ABUAD Journal of Engineering and Applied Sciences (AJEAS)

ISSN: 2992-5584

Volume 2, Issue 1, 1-9

Investigation of *Grewia mollis* Gum as Biopolymer Drag Reducing Agent

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Date Submitted: 25/12/2023
Date Accepted: 16/02/2024
Date Published: 25/06/2024

Abstract: *The hydraulic fracturing treatment relies heavy on fracturing fluid. Finding inexpensive polymer that lowers the pressure gradient in turbulent flow systems during hydraulic fracturing is crucial for the development of shale gas. Most researches focused on investigating the effects of polymers such as partially hydrolysed polyacrylamide, polyethylene oxide, guar gum, xanthan gum and synergies of mixtures as friction reducing polymers. The use of Grewia Mollis mucilage as the natural polymer as a potential friction reducer in slickwater fracturing treatment was the main focus of this study. The sample of Grewia gum was obtained and the mucilage was extracted from the inner stem bark by maceration in water at ambient temperature. The mucilage was the oven-dried at a temperature of 50 ^{o}C. Atomic Absorption spectrophotometer (AAS) and Fourier Transform Infrared (FT-IR) were employed to identify the elemental composition and the functional groups of the plant. The FT-IR spectrum exhibited typical peaks and bands characteristics of polysaccharide, while the ASS result shows the presence of minute quantity (0.219 mg/100 g) of lead (Pb) in the plant may show contamination (toxicity). The rheological measurements of the mucilage plotted on the shear viscosity graph exhibit shear-thinning or pseudo-plastic behaviour. Formulated slickwater fracturing fluid at various concentrations ranging from 200 to 500 ppm, were run in a constructed closed flow loop of 0.01905 m diameter of galvanised steel pipe, test section length of 2 m and entrance length of 2 m. Drag reduction was measured at flow rates (2.3, 2.8, 3.2, 3.6 and 4.0) m^3/hour at different concentrations (200 to 500) ppm. Percentage friction reduction of 37% was achieved at Reynolds number of 74269 by addition of 200 ppm of the fluid while 56% was achieved at Reynolds number 74269 by addition of 500 ppm. This indicates that Grewia Mollis mucilage exhibit drag reducing potential and can be applied in slickwater hydraulic fracturing application.*

Keywords: Friction reduction, polymers, Grewia mollis, slickwater, hydraulic fracturing.

1. INTRODUCTION

In current global energy landscape, natural gas is regarded as a transitional energy source towards more environmental sources. High permeability conventional gas reservoirs and low unconventional gas reservoirs, primarily shale gas reservoirs, are the major sources of natural gas. It is evident that hydraulic fracturing methods must be used to recover shale gas instead of the more traditional methods. The creation and connection of fracture networks through multiple stages of hydraulic fracturing treatments is the only way to achieve economic rates of hydrocarbon production from shale formations [4]. Slickwater hydraulic fracturing fluids are frequently used to create fracture networks that allow trapped gas in reservoirs to be released. Hydraulic fracturing is essential to the development of unconventional gas reservoirs.

Slickwater requires high pumping rates of about 50 to 100 bbl/min to deliver proppant to the fracture networks because it is much less viscous than gel fracturing fluids. Small amounts of drag-reducing agents (75 to 600 ppm) are frequently added to fracturing fluids to overcome the high pumping flowrates and to reduce the pressure drop. Pumping fracturing fluids into the well at high flowrates and pressures is necessary to cause fractures in the formation rocks. The initiation of fracture networks and their ultimate size dependent on the viscosity of the fracturing fluids [11].

Water makes up the majority of slickwater fracturing fluid, with small amounts of polymers (between 0.25 to 10 pounds per thousand gallons) [6, 21]. Low viscous fracturing fluids have gained popularity over the last decade due to the relatively low chemical loading of slickwater, the ease of clean-up, and the reduced damage that comes with using gel-based fluids. These factors make low-viscous fluids ideal for fracturing low-permeable formations [21].

Due to its lower viscosity, Slickwater is less able than viscous fluids to suspend and carry proppants to the fracture networks. Fracturing fluids are pumped at higher flowrates to compensate for the poor proppant transport, but this causes spectacular turbulence and frictional loss in the tubular pipeline [12, 21]. To reduce friction and compensate for surface pumping pressure loss when pumping at a higher flowrate, a small amount high molecular weight polymer is added to the fracturing fluid. These polymers act as a friction reducer.

In the process of making up slickwater hydraulic fracturing fluid, friction reducer usually polymer is the main additives. Drag reduction techniques of synthetic polymers have been widely adopted in large-scale engineering applications, such as marine drag reduction, but their broad use has been hindered by two main concerns: the high cost and environmental impact of synthetic polymers. High molar mass biopolysaccharides found in plant mucilaginous material are used as low-cost drag reducers in turbulent flow with a high Reynolds number in order to overcome consumable cost limits [23]. This research focuses on studying the effect of using *Grewia* gum mucilage, obtained from the inner stem bark of the *Grewia Mollis* plant, as a potential drag reducing agent in hydraulic fracturing treatment.

2. MATERIALS AND METHODS

2.1 Materials

2.1.2 Grewia mollis gum

The biopolymer polysaccharide used during the experiment in this study is *Grewia mollis* gum with average molecular weight of 5925 kDa, number average molecular weight of 3720 kDa and polydispersity of 1.6 [17]. The polysaccharide gum is made from the inner stem bark of the plant and its widely distributed species native to tropical Africa, Yemen, and Oman. The sample used for this study was obtained from Bayara market in Bauchi Local Government Area of Bauchi State, Nigeria.

2.2.2 Flow loop set up

The experimental flow loop system depicted in the diagram in Figure 1. The closed flow loop was designed to meet the objectives of the study. The system consists of galvanized steel pipe, storage tank, valves, a flow meter and pressure gauges. The fluid was cycled from the storage tank to the test section and back again using a centrifugal pump. To regulate the flowrate, a ball valve was employed. In order to measure a reasonable pressure along the test section, the entrance length of 2 m approximately 105 times the pipe's diameter (105D) was used for the fluid to develop fully turbulent. A flow meter was installed to measure flowrate. Two pressure gauges were mounted at a distance of 2 m, or approximately 105 times the pipe's diameter (105D) to ensure adequate pressure drop along the test section was to measure both upstream and downstream pressure.

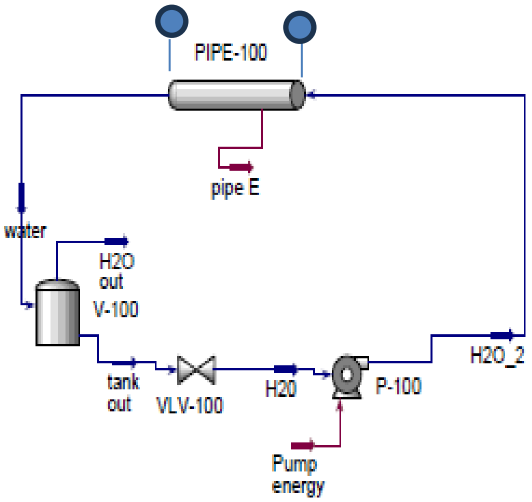

Figure 1: Schematic of the flow loop set-up

Figure 2: Constructed flow loop used for the experiment

Figure 2 depicts the built-in flow loop that was used in the study. A 60-litre storage tank, a 1-horsepower centrifugal pump (ATLAS) with pumping capacity of 6 to 69 litres/minute and 2850 rmp, a galvanized steel pipe of test section of 2 m long and internal diameter of 19.05 mm as well as a Sea Zhongjiang digital flow with model YF-B6S, a threaded stainless steel G3/4 Hall Effect flow sensor with a working pressure range from 0.05 to 1 MPa and a flow range of 2 to 30 litres comprises the setup. Using the pressure gauges, the pressure drop data along the test section was obtained, the fanning friction factor (f) at each given flowrate is found using Equation (1):

$$f = \frac{\Delta PD}{2\rho l u_b^2} \qquad (1)$$

Specification of the materials used in the construction of the flow loop is presented in Table 1

Table 1: Principal equipment component used and their features

Flow loop equipment	Features
Pipe	Material: stainless steel
Pump	(ATLAS 125) with a pumping capacity of 6-60 L/min
Flow meter	Sea Zhongjiang Digital Flow sensor with model YF-B6S, thread size of Stainless Steel G3/4 Hall Effect Flow Sensor, working pressure of 0.05- 1 MPa and flow range of 2-30 L
Mixing tank	60 L PVC Tank
Fann 35A Viscometer	It has rotational cylinder that is possible set at 600, 300, 100, 6 and 3 rpm

2.2 Methods

The design comprises three main sections: sample characterization, mucilage rheological testing, and drag reduction measurements using a flow loop setup. The elemental composition and functional group of the sample were ascertained using an atomic absorption spectrophotometer (AAS) and Fourier transform infrared (FT-IR) respectively. The shear and viscosity of polymer solutions were examined. Equations for the friction factor and generalized Reynolds number were used to measure and assess the flowrate and pressure drop.

2.2.1 Extraction procedure

The process of extraction involved macerating the sample in water at room temperature in order to extract the mucilage from the inner stem bark. The inner stem bark was soaked in water after it was chopped into pieces for a day. It is dried in an oven at 50 degrees Celsius for six hours after being filtered through a sieve with a 2000 micrometre mesh. For usage, the dried mucilage was group up and kept in an airtight [15]. An Agilent Technologies model Cary 630 FT-IR Spectrophotometer and background scans with a resolution of 4 cm^{-1}, Fourier Transform Infrared (FT-IR) was utilized to ascertain the functional group of the sample. Averaging 32 scans per spectrum produced by the machine was used to process the data. According to [24], *Grewia* Mollis sample's stem bark contained several heavy metals, including zinc, iron, copper, manganese, and magnesium. An atomic absorption spectrophotometer (AAS) AA320N was used to ascertain the heavy metals presence in the sample.

2.2.2 Sample formulation

A mechanical impeller was used to prepare the sample solution. In order to create a vortex and prevent the polymer from scissoring, the speed was first set low. To prevent lumps from forming in the solution, small amounts of water and polymer were added to the vortex in several intervals [26]. The speed was raised until the solution became viscous after the powdered polymer was added. For optimal hydration, the solution was stirred for two hours and left overnight [13]. 2000 ppm was used to prepare the master solution as shown in Figure 3, which was diluted during the experiment to the required concentrations of 200 to 500 ppm.

Figure 3: Prepared *Grewia* gum solution

2.2.3 Rheological measurement

In order to ascertain the rheological behaviour of each concentration of friction reducer solutions used, a profile of the shear rates to shear stress was made using a Fann 35A viscometer, which had different shear rates of 600, 300, 200, 100, 6, and 3 rmp. Dial readings were taken at 3, 6, 100, 200, 300, and 600 rpm at various concentrations were taken.

2.2.4 Friction reduction measurement

The flowrate was set at various values as Table 2 illustrates. For every flowrate, measurements of the pressure drop were made. This process was repeated for every concentration of polymer, and all calculations were made in the following manner as the flow through the test section became completely turbulent. The Reynolds number was calculated using Equation (2).

$$\mathrm{Re} = \frac{\rho u D}{\mu} \tag{2}$$

where ρ is fluid density, μ is dynamic viscosity, u is the section-averaged axial velocity (mean bulk velocity) and D is the pipe diameter.

For the turbulent pipe flow a dimensional analysis can give insight into the mean velocity profile in the near-wall region. The velocity, U can be obtained from Equation (3).

$$U = \frac{Q}{A} \tag{3}$$

Q is the flowrate and A is the cross-sectional area of the pipe as:

$$A = \frac{\pi D^2}{4} \tag{4}$$

The scaling parameters are u_τ, ρ and μ; where u_τ is the friction velocity as:

$$u_\tau = \sqrt{\frac{\tau_w}{\rho}} \tag{5}$$

With

$$\tau_w = \frac{D\Delta P}{4L} \qquad (6)$$

where ΔP is the measured pressure drop, L is the test section length, D is the pipe diameter and τ_w is the wall shear stress

Table 2: Experimental flowrate, velocities and Reynolds number used

Pipe Diameter, ID (m)	**Flowrate, Q (m^3/hr)**	**Velocities (m/sec)**	**Reynolds number**
0.0195	2.4	2.3	44561.40
	2.8	2.7	52359.65
	3.2	3.1	59043.86
	3.6	3.5	66842.11
	4.0	3.9	74640.35

Table 3: Properties of water used for the experiment

Water at 23 ºC	**Value**
Viscosity of water	10^{-3} Pa.s
Density of water	1000 kg/m^3

The Grewia gum biopolymer was run in a flow loop with a pipe diameter of 19.05 mm, at various concentrations and flowrates. The test section was located 2 m away from the entrance, or entrance length (Le), which limits pressure drop measurements in the fully developed region. Desiler equation was used to calculate as shown in Equation (7):

$$Le = 50d \qquad (7)$$

The type of flow and pipe wall roughness have a major influence on the relationship between friction factor and Reynolds number. When the Reynolds number is less than 2000, the flow is said to be laminar flow, and friction factor follows Boisuelle's equation shown in Equation (8):

$$f = \frac{16}{Re} \qquad (8)$$

Virk proposed an asymptote to represent the maximum fall in friction reduction, in which the relation between the Reynolds number and friction factor does not depend on the type of additives or pipe diameter [28], given by Equation (9).

$$f = 0.079Re^{-0.25} \qquad (9)$$

The percentage drag reduction was obtained by measuring the pressure drop, ΔP_a of water added polymers compared with pressure drop, ΔP_b solvent (water) at the same condition, as given by equation (10):

$$(\%FR) = \frac{\Delta P_b - \Delta P_a}{\Delta P_b} \times 100 = \frac{f_b - f_a}{f_b} \times 100 \qquad (10)$$

where ΔP_b the pressure drop is difference before adding additives, Nm^2 and ΔP_a is the pressure drop after adding additives, Nm^2 .

3. RESULTS AND DISCUSSION

3.1 Physio-Chemical Characterization

3.1.1 FT-IR analysis

Figure 5 depicts the mucilage's FT-IR spectrum after extraction from inner stem bark of the *Grewia mollis* plant. The usual characteristics peaks and bands of polysaccharides are visible in the spectrum of the mucilage. Hydroxyl (-OH) groups are indicated by the broad band observed at 3310 cm^{-1}. Stretching modes of the methyl group (-CH3)'s C-H bonds are shown by the peak that occurred at 2925 cm^{-1}. Sugar acids are typically present in natural gums, giving the gum macromolecule a mildly anionic character [17]. According to Okafor [19], the carboxylate group of the galacturonic acid residues is visible in the absorption band around 1595 and 1425 cm^{-1}. The acetyl group is found in the region spanning from 1500 to 1800 cm^{-1}. According to Nep [17] the finger print region for carbohydrates is represented by wave numbers ranging from is 800 to 1200 cm^{-1}. These values derived from the spectra agree with the results obtained by [17]. The monosaccharide composition of the gum was determined using gas chromatography. They reported that the primary structure of *Grewi*a gum may comprise five natural sugars: d-rhamnose, d-arabinose, d-xylose, d-galactose, and d-glucose [17].

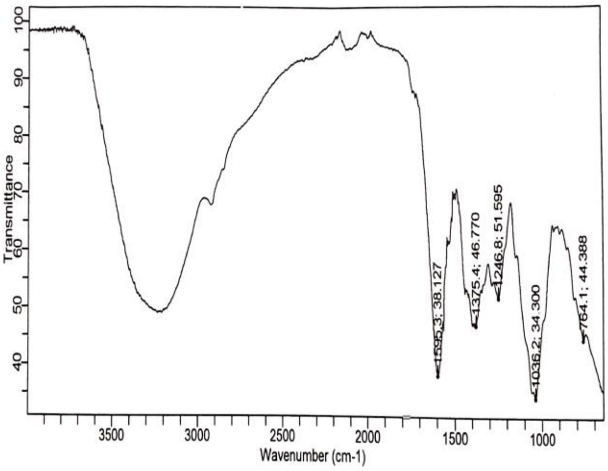

Figure 5: FT-IR spectra analysis of *Grewia mollis* gum

3.1.2 Mineral *analysis*

According to mineral analysis displayed in Table 4, *Grewia mollis* plant contain some heavy minerals such copper (0.059 mg/100 g), iron (0.316 mg/100 g), manganese (0.405 mg/100 g), lead (0.00219 mg/100 g), magnesium (1.559 mg/100 g) and zinc (0.458 mg/100 g). Lead (Pb) traces in the amounts in the inner stem which could indicate a certain level of toxicity. Although, this value is below FAO/WHO permissible value of 0.03 mg/100 g.

Table 4: Mineral composition of *Grewia* mucilage gum

Minerals	Composition (mg/100 g)
Manganese	0.405
Zinc	0.458
Magnesium	1.559
Iron	0.316

Minerals	Composition (mg/100 g)
Copper	0.059
Lead	0.219

3.2 Viscosity Measurement

3.2.1 Effect of shear rates on apparent viscosity

The fluid tested with shear rates ranging from 5.11 to 1021.80 s^{-1} is depicted in Figure 6 as a plot of the shear viscosity profile. Plotting the power law relationship on a double logarithmic scale, as illustrated in Figure 6, gives a linear appearance, accurately characterizing the shear-thinning region. This demonstrates that *Grewia* gum follows a power law and exhibits shear-thinning behaviour when the shear rate increases and their viscosities decrease. The polymer solution with 500 ppm clearly shows the highest shear-viscosity, whereas the solution with 200 ppm clearly shows the lowest shear-viscosity. The shear viscosity between the two ends with concentration s between 300 and 400 ppm lies in the middle. The shear-viscosities of all fluids decreased with corresponding increases in shear rate, exhibiting shear-thinning behaviour within the observed range of values from 1 to 200 s^{-1} [30].

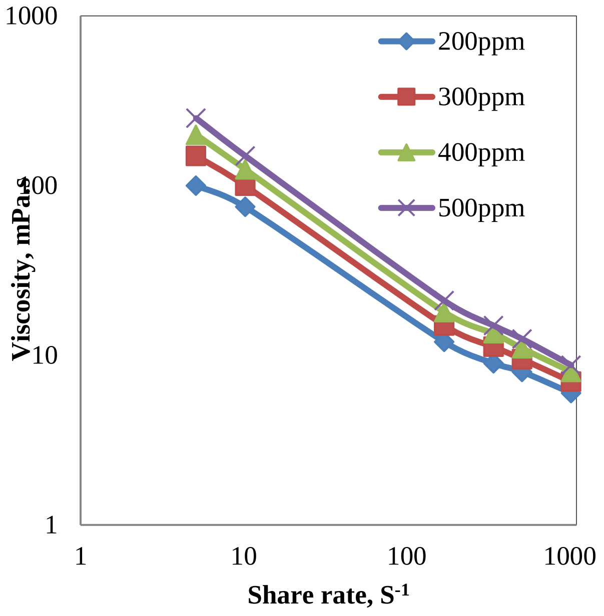

Figure 6: Viscosity vs, shear rate at various concentrations

3.2.2 Shear stress- shear rates

Potting shear stress versus shear rate for fluid concentrations ranging from 200 to 500 ppm as shown in Figure 7. It displays how the fluid's shear rate changes as the friction reducer concentrations varies (200, 300, 400 and 500 ppm). Shear stress increases nonlinearly with each plotted concentration. Shear-thinning or pseudo-plastic behaviour is seen at all concentrations.

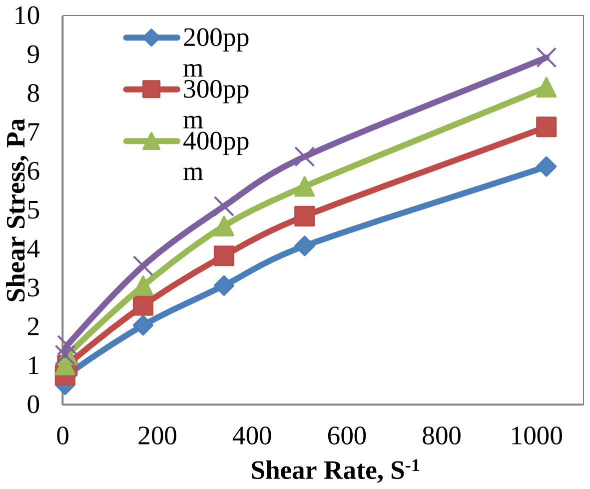

Figure 7: Shear stress vs. shear rate at various concentrations

3.2.3 Effect of viscosity on concentration

Figure 8 illustrates how the apparent viscosity of each tested solution increases as the concentration of Grewia gum increases at the same shear rate and ambient temperature. This suggests that the increase in the number of polymer chains in a given volume that interact with one another is responsible for the apparent viscosity, which is a function of concentration [30].

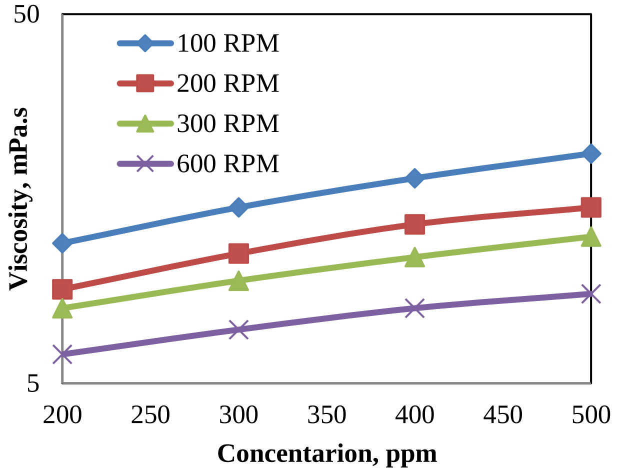

Figure 8: Effect of concentration on viscosity at different revolution per minute

3.2.4 Effect of concentration power law index consistency constant

Samples were taken at concentrations ranging from 200 to 500 ppm recorded shear stress and shear rate in order to assess the polymer power-law characteristics. Equations 3 and 4 are used to derive the power-law parameters, consistency constant and power law index, which are then shown as seen in Figures 9 and 10, respectively. *Grewia* gum solutions demonstrated non-Newtonian behaviour by showing that values of K increased and n values fell as concentration increased from 200 ppm to 500 ppm. *Grewia* gum solution's power law index values ranged from n = 0.48 to 058 at 500 ppm and 200 ppm, respectively. Because of a decrease in entanglements, the fluid's viscosity drops at high shear rates [10, 22].

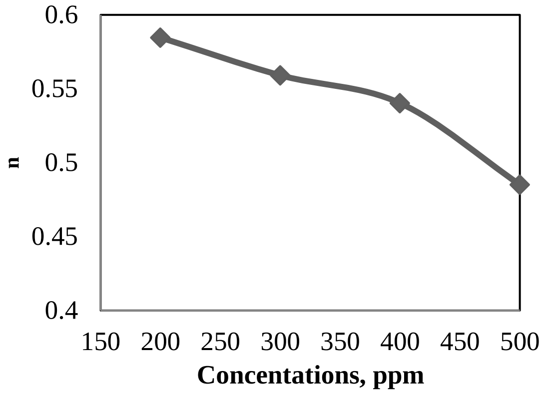

Figure 9: Power law parameter vs. concentrations

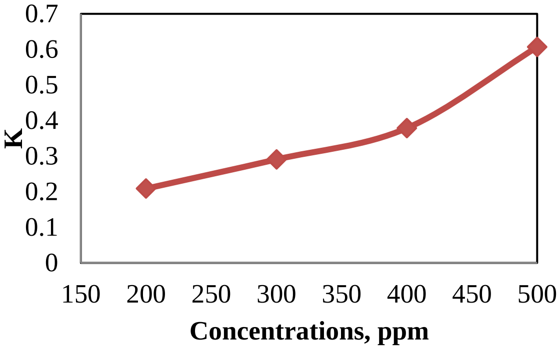

Figure 10: Power Law Parameter K vs. Concentrations

4.2.5 Effect of polymer concentration on drag reduction

A non-Newtonian behaviour was noted for all sample solutions. For all flow rates examined, Figure 11 demonstrates that the friction reduction increases as the polymer concentration rises. According to the results, at a flow rate of 4.0 m³/hour, an addition of 200 ppm reduced friction by 37%, while an addition of 500 ppm reduced friction by 56%. These results are consistent with those of [1]. Despite using aloe Vera as the biopolymer, they also noticed a consistent rise in friction reduction as the Reynolds number increased.

According to Dosumu [8], adding 200 ppm of Xanthan gum solution reduced friction by 40% in water flow at a Reynolds number of 35,775; at the same Reynolds number, adding the same concentration of Guar gum solution reduced drag by 33% in single-phase flow. Both Xanthan gum and Guar gum compare better than Grewia gum at equivalent concentrations, though the differences may be due to variations in the experimental setup.

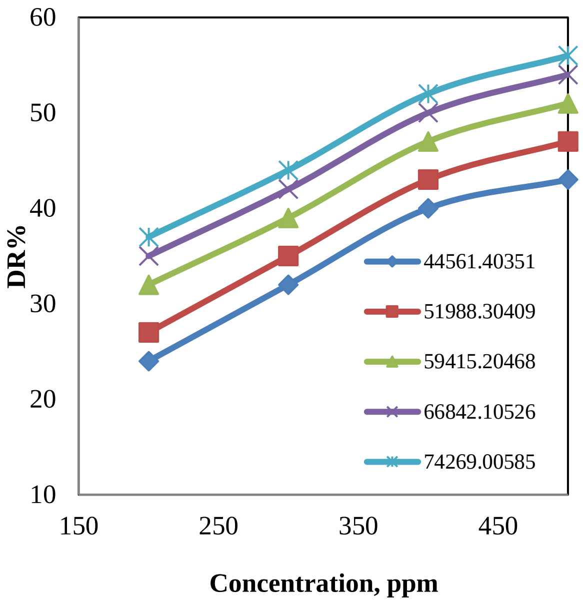

Figure 11: Effect of concentration on drag reduction

3.2.6 Effect of flow rates on Reynolds number

As demonstrated in Figure 12 for percentage drag reduction (%Dr), the influence of flow rates on the Reynolds number of *Grewia* gum as a friction reduction was conducted for five different flow rates (2.3, 2.8, 3.2, 3.6, and 4.0) m³/hour. In the pipe's test section, Figure 12 illustrates the solution's reduced friction as a function of flow rate at different concentrations. Prior to the injection of *Grewia* gum solutions into the main flow stream, the Reynolds numbers were calculated using the single-phase flow characteristics (pipe diameter, viscosity, density, and average velocity). Only when the turbulent flow reaches its maximum potential can friction be reduced. To guarantee that fluid flow rates remain in the turbulent regime, the minimum Reynolds number needed to be in this regime is 44,561, which is equivalent to the 2.3 m³/hour.

As the flowrate increases, it is evident that the amount of friction reduction increases. It is anticipated that an increase in flow rate will result in a rise in velocity or Reynolds number, and drag reduction will increase with these values [26, 29]. Additionally, it was observed that at moderate Reynolds numbers, the increase in effective viscosity improved the reduction of friction. At very high Reynolds numbers, on the other hand, drag reduction is caused by variations in effective viscosity as a function of the pipe wall due to turbulent activity [3].

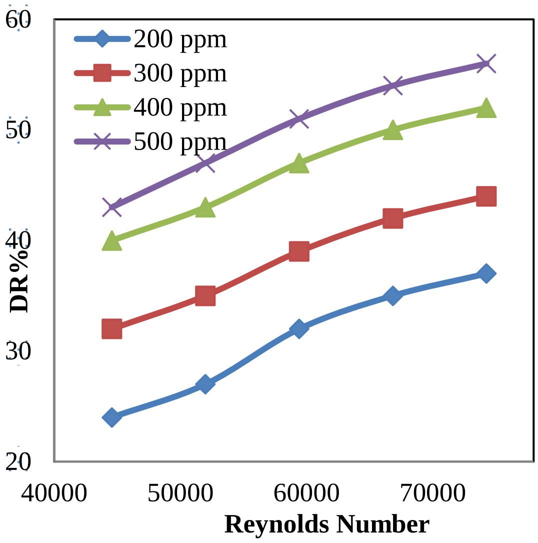

Figure 12: Effect of flow rates on drag reduction

3.2.7 Friction factor

When the Reynolds number is less than 2000, the flow is laminar [25], and the friction factor is determined by Boisuelle's equation (6). Figures 13 and 14 display the fluid solutions' features for reducing friction as the Reynolds number (Re) vs. the fanning friction factor (f). The relationship between the friction factor and Reynolds number for the Grewia gum solution at various concentrations in a pipe diameter of 0.01905 m was displayed in Figures 13 and 14. The Reynolds number rises as the friction factors decrease in the test section when the solution at different concentrations flows through it. The friction factor drops for the four concentration runs as the concentrations of *Grewia* gum (200, 300, 400, and 500 ppm) rise.

This suggests that the friction factor reduces and the friction reduction increases as the concentration of *Grewia* gum increases. The expected result is a drop in the friction factor values up to the Virk asymptote line when the polymer concentration is raised at higher Reynolds numbers. The maximum drag reduction asymptote (MDA) sets a limit on the effectiveness of polymer-induced friction reduction [10, 28, 29]. Until the MDA is reached, drag reduction rises as the drag reducing agent's concentration does. They found out that maximum drag reduction obtainable using drag reducing agent turbulent pipe flow is limited to MDR asymptote, which is a function of a Reynolds number. The magnitude of drag reduction increases with drag reducing agent concentrations of the system, until the maximum drag reduction asymptote is reached.

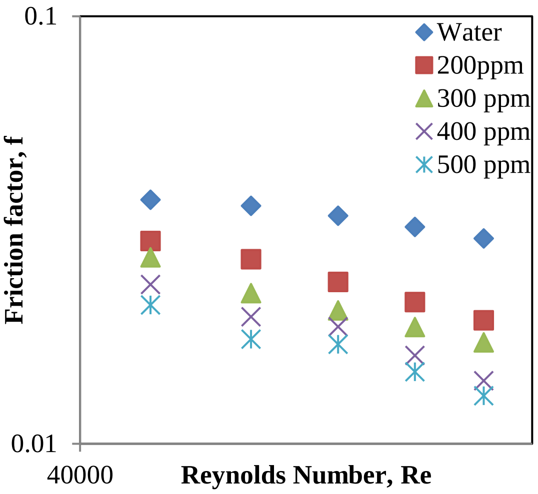

Figure 13: Fanning friction factor vs. Reynolds number

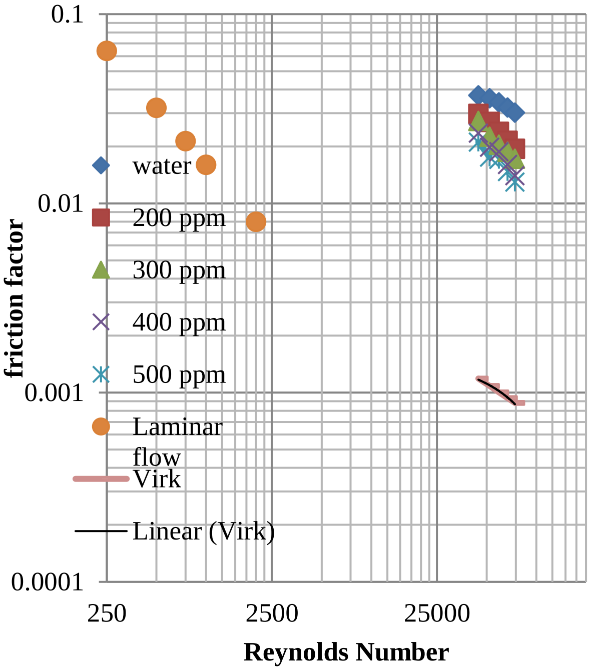

Figure 14: Fanning friction factor vs. Reynolds number

4. CONCLUSION

The study's FT-IR spectra of the mucilage taken from *Grewia mollis* reveal the presence of carboxylate, acetyl, methyl (-CH3), and hydroxyl (-OH) groups. These functional groups often accord well with spectra produced in other similar investigations [17] and are compatible with typical bands and peaks characteristics of the polysaccharides. The *Grewia mollis* mucilage-based slickwater fracturing fluid, shows Power Law shear-thinning at concentrations of 500 ppm to 200 ppm, with a power law index ranging from (n = 0.48 – 0.58). As the concentration of Grewia gum rises, so does the apparent viscosity of every solution test. This demonstrates how apparent viscosity depends on concentration and explains the rise in the apparent viscosity of every solution tested rises as the concentration of Grewia gum does. This demonstrates that, as noted by Zhang *et al.* (2019), the apparent viscosity is a function of concentration and can be related to the growth in polymer chains in a given volume that interact with one another. Experimental flow of the Grewia gum-based fluid through the flow loop was achieved with a maximum drag reduction performance of 56% at 500 ppm. *Grewia* gum concentration and the friction factor generally decreases with an increase in *Grewia* gum concentration in order ranging from 200 ppm, 300 ppm, 400 ppm, and 500 ppm. Generally, friction factor decreases as Reynolds number increases. This means that as the *Grewia* gum concentration increases, the friction factor decreases, and therefore, the drag reduction increases. This is not unexpected, as increasing the concentration of polymeric additives at higher Reynolds number results in a decrease of the friction factor values up to the Virk asymptote value. Hence, this can be potential alternative to synthetic polymers used as the drag reducing agent in the hydraulic fracturing treatment.

Future work needs to focus on the following:

- Effects of salinity and temperature on the performance of *Grewia* gum as friction reducer at different concentrations;
- Effects of different diameters test sections to observe the effect of diameter on the friction reduction for the *Grewia mollis* gum as drag reducing agent;
- Hydraulic fracturing operations use larger pipe sizes and high flowrates than those used in the laboratory, it is important to be able to scale up to predict drag reduction performance in large pipes diameter measurements.

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