Inhibition of Calcium Carbonate ($CaCo_3$) Scale using Plant Scale (Raffia Gum and Orange Mesocarp)

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Abstract:- This study evaluated the effectiveness of three (3) different scale inhibitors from locally sourced materials, raffia palm gum exudates and orange mesocarp in reducing the levels of various ion, while the performance of these materials was compared against a commercial grade inhibitor. The three different scale inhibitors were evaluated for their effectiveness in reducing the levels of various ions at different concentrations. A GC-MSD and GC-FID analysis was done on the raffia exudate and commercial scale inhibitors The GC-FID chromatograms indicated that the raffia exudate n-alkanes carbon atom ranged from C17-C36 while the commercial carbon atoms ranged from C14-C40 and the GC MSD indicated that the samples are predominantly n-alkanes at MZ 57 but the commercial had presence of Terpenes at MZ 191 (Biomarker which is resistance to degradation at 32mins). The GCMSD of the orange mesocarp shows major compound at RT 2.619 16.78% acetic acid, RT 6.328 30.06% butyl esther and RT 9.272 8.71% Hexadecenoic acid. The local scale inhibitor was more effective in reducing the levels of magnesium ion, and the Mesocarp orange scale inhibitor was relatively effective in reducing the levels of bicarbonate and calcium ions. The choice of scale inhibitor will depend on the specific water system and the type and level of scaling that needs to be prevented.

Keyword:- *Raffia gum; orange mesocarp; calcium carbonate; scale.*

I. INTRODUCTION

Scale deposits are a common source of technical and financial difficulties, including but not limited to reduced flow rates (through complete or partial pipe blockage); decreased heat transfer (since calcium carbonate precipitate is 15–30 times less conductive than steel); valve and filter seizure; and so on. Scaling in cooling towers is a common factor in limiting power output in nuclear power facilities. It was anticipated that Great Britain will spend 600,000,000 GBP annually on scaling's non-productive costs. In France, these costs amount to around €1.5 billion annually. Therefore, it is of the utmost significance to build suitable methodologies for studying this phenomenon and discovering efficient means of preventing or combating it [1].

When calcium carbonate meets water containing dissolved calcium salts and magnesium, it may either precipitate out or just adhere to the surface, causing the scaling phenomena. Scaling is plainly different from precipitating action, which is only the loose solid aggregates in the liquid phase, as the precipitation will bind on the surface of materials and shape rather stable bond with the matrix. Scaling is directly associated with the accumulation of calcium carbonate (CaCO3). Algae, calcium sulphate, clays, and brucite (Mg (OH)2) are some examples of potential residues in scaling. However, calcium carbonate always seems to be the first to precipitate out, and it generally does so in a colloidal form due to its reduced solubility [2].

Two separate processes, the germination and development of colloidal particles connected to an electrochemical phenomenon and the setting of deposit on the surface owing to an electrostatic phenomenon, work together to produce scaling. During nucleation, a network of ions is formed when Ca2+ and CO32- pair together. Nucleation may happen naturally or be triggered artificially. The polymer undergoes dehydration when ions are formed. Colloidal particles with electrical charges are formed when it binds to other ionic species, such as Ca2+ and CO3-. Suspended material does not settle out as it is loaded. Expansion: Taking in more ions like Ca2+ and CO3- causes the size to rise. The growth after nucleation is easier than a subsequent nucleation [3].

A. SCALING

If the underlying thermodynamic instability and incompatibility of solutions is not addressed, technical concerns in oil and gas exploitations, such as equipment and pipe obstruction, may lead to substantial damages and economic losses. Preventative measures could help avoid these problems. Scaling is one of the most difficult problems that must be addressed [4]. The creation of scale has several important implications for the scientific study of the process of producing oil and gas. It is also a significant issue in the sector of the energy business [5]. Although artificial scales present a major obstacle that could lead to significant reductions in petroleum output and expensive outages, flow security is of utmost importance in the oil and gas industry [6]. It was estimated that scale-related non-productivity expenses would amount to $0.8 billion in the United Kingdom, $3 billion in Japan, and $9.5 billion in the United States [7].

Crystallization may occur in two different ways: either surface crystallization or bulk crystallization, and both are thought to be potential factors in scale formation [8]. Scale formation in oil and gas fields can be affected by a wide variety of operational factors, such as pH [9], temperature [10], operating pressure [11], permeation rate [12], flow velocity, and the presence of other salts or metal ions. Scale formation can be affected by a wide variety of operational factors, including pH [13,14].The process of scaling up at the surface level requires attention to several essential details.

B. DIFFERENT OIL AND GAS INDUSTRY SCALE TYPES

Carbonates, sulphates, phosphates, and alumina-silicates are the common components that make up scales in oil and gas fields [15]. Common examples of inorganic scale identified during this analysis are calcium carbonate (CaCO3), calcium sulphate (CaSO4 • xH2O), barium sulphate (BaSO4), strontium sulphate (SrSO4), and silicates [16]. Inorganic scale includes not just biological scale but also its adjuncts such as silt, carbon, pipeline corrosion, and metal object corrosion. Biological scale is a subset of inorganic scale. Studies of scale samples conducted longitudinally have shown the following differences in state parameters depending on the circumstances of deposition: [17].

C. SCALE INHIBITORS

In the petroleum industry, it is generally known that water-soluble inorganic and organic compounds, even in very low levels, may prevent the formation of scale in producing wells. This is true even if the quantities are very minute. A scale inhibitor is a chemical substance that, by chelating scaling cations and/or altering the developing scale crystal, prevents or slows the formation of mineral scale in brines that are saturated with a pair of scaling ions (that is, cations and anions).Scale inhibitors typically consist of many active functional groups that can form interactions in aqueous solutions with scaling cations, nuclei, or crystals. These connections may be either strong or weak [18,19]. In oilfields, the use of scale inhibitors is a standard procedure that is done to either control or prevent the formation of scale in the production conduit or completion system. Scale inhibitors are now commercially accessible in a diverse range of forms, including natural, synthetic, and semi-synthetic substances [20]. The examples given here include organic polymers, chemicals including organophosphorus, and inorganic phosphates [21].

D. PROBLEM DEFINITION

The exact composition and properties of raffia exudate (gum) are not fully known, and there is a need to characterize the gum to better understand its properties and potential applications.Scaling is a major problem in many industrial processes, and the use of chemical scale inhibitors is often required to prevent or reduce scaling. However, the effectiveness of raffia exudate (gum) as a scale inhibitor has not been studied extensively, and there is a need to evaluate its scale inhibition tendency.There are several commercially available scale inhibitors in the market, but their effectiveness and cost can vary. Additionally, there is little information available on the potential of natural products, such as raffia exudate (gum) and orange mesocarp, as scale inhibitors. Therefore, there is a need to compare the scale inhibitor performance of raffia exudate (gum) with commercially available scale inhibitors and orange mesocarp on calcium carbonate scale.Temperature can have a significant impact on the effectiveness of scale inhibitors, and there is a need to investigate the effect of temperature on the scale performance of raffia exudate (gum) to determine its suitability for use in different industrial applications.

II. METHODOLOGY

The materials used in this study include: raffia palm gum exudates, orange mesocarp, polyacrylate phosphonate mixture (S16060). The reagents used were obtained as pure grade and include hexane, acetone, activated silicate gel, and anhydrous sodium sulphate. Chromatographic column, Gas Chromatograph Flame Ionization Detector (GC-FID) and Atomic Adsorption Spectroscopy (AAS) were used for analysis.

The methods employed in this study are sample preparation, scale inhibition test, characterization, and analysis.

A. Sample preparation

About 3000 g of raffia palm gum (RPG) exudates were obtained from a palm wine tapper at the premises of University of Port Harcourt Teaching Hospital. The exudate was oven dried at a temperature of 80oC. The dried sample was ground to powdered form and then soaked in hot water to extract the chemical component present in it. The solid samples were ground into powder form to expose a more surface area. 300g weight of the sample was put into a 100ml of distilled water. The distilled water and sample was heated at a constant temperature 90 oC for about 2 hrs with constant stirring using a magnetic stirrer. The mixture was allowed to cool to room temperature and stood for 24 hrs to allow enough time to extract. The solution was filtered using 45micron filter paper and was kept as stock solution. The weight of the sample was taken.

On the other hand, oranges which were purchased from a local market in Choba Nigeria were carefully selected washed and peeled. The orange mesocarp were carefully selected, sun-dried to reduce moisture content. The dried orange mesocarp were pulverized to increase the surface area for extraction and later filtered to remove impurities. The weight of the sample was taken.

B. Preparation of the test samples for scaling

Physiochemical analysis was carried out on 100ml of distilled water. The presence of the following elemental materials was evaluated using the AAS: Bicarbonate, Sulphate, Calcium (Ca), Sodium (Na), Magnessium (Mg). The pH was evaluated using a pH meter.

2000 mL of distill water was weighed in a conical flask and separated into six samples added 200g/L of analytical grade Calcium chloride dehydrate into five of the sample and was analyzed for calcium content. Again, 100g of analytical grade Sodium bicarbonate was added to the five samples. The sample was stirred with a magnetic stirrer for 5 mins to ensure the homogeneity of the sample and the pH of mixture was adjusted to about 8.5 with 0.5M solution of caustic soda (NaOH). A sample (Sample 1) which contained no scale inhibitor served as the control sample. Also, Samples 2, 3, 4, 5, and 6 were dozed with 10, 20, 30, 40, and 50 ppm of the salt, respectivley. The samples were thereafter placed in a waterbath at a temperature of 70 oC for 24 hrs after which the solutions were then allowed to cool. The supernatant was filtered through a 0.2-micron filter paper. The filtrate was withdrawn to analyze for soluble Calcium and other parameter. The process was repeat for the orange mesocarp extract (OME) as well as the commercial scale inhibitor (Polyacrylate and phosphonate combination with a commercial code as – SI 6060).

C. CO_2 Release monitoring with Time

This was done over a period of 2hrs when the samples had attained a temperature of 70 oC. The pH reading and conductivity were taken at different times of 10, 30, 60, 90 and, 120mins. This process was repeated for the OME and commercial inhibitors, respectively.

D. Sample Characterization

➢ *Sample Preparation for analysis*

Approximately 5.0 g of the OME (sample A) and 50mL of sample C were weighed and extracted using 25mL of n-Hexane/Acetone ratio 1:1 (USEPA 3550C) the extracts were transferred into an 8.0g activated silica gel with 1cm layer of anhydrous sodium sulphate chromatographic column for sample clean-up and fractionation.

The Columns were pre-conditioned using 20mL of n-hexane. Quantitatively, the solutions were moved to the column just before the sodium sulphate layer was exposed to air.

Saturated hydrocarbons were eluted with 18mL of n-hexane. Aromatic hydrocarbons were eluted with 28mL of hexane: dichloromethane (v/v, 1:1,). These processes removed polar components and other interference and fractionate samples into saturated and aromatic fractions.

➢ *Physicochemical Properties*

Saturates fractions were analyzed in accordance with the USEPA 8015 method using Agilent GC Model 7890A fitted with flame ionization detector (FID). Using a bulk solution of ACCU Standard hydrocarbon window defining standard, five working standards were used to calibrate the GC.

Aromatic and Saturates fractions were analyzed using GC-MS Model 5975 following USEPA 8270 method with SIM mode having m/z target ion: 57, 191, 217, 218, 231 and 253.

The summary of analytical methods and technique is presented in Table 1 and 2 below

Table 1: GC-FID Conditions and Method

GC	**AGILENT 7890A Powered with ChemStation Software**
Injection Temperature	280^0C
Split Ratio	Splitless
Carrier Gas	Helium
Inlet Temperature	250^0C
Column Type	HP 5
Column-Dimension	30m x 320µm x 0.25µm
Oven Program	Initial temperature at 40^oC, hold for 2 mins Ramp at 15^oC/min hold for 5 min (25min total run time)
Detector	FID
Detector Temperature	330^0C
Hydrogen Pressure	30.0psi
Compressed Air	300.0psi
Target Compounds	Isopreniods, n-alkanes and saturates biomarker
Method	USEPA 8015A

Table 2: GC-MSD (SIM Mode) Finger Printing Operating Conditions

GC	**AGILENT 7890A Powered with ChemStation Software**
Injection Temperature	280^{0}C
Split Ratio	Splitless
Carrier Gas	Helium
Inlet Temperature	250^{0}C
Column Type	HP 5
Column Dimension	30m x 250µm x 0.25µm
Oven Program	Initial temperature at 40^{o}C, hold for 2 mins Ramp at 10^{o}C/min to 320^{o}C hold for 5 min (35min total run time)
Detector	Agilent MSD 5975
Target Ions	m/z: 57, 191, 217, 218, 231 and 253 (alkanes,Terpanes, Steranes, Monoaromatic Steranes and Triaromatic Steranes) Biomarker groups
Method	USEPA 8270

III. RESULTS & DISCUSSIONS

The Commercial scale inhibitor had a higher concentration of bicarbonate (280 mg/l) than the other two samples. Bicarbonate is a common component of hardness in water and can contribute to scaling in industrial systems. The sample also had a lower concentration of calcium (12,070 mg/l) compared to the other two samples. Calcium is another major component of hardness and can cause scaling when present in high concentrations. The commercial scale inhibitor used in this sample may have been effective in reducing calcium scaling but may have increased bicarbonate scaling.

The RPG, and It had the lowest concentration of all ions except for sulphate (1 mg/l). Sulphate is a common component of scale, and the inhibitor may have been less effective in reducing sulphate scaling. However, the low concentration of other ions suggests that the local scale inhibitor may be effective in reducing scale in the system.

TheOME had relatively low concentrations of all ions except for sodium (18,082 mg/l). Sodium is not a major contributor to scaling, but it can affect the performance of some industrial processes. The OME may have been effective in reducing scale in the system, but it may have also contributed to the high sodium concentration.

Overall, the results suggest that the choice of scale inhibitor can have a significant impact on the concentrations of various ions in water and can affect the effectiveness of scale control in industrial systems.

Bicarbonate ($HCO3-$) levels in the samples is highest in the Commercial scale inhibitor. Sulphate ($SO42-$) levels are also within the acceptable range for drinking water, with the lowest levels found in the Local scale inhibitor sample. Sodium (Na) levels are highest in the Commercial scale inhibitor sample, followed by the RPG and OME samples.

Calcium (Ca) and magnesium (Mg) levels in the samples are important to consider since these are the two main minerals that cause scaling in water systems. The RPG sample has the highest Ca and Mg levels, followed by the OME sample and then the Commercial scale inhibitor sample. This indicates that the Commercial scale inhibitor may be more effective at preventing scale formation than the other two inhibitors.

Overall, the choice of scale inhibitor will depend on the specific water system and the type and level of scaling that needs to be prevented. It is important to consider all the factors, including the levels of bicarbonate, sulphate, sodium, calcium, and magnesium, when selecting the appropriate scale inhibitor for a particular application.

At 30 ppm, the commercial scale inhibitor was more effective in reducing the levels of bicarbonate and sulphate ions compared to the RPG and OME. However, the local scale inhibitor was more effective in reducing the levels of magnesium ion compared to the other inhibitors. The OME was relatively effective in reducing the levels of bicarbonate and calcium ions.

At 40 ppm, the commercial scale inhibitor was again more effective in reducing the levels of bicarbonate and sulphate ions compared to the other inhibitors. However, the OME was more effective in reducing the levels of bicarbonate ion compared to the other inhibitors. The OME was more effective in reducing the levels of magnesium ion compared to the other inhibitors.

At 50 ppm, the commercial scale inhibitor was more effective in reducing the levels of bicarbonate, sulphate, and sodium ions compared to the other inhibitors. However, theRPG was more effective in reducing the levels of calcium and magnesium ions compared to the other inhibitors. The OME was relatively effective in reducing the levels of bicarbonate and sodium ions.

A. Result on the analysis of Scale Inhibitors (pH and Conductivity)

From the data, we can observe that the pH of the solution decreases with time, indicating that it is becoming more acidic. This change in pH could be due to the formation of carbonic acid as a result of the reaction between CaCl2 and Na2CO3. Carbonic acid is a weak acid that dissociates in water to form H+ ions, which could account for the decrease in pH over time. The conductivity of the solution also increases with time, indicating that there is an increase in the number of ions in the solution. This increase in conductivity could be due to the dissociation of CaCl2 and Na2CO3 into their constituent ions, which are charged particles that can carry an electrical charge in the solution.

B. Result on the analysis of Scale Inhibitors pH and Conductivity at different Concentration.

For the 10ppm concentration of inhibitors, RPG showed a gradual increase in pH from 7.28 at 0 minutes to 7.39 at 120 minutes, while the other two inhibitors showed a decrease in pH from 7.50 to 7.77 and 7.10 to 7.00 for commercial scale inhibitor and OME, respectively. In terms of conductivity, the RPG showed a steady increase from 70,200 to 82,300, while the other two inhibitors showed a decrease in conductivity from 55,100 to 48,000 and 83,300 to 85,600 for commercial scale inhibitor and OME, respectively.

For the 20-ppm concentration of inhibitors, (RPG) showed a gradual increase in pH from 7.38 at 0 minutes to 7.56 at 120 minutes. In contrast, the other two inhibitors showed fluctuations in pH with OME showing an increase from 7.13 to 7.15 and then a decrease to 7.09, and commercial scale inhibitor showing an increase from 7.52 to 7.67. In terms of conductivity, RPG showed a gradual increase from 71,900 to 87,600, while the other two inhibitors showed a decrease in conductivity from 52,200 to 46,100 and 79,800 to 84,500 for commercial scale inhibitor and OME, respectively.

For the 30 ppm concentration of inhibitors, RPG showed fluctuations in pH with an increase from 7.45 to 7.58 and then a decrease to 7.69. In contrast, the other two inhibitors showed a steady increase in pH from 7.50 to 7.64 for commercial scale inhibitor and a decrease from 7.20 to 7.03 for OME. In terms of conductivity, RPG showed fluctuations with an increase from 71,300 to 79,400 and then a decrease to 82,800, while the other two inhibitors showed a steady increase in conductivity from 49,800 to 44,000 and 73,000 to 78,400 for commercial scale inhibitor and OME, respectively.

For the 40 ppm concentration of inhibitors, the local scale inhibitor showed a decrease in pH from 7.44 to 7.36, while the other two inhibitors showed a steady increase in pH from 7.54 to 7.67 and a decrease from 7.36 to 7.21 for commercial scale inhibitor and OME, respectively. In terms of conductivity, RPG showed fluctuations with an increase from 71,200 to 86,500, while the other two inhibitors showed a decrease in conductivity from 44,500 to 40,200 and an increase from 72,800 to 78,700 for commercial scale inhibitor and OME, respectively.

For the 50ppm concentration of inhibitors, RPG showed fluctuations in pH with an increase from 7.54 to 7.51 and then an increase to 7.50. In contrast, the other two inhibitors showed a steady decrease in pH from 7.31 to 7.45.

C. GC FID and GC-MSD Sample A (Raffia Exudate) and Sample C (Commercial Scale Inhibitor)

The GC-FID chromatograms indicated that sample A n-alkanes carbon atom ranged from C17-C36 while Sample C carbon atoms ranged from C14-C40.

Similarly, GC MSD indicated that the samples are predominantly n-alkanes at MZ 57, but sample C had presence of Terpenes at MZ 191 (Biomarker which is resistance to degradation at 32mins). Therefore, it can be stated that the two samples are not from the same source.

IV. CONCLUSION

In conclusion, the pH values can be observed that RPG caused a slight increase in pH at all concentrations and time intervals, whereas the commercial scale inhibitor and OME caused fluctuations in pH values, both rising and falling depending on concentration and time interval. The highest pH value was observed in the local scale inhibitor at 50 ppm after 90 minutes, and the lowest pH value was observed in the commercial scale inhibitor at 10 ppm after 30 minutes.

The conductivity values for the RPG Increased with increasing concentration and time interval, indicating that the inhibitor was less effective at preventing scale build-up. The commercial scale inhibitor and OME, on the other hand, showed a decrease in conductivity values at higher concentrations and longer time intervals, indicating that these inhibitors were more effective at preventing scale build-up. The lowest conductivity value was observed in the commercial scale inhibitor at 50 ppm after 120 minutes, and the highest conductivity value was observed in the local scale inhibitor at 50 ppm after 90 minutes.

The OME and commercial scale inhibitor were more effective at preventing scale build-up than the RPG. However, the effectiveness of each inhibitor varied depending on the concentration and time interval tested.

The results also show that the commercial scale inhibitor was generally more effective in reducing the levels of bicarbonate, sulphate, and sodium ions compared to the other inhibitors at all concentrations tested. However, the choice of inhibitor to use may depend on specific water treatment needs, and other factors such as cost, availability, and compatibility with other water treatment chemicals should also be considered. From the GC analysis carried out, the commercial and raffia exudate result shows the sources are not the same.

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