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Empirical Study of *Grewia* Gel as an Alternative Thickening Agent in Hydraulic Fracturing Fluid

Salihu MUBARAK[1], Usman HASSAN[1], Usman Dadum HAMZA[2]

[1]*Department of Petroleum Engineering, Abubakar Tafawa Balewa University Bauchi, Nigeria*
mubaraksaleehu@gmail.com/uhassan@atbu.edu.ng
[2]*Department of Chemical Engineering, Abubakar Tafawa Balewa University Bauchi, Nigeria*
dhusman@atbu.edu.ng

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***Abstract**: This research work studied a new fracturing fluid material (Grewia gel) that can be used in stimulating oil and gas wells. The local base bio-polymer was sourced within the Bayara community in Bauchi, Bauchi State. The gel was extracted by hydrating the dried Grewia sample in water for 24 hours. The mixture was then stirred and passed through a muslin bag to remove extraneous materials. The filtrate was dried in an oven at 50 °C for 24 hours and subsequently dry-milled. The dried product was passed through a 1.0 mm sieve and stored in an air-tight container. A combination of analytical techniques such as Fourier Transform Infrared Spectroscopy (FT-IR), Filtration Control Test (FCT), and Thermogravimetric Analysis (TGA) was used to characterise the sample. The experiment was designed using the Box Behnken Design (BBD) model, where independent variables such as pH, gel, and cross-linker concentration and the dependent variables of viscosity and gel strength were considered. The effect of the independent variables on the responses was studied. The optimum composition of the fluid sample was found to be 29.91 pptg of the gel sample and 5.16 pptg of borate crosslinker at a pH condition of 9.59. Experimental results showed that the rheological characteristic of the gel is comparable with guar gum over the shear and temperature ranges under study and suitable for temperatures up to 160 °F. In addition, the gel has a better aqueous solubility (92%) when compared to the conventional guar gum with 85%. The Grewia gel can be considered a promising alternative for its application in the petroleum industry as a fracturing fluid gelling agent owing to its improved solubility, rheology, thermal stability, proppant suspension ability, and cheaper cost when compared to the imported guar gum.*

***Keywords:** Fracturing fluid, Grewia gel, rheological characteristics, petroleum industry, thickening agent.*

1. INTRODUCTION

Well stimulation refers to the range of activities employed in order to increase well productivity by increasing the permeability of the near wellbore region. It is employed onto a well if the reservoir permeability is impaired by either the drilling processes or the undisturbed natural permeability of the reservoir is not economically enough [11]. According to Economides, there are three different methods available for reservoir stimulation namely: Hydraulic fracturing, Matrix Acidizing and Acidic fracturing. However, the recent technological advancement in Hydraulic fracturing (HF) has made it the most promising technique in countries with unconventional reservoirs and is creating considerable excitement worldwide. This has helped gain access to hydrocarbon deposits which were earlier inaccessible to conventional drilling operations [8]. It entails the high-pressure injection of a base fluid (water), propping agent (silica sand) and some chemical additives to stimulate and enhance the flow of the reservoir fluid into the wellbore. The main essence of the fluid is to initiate the creation of these fracture networks and to suspend the proppants till the fracturing process is complete. These fracture networks are kept open using proppants, which prevent the closure of those fractures due to overburden stresses acting on the formation [13, 19].

After the completion of the process, the injected fluids are broken into low-viscosity liquids using breakers to facilitate the flow back of the fluid to the surface. This flow back fluid that remains underground is a great threat to the environment and is estimated that only 5 - 60% of the fracturing fluid flows back to the surface and the rest remains underground [7, 12].

HF is employed in permeability-impaired formations such as low-permeability organic-rich shale formations and tight-gas reservoirs [10], it significantly improves the productivity of the wells and the overall recovery factor. It is also widely used in moderate permeability reservoirs (up to 50 mD for oil and 1 mD for gas) with large skin around the vicinity of the wellbore [9]; thus, bypassing the damaged zone without negatively impacting the formation reserves.

A typical fracturing fluid usually consists of a gelling agent, corrosion inhibitors, iron control additives crosslinkers, buffers, clay stabilizers, gel stabilizers, surfactants, and breakers mixed with fresh water [8]. These various chemical additives are used to prevent damage resulting from such operations or to enhance it beyond the aim of fracturing operation [2, 12]. Several types of fracturing fluids have been used in the oil and gas fields which include linear and cross-linked polymer gels, viscoelastic surfactants, and foam-based fluids [10]. The viscoelastic surfactant-based fracturing fluid is thermally stable though shear thinning behaviour and water solubility are the two important characteristics behind increasing interest in their use in fracturing operations when compared to the polymer-based gel [6]. However, the rheological properties of viscoelastic gels are severely affected by temperature and surfactant concentration, they have more fluid leak-off due to low molecular weight and the absence of filter cake [13].

Conventionally, water-soluble polymers like Guar and its derivatives like Carboxyl Methyl Hydroxyl Propyl Guar (CMHPG) in linear or metal ions-cross-linked form are being used as gelling agents to develop sufficiently high viscosity to fracture the formation and to place the proppant into the fracture [10]. They are thermally stable and have good proppant suspension ability for varying reservoir permeability. At high pressure, filter cake formation further reduces the leak-off of fluids into the formation [13]. However, the high residue deposited within the fracture after the fracturing operation is a major disadvantage of the polymer-based fracturing fluid [14]. Despite its inherent drawbacks (in-soluble residue, its high demand, and limited availability), Guar gum is one the most popular polymers used in aqueous-based fracturing fluids since it has great viscosifying properties [14, 16]. Therefore, a fracturing fluid with a more soluble gelling agent, better rheological properties, thermal stability, proppant suspension ability, and less leak-off is required.

This research work studied a new fracturing fluid material *Grewia bicolor* gel that can be used as a gelling agent in fracturing tight and conventional reservoirs. In Nigeria, the *Grewia* plant grows abundantly in the Northern region of the country where it is found growing wild or cultivated [4]. The *Grewia* gel is extracted from the stem bark of the *Grewia* plant which slowly hydrates in water, dispersing and swelling to form a highly viscous dispersion exhibiting pseudoplastic flow behaviour [20]. The polysaccharide gel is used as a stabilizer or suspending agent in foods, cosmetics, and pharmaceutical industries [14, 17].

2. MATERIALS AND METHOD

The following materials were used in the course of conducting this study as shown in Table 1.

Table 1: Laboratory materials used for this research work

Materials	Uses
Weighing balance	Weight Measurement of *Grewia* gel Sample
Water bath	Heating and temperature control of fluid samples
Thermometer	Temperature Measurement of fluid Samples
Sieve	Sieving of *Grewia* gel
Oven	Drying of *Grewia* gel
Viscometer	Viscosity measurement of fluid Samples
pH Meter	pH Measurement of fluid Samples
Stirrer	Stirring of fluid Samples
Whatman paper	Filtration Control Test
Beaker	Mixing vessel
Sodium hydroxide	Buffer
Borate crosslinker	Crosslinker

2.2 Method

The methods followed in conducting this research work are summarized in the chart below (Figure 1).

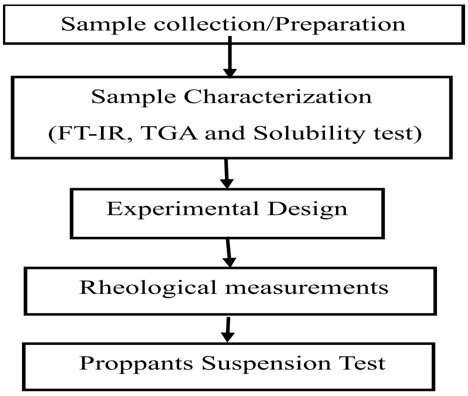

Figure 1: The methodology flow diagram

2.2.1 Sample Collection/Preparation

The *Grewia bicolor* sample was sourced within the Bayara community in Bauchi, Bauchi state, Nigeria. The dried sample (Plate 1) was immersed in water and allowed to hydrate for 24 hours. The mixture was then stirred and passed through a muslin bag to remove extraneous materials. The filtrate was dried in an oven at 50 °C for 24 hours and subsequently dry-milled. The dried product was passed through a 1.0 mm sieve, weighed and stored in an air-tight container. Plate one (1) illustrates the *Grewia bicolor* sample that was used in conducting this research work.

Plate I*: Grewia* plant

The *Grewia* sample is characterized in terms of its solubility in water using filtration control test (FCT), functional group responsible for thickening and breaking of the gel using Fourier Transform Infrared Spectroscopy (FT-IR) and Thermal stability test using thermogravimetric analysis (TGA).

2.3.1 Aqueous Solubility

The aqueous solubility of the Bio-polymer was determined gravimetrically by hydrating 0.5 g into 500 ml of water at room temperature for 24 hours. The solution was then filtered through a pre-weighed filter paper of medium porosity. The residue on the filter paper was weighed after drying in an oven at 50 °C for 24 hours and the solubility was determined.

2.3.2 Infrared Spectroscopy

The Infrared spectrum of the *Grewia* sample was observed within 4000–400 cm^{-1} wavelength and a transmittance range of 60-130 using a Cury 630 FT-IR spectrophotometer. About 0.5 g of the polymer sample was scanned and the representative signature was collated and analysed.

2.3.3 Thermal Analysis

TGA 8000 Thermo-gravimetric analyser was used to study the thermal degradation of the polymer sample under a nitrogen atmosphere. One (1) mg of the sample was placed into the sample pan and heated at a rate of 10 °C per minute up to 890 °C. Results were obtained in triplicate and the representative plot was collated and analysed.

2.4 Experimental Design

In this study, Box-Behnken design was used to generate the experimental design. The experiments were performed considering different combinations of independent variables. Three process parameters; pH, amount of gel, and cross-linker were chosen as the independent variables while the viscosity and gel strength were the dependent response variables. Linear gels were prepared at varying polymer concentrations as shown in Table 2 with continuous stirring for improved solubility. The solutions were left for 1 hour for complete dissolution before heating to 80 °F (26.7 °C) to prepare the solution for cross-linking. The cross-linker (borate) was mixed with the polymer solutions at variable concentrations and pH conditions. Table 1 shows different levels of independent variables used in this work

Table 2: Experimental design

Factor	Name	Unit	Low Actual	High Actual	Low Coded	High Coded
A	*Grewia* gel	pptg	20.0	40.0	-1.0	+1.0
B	cross linker	pptg	4.0	6.0	-1.0	+1.0
C	pH	-	8.0	11 .0	-1.0	+1.0

Source: (API RP 39, Muhammad *et al.*, 2018)

2.5 Proppants Suspension Test

Laboratory testing for proppants suspension ability of the fluid was made under static mode. The optimized fluid sample was poured into a calibrated cylinder. Subsequently, 100 mesh sizes of proppants with a loading rate of 1 pounds of proppants added (ppa) were poured gently into the cylinder and the percentage Settling rate over time was determined.

3. RESULTS AND DISCUSSION

3.1 Aqueous Solubility

The bio-polymer (*Grewia*) hydrates and swells slowly in water. The yield of the sample was found to be 32% after extraction from the inner stem bark of the plant. The low yield of the gel is attributed to the insoluble cell wall materials making up a larger proportion of the Sample [3, 15]. The aqueous solubility of the gel is determined as related in equation one (1).

$$\text{Solubility} = (W_s - W_r)/ V \qquad (1)$$

Where W_s = weight of sample
W_r = weight of residue
V = volume of water used

The gel has an aqueous solubility of 0.92 g/L (92%). This indicates that it will leave a lower insoluble residue after breaking when compared to the conventional guar gum with 82 to 85% depending on the mode of extraction of the gum [13]. This will result in a reservoir with better conductivity.

3.2 Functional Groups

The FTIR spectra of the *Grewia* gel is depicted in Figure 2. The gel on hydration has major peaks at 3265 cm^{-1} and 2914 cm^{-1}, the wide peak at 3265 cm^{-1} corresponds to the self-association of the hydroxyl group by hydrogen bonding [4]. The peak obtained at 2914 cm^{-1} is as a result of the stretching modes of the C-H bonds of methyl group which reveals that bonding has happened between water and polymer molecules [3]. Absorption peak around 1599 cm^{-1} depicts the presence of a carboxylate group of the galacturonic acid residues as reported by Okafor and co-workers [15]. However, absorption peaks at 1388 cm^{-1} and 1243 cm^{-1} are typical of acetyl groups. The peaks between 766 and 1028 cm^{-1} represent the fingerprint region for carbohydrates [4]. Therefore, the peak at 1028 cm^{-1} represents the starch content in the *Grewia* sample dissolved in the polymer gel system representing the breaking of the inter-molecular starch bonds upon dilution with water and followed by the formation of the hydrogen bond with water molecules leading to swelling of the polymer [5].

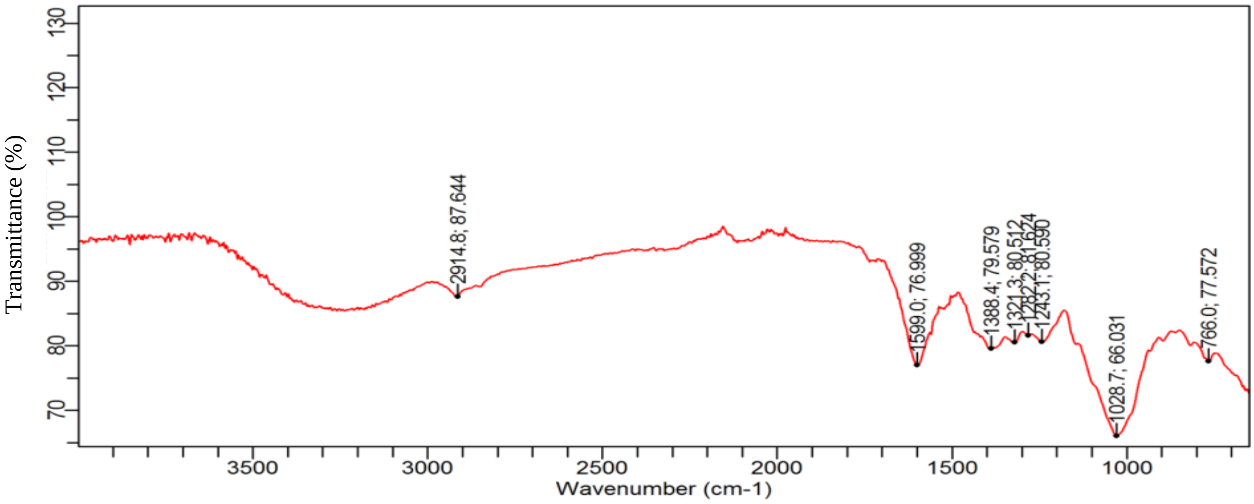

Figure 2*:* FT-IR spectra of *Grewia* gel showing the various absorption peaks

3.3 Thermogravimetric Analysis

The representative plot of the thermogravimetric analysis carried out on the *Grewia* gel sample under a nitrogen atmosphere is shown in Figure 3. From the details of thermal stability data according to the thermogram for the gel, it shows that heating at a rate of 10 ºC per minute from 30 ºC to 890 ºC results in three mass loss events. The first mass loss of about 5%, taking place between 30 to 100 ºC is due to the volatilization of absorbed water by the polymer [16]. The mass loss between 100 – 355 ºC is due to the loss of adsorbed and structural water of biopolymers as related by Elijah and co-workers [3] or due to the desorption of moisture as hydrogen-bound water to the polysaccharide structure [18]. Also, the mass loss event from 400 ºC to 500 ºC which resulted in a mass loss of about 40%, is attributed to the decomposition of the gel. Finally, the mass loss from 500 ºC and above is attributed to the continuous decomposition of the gel sample due to excess heat. The mass loss onset (representing the onset of decomposition) of 370 ºC suggests that *Grewia* polysaccharide gel has good thermal stability.

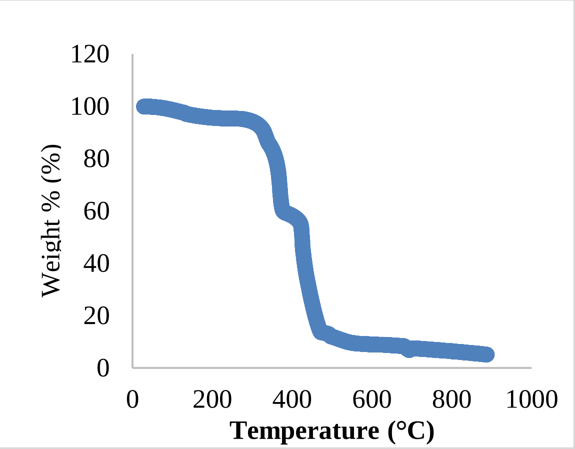

Figure 3: Thermo-Gravimetric plot of *Grewia* gel

3.4 Experimental Design Matrix

Box-Behnken design was used to generate the experimental design. A total of seventeen (17) experimental runs were generated and the result is shown in Table 3. The Box–Behnken model does not contain combinations of all the factors at their highest or lowest values at the same time and it can avoid extreme treatment combinations. The design matrix used in Table 3 was generated based on the levels of factors in Table 2. Effect of the factors on the responses is discussed in the succeeding section below.

Table 3: Input and responses based on Box-Behnken design

Run	Factor A *Grewia* gel	Factor B Cross linker	Factor C pH (pptg)	Response 1 Viscosity (cP)	Response 2 Gel Strength (lb/100ft^2)
1	30	5	9.5	153.4	10.9
2	20	5	11	117	8.5
3	30	6	8	137.2	9.5
4	40	6	9.5	172	11.3
5	40	5	8	149.2	10.3
6	20	4	9.5	118.4	8.8
7	20	5	8	112.3	8.3
8	30	4	8	135.4	9.5
9	30	4	11	136.5	9.5
10	20	6	9.5	120	9
11	30	5	9.5	153.4	10.9
12	30	5	9.5	153.4	10.9
13	30	6	11	138.6	9.7
14	40	5	11	162.6	11.1
15	30	5	9.5	153.4	10.9
16	40	4	9.5	168	11.2
17	30	5	9.5	153.4	10.9

3.5 Effect of Factors on Apparent Viscosity

The effect of the independent variables (pH, gel, and crosslinker concentration) on the Apparent viscosity of the fluid is shown in Figure 4. Figure 4a shows that the gel concentration has a significant effect on the apparent viscosity of the fluid i.e., as the gel concentration increases, the viscosity equally increases while the crosslinker concentration has a minor effect on the fluid viscosity as it remains relatively static with a relatively small decrease in viscosity from 4.2 pptg of the cross-linker and below. From Figure 4b, it is observed that the two major factors that affect the viscosity of the fracturing fluid are gel concentration and pH level. The viscosity of the fluid significantly increases as the gel concentration alongside the pH level.

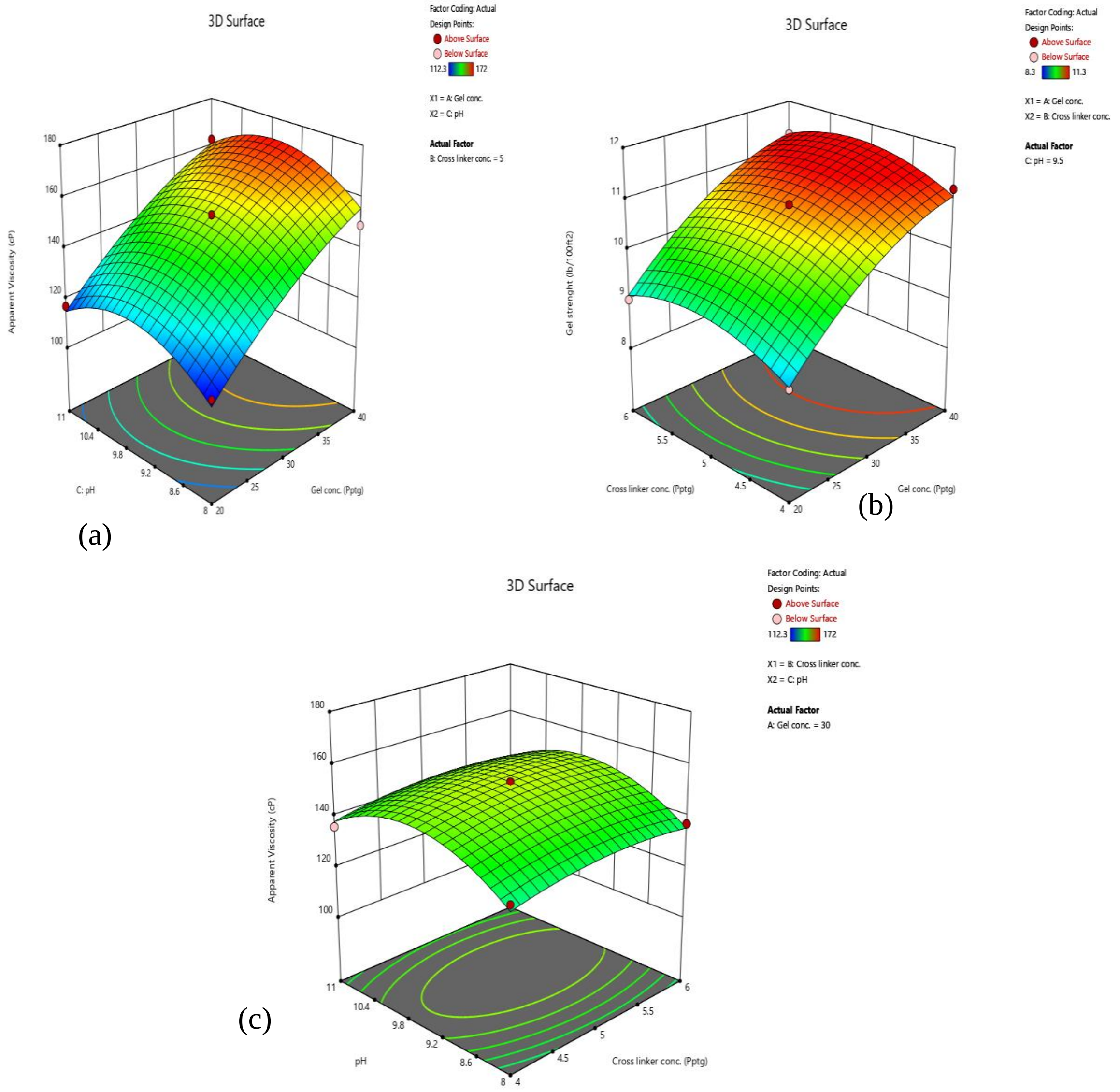

Figure 4: 3D surface curve plot for the effect of (a) pH and gel concentration on viscosity (b) Gel and cross-linker concentration on Viscosity (c) cross-linker concentration and pH on Viscosity

The Model equation for the apparent viscosity of the fluid sample is given in Equation two (2)

$$\text{Apparent viscosity } (\mu) = -595.14 + 5.40 \times A + 37.61 \times B + 111.15 \times C - 0.05 \times A^2 - 3.64 \times B^2 - 5.76 \times C^2 \quad (2)$$

Where A = Gel concentration, B = Cross-linker concentration, and C = pH

3.6 Effect of Factors on Gel Strength

The effect of the independent variables (pH, Gel, and crosslinker concentration) on the gel strength of the fluid is shown in Figure five (5). Figure 5a indicates that the gel concentration has a significant effect on the gel strength of the fluid i.e., as the gel concentration increases, the gel strength equally increases while the crosslinker concentration has a minor effect on the gel strength. Figure 5b depicts that the gel strength of the fracturing fluid is pH sensitive as high values of gel strength are observed between the pH range of 9.2 to 10. At pH values out of this range, the gel strength tends to decrease, Finally, gel concentration has been found to have a significant impact on the gel strength of the fracturing fluid within the pH range of 9.2 – 10. as depicted in Figure 6c

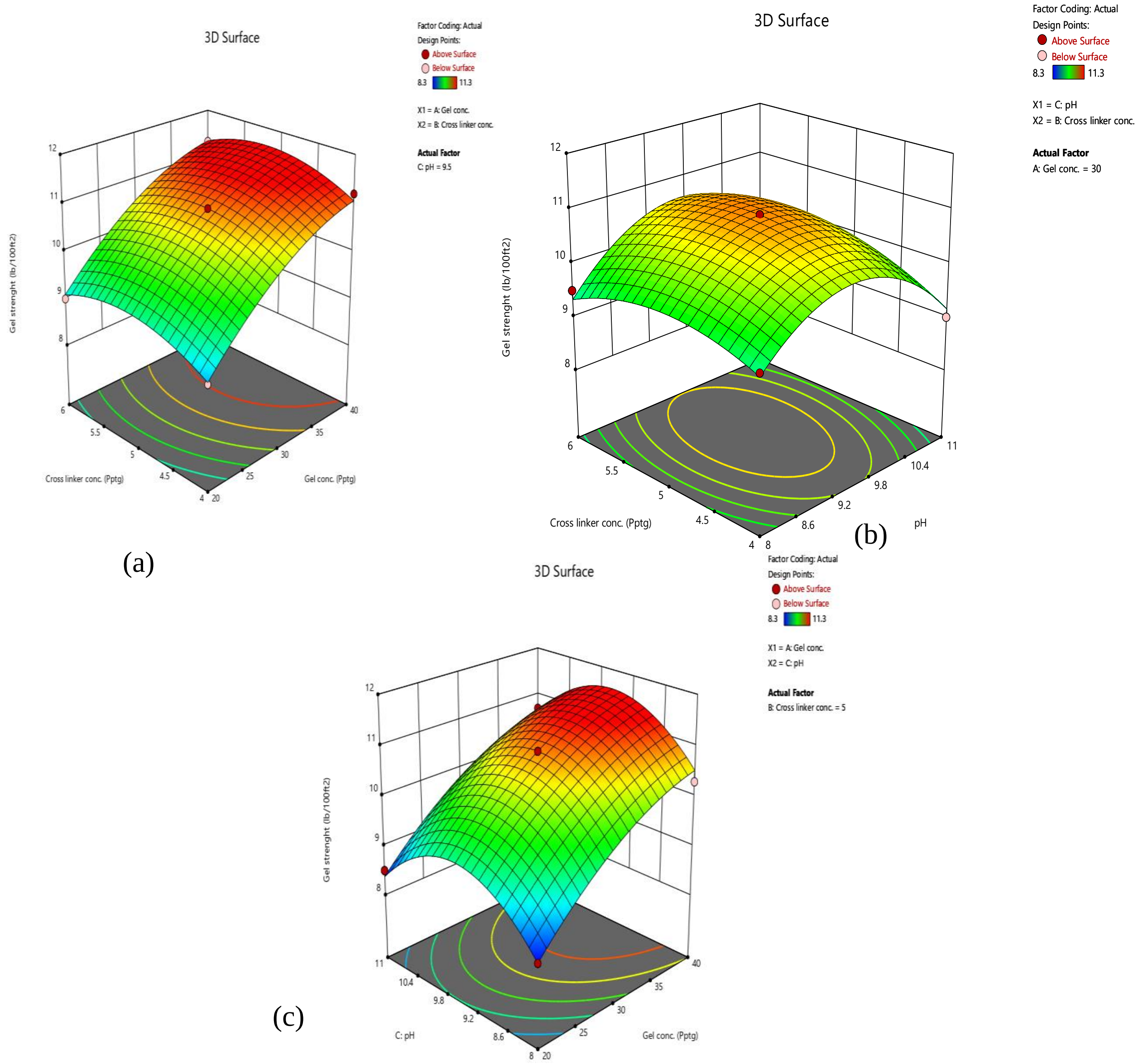

Figure 5: 3D surface curve for the effect of (a) Gel and cross-linker concentration on gel strength; (b) pH and cross-linker concentration on gel strength; (c) Gel concentration and pH on Gel strength

The Model equation for the gel strength of the fluid sample is given in Equation 3

$$\text{Gel Strength} = -44.1 + 0.35 \times A + 3.27 \times B + 8.31 \times C + 0.12 \times B \times C - 0.004 \times A^2 - 0.43 \times B^2 - 0.47 \times C^{2-} \quad (3)$$

Where A = Gel concentration, B = Cross linker Concentration and C = pH

3.7 Optimization of the process

After the experiments were conducted, the experimental results were analysed and subsequently optimized according to some goals (maximizing the apparent viscosity and gel strength, minimizing the gel concentration and finally keeping the cross-linker concentration and pH within range). The model was a significant model with an insignificant lack of fit. It has an F-Value of 88.55 It is also characterized by a predicted R^2 value of 0.7659 which is reasonably in agreement with the adjusted R^2 value of 0.9666. The desirability of the model has been noted as 0.6703 from the contour plot. From the study of the contour plot, the dependent variables of apparent viscosity are 153.429 cP and gel strength of 10.904 lb/100 ft^2. Figure 6 illustrates the contour plot for the desirability from dependent variables at varying gel and crosslinker concentration.

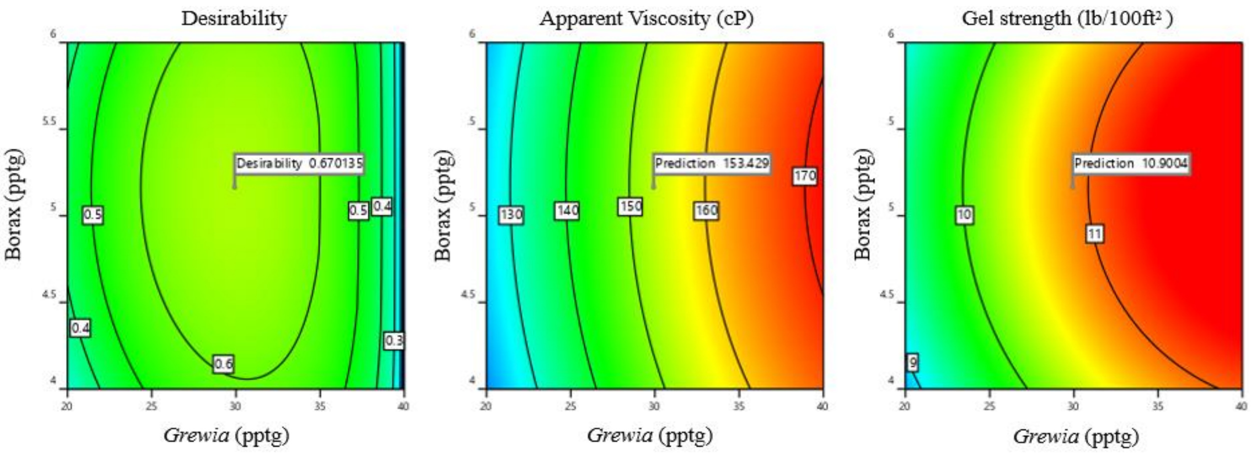

Figure 6: Contour plot for the desirability from dependent variables at varying gel and crosslinker concentration

3.8 Effect of Shear Rate on Apparent Viscosity

The effect of shear rate on the apparent viscosity of the optimized fluid sample can be seen as the viscosity of the optimized gel dispersion decreases with an increase in shear rate. This is indicative of pseudoplastic flow behaviour [7]. At high shear rates, the decrease in viscosity can be attributed to a decreasing number of chain entanglements [10]. However, from the result in Figure 7, the fluid sample can maintain comparable viscosity to guar gum due to the presence of the acetyl group which promotes interactions among polymeric chains and the formation of a gel network [7].

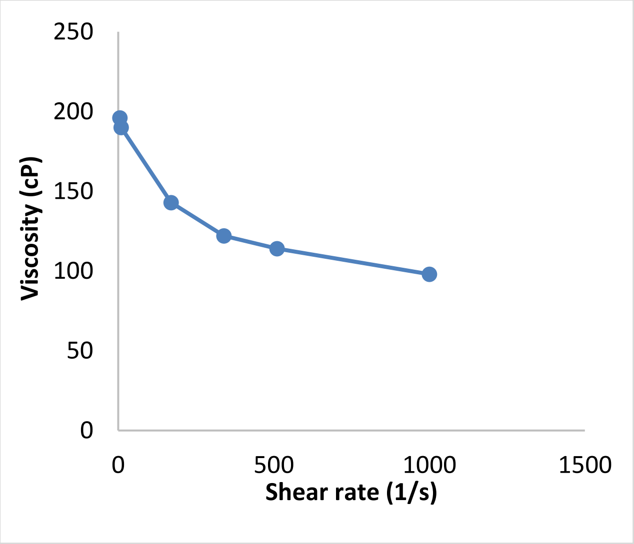

Figure 7: Viscosity variation with shear rate at 40 ºC

3.9 Effect of Temperature on Viscosity

According to the experimental result, at a shear rate of 100 s^{-1}, the viscosity of fluid decreases with a slight increase in temperature (30 ºC to 70 ºC), the increase in temperature results in thermal thinning of the gel samples. However, from the results in Fig. 8, it is evident that the fluid sample can maintain the required 90 cP viscosity beyond 70 °C. The required 90 cP at 100 s^{-1} comes from the rule of thumb applicable for fracturing fluids to suspend proppants and maintain required rheological properties during fracturing operations [1, 5].

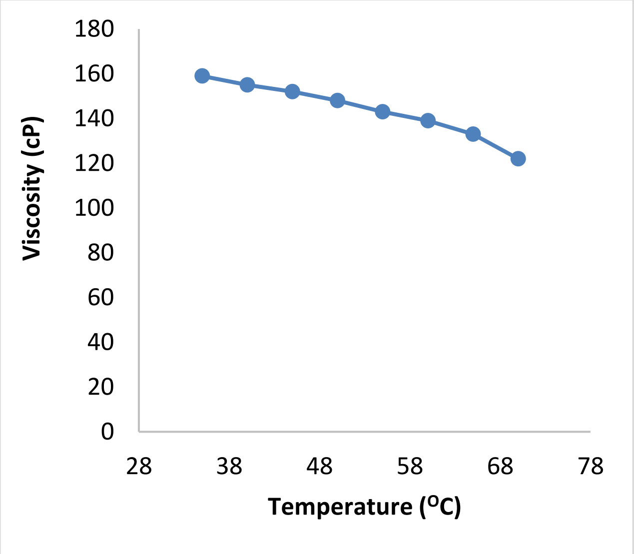

Fig. 8: Viscosity Variation with Temperature at 100 s^{-1}

3.10 Proppant Suspension Test

Figure 9 below illustrates the result of a proppants suspension test of the optimized polymer gel slurry using a load of 1 pound of proppant added (ppa) per gallon of 100 mesh size. The proppants remain suspended for 17 minutes as shown in the first cell. After this time, it then begins to settle as seen by the accumulation of particles at the bottom of the second cell (partial settling where some portions of the proppants have settled at the bottom while some are halfway suspended by the fluid and the rest of the base fluid remains topmost as shown in the second cell. The Blue Zone (base fluid) and orange zone (proppants) observed after 30 minutes in the third cell indicate that the proppants have settled and the upper part of the cell is occupied mainly by the base fluid.

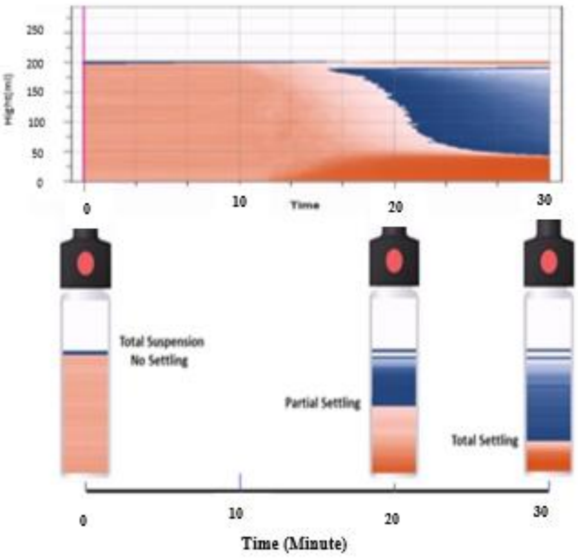

Figure 9: Proppants suspension test of the optimized polymer gel slurry

3. CONCLUSION

The bio-polymer has been extracted from the stem bark of the *Grewia bicolor* plant with a yield of 32% and a solubility of 0.92 g/L. The analysis and optimization of the rheological properties of the gel for minimal operational condition have been investigated using the Response Surface Methodology (RSM). From the analysis of variances (ANOVA), it could be concluded that all dependent variables are significant with $p < 0.05$ and good model fit with high R^2 values (>0.9). The good thermal stability of the gel as evidenced by the high deformation onset of 370 ^{o}C indicates that the gel can be used as a gelling agent even under high temperature condition. Further to this, the fluid has comparable viscosity (122 cP) and gel strength (9.0 lb/100 ft^2) at a shear rate of 100 s^{-1} and temperature of 70 ^{o}C to the conventional guar gum Hence, it can be used as an alternative to guar gum and can compete favourably with other gelling agents in terms of proppant suspension as the proppants remain suspended (No settling) for 17 minutes while total settling of the proppants was noted after 30 minutes.

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APPENDIX A

The solubility of the *Grewia* gel is calculated as shown in the equation below

$$\text{Solubility} = (W_s - W_r) / V \qquad \text{(A-1)}$$

Where

w_s = Weight of sample (w_s) = 0.50 g
w_f = Weight of filter paper = 0.50 g
w_f = Weight of filter + residue = 0.54 g
w_r = Weight of residue = 0.04 g
V = Volume of water used = 500 ml

$$\begin{aligned}\text{Solubility} &= (W_s - W_r) / V \\ &= (0.5\text{-}0.04) / 500\ \text{ml} \\ &= 0.46\ \text{g} / 500\ \text{ml} \\ &= 0.92\ \text{g/L}\ (92\%)\end{aligned}$$