



Application of rice husk and gum Arabic hybrid as loss control additives in hydraulic fracturing fluids

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Abstract

In order to build cracks that will allow the hydrocarbon to flow into the well bore, hydraulic fracturing involves injecting fluid, known as hydraulic fracturing treatment fluid, mixed with additives at a high rate and pressure through well perforations into reservoir formation. By analyzing the effects of process variables like the amount of locally sourced material (rice husk and gum Arabic) and performing comparative analysis between the developed and API standard hydraulic fracturing fluids, the optimal values of the responses (filter loss volume, filter cake thickness, filter cake permeability, and pH) were determined at the end of the thesis. A combination of analytical techniques of X-ray fluorescence (XRF) and Atomic absorption spectrum (AAS) were employed to characterise Rice Husk and Gum Arabic samples. From the XRF analysis, the most abundant oxides in both samples are SiO₂ and Al₂O₃. The silicon oxide (SiO₂) was 71.70%, alumina (Al₂O₃) was 3.910%, iron (Fe₂O₃) was 1.860% in rice husk with (SiO₂) 33.00%, (Al₂O₃) 15.00% (Fe₂O₃) 9.80% obtained in gum Arabic. The AAS analysis indicates an insignificant amount of heavy metals for both samples. In rice husk, nickel has the highest trace with 0.058 %, while in gum Arabic; the trace was recorded in arsenic with 0.089%. The research was conducted based on the Box-Behnken design of experiment, a feature of the Response Surface Methodology (RSM). Half-hourly sampling was carried out for the determination of the dependent variable. In addition, an amount of zirconium (14-86g) was added to serve as a cross-linker. At the end of the experiment, result validation was carried out after optimisation of the responses and comparison of the result using API standards. The results show that rice husk and gum Arabic improved the filtration characteristics by 71% and 33% respectively, suggesting their applicability to be used as an excellent fluid loss control agent with gum Arabic addition resulted in an enhanced viscosity performance.

Keywords: Petroleum engineering, hydraulic fracturing, locally sourced materials, fluid loss control additives, fluid loss volume, filter cake thickness, filter cake permeability, pH.

Introduction

Nowadays petroleum products contribute to a higher percentage of the world energy supplies (Chala *et al.*, 2018). Following the drilling of the oil well to the depth of interest where hydrocarbon found, well needs to be returned to production to get cash flow from extracted black gold and compensate the drilling cost through production. Some wells deliver hydrocarbons easily and others do not, for which treatment called well stimulation is vital (Kurison *et al.*, 2019). The rock formation producing hydrocarbons has in nature bulk of rock with spaces or voids between rock grains (Liu *et al.*, 2020). The voids in the reservoir store some hydrocarbons in the form of liquid/gas phases. In several cases, production of hydrocarbons is hampered by obstacles known as formation damage in petroleum industry. This damage blocks voids that are close to the wellbore section. In this line, hydraulic fracturing stimulation helps to bypass the damaged region and induce new paths to succour the flow of hydrocarbons from rock formation to wellbore to expedite well production (Rigzone, 2020; Nyugen *et al.*, 2020). Hydraulic fracturing is a technique of injecting fluid mixed with additives called hydraulic fracturing treatment fluid at both high rate and pressure through well perforation inside reservoir formation to create paths/cracks for hydrocarbons to flow again into wellbore (Huerta *et al.*, 2020; Bai *et al.*, 2020; Chala *et al.*, 2018; Zeng *et al.*, 2020). The paths should have large

contact area inside the reservoir formation to be distributed in the formation matrix to drain the remaining hydrocarbons efficiently. After the stoppage of injecting hydraulic fracturing fluid, the initiated cracks tend to close as the injection pressure drops, leading to lowering the permeability. In this case, a material called proppant is injected with the treatment to hold cracks and remain them open.

Both types of hydraulic fracturing either water-based, oil based, foamed fluid or polymer free fluids, would have similar aim of creating better fracturing conductivity inside the formation for better well productivity. For Acid hydraulic fracturing, the acid is mixed with viscous fluid normally to be obtained by mixing water with certain amount of polymer and this is injected in formation with a pressure higher than formation fracturing pressure, which lets the fluid leak inside the formation and start etching process. The etched surface should not be smooth and soft so that it remains open after formation is closed due to release of injected pressure (Li *et al.*, 2020).

Materials and Methods

1. Sample preparation

The rice content of the rice husk, which was sourced from a mill in Bauchi Local Government area of Bauchi State, was separated from the husk by spreading it out on a sieve. After that, to remove any remaining moisture, the rice husk was

heated to 40°C in a vacuum oven for 5 hours. To get fine particles, the dry ingredient (rice husk) was grounded into fine particles and sieved through sieve No. 125µm in order to prevent contamination, this sample was kept in airtight containers. Gum Arabic (Acacia Senegalese) was obtained from a local market in Alkaleri local government area of Bauchi State. The gum Arabic (Acacia Senegalese) was dried in an oven at a temperature of 100°C and crushed to fine particles using a rock crushing machine. Distilled water was used to dissolve gum Arabic and sieved to remove coarse particles.

2. Hydraulic fracturing fluid preparation

Seventeen (17) fracturing fluid samples were prepared (formulated) via Box-behnken design and statistical tool. Details of the data obtained is as shown in table 6. The experiment was designed using the response surface methodology (RSM), the experimental factors were, zirconium as a crosslinker and hybrid mixture of Rice husk and Gum Arabic as (additives) and the levels are Lower(-), Middle (0) and the Upper(+). The experimental responses are fluid loss volume; filter cake thickness, pH and filter cake permeability. The detailed experimental design is presented in Table 6 respectively.

Table 1: Experimental factors and level of variables

Factor(s)	Level of Variables		
	Lower (-)	Middle (0)	Upper (+)
Zirconium (g)	14	50	86
Rice husk (g)	16	66	76
Gum Arabic (g)	16	66	76

3. Filtration Test Procedure

The API suggested practice, API 13B-1, served as the foundation for the filtering test to assess the locally obtained material's ability to control fluid loss. The standard advises performing the lower temperature lower pressure (LTLP) filtration test in the water-based fracturing fluid at 100 psi (about 700 kPa) pressure for 30 minutes (i.e., 1800 s). The LTLP filter press used in this study was a cylindrical cell with a 3 inch internal diameter and a 5 inch height that is used to hold the fracturing fluids.

3.1 Fracturing fluid filter cake characteristics

According to API, fracturing fluid filter cake has the following qualities: it is solid, slippery, smooth, soft, sticky, etc (Agwu *et al.*, 2019). Therefore, based on API description, the qualitative properties of the hydraulic fracturing fluid cake were established from the appearance and texture of the filter cakes of the formed fracturing fluid samples.

3.2 Filter Cake Permeability

Both static and dynamic filtration are controlled by the mud filter cake's permeability (Belhaj, 2021) [4]. It determines if the mud cake that formed around the wellbore during the hydraulic fracturing procedure is permissible or transmissible. Filter cake permeability indicates, whether the created mud cake can either block or permit fluid (i.e., mud filtrate) to travel through it and into the formation, it is often represented by;

$$K = q_w q_c \frac{\mu}{2\tau\Delta} A^2 \tag{1}$$

Where

K = mud cake permeability (Darcies, D)

q_w = mud filtrate (fluid loss) volume (cubic centimeter, cm³)

q_c = mud cake volume (centipoise, cP)

μ = mud filtrate viscosity (centipoise, cP)

t = time (seconds)

Δp = pressure differential (atmospheres)

A^2 = filter cake area (centimeter, cm³) But, using conventional filter press data reported by Reutela (2019) presented in equation (1) is given by;

$$K = 19.9 q_w q_c \mu \tag{2}$$

Expressed Eq. (1), but regrettably that, the drawback of Eqs. (1) and established by Lomba (2020) in Agwu *etal.*(2019) was used to evaluate the formulated hydraulic mud samples, and cake permeability. Thus;

$$K = 8.95 \times 10^{-5} q_w \epsilon \mu \tag{3}$$

where ϵ is the filter cake thickness and q_w is the filtrate volume; all measured in millimeter (mm) while μ can be determined from equation 2.

3.3 XRF Procedure for Gum Arabic Analysis

The gum Arabic was first dried in the oven after selection at a temperature of 100°C to allow it to be crushed to a fine particle using a crushing machine to reduce its size. The crushed particles were then turned into powdered particles using Mortar and pestle. The powdered particle was dissolved into a molten glass forming flux at high temperature for homogeneity. A ratio of 1 part gum to 2 parts water was used (distilled water) and allowed to cool to 140°F (60°C) and the gum Arabic was added, stirred to make sure there was no lumps. The mixture was continuously warmed at this temperature until the gum Arabic dissolved completely. The sample holders containing the sample which were run in a vacuum or air for 10 minutes and they were inserted into the XRF spectrometers for the elemental oxide composition analysis

Results and discussion

Based on the API specifications for the common polymeric materials: CMC and PAC utilised in the industry, the performance of the locally available materials as fluid loss control additives in water-based fracturing fluid was assessed. Table 3 provides these parameters. Filter loss volume, filter cake thickness, and mud cake permeability were the hydraulic fracturing fluid filtration parameters that were assessed to determine the efficacy of the locally available materials as fluid loss control additives.

1. X-Ray Fluorescence (XRF) results

1.2 XRF Result of rice husk

The XRF analysis was conducted to determine the elemental oxides composition of the rice husk. The XRF result for the rice husk samples is shown in table 4

Table 2: XRF analysis results of rice husk sample

No.	Component	Result (mass %)	API Standard
1.	Na ₂ O	0.880	1.00
2.	MgO	2.220	3.0-3.3
3.	Al ₂ O ₃	3.190	4.5-5.0
4.	SiO ₂	71.70	44.0-50
5.	P ₂ O ₅	11.20	20.5-21.0
6.	K ₂ O	5.210	5.0-6.0
7.	CaO	1.810	2.00
8.	MnO	0.254	1.00
9.	Fe ₂ O ₃	1.860	3.00
Total			98.324

The chemical composition of rice husk showed better cementing potential when compared to conventional bentonite. The potential of a material to control fluid loss is dependent on the amount of silicon oxide composition of the sample. The silicon oxide in rice husk from the XRF analysis showed a composition of 71.70% which is higher when compared with the 48.16% composition of silicon oxide of bentonite from Ashaka Cement Company in Gombe state, Nigeria.

2. XRF results of Gum Arabic

The XRF analysis was conducted to determine the elemental oxides composition of the gum Arabic.

Table 3: XRF results of Gum Arabic

No.	Component	Result (mass %)	API Standard	e
1.	Na ₂ O	2.210		
2.	MgO	4.280		
3.	Al ₂ O ₃	15.00		
4.	SiO ₂	33.00		
5.	P ₂ O ₅	1.470		
6.	K ₂ O	10.20		
7.	CaO	20.60		
8.	MnO	2.360		
9.	Fe ₂ O ₃	9.800		
Total		98.92		

The chemical composition of gum Arabic showed 33.0% of SiO₂ which imply a good cementing potential. The potential of a material to control fluid loss and transport proppants is dependent on the amount of silicon oxide composition of the sample and the sodium oxide composition present. The sodium oxide in gum Arabic from the XRF analysis showed a composition of 2.210% which shows a significant amount to be considered as a viscosifier.

Table 7: AAS results for rice husk

No	Element	Concentrations in (mg/L)	Maximum permissible limit (mg/L) by (WHO) 2021
1	Ni	0.058	2.00
2	Mn	0.016	0.05
3	As	0.037	0.05
4	Ag	0.131	1.00
5	Cd	0.056	0.003
6	Cr	0.036	0.05
7	Cu	0.149	2.50
8	Co	0.027	1.00
9	Pb	0.047	0.01
10	Hg	0.035	0.01

From the atomic absorption spectroscopy (AAS) analysis, insignificant amounts of the heavy metals were detected in

Table 4: Result of analysis using Box-behnken design and statistical tool

Std	Run	ZC	RH	GA	FLV	FCT	FCP	pH
		G	G	g	mL	Mm	mD	-
15	1	14	42.865	71.43	13.70	1.87	2.5	9
11	2	86	42.865	14.3	13.40	2.36	2.94	9.5
17	3	50	42.865	42.865	13.10	1.4	1.63	8.5
8	4	50	71.43	14.3	12.30	1.55	1.76	7.6
4	5	50	14.30	71.43	8.30	1.22	0.92	8.5
14	6	14	14.30	42.865	28.10	1.68	4.48	8.5
1	7	50	14.30	14.3	15.10	1.4	1.97	5.7
5	8	50	71.43	71.43	11.50	1.52	1.61	7.5
10	9	14	71.43	42.865	13.10	1.8	2.15	8.5
6	10	86	14.30	42.865	12.50	1.6	1.84	6.5
16	11	50	42.865	42.865	29.60	1.71	5.3	9.5
7	12	86	71.43	42.865	14.10	1.52	2.05	6.8
3	13	50	42.865	42.865	13.07	1.52	1.9	7.6
9	14	50	42.865	42.865	13.10	1.55	1.93	7.8
13	15	14	42.865	14.3	14.00	1.57	1.99	6.5
12	16	50	42.865	42.865	12.70	2.51	2.94	8.5
2	17	86	42.865	71.43	11.00	1.39	1.64	8.5

Table 5: Optimum values for the factors and the responses Sixty five solution were found and the optimum condition was selected by the software.

ZC	RH	GA	FLV	FCT	FCP	pH
14.00	42.865	65.860	12.614	1.655	2.035	6.556

The selected optimum values obtained were used for the validation of the responses. Table 9 shows the validated results of the responses after optimisation of the responses.

Table 6: Validated result after optimisations

ZC	RH	GA	FLV	FCT	FCP	pH
14.00	42.865	65.860	13.14	1.69	2.10	7.50

Percentage error is obtained using the relationship as

$$\frac{\text{actual value} - \text{predicted value}}{\text{actual value}} \tag{4}$$

Equation (4) used to calculate the percentage error obtained after validation of the responses.

Table 6: Percentage errors calculated

ZC	RH	GA	FLV	FCT	FCP	pH
0	0	0	4.003%	2.07%	3.09%	12.60%

Atomic Absorption Spectroscopy (AAS) results

1. AAS results for Rice husk

The AAS result of the rice husk is shown in table 11

the rice husk sample, while Copper has the highest trace with a composition of 0.149% followed by Gold with a

composition of 0.131% which are all within the permissible limit recommended by WHO (2021). Manganese has the least composition of 0.016%, which is also within the WHO (2021) recommendation. In view of this, the sample can be as considered safe for applications in the oil and gas industry as compatible additives in hydraulic fracturing.

Table 8: AAS results of Gum Arabic

No	Element	Concentrations in (mg/L)	Maximum permissible limit (mg/L) by (WHO) 2021
1	Ni	0.024	2.00
2	Mn	0.057	0.05
3	As	0.087	0.05
4	Ag	0.061	1.00
5	Cd	0.040	0.003
6	Cr	0.014	0.05
7	Cu	0.78	2.5
8	Co	0.021	1.00
9	Pb	0.043	0.01
10	Hg	0.007	0.01

From the atomic absorption spectroscopy (AAS) analysis, insignificant amounts of the heavy metals were detected in the gum Arabic sample. In gum Arabic, copper has the highest trace with 0.78% composition followed by arsenic with 0.087% composition. While the percentage composition of copper is within the acceptable limits recommended by WHO (2021), the composition of Arsenic is beyond the recommended limit for the metal. Mercury has the least composition of 0.007%, which is within the WHO (2021) recommendation. In view of this, the sample is considered safe for applications in the oil and gas industry as compatible additives in hydraulic fracturing.

6. Fluid loss results

The fluid loss test was conducted to determine the amount of fluid that was lost after the filter cake was developed. Excessive fluid loss prevent fracture propagation because of insufficient fluid volume accumulation in the fracture. The fluid loss volume for the hybrid mixture of rice husk and gum Arabic additives is showed in Figure 1, according to the experimental data, run 2 in Table 6 showed that, a fluid loss value of 12.70 mL was noted for rice husk at a concentration of 42.865 g/mL .When the same result for run 2 is compared with that of run 5 with 71.43 g/mL concentration of rice husk (about 40% increase in concentration), the value of fluid loss volume was 11.50 mL indicating a 9.45% decrease in fluid loss volume. This showed that with increase in rice husk concentrations, fluid loss of the fracturing fluid is reduced. The fluid loss volume was reduced from 12.70 mL in run 2 with 50 g/mL of zirconium to 11.00 mL when the concentration of zirconium was increased to 86 g/mL with a constant concentration of rice husk at 42.865 g/mL in run 7, as can be seen from the results in table 6. However, the concentration of zirconium as a cross-linker also contributes to reducing the fluid loss volume. The fluid loss volume of 12.30 mL in run 9 when compared with the fluid loss volume of 11.50 mL in run 8 at constant concentrations of Rice husk and zirconium show the influence of Gum Arabic in the reduction of fluid loss,as Gum Arabic concentration increases from 14.30 g/mL to 71.43 g/mL

Filter cake thickness

Fig 2: Filter cake thickness for the hydraulic fracturing fluids samples at more varying contents of the control additives at a given room temperature.

The result in Table 6 shows that filter cake thickness formed by fracturing fluid decreases as the concentration of gum Arabic increases as observed from run 2 and run 9. Result from run 4 and 5 indicate that filter cake thickness decreases with decrease in concentration of rice husk. Likewise, the filter cake thickness decreases with decrease in concentration of zirconium as a cross-linker in the fracturing fluid as recorded in run 1 and run 6

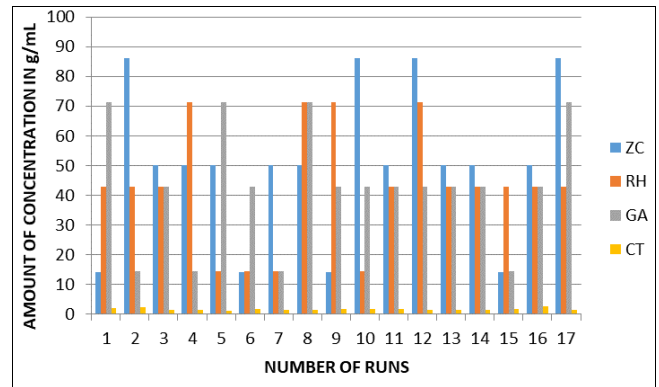


Fig 1: Filter cake thickness (mm)

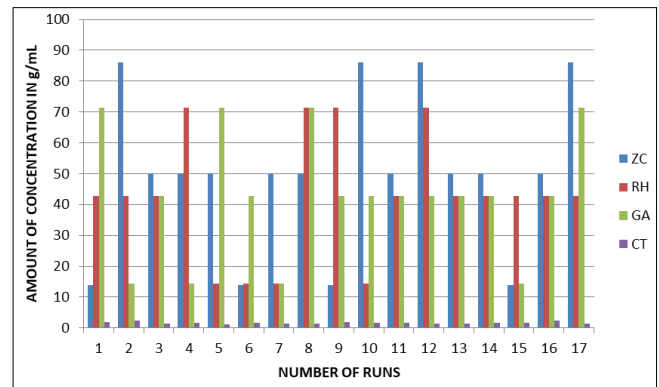


Fig 2: Filter cake thickness (mm)

8 Filter cake Permeability result

8.1 Filter cake permeability

Figure 3: Filter cake permeability for the hydraulic fracturing fluids samples at varying composite contents of the control additives at a room temperature.

From the result in Table 6, the relationship between the filter cake permeability (FCP) and fluid loss volume (FLV) values is linear such that, the filter cake permeability is a measure of the fluid loss through the medium. An average fluid viscosity of 1.12cP and 1.70cP for gum Arabic and a hybrid mixture of gum Arabic and rice husks, respectively, was determined from the filter cake permeability findings obtained for the various fracturing fluid samples. As the additive level in the fracture sample increases, it is shown that the filter cake permeability values drop. This finding explains why low additive content in the hydraulic fracturing fluid resulted in low filter cake thickness values and high filter loss volume values. In order to further evaluate the locally sourced material, it is important to determine the fluid loss control additive

content and filter cake permeability based on the results of the static filtering test.

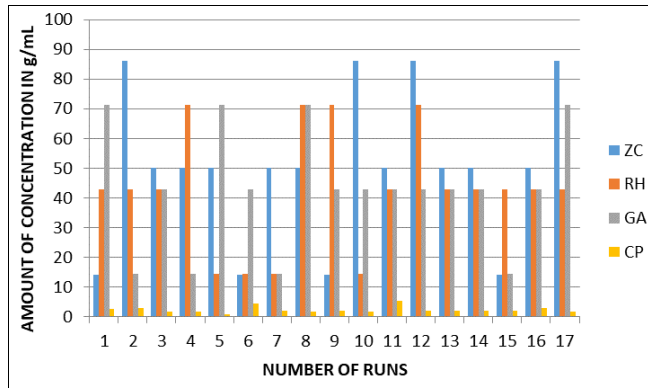


Fig 3: Filter cake permeability (mD)

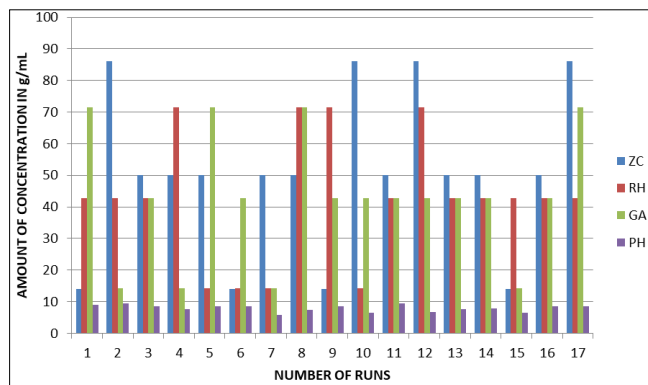


Fig 4: Fluid control additives against pH values

For successful hydraulic fracturing operations, fracturing fluids “the blood of the operation” is expected to performed critical functions. One of these functions is to seal the wall of the formation being fractured to prevent filtrations.

pH value results

pH value

Figure 4: pH for the hydraulic fracturing fluids samples at varying contents of the control additives at a given room temperature.

From the result in Table 6, rice husk has influence on the pH of the fluid as it can be seen that the pH of the experimental run is almost alkaline all through and rice husk is the only alkaline factor used in the experiment. Fourteen (14) out of the seventeen (17) runs show alkaline value for pH, Ranging from (8-9.5) while only three (3) runs indicate acidic values for pH, indicating a superior influence of rice husk in the composite mixture.

Conclusions

1. The two agro - based materials were characterised by XRF and AAS techniques.
- a. It has been observed that, from XRF analysis, the gum Arabic increased the viscosity and viscoelasticity of the hydraulic fracturing fluids while the rice husk increased the ability to control fluid loss due to high percentage of silicon oxide present SiO₂ (> 71.70 %) with 33.0% recorded in gum Arabic.
- b. It has also been observed that, from (AAS) analysis, most of the heavy metals detected from the two (2) samples (rice husk and gum Arabic) were within the

maximum permissible limit set by (WHO) in water, (eg) Ni with 0.024 mg/L obtained from the analysis has a maximum permissible limit of 2.0 mg/L set by (WHO) 2021, while Cu was 0.078mg/L and the maximum limit is 2.50 mg/L.

2. API standard hydraulic fracturing fluid using locally source materials i.e. (rice husk and gum Arabic was formulated. The fluid loss control capability of the proposed additives was noticeably increased when the concentration of rice husk increased from 14 g to 60 g while fluid loss volume decreased linearly from 25 ml to 13 ml respectively.
3. An optimum value of the responses such as fluid loss volume (12.614ml), filter cake thickness (1.655mm), filter cake permeability (2.035mD) and pH (6.6) was obtained best on the Response Surface Methodology (RSM) experimental design.
4. Comparative analysis between the formulated additives and the commercial additives was done using API standard method. The API standard values for the responses such as fluid loss volume (25 mg/L), filter cake thickness (2.0 mm), filter cake permeability (5.0 mD) and pH all fall within the standard specification.

Recommendation

Further work is recommended to establish the thermal stability of rice husk as filtration (Fluid loss) control additives and gum Arabic as viscosifier using dynamic conditions. However, a further study on the effect of temperature on the formulated hydraulic fracturing fluids will help assess the performance of rice husk and gum Arabic at high temperature and high pressure conditions.

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