

PRELIMINARY EVALUATION OF CASHEW GUM EXUDATE AS A GREEN SCALE INHIBITOR

ABSTRACT

This preliminary study explores the potential of cashew gum as a sustainable, effective inhibitor of calcium carbonate scale by characterization analysis to determine its composition, temperature-tolerance and crystallinity in relation to conventional scale inhibitors. The use of green materials has garnered attention as a promising natural compound for industrial processes. Scale formation, a pervasive issue in oil production leads to reduced efficiency and increased maintenance costs caused by blockages in pipelines etc. However, scale inhibitors have been used to control various scale types. Cashew gum, a natural polysaccharide, with its biodegradable and eco-friendly characteristics aligns with global emphasis on sustainable, green chemistry. The preliminary analysis of the scale-inhibitory effect of cashew gum was evaluated using Thermogravimetric analysis (TGA), X-ray diffraction (XRD), Fourier-transform-infrared spectroscopy (FTIR), and Gas chromatography-mass spectrometry (GC-MS) to analyze their crystal structure, thermal stability, identify and quantify the chemical compounds. Results showed an onset degradation at 327.3 °C temperature with a 29% decline in mass of the sample and a calcination temperature of 525 °C. The XRD showed a single peak at 19.20° indicating poor crystallinity of the extract, thus a mixture of crystalline and amorphous phases was proposed. The FTIR spectra showed a symmetrical stretching vibration of the O-H bond, characteristic of glucoside ring. The presence of C-O bonds and carbonyl moiety was observed at several low peak intensities indicating a low composition of these functional groups. The chromatogram identified six compounds linked to a functional group with 7-octadecenoic acid methyl ester having the highest peak area of 63.52 %, indicating that fatty acids are the dominant constituents in cashew gum. Its compatibility with diverse water compositions makes it a potential solution for scale inhibition. The study shows that cashew gum is a viable, eco-conscious option for mitigating scales based on its chemical composition and thermal stability.

Keywords: Calcium carbonate, Scale formation, FTIR, cashew gum, green scale inhibitor, TGA

1. INTRODUCTION

Over the years, the oil industry has remained plagued by the severe problems resulting from associated scale formation such as flow assurance issues, formation damage, pressure reductions, equipment damage, loss of energy, time, cost, and production. Scale is a solid layer attached to

equipment surfaces and piping systems through a process called scaling or encrustation, in which some materials, originally dissolved in fluids, are deposited on these surfaces (Hoang, 2015). The term “scale” widely describes a dense solid deposit that grows over time inhibiting the flow of fluids in pipelines, valves etc. resulting in significant damage to such equipment and a decline in the rate of production (Wayne, 2008). Scales are formed during several industrial processes such as crystallization, distillation, dilution, cooling, or heating of liquids and improved oil recovery. Zhu *et al.* (2018) noted that scales could be formed anywhere along surface equipment such as surface water injection facility, production wells and facilities, tubing resulting in severe technical issues such as declined production capacity, increased corrosion, blockage on flow lines and equipment which will negatively affect the overall cost of the process. Chaussemier *et al.* (2015) stated that the related costs of scaling is estimated at billions of dollars annually in developed industrialized nations. Bin Merdhah *et al.* (2010) mentioned three mechanisms that can lead to scale deposition in offshore and onshore environment. These include the blending of incompatible waters, temperature and pressure changes, and evaporation of formation brine. Scales deposited due to water incompatibility take place when two different waters, such as seawater (used as injection water during oil recovery) and formation water from the reservoir, are mixed. This causes oversaturation of scale components in the produced water due to mineral precipitation. Seawater, with its high sulfate ion concentration, combines with formation water high in calcium and barium ions, leading to the precipitation of sulfate scales such as barium sulfate and calcium sulfate (Olajire, 2015; Bin Merdhah *et al.* 2010). Amiri and Moghadasi (2012) in their study stated that calcium carbonate scales found in oilfield operations are formed from the combination of calcium and bicarbonate ions. This is because small percentage of bicarbonate ions is dissociated at the pH values found in most injection waters to form H^+ and

CO_3^{2-} . The studies revealed that carbonate scales are also formed when connate or aquifer water passes through the bubble point and carbon dioxide is evolved, resulting in a reduced solubility and subsequent precipitation with divalent ions, such as iron and calcium.

Numerous research endeavors have suggested various approaches to alleviate formation damage induced by scales, aiming to enhance the productivity and injectivity of oil and gas wells. Among these, scale inhibition has emerged as the favored downhole treatment method for preventing or managing scale formation and deposition. Scale inhibition entails chemically preventing the hindrance of fluid flow by scale formation (Alzahrani *et al.* 2014). This treatment is employed to control or minimize the occurrence of scales within a well.

This is accomplished through the adsorption of scale inhibitor on the crystalline surface, thereby disrupting the continued growth of the crystal and the deposition of minerals that form scales. In certain instances, scale inhibitors hinder the adherence of scale crystals to solid surfaces like piping. Effective scale inhibitors are imperative for preventing the formation and deposition of scales. Conventional scale inhibitors are hydrophilic, meaning they dissolve in water. According to Kelland (2009), the most commonly used commercial scale inhibitor chemicals in the oil industry include inorganic phosphates, organophosphorous compounds, and organic polymers, particularly PPCA (Poly-phosphono-carboxylic-acid) and DETPMP (diethylene-triamine-penta-methylene-phosphonic-acid). These conventional scale inhibitors often consist of synthetic chemicals that are highly toxic and non-biodegradable. Hence, the growing interest in exploring natural, bio-based, eco-friendly alternatives as scale inhibitors.

Green scale inhibitors are natural-based inhibitors that exhibit voluntary biodegradation, demonstrate high efficacy, with zero toxicity level (Jing and Tang, 2011). The use of cationic polymers as a green scale inhibitor has recently been the subject of numerous inhibition studies.

Other novel promising green scale inhibitors include poly-maleates (PMA), poly-aspartic acid (PASP), and poly-epoxy-succinates (PESA), as well as their various derivatives including copolymers with polyacrylates (PA) owing to their exceptional efficacy against sulfate and carbonate scales of calcium and their eco-friendly properties. In analyzing the various factors that influence scale inhibition, Hoang (2015) stated that retention time and chemical structures have an inhibitory effect with a significant reduction in the scale inhibitory efficiency with respect to time. Kamal *et al.* (2018) also added that pH sensitivity, temperature, and concentration of the inhibitor have an effect on the efficacy of the scale inhibitors. They noted that as pH value increases from 3 to 8, the efficiency of the scale inhibitor increases, preventing calcium carbonate precipitation or deposition. They further noted that higher temperatures reduce the efficacy of scale inhibitors because high temperatures reduce the solubility of calcium carbonate scales and accelerate scale formation thus requiring higher concentration of the inhibitor or more potent inhibitor. However, there's a need to develop fit-for-purpose green scale inhibitors in order to attain a balance between efficiency and eco-friendly option.

Cashew gum exudate is a natural polysaccharide obtained from the exudate of the cashew tree (*Anacardium occidentale*). It is composed mainly of galactose, arabinose, and glucuronic acid units. Cashew gum exudate has drawn attention as a potential green alternative in various fields due to its biodegradability, non-toxicity, and renewable nature. Studies have demonstrated its potential applications in food, pharmaceutical, and cosmetic industries. This **original research paper** aims to investigate the use of cashew gum exudate as a potential green scale inhibitor for calcium carbonate scales by conducting preliminary assessment of its chemical composition, crystalline structure, thermal stability over time, pH, and compatibility tests to assess the effects of calcite scale deposition in the absence and presence of cashew gum exudate.

2. MATERIALS AND METHODS

2.1. Materials

The equipment and reagents used in this study include beakers, glass rod, pyrometer bottle, 75 μ m mesh sieve, funnel, weighing balance, digital roto-viscometer, electric oven, pH meter (HI9813-6), blender, ethanol ($\text{CH}_3\text{CH}_2\text{OH}$), distilled water, chloroform (CHCl_3), Diethyl ether (CH_3CH_2) $_2\text{O}$, Acetone, and cashew gum exudate.

2.2. Methods

Extraction of cashew gum exudate

Cashew gum exudate (CGE) used in this research work was obtained from the bark of an incised Cashew Gum trees found within the University of Port Harcourt, Choba, Rivers state, Nigeria with coordinates 4.9070°N, 6.9162°E. The gum exudates were obtained by creating an incision into the cashew tree which was then allowed to settle for seven days after which the gum exudate was formed. The gum exudate was handpicked to ensure the absence of any impurities (possibly from the bark of the cashew tree), sun-dried, and pulverized using a blender. Thereafter, the exudate was dehydrated in a chloroform-water mixture (ratio 70:30) over five days, then filtered through a 75-micron mesh to acquire a particulate-free slurry, which was left to settle. Subsequently, the gum was extracted from the slurry using pure ethanol, filtered, and defatted with diethyl ether. The resulting precipitate was then dried again at 40°C for 4 days, then pulverized and stored in an airtight container.

Characterization Studies

pH value

For determination of pH, 10 grams of the gum exudate was weighed and introduced into deionized water to obtain a 50 ml solution. This was made up to prepare the required solution and subsequently its pH was determined with the aid of the pH meter (HI9813-6) Hanna Instrument USA. The probe was dipped into the solution sample and the measured pH value was displayed on the led screen of the pH meter.

Specific gravity and density

To determine the specific gravity and density of the sample, the pycnometer bottle underwent washing, thorough drying, and subsequent weighing. It was then filled with water and weighed again. Afterwards, the water was drained out of the bottle, re-filled with gum exudate, and weighed once more.

The obtained values were inputted and calculated using equations 1 and 2.

$$\text{Specific Gravity} = \frac{\text{weight of 50 ml of oil}}{\text{weight of 50 ml of water}} \quad (1)$$

$$\text{Density (g/l)} = \frac{\text{weight of 50ml of oil}}{\text{Volume of 50ml of oil}} \quad (2)$$

Viscosity

This was measured using a digital roto-viscometer. The gum exudate was put into a 50 ml container of the instrument. The exudate was properly stirred using a stir rod, while laboratory temperature was maintained. Subsequently, the container carrying the sample was positioned on a processing ring on top of the turntable, and the lever was lowered to activate the motor automatically. The disc was allowed to rotate till it attained a steady state, typically within 5

minutes. Once the required time had elapsed, the viscosity was recorded in millipascal seconds (mPas). Triad measurements were conducted on the sample, and the mean value was calculated and documented.

Compatibility

The compatibility between the gum exudate and brine containing divalent ions; calcium carbonate was analyzed at varied temperatures of 27 °C and 100 °C for 7 days.

Solubility

Solubility denotes the highest quantity of a substance that can dissolve in a solvent at a specific temperature, typically measured in grams per 100 grams of solvent. A 0.5g portion of the sample was introduced into 100 ml of distilled water and thoroughly mixed using a magnetic stirrer. Following this, the solubility of the sample was visually assessed.

This quantity continues in 0.5g increment until the solution becomes saturated. The procedure was conducted at both room temperature and at 100°C.

Fourier Transform Spectroscopy (FTIR)

Analysis of CGE extract was conducted with an Agilent spectrophotometer scanning in the 4000 – 1000 cm^{-1} range.

Thermogravimetric Analysis (TGA)

The TGA test measures the weight change of the sample as a function of temperature and time, allowing the determination of thermal properties such as the degradation temperature and

thermal stability of the material such as polymers. A known weight of cashew gum exudate is placed in a TGA pan and heated in a controlled environment, typically under an atmosphere of nitrogen or other inert gas. The temperature of the CGE sample is ramped at a controlled rate, typically in the range of 5 to 10°C/min, while the sample weight is continuously monitored. The TGA data is plotted as a weight versus temperature curve, and the degradation temperature, residual weight, and weight loss percentage of the sample can be determined from the curve. The TGA results can be used to determine the thermal stability and thermal behavior of the cashew gum exudate, as well as to estimate its purity, crystallinity, and the presence of any impurities.

XRD Analysis

A powdered gum sample was subjected to X-ray diffraction analysis using an XPERT-PRO diffractometer. The X-ray diffraction pattern was generated at ambient temperature, employing Cu as the anode material, with an operating voltage of 45KV and a current of 40mA. The analysis covered a diffraction angle range from 5° to 50°.

GC-MS Analysis

The sample was injected into the gas chromatograph which separates the different compounds within the sample by channeling the sample via the column aided by helium gas. The various components are then placed inside the mass selective detector which produces a spectrum containing peaks that identifies the components based on the retention time.

3. RESULTS AND DISCUSSION

The results obtained for the preliminary analysis of the cashew gum exudate (CGE) as calcium carbonate scale inhibitor was discussed in this section. To gain more insights into the behaviour of the cashew gum extract in aqueous solution, some basic physio-chemical analyses were done on the sample and the results are presented in Table 1. The CGE sample was observed to be insoluble in water but sparingly soluble in both acetone and toluene which are organic solvents. This could infer that the sample is more organic in nature than not hence its slight solubility in like solvents. The pH of 5.3 observed is indicative of the acidic nature of the CGE. A moisture content of 3.86 % and water absorption capacity of 1.93 demonstrates that the material contains some water of hydration, an observation which is corroborated by results of FTIR which showed vibrational frequencies associated with O-H of water molecules. The sample also showed a density of 0.96 mg/L and specific gravity of 0.96 which are expected thresholds for these kinds of materials. A high viscosity of 33.3 mPa.s was also observed at laboratory conditions, this suggest that the material is highly viscous, indicative of its high thickness and consequently its high resistance to flow, and deformation (Amjadet *al.* 2011; Obuebiteet *al.* 2021).

Table 1: Physio-chemical characterization of CGE

S/N	Parameter	Value	Unit
1	pH	5.30	-
2	Moisture content	3.86	%
3	Density	0.94	mg/l
4	Specific gravity	0.96	-
5	Viscosity @ 27°C	33.3	mPa.s
6	Water absorption	1.93	-

7	Solubility in water	Insoluble	-
8	Solubility in acetone	Sparingly soluble	-
9	Solubility in toluene	Sparingly soluble	-

Compatibility test carried out between the gum exudate and brine containing divalent ions; calcium carbonate resulted in slightly cloudy solutions at concentrations less than 0.5%w/v as shown in Table 2. This is due to the effect of divalent ions as reported by Obuebite *et al.* (2021). As the temperature increased to about 100 degrees celsius, the solution produced clear, compatible solution devoid of any precipitation.

Table 2: Compatibility of cashew gum in brine solution

Sample	Concentration (%)	Result @ 27°C temp	Result @ 100°C temp
Cashew gum	0.2, 0.5, 1.0, 1.5, 2.0	<ul style="list-style-type: none"> • Slightly cloudy solutions at low concentration • Clear solutions at concentrations above 0.5%. 	Clear, compatible solutions across all concentrations.

Result of FTIR Analysis

The FTIR spectrum of CGE is presented in Figure 1 with the most important peaks assigned and labelled. The peak between 3800-3600 cm⁻¹ in the spectrum of the sample indicate symmetric stretching vibration of the O-H bond which is characteristic of glucoside ring in the sample as reported by earlier studies for similar kind of materials (Ibekwe *et al.* 2017; Nair *et al.* 2020)

while C-H vibration peaks appear between 2900-3000 cm^{-1} . In addition, the symmetrical vibration of the carbonyl moiety (sy -COO-) occurred between 1700 and 1780 cm^{-1} whereas the asymmetrical vibration was observed between 1500 and 1540 cm^{-1} . Another band observed between 1020-1070 cm^{-1} in the spectrum of the sample has been ascribed to the vibration of C-O bonds. An overlapping band occurring at ca. 1610 cm^{-1} which is often ascribed to O-H scissors vibrations from water molecules. It is important to point out that, the peak intensities are low which could be a suggestion that CGE has low composition of the chemical compounds possessing these functional groups as noted by Hua and Wang, (2009).

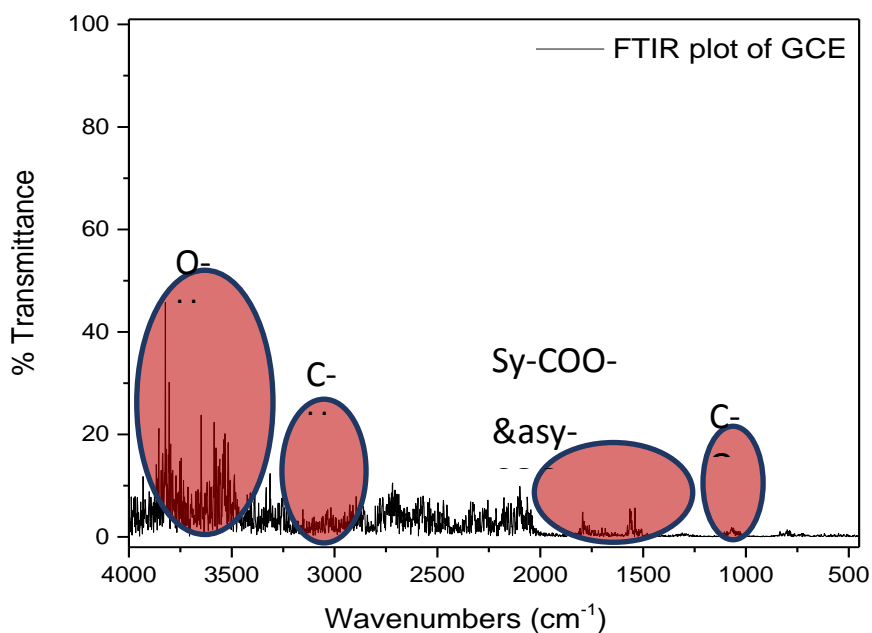


Figure 1: FTIR Plot of Cashew Gum Exudate

Result of GC-MS Analysis

The chemical composition of CGE was studied using a Gas Chromatography-Mass Spectrometer (GC-MS) system. The GC-MS analysis of CGE indicated similar profiles as shown in Figure 2 and Table 3 respectively. Six (6) compounds were identified. These include decanoic acid, methyl tetradecanoate, hexadecanoic acid methyl ester, 7-octadecenoic acid methyl ester, cis-9-octadecenoic acid, propyl ester, and tetracosane.

Table 3: GC-MS results of CGE

RT	Compound	Molecular Formula	MW gmol ⁻¹	Peak Area %
5.56	Decanoic acid	C ₁₀ H ₂₀ O ₂	172	2.30
6.47	Methyl tetradecanoate	C ₁₅ H ₃₀ O ₂	242	1.95
7.60	Hexadecanoic acid methyl ester	C ₁₇ H ₃₄ O ₂	270	24.70
8.54	7-Octadecenoic acid methyl ester	C ₁₉ H ₃₆ O ₂	296	63.52
8.86	cis-9-Octadecenoic acid, propyl ester	C ₂₁ H ₄₀ O ₂	320	5.67
10.01	Tetracosane	C ₂₄ H ₅₀	338	1.86

Comparisons of MS fragmentation patterns were used to preliminarily identify the samples by matching them with an NIST library (Simon-Manso *et al.* 2013; Farahani *et al.* 2022). The most prevalent compound present in the extract was identified to be 7-octadecenoic acid methyl ester which gave the highest peak area (63.52 %) at 8.54 retention time. However, tetracosane was shown to be the least present compound in CGE with a 1.86 % peak area at 10.01 min retention

time. This is an indication that fatty acids are the dominant constituents of the extract under investigation (Maia *et al.* 2000). It is important to note that the sample contain hexadecanoic acid methyl ester and octadecanoic acid methyl ester at similar retention indices, an observation that rationalizes the similarity in the chemical constituents of CGE (Rodrigues and Grosso, 2008).

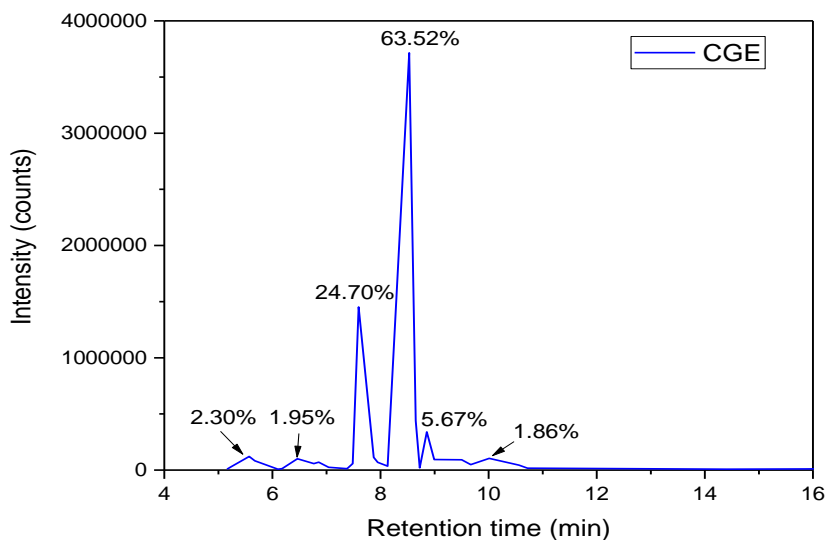


Figure 2: GC-MS Chromatogram of CGE

Result of XRD Analysis

XRD helps to give valuable information on the crystalline or amorphous nature of a material (Bunaciu *et al.* 2015). The diffractogram of the studied material, CGE is presented in Figure 3. The material showed single peak at relatively low 2θ values which is associated with the 100 Miller planes typical of these kinds of materials. The peak appeared at 19.20° at a relative intensity of ca. 2500 a.u. indicating a poor crystallinity of the material. The d-spacings and intercolumnar distances in the material was observed at 0.462 and 0.533 nm respectively, typical of materials with similar textural and structural properties. Consequently, a mixture of both

crystalline and amorphous phases has been proposed for the material. This agrees with earlier published literature (Veluraja and Atkins, 1989).

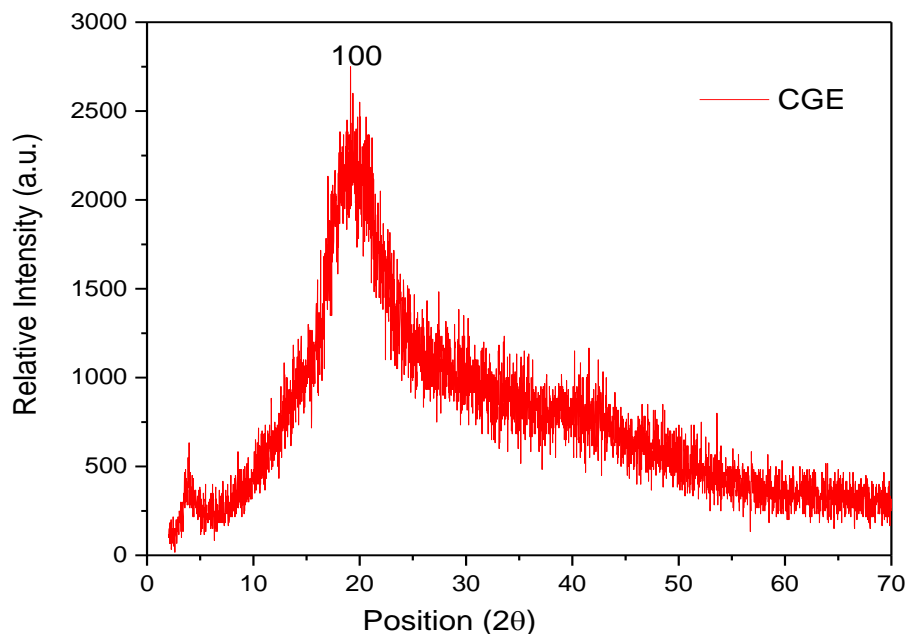


Figure 3:XRD Plot of Cashew Gum Exudate

Result of TGA Analysis

The assessment of how the mass of a sample changes with temperature variation is conducted through thermogravimetric analysis (Han *et al.* 2004). In order to elucidate the alterations undergone during the heat treatment of the cashew gum extract sample, thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were executed within a temperature range of 30 to 800 °C under atmospheric conditions. Figure 4 depicts the TG curve of the CGE. Notably, the onset degradation temperature was approximately 327.3 °C, indicating the observation of moisture and other impurity loss until this point, resulting in a mass reduction of

7%. The steep decline in the curve of GCE demonstrates an abrupt decrease in mass of 29% initially observed up to 376 °C, followed by another decrease up to 460 °C, culminating in the formation of residues.

Consequently, 460 °C has been assigned as the calcination temperature for the two samples, with major part of the weight loss occurring above 300 °C.

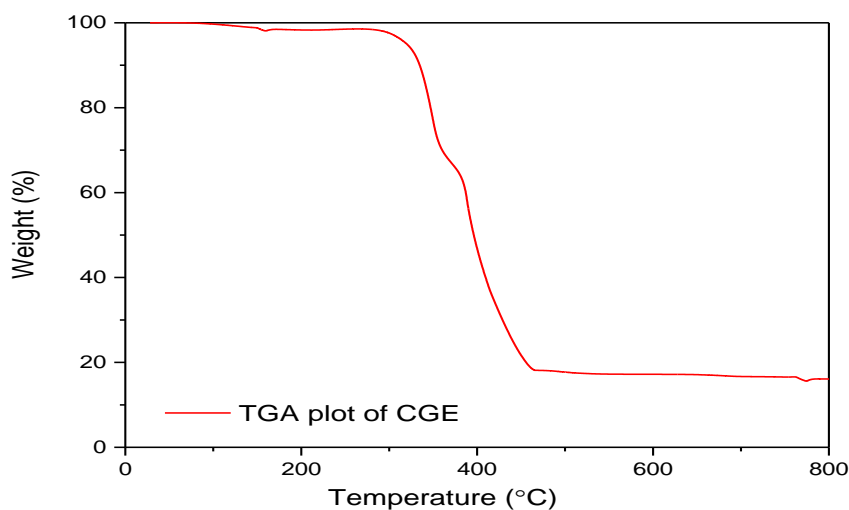


Figure 4: TGA Plot of Cashew Gum Exudate

Result of DTA Analysis

From the DTA plot (see Figure 5), the exothermic and endothermic occurrences within the samples were examined across a predetermined temperature range. In the DTA thermogram, during the endothermic process, the temperature of CGE dropped below the target temperature, manifesting as a downward peak. Conversely, in the exothermic process, the temperature of the gum surpasses the benchmark temperature, resulting in a minimum as seen on the plot. The

analysis encompassed a temperature range spanning from 30 °C to 800 °C, with air serving as the atmosphere for all samples. The DTA plot of CGE appeared to have an endothermic peak assignable to absorbed water evaporation at about 260 °C and two other endothermic peaks at 320°C and 388 °C attributed to the decomposition of organic residues. Furthermore, two exothermic peaks appeared at about 440°C and 525 °C, this is attributed to degradation of impurities and completion of the reaction respectively. The peak at 525 °C thus forms the calcination temperature of the CGE sample.

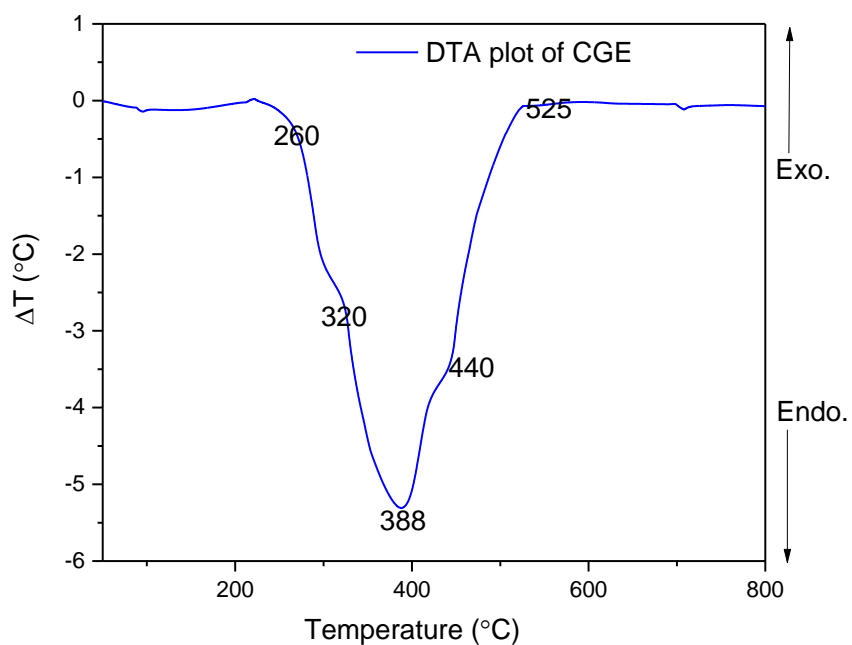


Figure 5: DTA Plot of Cashew Gum Exudate

The mechanism of scale inhibition by cashew gum exudate is not fully understood. However, it is believed that the polysaccharide molecules present in cashew gum exudate adsorb onto the

crystal surfaces preventing further crystal growth and aggregation. The large molecular size and hydrophilic nature of cashew gum exudate contribute to its ability to inhibit scale formation.

4. CONCLUSION

In this original research article, Cashew gum exudate showed promise as a green scale inhibitor for calcium carbonate scales. Its biodegradability, non-toxicity and renewable nature makes it an attractive alternative to synthetic scale inhibitors. There is growing interest in the use of natural polymers as scale inhibitors in the oil and gas industry, including their potential for inhibiting the formation of calcium carbonate scale. Natural polymers have several advantages over synthetic polymers, including their biodegradability, low toxicity, and high availability. Several studies have investigated the effectiveness of natural polymers such as chitosan, guar gum, and xanthan gum in inhibiting scale formation. While the results have been promising, there is still much to learn about the mechanisms by which these natural polymers inhibit scale formation and how to optimize their performance under different conditions. One potential advantage of natural polymers is that they may be more effective in high-temperature, high-salinity environments than synthetic polymers. Additionally, natural polymers may be less likely to interact with other chemicals in the oil and gas production process, which could reduce the risk of scale formation. **Further research is needed to optimize its performance, understand and evaluate its mechanism of action as an effective green scale inhibitor.** Incorporating cashew gum exudate into scale inhibition strategies can contribute to sustainable and environmentally-friendly practices in various industries.

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Authors' Contributions

Amalate Ann Obuebite (AAO) designed the study, wrote the protocol, conducted and interpreted the analyses and reviewed the final manuscript.

Obumneme Okwonna (OO) reviewed the various literatures and wrote the initial draft of the manuscript. All authors read and approved the final manuscript.

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