

# PRELIMINARY EVALUATION OF CASHEW GUM EXUDATE AS A GREEN SCALE INHIBITOR

## ABSTRACT

The study explores the potential of cashew gum as a sustainable, effective inhibitor of calcium carbonate scale. The use of green materials has garnered attention as a promising natural compound for industrial processes. Scale formation, a pervasive issue in oil production and water treatment, leads to reduced efficiency and increased maintenance costs caused by blockages in pipelines, oil wells etc. However, the use of scale inhibitors has been used for the control of various scale types including calcium carbonate scale. Cashew gum, a natural polysaccharide, with its abundance, bio-degradable and eco-friendly characteristics aligns with global emphasis on sustainable and green chemistry. The gum exudate was extracted from the bark of a cashew tree. 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 concentration of the compounds present. 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 also 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 the major constituent being 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 high compatibility with diverse water compositions makes it a potential solution for scale inhibition. The study has shown 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

## 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, injection and production wells, production facilities, pipelines, tubing resulting in severe technical problems such as the reduction in production capacity, reservoir choking, accelerated corrosion, blockage on flow lines and equipment which will negatively affect the overall cost of the process. Chaussemier *et al.* (2015) estimated the associated expenses due to scaling at some billion dollars per year in major developed industrialized countries. Bin Merdha *et al.* (2010) mentioned three mechanisms that can lead to the formation of scale deposits both in offshore and onshore environment. They include: mixing of two incompatible brines, change in conditions (temperature and pressure), and brine evaporation. Scale deposits from incompatible brine-mix occurs when two incompatible waters such as formation water (in situ in the reservoir) and seawater injected into the reservoir during water flooding enhanced recovery operations gets mixed downhole. The produced water is thus oversaturated with scale components (due to the precipitation of minerals when mixed) because seawater is composed of a high concentration of sulfate ( $\text{SO}_4^{2-}$ ) ions while formation water is rich in ions such as calcium ( $\text{Ca}^{2+}$ ) and barium ( $\text{Ba}^{2+}$ ). A mixture of these two waters leads to precipitation of sulfate scales, such as barium sulfate ( $\text{BaSO}_4$ ) and calcium sulfate (Olajire, 2015; Bin Merdha *et al.* 2010). Invariably, water is the major source of all scales. 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  $\text{H}^+$  and  $\text{CO}_3^{2-}$ . 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.

Several studies have been proposed to mitigate formation damage caused by scales in order to improve the productivity and injectivity of oil and gas wells. The process of scale inhibition has been adjudged the most preferred downhole treatment for the prevention or control of scale formation and deposition. Scale inhibition is the process of preventing the formation of scale from hindering fluid flow (Alzahrani *et al.* 2014). It is a chemical treatment used to control or reduce scales from forming in a well. This is typically achieved by the absorption of the chemicals (scale inhibitor) onto the crystal surface thus interfering with the further growth of the crystal and deposition of scale-forming minerals. In some cases, scale inhibitors prevent the scale crystals from adhering to solid surfaces such as piping. Effective scale inhibitors are necessary to prevent the formation and deposition of scales. Conventional scale inhibitors are hydrophilic, that is, they dissolve in water. The most common commercial scale inhibitor chemicals used in the oil and gas industry as noted by Kelland (2009) are inorganic phosphates, organo phosphorous compounds, and organic polymers especially PPCA (Poly-phosphonocarboxylic-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 have voluntary biodegradability, possess high efficiency, and are nontoxic (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) due to their excellent performance on sulfate and carbonate scales of calcium  $\text{Ca}^{2+}$  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 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.

## **MATERIALS AND METHODS**

### **Materials**

The equipment and reagents used in this study include beakers, glass rod, pyrometer bottle, 75  $\mu\text{m}$  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 ( $\text{CHCl}_3$ ), Diethyl ether ( $\text{CH}_3\text{CH}_2)_2\text{O}$ , Acetone, and cashew gum exudate.

### **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 tree 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 hand-picked to ensure the absence of any impurities (possibly from the bark of the cashew tree), sun-dried, and pulverized using a blender. Thereafter, it was dehydrated in chloroform-water ratio (70:30) for five days and then strained through a 75-micron mesh sieve to obtain particulate free slurry which was allowed to sediment.

Thereafter, the gum was precipitated from the slurry using absolute ethanol, filtered and defatted, using diethyl ether. The precipitate was re-dried at 40°C for 48 hours. The dried flakes were pulverized using a blender and stored in an airtight container.

## CHARACTERIZATION

### pH value

For determination of pH, 10 grams of the powdered sample was weighed and dissolved in deionized water and made up to 50 ml. This was made up to prepare the required solution. The pH of the solution was determined using a 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 was washed, properly dried, and weighed. The bottle was then filled with water and re-weighed. Afterwards, the bottle was emptied, dried, and filled with the watermelon seed oil and re-weighed. The obtained values were inputted and calculated using equations 1 and 2.

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

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

### Viscosity

The viscosity of the sample was determined using a digital roto-viscometer. The 50 ml tin sample container of the instrument was filled with sample under test to 0.3 mm of the top rim of the depth gauge. The sample was stirred with a stir rod and the temperature was maintained at 27°C. The container with the sample was placed in the processing ring on the turntable and the lever pulled down to switch on the motor automatically. The disc was then allowed to run until a steady state was reached (5 minutes). At the end of the required time, the viscosity was recorded in millipascal seconds (mPas). Triplicate measurements were made for each sample and the mean value determined and recorded.

### 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 indicates the maximum amount of a substance that can be dissolved in a solvent at a given temperature, and it is measured in grams per 100 g of solvent (g/100g).

0.5g of sample was turned into 100 ml of distilled water and mixed thoroughly using a magnetic stirrer. Thereafter, the solubility of the sample was visually determined. This quantity continues in 0.5g increment until the solution becomes saturated. The procedure was conducted at both room temperature and at 100°C.

**Standard test method for water absorption of plastics (Designation: D 570 – 98) Twenty-four-hour immersion:**

The conditioned specimen was placed in a container of distilled water maintained at a temperature of 26 °C and was rest on edge and entirely immersed. At the end of 24, +1/2, -0 h, the specimens were removed from the water one at a time, all surface water wiped off with a dry cloth, and weighed to the nearest 0.001 g immediately.

**Fourier Transform Spectroscopy (FTIR):** Fourier Transform InfraRed (FTIR) analysis of the CGE extract was conducted with an Agilent spectrophotometer scanning in the 4000 – 1000  $\text{cm}^{-1}$  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°.

**GS-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.

## **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.

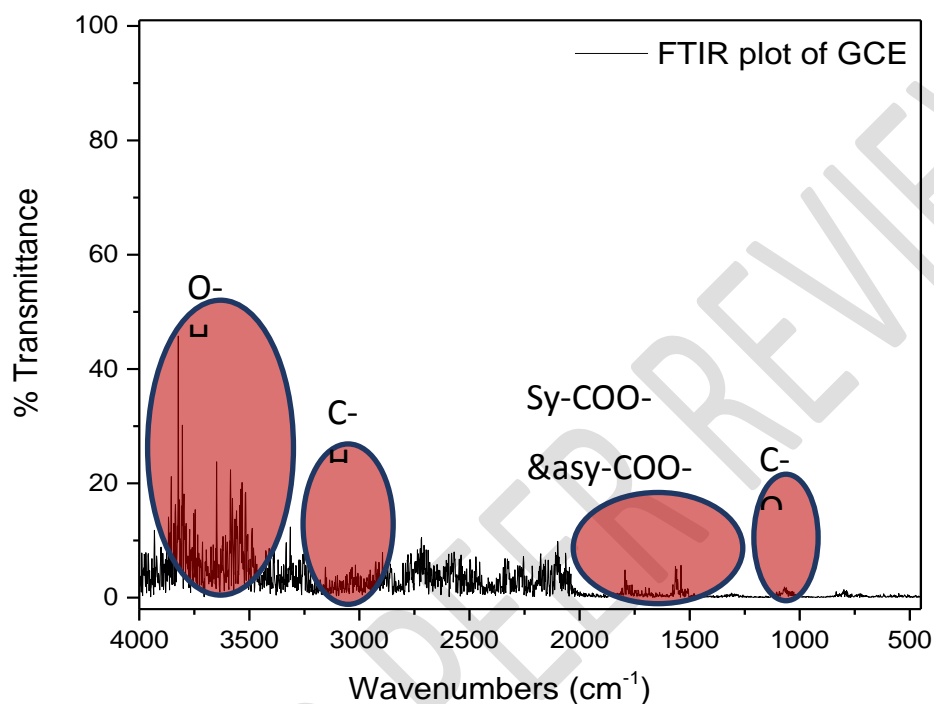
**Table 2:** Compatibility of cashew gum in brine solution

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"> <li>Slightly cloudy solutions at low concentration</li> <li>Clear solutions at concentrations above 0.5%.</li> </ul>	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<sup>-1</sup> 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<sup>-1</sup>. In addition, the symmetrical vibration of the carbonyl moiety (sy -COO-) occurred between 1700 and 1780 cm<sup>-1</sup> whereas the asymmetrical vibration was observed between 1500 and 1540 cm<sup>-1</sup>. Another band observed

between 1020-1070  $\text{cm}^{-1}$  in the spectrum of the sample has been ascribed to the vibration of C-O bonds. An overlapping band occurring at ca. 1610  $\text{cm}^{-1}$  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

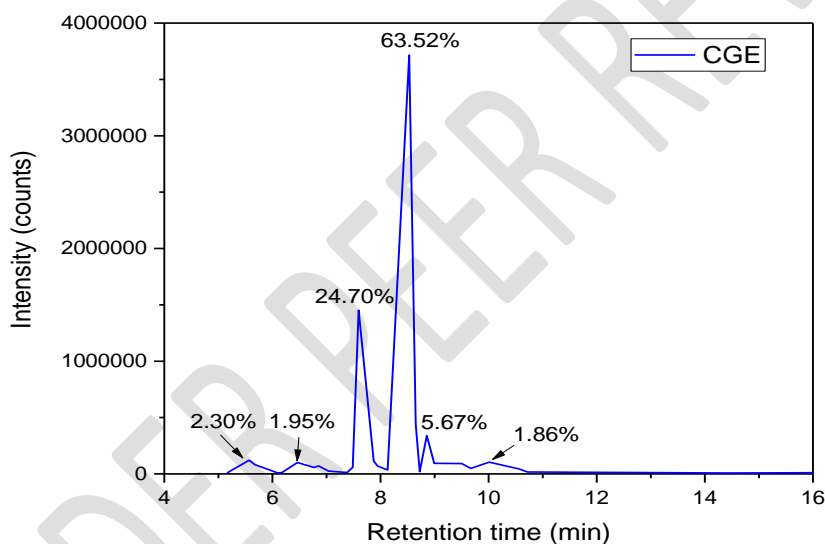
The chemical composition of CGE was studied using a Gas Chromatography-Mass Spectrometer (GC-MS) system. The GC-MS analysis of CGE indicated similar profiles as shown in Figure 2 and Table 3 respectively. Six (6) compounds were identified. These include decanoic acid, methyl tetradecanoate, hexadecanoic 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 $\text{gmol}^{-1}$	Peak Area %
5.56	Decanoic acid	$\text{C}_{10}\text{H}_{20}\text{O}_2$	172	2.30
6.47	Methyl tetradecanoate	$\text{C}_{15}\text{H}_{30}\text{O}_2$	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

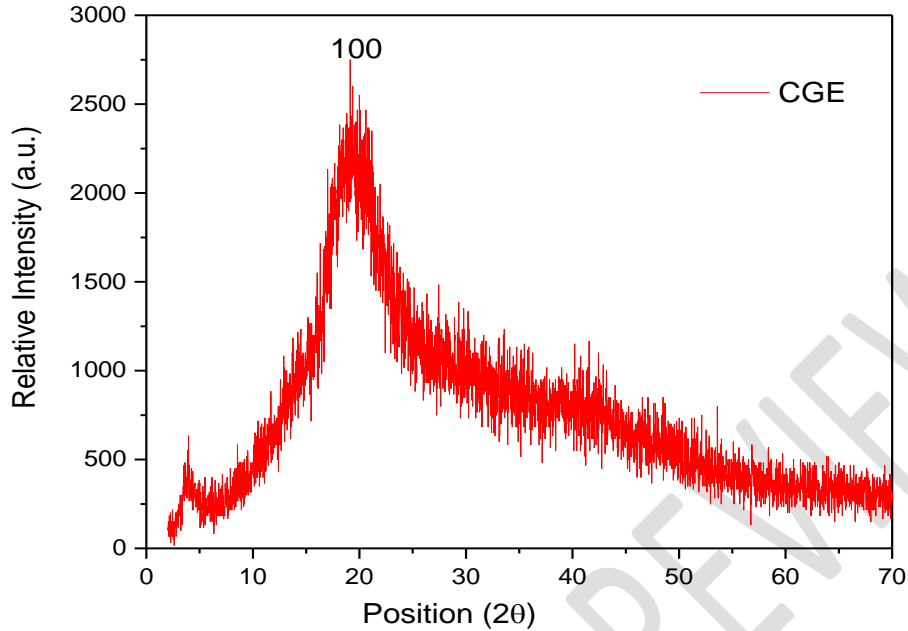
Comparisons of MS fragmentation patterns were used to preliminarily identify the samples by matching them with an NIST library (Simon-Manso *et al.* 2013; Farahani *et al.* 2022). The most prevalent compound present in the extract was identified to be 7-octadecenoic acid methyl ester which gave the highest 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

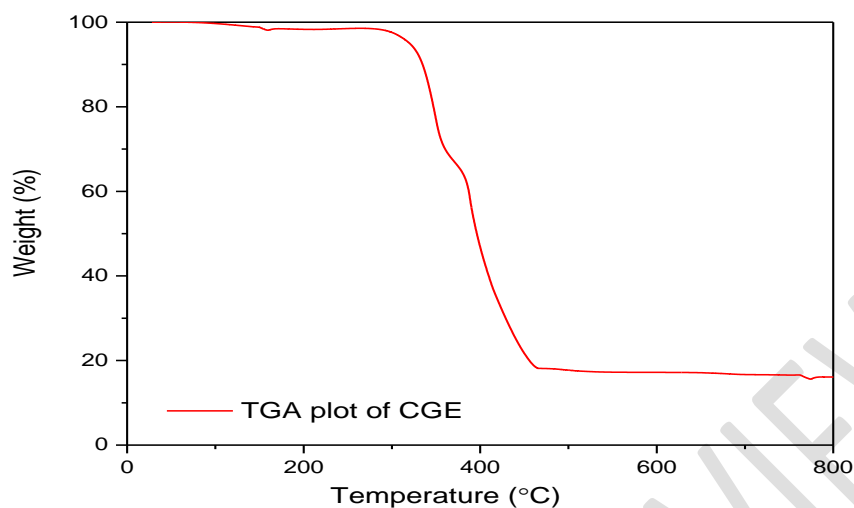
XRD helps to give valuable information on the degree of crystallinity or amorphous nature, and crystalline phase of materials (Bunaciu *et al.* 2015). The diffractogram of the studied material, CGE is presented in Figure 3. The material showed single peak at relatively low  $2\theta$  values which is associated with the 100 Miller planes typical of these kinds of materials. The peak appeared at  $19.20^\circ$  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**

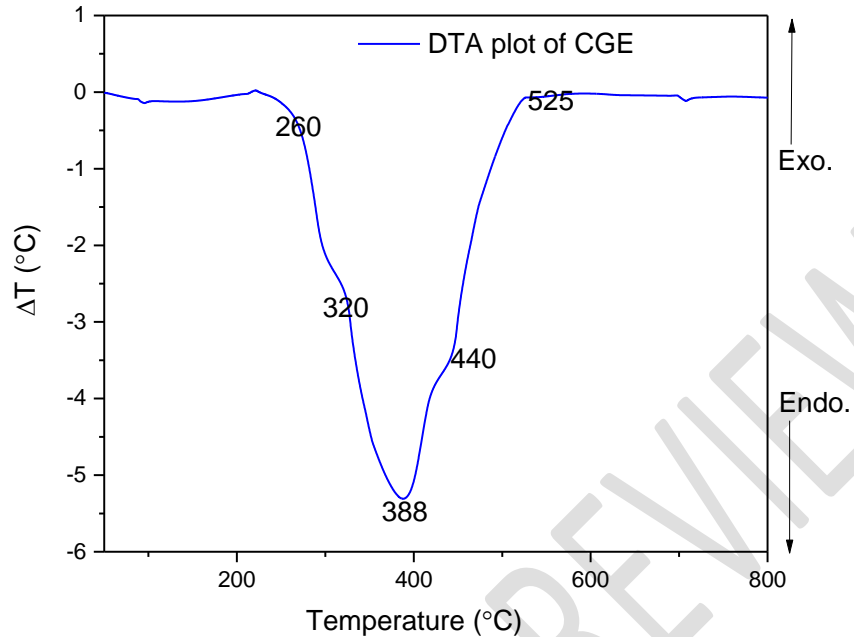
The interpretation of the variation in mass of a sample with change in temperature is done by thermogravimetric analysis (Han *et al.* 2004). To determine the changes that occurred during heat treatment of the cashew gum extract sample, thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were carried out from 30 to 800 °C in atmosphere. The TG curve of the CGE is shown in Figure 4. It can be observed that the onset degradation temperature occurred at ca. 327.3 °C implying that moisture and other impurity loss were observed until 327.3 °C with a mass loss of 7 %. The steep curve of GCE shows a sudden decrease in the mass of 29 % seen first up to 376 °C after which another decrease occurred up to 460 °C followed by 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 as seen in Figure 5, exothermic and endothermic events taking place within the samples have been analysed over a programmed range of temperature. During endothermic process in the DTA thermogram, the temperature of a sample fall behind the reference temperature and a down peak was observed whereas in exothermic process, the sample temperature exceeds the reference temperature, and a minimum is observed on the graphical plot. The analysis was done between 30 °C and 800 °C with air 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 ca. 320°C and 388 °C which can be ascribed to the decomposition of organic residues. Two exothermic peaks appear at about 440°C and 525 °C which can be assigned 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.

## CONCLUSION

Cashew gum exudate shows 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 and understand its mechanism of action. Incorporating cashew gum exudate into scale inhibition strategies can contribute to sustainable and environmentally-friendly practices in various industries.

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