosity, Pa.s

Eq. (2) was modified to calculate the inner diameter of the test section conduit based on pressure drop data gathered at a constant flow rate.

$$D = \sqrt[4]{\frac{128LQ\mu}{\pi\Delta P}} \quad (2)$$

The thickness of the deposited material at every point was calculated using Eq. (3) based on the diameter values obtained.

$$\delta = \frac{D_o - D_n}{2} \quad (3)$$

where,

D_o = Original pipe diameter
 D_n = new pipe diameter

2.5 Measurement of Wax Deposition

This simple lab experiment was performed by simplifying the cold finger test. This was performed to prove that okra mucilage was effective as a wax inhibitor.

The effectiveness was calculated by using Eq. (4) below.

$$\text{Effectiveness \%} = \frac{W_{Base} - W_{Case}}{W_{Base}} \quad (4)$$

where,

W_{base} = Weight of precipitate wax for base case (without okra mucilage)
 W_{case} = Weight of precipitate wax for different case (with okra mucilage or okra powder)

The data gathered was used to make a correlation for the result of using diluted okra powder by comparing it to the past research data using a flow loop setup.

3. Results

3.1 Qualitative Analysis

3.1.1 FTIR analysis

The Okra functional group and main component were identified using FTIR results, as shown in Figure 1. The spectrum is separated into two major regions, with the main functional group region ranging from 1500 cm⁻¹ to 4000 cm⁻¹ and the fingerprint group region ranging from 400 cm⁻¹ to 1500 cm⁻¹. As shown in the Figure 1, a strong and broad peak of frequency of 3434.24 cm⁻¹ from the spectrum indicates the presence of the group of O-H [22,24]. This indicates that the three primary components of okra contain aromatic sugar groups with O-H as the main functional group. The

medium peak observed at 2928.55 cm^{-1} indicates the group of C-H stretch found in galactose and rhamnose [25].

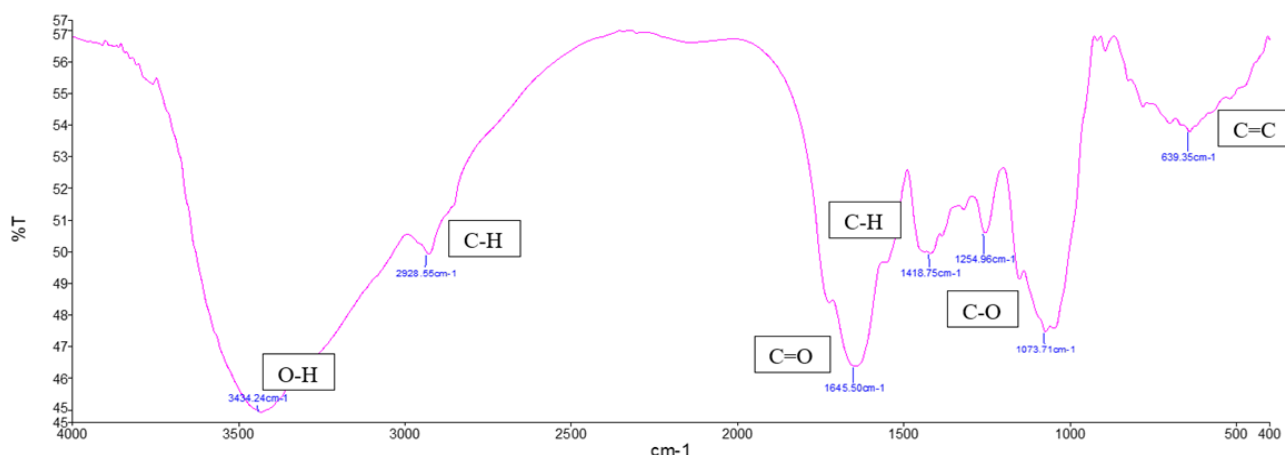


Fig. 1. Analysis of the polymer made from okra

The small peak at 1645.50 cm^{-1} indicates the presence of the C=O stretch in galacturonic acid, while the smaller peak at 1418.15 cm^{-1} indicates the presence of the C-H curve in galactose and rhamnose [26,27]. The existence of C-O stretch bonds in aromatic molecules like galactose rhamnose and galacturonic acid is indicated by a frequency of $1200\text{-}1000\text{ cm}^{-1}$. At the end of the spectrum of frequency of 639.34 cm^{-1} indicated the presence of the alkane group of C=C bending [28]. This leads to the conclusion that galactose, rhamnose, and galacturonic acid are the primary components of Okra. These sugar components shown are the causes of Okra being slippery and slimy. The wavelength with the functional groups' result has been summarized in Table 1.

Table 1

Summary of FTIR analysis

Types	Absorption frequency cm^{-1}	Group
Main functional groups	3434.24	O-H stretch
	2928.55	C-H stretch
Fingerprint group	1645.50	C=O stretch
	1418.15	C-H bend
	1200-1000	C-O stretch
	639.35	C=O bend

3.1.1 XRD analysis

As shown in Figure 2, the XRD analysis of Okra revealed that it has an amorphous and crystalline structure. The wide distribution observed in the X-ray diffraction pattern shows the polymer's amorphous nature. This is because of the X-ray scattering by the atoms randomly scattered throughout an extensive range, resulting in the development of a broad spike [29]. A big spike can be observed at the lower angle of the Analysis. This indicates the crystalline peak. When X-ray light hit the lattice planes, crystalline structures are formed by aligned atoms in repetitive patterns, resulting in high-intensity peaks.

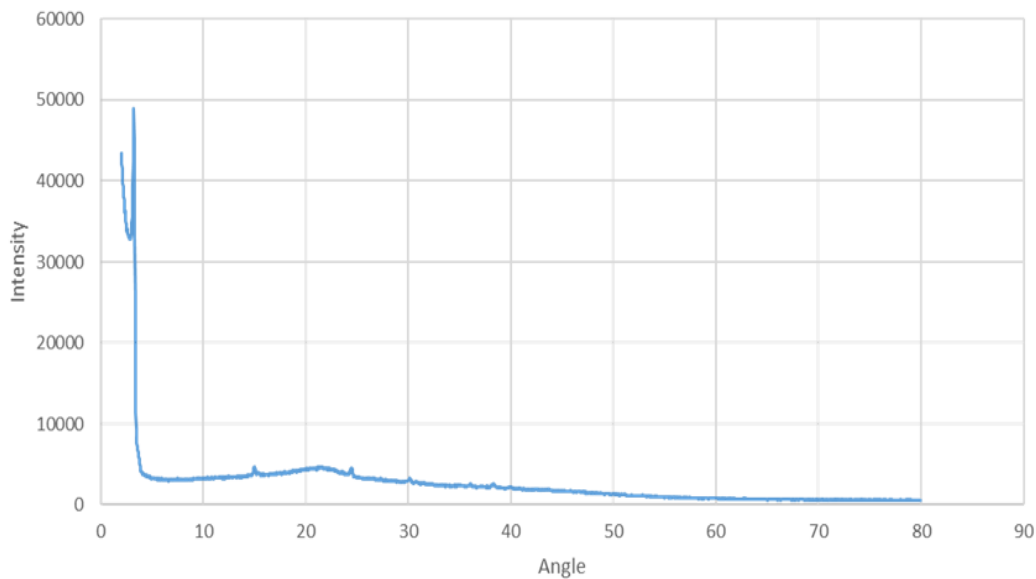


Fig. 2. Okra X-ray diffraction study

3.2 Wax Deposition Analysis

According to the research of Theyab [30], there are six main factors affecting wax deposition. The factors are temperature differential, crude oil composition, experimental time, flow rate, pressure, and pipe surface properties. However, it is important to note that the wax deposit raised relatively by enhancing experimental time, but in the oil fields, the deposition of wax increases regularly because more paraffin is deposited from fresh crude oil on the hydrocarbon pipeline, creating more possibilities for accumulation upon the cold surface [31]. In this study, the flow loop was supposed to investigate the use of okra mucilage as wax inhibition. The deposited wax weight for the base case, case 1 (using Okra mucilage), and case 2 (using diluted Okra powder) were presented in Table 2.

Table 2
 The average of wax deposition by weight

Condition	Weight (g)
Without wax inhibitor	0.09
With okra mucilage	0.04
With diluted okra powder	0.02

From the results it was revealed that diluted okra powder work as better wax inhibitor compared to the okra mucilage.

3.3 Determination of Inhibitor Effectiveness

The average wax weight and the effectiveness of Okra mucilage and Okra powder as wax inhibitors are shown in Table 3. As shown in Table, using okra powder gives a percentage of 20 % improvement from using okra mucilage. This data correlated with the past research data using the flow loop setup. The data in Table shows that the efficiency of okra mucilage is 60.53%. From this result, it was safe to assume that the ratio between the simple lab experiment result and the past result is 1:2. Since the effectiveness of using okra powder in the simple lab experiment is 81.58%, the effectiveness by using a flow loop setup result was approximately about 60.53%.

Table 3

The average of wax deposition by weight

Condition	Effectiveness (%)
Without wax inhibitor	10.00
With okra mucilage	60.53
With diluted okra powder	81.58

4. Conclusions

In this study, the synthesized okra mucilage has been characterized by using FTIR analysis and knowing its structure type by using the XRD analysis. Based on the results of the analysis, it can be concluded that galactose, rhamnose, and galacturonic acid are the primary components of okra. The component stated is the cause of the okra mucilage being slimy and slippery. XRD results demonstrated that okra has a crystalline and amorphous structure. Moreover, it has been proven that using okra mucilage prevents wax from precipitating in the pipeline. This is because it has the characteristics of being slimy and slippery, which will help reduce the surface roughness. Additionally, using diluted powdered okra mucilage gives better results than using okra mucilage straight up. Based on the correlation made, using diluted powdered okra mucilage performed better in effectiveness in the flow loop setup.

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