Parametric Evaluation on the Interfacial Tension Response of Agro-Surfactant

Chukwuebuka Francis Dike*, Nkemakolam Chinedu Izuwa, Anthony Kerunwa, Nzenwa Dan Enyioko, Enyang Lilian Ndoma-Egba, Chukwuebuka Gabriel Mbah, Ogonnaya Michael Ogbuatu, Federal University of Technology, Owerri, Nigeria

Abstract

The introduction of surface active agent such as surfactants reduces interfacial tension (IFT) between the oil-water systems to yield higher oil recovery. This reduction continues with surfactant concentration until the critical micelle concentration is attained. The IFT reduction capacity of surfactant in brine-oil system is impacted by the surfactant concentration, salt concentration, temperature variation and polymer concentration. In this study, parametric evaluation was conducted to determine the impact salinity, temperature and polymer on the IFT value of costus afer extracts (CAE), vernonia amygdalina extract (VAE), carica papaya extract (CPE) and sodium lauryl sulfate (SLS). From the result of IFT at varying salinity, CAE, VAE and CPE is not suitable for high saline environment. From the result of IFT at varying temperature, CAE and CPE have dominant nonionic properties, while VAE showed dominant anionic properties. From the result of IFT at varying polymer, polymer introduction reduces the IFT value of the surfactants.

Introduction

Chemical Enhanced Oil Recovery (CEOR) is the most widely used Enhanced Oil Recovery (EOR) approach (Sedaghat et al. 2013), and deals with the introduction of surfactant, alkaline, polymer or their hybrid in improving oil recovery from reservoir rock (Izuwa et al. 2021a). CEOR improves oil recovery using mechanisms such as wettability alteration, interfacial (IFT) reduction, mobility control, polymeric viscoelastic and permeability reduction (Afolabi 2015). Of the chemicals utilized in CEOR are surfactant which reduces the IFT between brine and water (Kerunwa 2020). Surfactant comprises of hydrophobic and hydrophilic group which influences its behavior in a brine-oil system. Based on the hydrophilic head, surfactants can be categorized into zwitterionic (+ve and –ve), cationic (+ve), anionic (-ve_ and non-ionic (neutral) (Schramm 2000). The anionic and nonionic surfactants are the most widely accepted for CEOR (Coung et al. 2017). The anionic surfactant are classified into sulfate, sulfonate, phosphate and carboxylate, while non-ionic surfactants are ether, ester, phenol, hydroxyl and amine (Nikunji and Tejas 2017). Anionic surfactants are the most preferred for CEOR due to their high effectiveness in lowering IFT, low adsorption on sandstone and high temperature stability (Jeirani et al. 2014). The combination of anionic and non-ionic surfactant enhances their tolerance in formation water with high salt concentration (Sheng 2011). IFT responses of surfactant in a brine and oil system can be impacted upon by parameters such surfactant concentration, alkaline concentration, salt concentration, temperature variation and polymer concentration. The introduction of surfactants concentration into brine-oil system reduces their IFT until a point which further surfactant concentration increase yields no reduction in IFT (Ali et al 2020). This surfactant concentration is referred to as critical micelle concentration (CMC), and can be used to identify the best surfactant at low concentration (Onykonwu and Akaranta 2016). After the CMC IFT values stabilizes or in some cases increase depending on the type and nature of the surfactant utilized. For crude oil with high total acid number (TAN), the concentration of alkali in the solution

*Corresponding author: skippin1994@yahoo.com

determine the percentage of in-situ surfactant which might or might not reduce IFT. The ability of an alkali material to reduce IFT is tied to its pH value (Krumrine et al. 1982). When combined with surfactant, alkali further reduces the IFT between oil-water systems. The salinity level of water-oil system particularly at low salt concentration could potentially reduce IFT (Obuebite et al. 2020). For solutions containing surfactant-containing solution, IFT value drops with increase in salt concentration until optimal salinity stage before further increase in salinity starts to increase IFT (Bera and Mandal 2015). The introduction of polymers tends to increase viscosity of solutions and influence IFT value. Polymer have tendency to reduce IFT at low concentration with surfactants (Izuwa et al 2021b). The introduction of heat to surfactant based solutions, yield further reduction in their IFT values (Jiramet et al. 2017). In the study the IFT response of the selected surfactants were characterized using fourier transform infrared spectroscopy (FTIR) before been evaluated at varying salinities, varying temperature and varying polymer concentrations. Costus Afer Extract (CAE), Vernonia Amygdalina Extract (VAE), Carica Papaya Extract (CPE) and Sodium Lauryl Sulfate (SLS) were used as Surfactant while Araucaria Columnaris Extracts (ACE), Terminalia Mantaly Extracts (TME) and Xanthan Gum (XG) were utilized as Polymers.

Materials and Methods

Materials. The material used for the study includes; 3 Locally Sourced Agro-Surfactant; Carica Papaya extracts (CPE) and Vernonia Amygdaline extracts (VAE), Conventional Surfactant: Sodium Lauryl Sulphate (SLS), Biopolymer: Xanthan Gum (XG), Terminalia Mantaly (TME) and Araucaria Columnaris (ACE). Industrial Salt (NaCl), Agilent 19091S-433UI gas chromatograph (GC) system, Attensio Sigma 702/702ET Tensiometer, Beaker, Test-tube, Syringe, Weigh balance and Crude Oil. The Crude Oil was gotten from a field in the Niger-Delta. The crude oil has API gravity of 34.97^o, specific gravity of 0.84 and dynamic viscosity of 3.752cp (at room temperature).

Preparation of Materials. The carica papaya extract and vernonia amygdaline extract was recovered from the tree, washed thrice with deionized water to remove unwanted materials and dried for 24hrs at room temperature. The dried leaves were crushed into smaller particles. The pulverized leaves were soaked in water (%wt concentration of the required CEOR fluid) for 4hrs, before utilization for the lab evaluation. The costus afer stems were washed with deionized water three times to effectively remove unwanted material. The top, bottom and outer body of the stem were effectively removed to ensure that only the inner component of the costus afer stem. The inner component of the costus afer stem were sliced into smaller sizes before mechanical press was conducted to recover extracts. The extract were purified by filtration using API filter paper.

Gas-Chromatography. The crude oil sample was evaluated using Agilent 19091S-433UI gas chromatograph (GC) system fitted to a fused silica capillary column (30m x 0.5mm ID) and connected to the Agilent mass selective detector (MSD). 1µ was introduced into the GC system using the automatic liquid sampler (ALS). The oven temperature was sustained for 0min at 50°C, 50-200°C at 15°C/min, 200-250°C at 10°C/min, 250-280°C for 1min. The mass spectrometer made use of 70eV electron energy, ion source temperature of 250°C and interface temperature of 280°C

FTIR Evaluation. Fourier Transform Infrared (FTIR) evaluation was used for this study. M530 modelled bulk scientific infrared was used for the FTIR experimental analysis. 0.5g of the local samples were mixed with 0.5g of potassium bromide (KBr) nanomaterial, after which 1ml of nujol (a solvent for preparation of sample by the spectrophotometer) was introduced into the samples using syringe to form a paste before introducing it to the apparatus and a wavelength of $600\text{-}4000cm^{-1}$ is used to derive spectra heights. The spectroscopy yields chart in absorbance spectra form, which indicates the molecular structure and chemical bonds present in the sample. The analytical spectra derived for each substance were then compared with the catalogue of the instrument to determine the functional group present.

IFT Test. The IFT analysis was used in the determination of the critical surfactant concentration of the locally sourced surfactants. Attension Sigma 702/702ET Tensiometer was utilized for the study. The procedures

utilized was obtained from operation manual of the Tensiometer. The IFT between the oil-brine systems was first determined before the introduction of surfactant with concentration depicted in **Table 1**. The impact of salt concentration on surfactant's IFT was studied with brine formulation depicted in **Table 2**. The impact of temperature variation on surfactant's IFT was studied with temperature ranges depicted in **Table 3**. The impact of varying polymer concentration on surfactant's IFT was studied with formulation depicted in **Table 4**.

Table 1—Surfactant Formulation for IFT Test.

S/N	Surfactant	Surf. Conc. (%wt)	Salt Conc. (%wt)
1	CAE	1%wt	5000ppm
2	VAE	1%wt	5000ppm
2	CPE	1%wt	5000ppm
3	SLS	1%wt	5000ppm

Table 2—Brine Formulation for IFT Test (NaCl).

S/N	Surfactant	Surf. Conc. (%wt)	Brine Formulation
1	CAE	1%wt	10,000pm, 15,000ppm, 20,000ppm, 25,000ppm and 30,000ppm
2	VAE	1%wt	10,000pm, 15,000ppm, 20,000ppm, 25,000ppm and 30,000ppm
3	CPE	1%wt	10,000pm, 15,000ppm, 20,000ppm, 25,000ppm and 30,000ppm
4	SLS	1%wt	10,000pm, 15,000ppm, 20,000ppm, 25,000ppm and 30,000ppm

Table 3—Temperature Variation for IFT Test.

S/N	Surfactant	Surf. Conc. (%wt)	Salt Conc. (%wt)	Temperature Variation
1	CAE	1%wt	5000ppm	27, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95 and 100
2	VAE	1%wt	5000ppm	27, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95 and 100
3	CPE	1%wt	5000ppm	27, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95 and 100
4	SLS	1%wt	5000ppm	27, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95 and 100

Table 4—Polymer Variation for IFT Test.

S/N	Polymer	Polymer Concentration (%wt)
1	ACE	0.25%wt, 0.5%wt and 1%wt
2	TME	0.25%wt, 0.5%wt and 1%wt
3	XG	0.25%wt, 0.5%wt and 1%wt

Results and Discussion

Gas-Chromatography. Table 5 shows the various component present in the crude oil. As shown in Table 5, the crude oil comprised majorly of naphthalene, iso-alkane, n-alkane, alcohol, alkyl-alkanes, anhydrites, ester, carboxylic acid and aldehydes. The aldehyde recorded 24% of the crude, carboxylic acid recorded 15%, alkyl-alkane recorded 26%, iso-alkane recorded 13.37% while anhydride recorded close to 7.5% percent composition. The crude oil contained 51.819% paraffin, 32.293% naphthalene, 32.293% carboxylic acid and 0.9839% other compounds. Eser (2013) grouped crude oil into paraffinic, paraffinic-napthaenic, naphthenic, aromatic-naphthenic, aromatic-asphaltic and aromatic-intermediates based on the relative abundance of paraffin, aromatics and naphthene compounds. The relative abundance of alkane and alkane related compounds in the crude oil, shows that the crude oil is a paraffinic crude oil and its in-line with its high api gravity result.

Table 5—Compositional Analysis of all components of Crude Oil.

Pk#	RT	Area%	Library/ID
1	5.212	0.08	Decahydro-8a-ethyl-1,1,4a,6-tetram ethylnaphthalene
			Bicyclo[3.1.1]heptane-2-carboxalde hyde, 6,6-dimethyl-
			Cyclohexane, 1-(cyclohexylmethyl)-2-ethyl-, trans-
2	5.630	0.20	Undecane
			Octadecane
			Decane, 2-methyl-
3	5.630	0.31	Dodecane
			Carbonic acid, prop-1-en-2-yl tridecyl ester
			Carbonic acid, prop-1-en-2-yl tetradecyl ester
4	5.733	0.17	4-Methyl-trans-3-thiabicyclo[4.4.0] decane
			Naphthalene, 1,4,5-trimethyl-
			Naphthalene, 1,4,6-trimethyl-
5	5.909	0.31	Decahydro-8a-ethyl-1,1,4a,6-tetramethylnaphthalene
			3,7-Dimethyl-6-nonen-1-ol
			3-Cyclohexene-1-carboxaldehyde, 1,3,4-trimethyl
6	5.991	0.21	4-Methy-trans-3-thiabicyclo[4.4.0]decane
			Naphthalene, 2,3,6-trimethyl-
			Naphthalene, 1,6,7-trimethyl-
7	6.153	0.59	p-Menth-8(10)-en-9-ol, cis-
			Octatriacontyl pentafluoropropionate
			E-2-Tetradecen-1-ol
8	6.431	1.54	Tridecane
			2-Piperidinone, N-[4-bromo-n-butyl]-
			Pentadecane
9	6.820	2.17	Tridecane
			Methoxyacetic acid, 2-tridecyl ester
			Dodecane, 4,9-dipropyl-
10	6.911	0.82	Cyclopentane, 1-pentyl-2-propyl-
			3-Methyl-4-(methoxycarbonyl)hexa-2, 4-dienoic acid

			1,2-Cyclohexanediol, cyclic sulfite, trans-
11	7.254	19.97	Tridecane, 7-hexyl-
			Dodecane, 2-methyl-8-propyl-
			Decane, 2-methyl-
12	7.922	0.26	Pentadecane
			Pentadecane
			Pentadecane
13	7.996	0.37	Dodecane, 2,6,10-trimethyl-
			Methoxyacetic acid, 4-tetradecyl ester
			Methoxyacetic acid, 2-tridecyl ester
14	8.618	1.73	Nonadecane
			Hexadecane, 1-chloro-
			Hexadecane, 1-chloro-
15	9.282	4.84	Octadecane, 1-chloro-
			Carbonic acid, hexadecyl prop-1-en-2-yl ester
			Tritetracontane
16	9.822	13.37	1-Octadecene
			1,2-Benzisothiazole, 3-(hexahydro- 1H-azepin-1-yl)-, 1,1-dioxide
			Z-8-Methyl-9-tetradecen-1-ol acetate
17	9.917	6.70	Nonadecane, 2-methyl-
			Batilol
			1-Octadecanesulphonyl chloride
18	23.117	2.7	2-Dodecen-1-yl(-)succinic anhydrid
			Squalene
			17-Pentatriacontene
19	24.77	14.90	Propionic acid, 3-iodo-, heptadecyl ester
			1-Docosene
			8-Hexadecenal, 14-methyl-, (Z)-
20	25.005	4.71	2-Dodecen-1-yl(-)succinic anhydrid
			1-Hexacosene
			Aspidospermidin-17-ol, 1-acetyl-19, 21-epoxy-15, 16-dimethoxy-
21	35.46	24.06	Cyclopropaneoctanal, 2-octyl-
			Erucic acid
			Octadecane, 1-(ethenyloxy)-

FTIR Characterization. **Figures 1** to **4** provide a comprehensive evaluation through Fourier Transform Infrared Spectroscopy (FTIR) of both agro-surfactants and synthetic surfactants. This analysis unveiled the hydrophobic and hydrophilic compositions of these chemicals, revealing a spectrum of functional groups.Arranged in ascending order of wavelength, the identified functional groups include C-Br, C-Cl, R-O-R, H2C=CH3, H2C=CH, RNH3, RCOOH, R-C $\equiv$ N, CH2, R-S-C $\equiv$ N, RCHOH, R2CHOH, R2NH, R3N, and R3CHOH. Remarkably, agro-surfactants such as CPE, VAE, and CAE demonstrated a striking similarity in their hydrophilic components (ether, amine, nitriles, carboxylic, and hydroxyl) as well as their hydrophobic tails

(Methylene and Ethene) when compared to the conventional surfactant SLS. Upon scrutinizing the FTIR Spectra of SLS and CPE, it was noted that the ester functional group was absent, whereas VAE and CAE exhibited its presence. This observation underscores a notable resemblance in composition between synthetic and agro-surfactants. Ahmed et al. (2019) categorically classified anionic surfactants into carboxylate, sulfate, sulfonate, and phosphate groups, while non-ionic surfactants predominantly comprise ether and hydroxyl groups. The presence of ester, hydroxyl, carboxylic, ether, and amine groups in the agro-surfactants suggests their potential to exhibit both non-ionic and anionic surfactant behavior. This versatility makes them suitable for applications in sandstone reservoirs and high salt concentration environments, aligning closely with the findings of Tadros (2014).
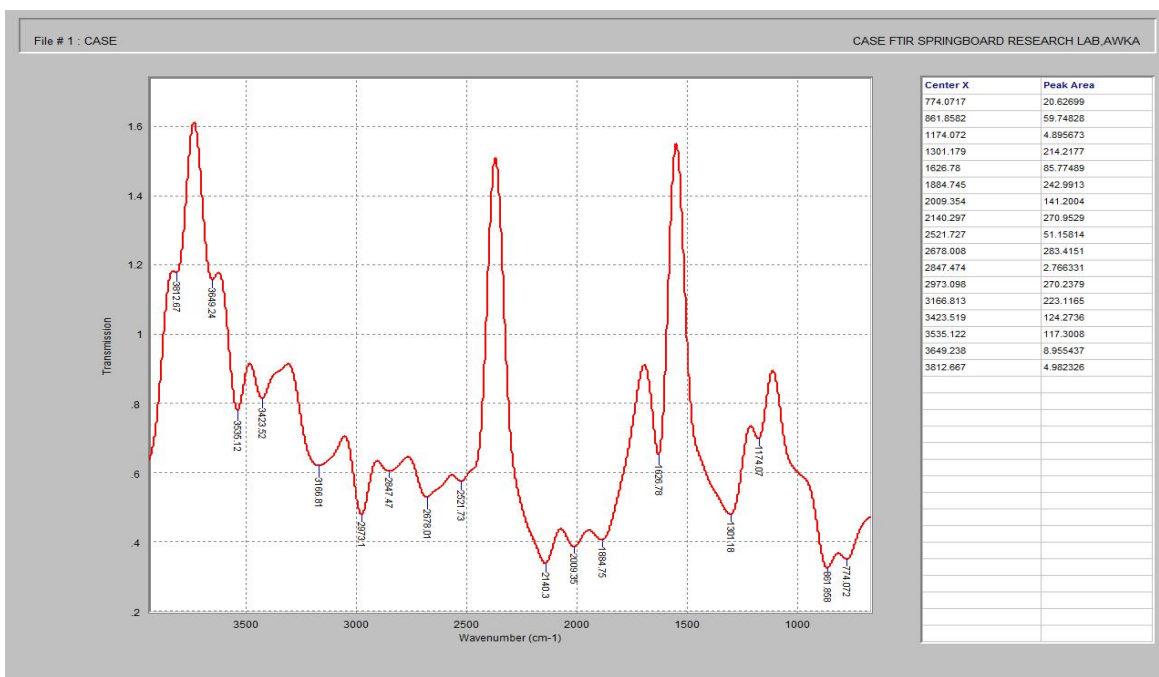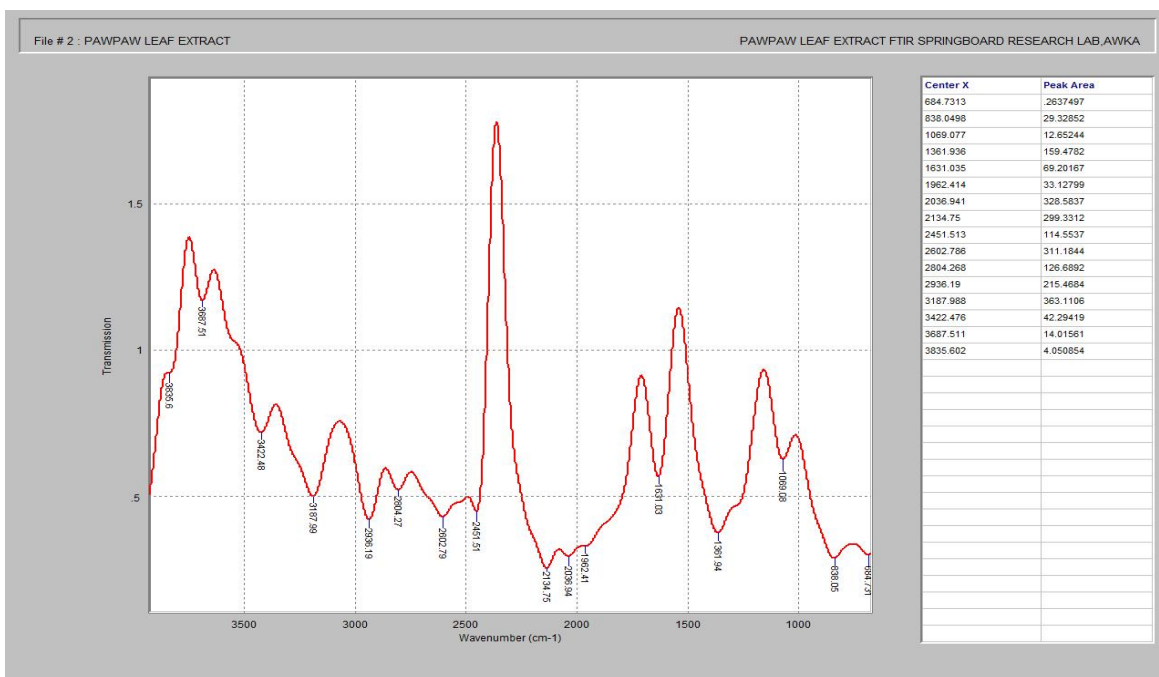

Figure 1—FTIR Spectra for CAE.
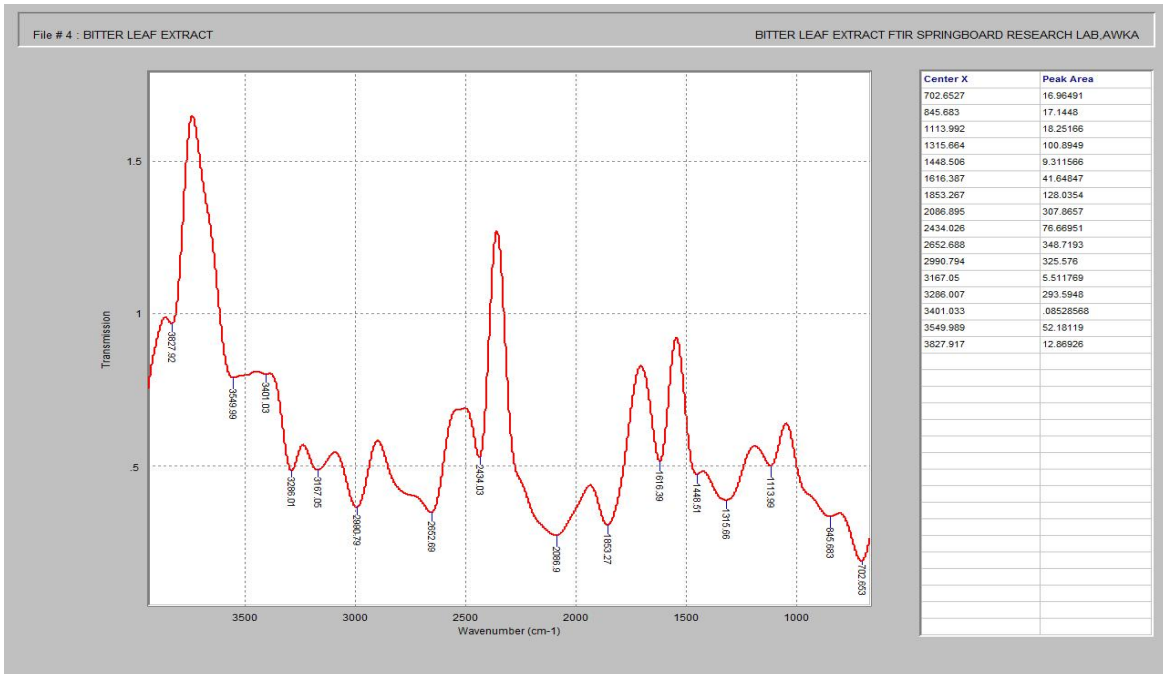

Figure 2—FTIR Spectra for CPE.

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Center X	Peak Area
702.6527	16.96491
845.683	17.1448
1113.992	18.25166
1315.664	100.8949
1448.506	9.311566
1616.387	41.64847
1853.267	128.0354
2086.895	307.8657
2434.026	76.66951
2652.688	348.7193
2990.794	325.576
3167.05	5.511769
3286.007	293.5948
3401.033	.08528568
3549.989	52.18119
3827.917	12.86926

Figure 3—FTIR Spectra for VAE.

FTIR SPECTRA OF SAMPLE 5   None

Center X	Peak Area
701.885	40.56517
836.5978	5.185813
945.5425	9.741814
1075.075	4.15203
1225.211	22.67766
1416.241	43.48038
1630.799	30.9653
2069.031	480.8788
2461.876	86.7017
2628.405	179.3116
2811.607	100.4909
2917.902	176.1442
3198.601	153.9689
3367.33	1.471542
3431.459	237.8312
3692.711	1.869153
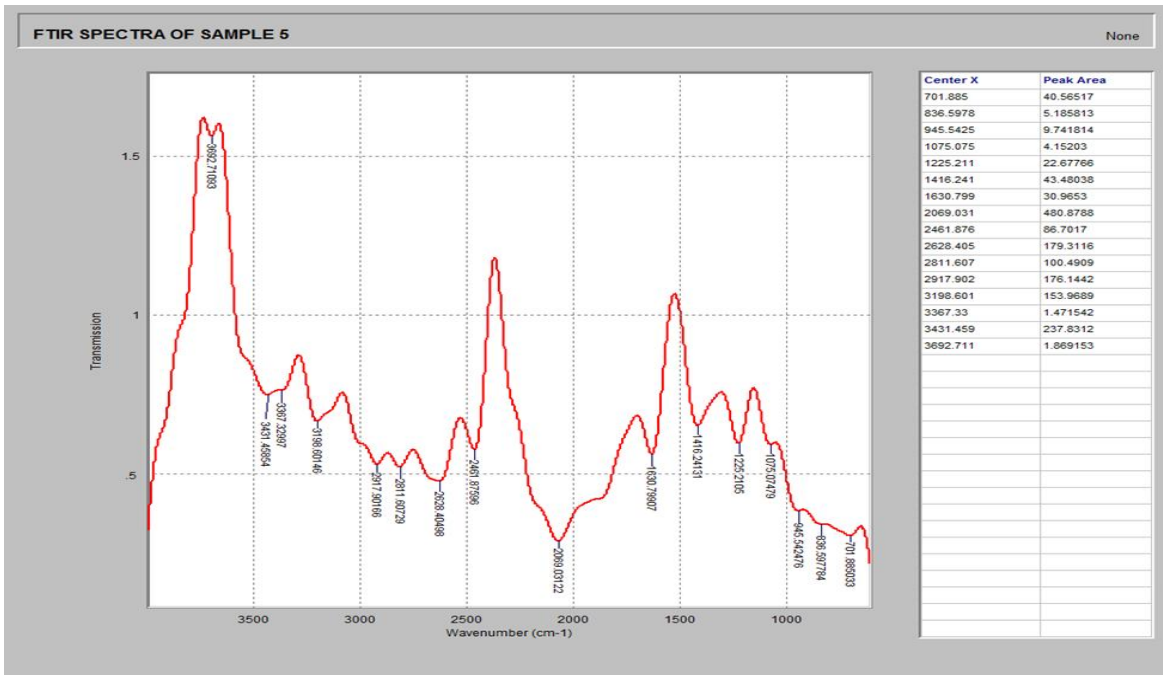
Figure 4—FTIR Spectra for SLS.

Interfacial Tension. In **Figure 5**, we delve into the Interfacial Tension (IFT) responses of CAE, VAE, CPE, and SLS. The data showcased reveals initial IFT values of 12.43 mN/m, 9.98 mN/m, 11.35 mN/m, and 6.93 mN/m respectively for these surfactants. Upon their introduction into the brine-oil system, a significant reduction in IFT was observed. Specifically, CAE, VAE, CPE, and SLS contributed to reductions of 53.96%, 63.04%, 57.96%, and 74.33% respectively, when compared to the initial IFT of the brine-oil system. These findings align closely with the research conducted by Kerunwa (2020), which emphasizes the effectiveness of surfactants in diminishing the IFT between brine-oil systems. Moving forward, in Figure 6, we further explore the impact of salinity on the IFT responses of CAE, VAE, CPE, and SLS.
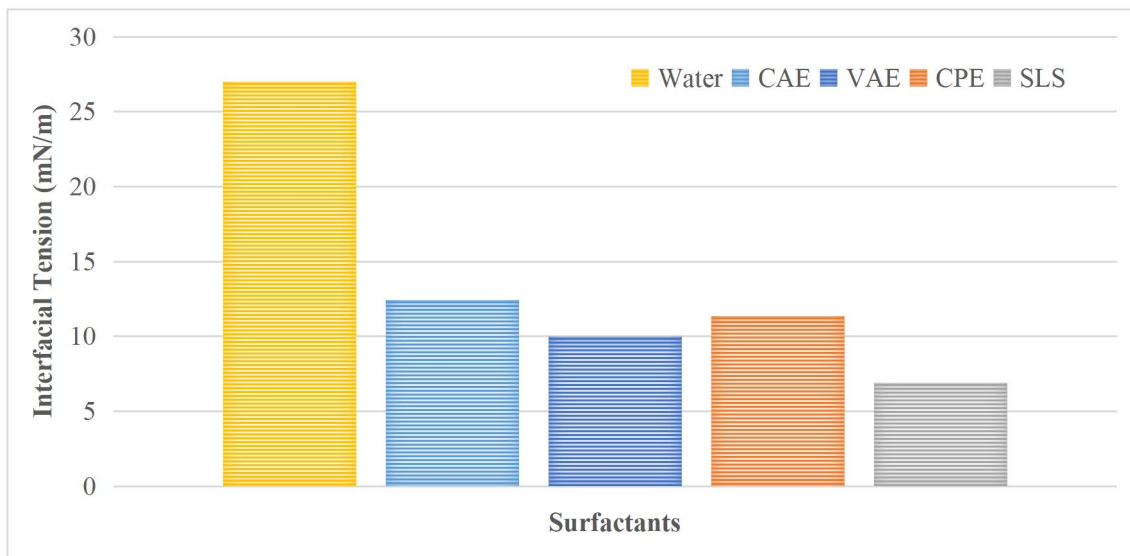
Figure 5—Interfacial Tension (IFT) of the Surfactants.

As illustrated in **Figure 6**, the Interfacial Tension (IFT) dynamics of various surfactants, including CAE, VAE, CPE, and SLS, exhibit intriguing responses to changes in salinity levels. For CAE, as salinity escalates from 5000 ppm to 30000 ppm, we observe a noteworthy rise in IFT, increasing from 12.43 mN/m to 16.65 mN/m. However, there's a slight deviation in this trend as the salinity peaks at 20000 ppm, resulting in a temporary decrease in IFT before resuming its upward trajectory. On the other hand, VAE displays a consistent uptick in IFT with increasing salinity, from 9.98 mN/m to 11.04 mN/m as salinity climbs from 5000 ppm to 30000 ppm. Similarly, CPE records a progressive increase in IFT from 11.35 mN/m to 12.92 mN/m over the same salinity range. Interestingly, SLS exhibits a nuanced response, with an initial increase in IFT from 6.93 mN/m to 7.2 mN/m as salinity escalates from 5000 ppm to 10000 ppm, followed by a subsequent reduction in IFT down to 6.16 mN/m as salinity further increases to 30000 ppm. These observations resonate with findings from Bera and Mandal (2014), suggesting that the relationship between salinity and surfactant IFT values can vary, with both increases and decreases in IFT being possible outcomes of salinity variations.
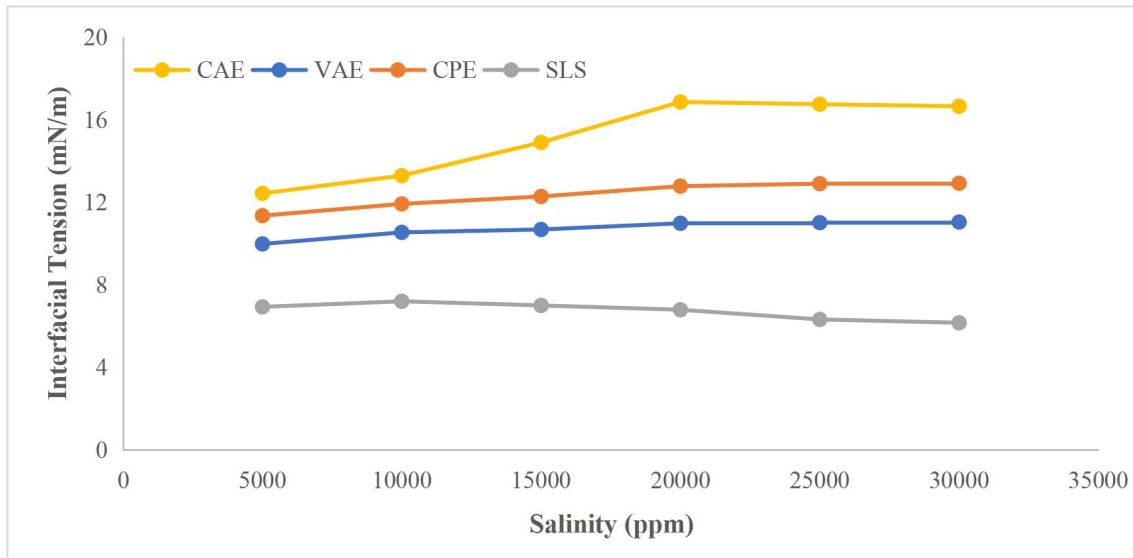
Figure 6—Impact of Salinity on the IFT of the Surfactants.

In **Figure 7**, we delve into the interplay between temperature fluctuations and Interfacial Tension (IFT) responses across various surfactants, namely CAE, VAE, CPE, and SLS. The data reveals intriguing trends as temperatures climb from 27°C to the boiling point at 100°C. For CAE, we observe a gradual decline in IFT values from 12.43 mN/m at 27°C to 7.98 mN/m at 100°C. Similarly, VAE experiences a reduction from 9.89 mN/m to 7.75 mN/m over the same temperature range. CPE showcases a decline from 11.26 mN/m to 7.84 mN/m, while SLS demonstrates a more pronounced drop from 6.82 mN/m to 4.89 mN/m.

Notably, non-ionic surfactants exhibit greater sensitivity to temperature changes compared to anionic surfactants. This aligns with findings by Izuwa et al. (2021b), suggesting that the composition of surfactants influences their response to temperature variations. The response of CAE and CPE to temperature variation change could be attributed to it having more non-ionic surfactant properties. VAE and SLS recorded similar IFT reduction pattern and comprised of more anionic surfactant features. The reduction in IFT value of the surfactant with increase in temperature is in-line with Jiramet et al. (2017) study, which showed that increase in temperatures yields a drop in IFT for define surfactant concentration.
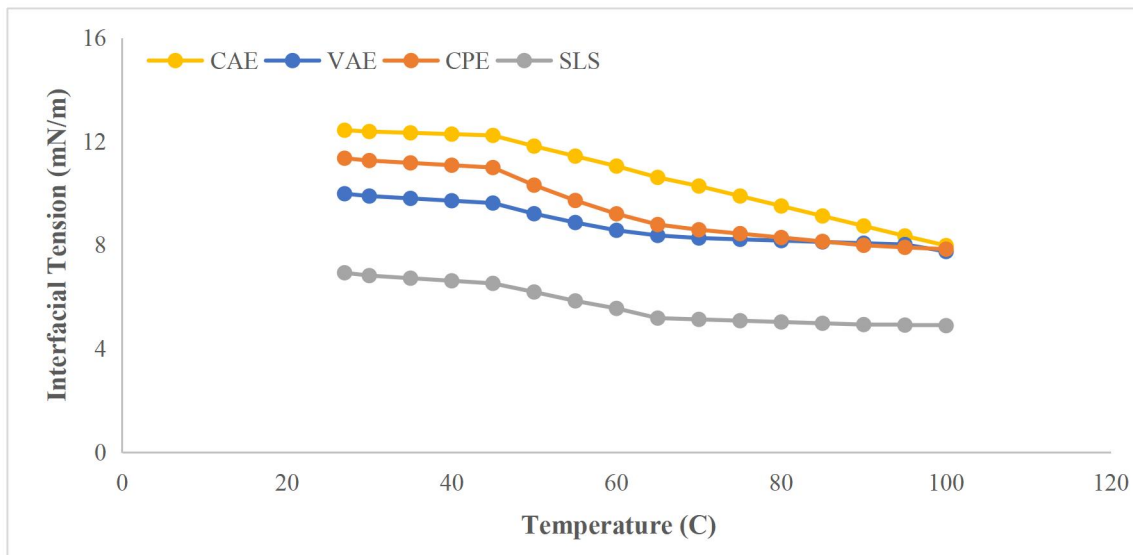

Figure 7—Impact of Temperature on the Surfactant IFT behavior.

Figures 8-10 depicts the impact of CAE, TME, ACE and XG polymer on the IFT responses of VAE, CPE and SLS. As depicted in Figure 8, the results from the CAE tests indicate a noticeable decrease in IFT from 12.43 to 8.78 mN/m and 8.71 mN/m with the introduction of 0.25% wt and 0.5% wt of TME respectively. However, with a further increase in TME concentration to 1% wt, there was a subsequent rise in IFT from 8.71 to 10.33 mN/m. Moving on to the VAE trials, a decline in IFT was observed from 9.98 to 9.47 mN/m and 9.06 mN/m with the addition of 0.25% wt and 0.5% wt of TME respectively. Yet, when TME concentration was increased to 1% wt, there was an increase in IFT from 9.06 to 9.7 mN/m. Similarly, in the case of CPE experiments, there was a decrease in IFT from 11.35 to 9.4 mN/m and 8.58 mN/m with 0.25% wt and 0.5% wt of TME respectively. However, with a further increase in TME concentration to 1% wt, there was a rise in IFT from 8.58 to 8.83 mN/m. Lastly, SLS tests demonstrated a decline in IFT from 6.93 to 5.78 mN/m and 5.67 mN/m with the inclusion of 0.25% wt and 0.5% wt of TME respectively. Yet again, with an increase in TME concentration to 1% wt, there was a rise in IFT from 5.67 to 5.73 mN/m.
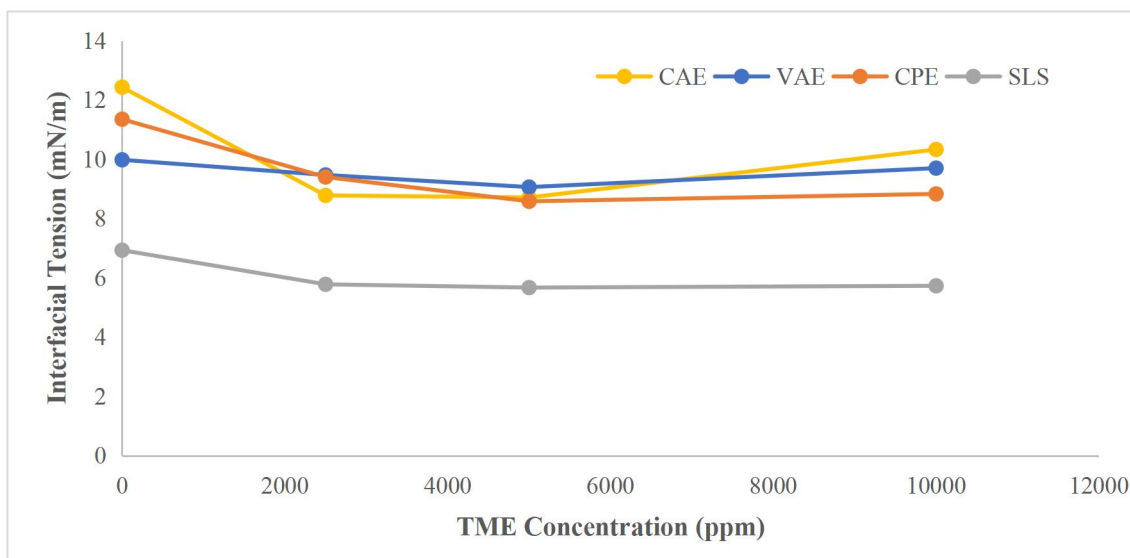

Figure 8—Impact of TME Polymer on the Surfactant IFT behavior.

As shown in **Figure 9**, CAE recorded IFT drop from 12.43mN/m to 10.47mN/m and 8.78mN/m when 0.25%wt and 0.5%wt TME was introduced. Further increase in ACE concentration to 1%wt yielded IFT rise from 8.78mN/m to 8.79mN/m. VAE recorded IFT drop from 9.98mN/m to 9.74mN/m when 0.25%wt ACE was introduced. The IFT however increased to 10.15mN/m at 0.5%wt ACE before dropping to 9.68mN/m at 1%wt ACE respectively. CPE recorded IFT drop from 11.35mN/m to 10.12mN/m, 10mN/m and 9.91mN/m when 0.25%wt, 0.5%wt and 1%wt ACE respectively. SLS recorded IFT reduction from 6.93mN/m to 6.02mN/m when 0.25%wt ACE was introduced. Further introduction of ACE up to 0.5%wt and 1%wt concentrations, yielded a rise from 6.02mN/m to 6.09mN/m, and a drop from 6.09mN/m to 5.96mN/m respectively.
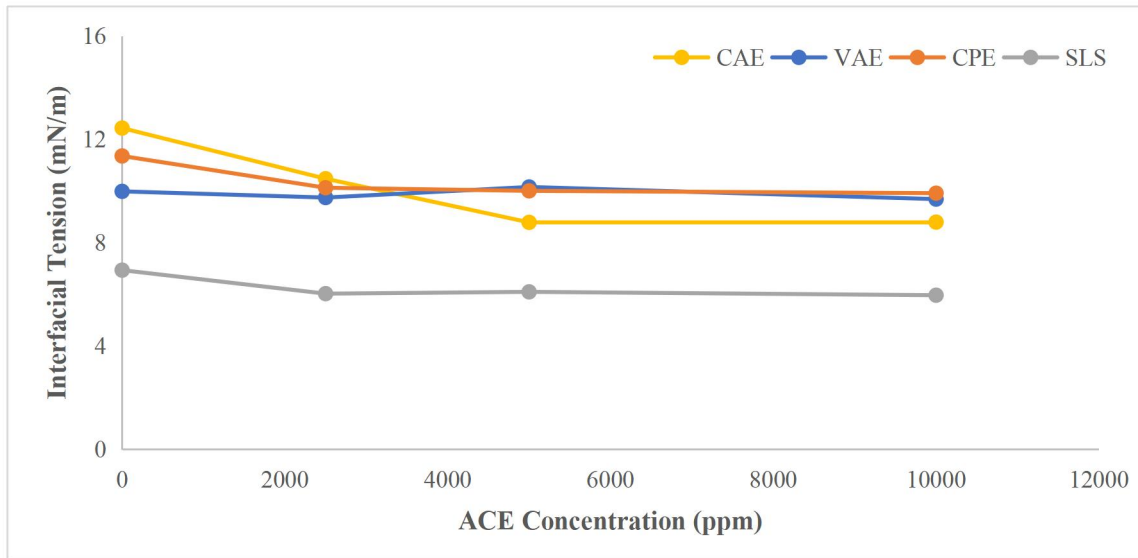
Figure 9—Impact of ACE Polymer on the Surfactant IFT behavior.

In **Figure 10**, CAE recorded IFT drop from 12.43 to 10.94 mN/m when 0.25% wt XG was introduced. Further XG introduction from 0.5% wt and 1% wt increased IFT from 10.94 to 11.28 mN/m, and reduced IFT from 11.28 to 10.9 mN/m. VAE recorded IFT dropping from 13.34 to 10.19 mN/m and 9.66 mN/m at 0.25% wt and 0.5% wt XG concentration, respectively. Further XG increase to 1% wt concentration yielded IFT increase from 9.66 to 9.77 mN/m. CPE recorded IFT increasing from 10.7 to 10.84mN/m and 10.98 mN/m for 0.25% wt and 0.5% wt XG concentration, respectively. Further XG concentration introduction from 0.5% to 1% wt reduced IFT from 10.98 to 10.72 mN/m. SLS recorded an IFT rising from 6.24 to 6.95 mN/m when 0.25%wt XG was introduced. Further ACE concentration yielded IFT reduction from 6.95 to 6.92 mN/m and 6.83 at 0.5%wt and 1%wt polymer concentration, respectively. The reduction in IFT of surfactant with polymer introduction was in-line with the observation of Izuwa et al (2021b) study which indicated that polymer chemical tend to lower IFT at low concentration with surfactants. This is also in agreement with Abhijit et al (2011) study which confirmed the interaction of surface active agents and polymers, and also noted that further polymer concentration increase results in an increase in IFT value.
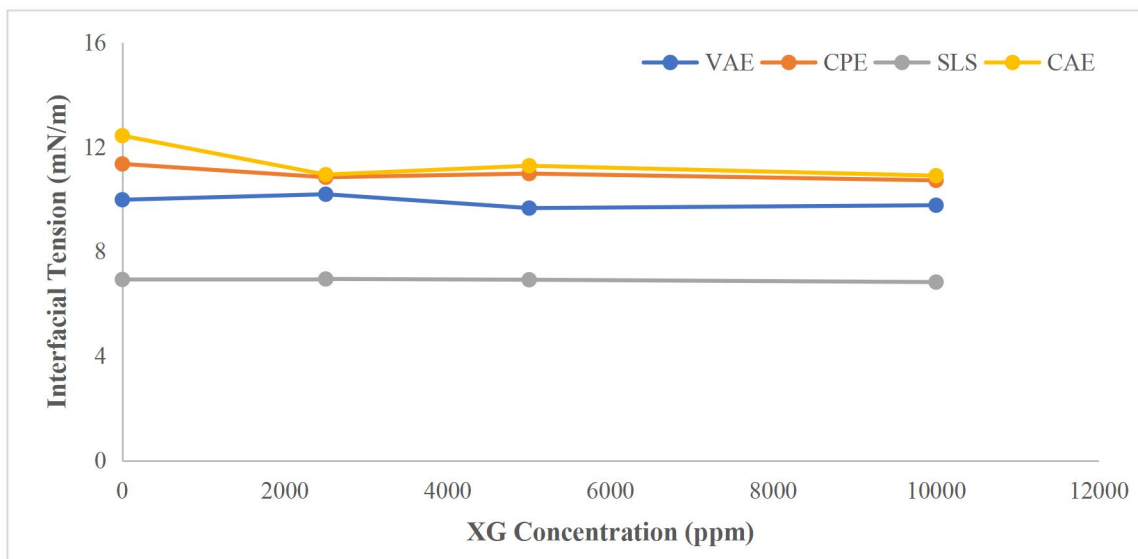

Figure 10—Impact of XG Polymer on the Surfactant IFT behavior.

Conclusions

From the experimental analysis, the following conclusions can be made.
1. The presence of more alkane and alkane-related compounds characterized the crude oil as a paraffinic crude.
2. The surfactants reduced IFT between brine-oil systems. SLS recorded the least IFT value of the surfactants utilized.
3. The local surfactants are not suitable for high saline environment.
4. CPE and CAE have more non-ionic surfactant properties and are suitable for high temperature environment while VAE have more anionic surfactant properties.
5. Polymer at certain concentration reduces the IFT values of the surfactant solution.

Conflicting Interests

The author(s) declare that they have no conflicting interests.

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Chukwuebuka Francis Dike is a Master student in the Department of Petroleum Engineering, Federal University of Technology Owerri with research interest in enhanced oil recovery and reservoir engineering.

Nkemakolam Chinedu Izuwa is an Associate Professor in the Department of Petroleum Engineering, Federal University of Technology Owerri with research interest in reservoir engineering, enhanced oil recovery and gas engineering. Izuwa holds a bachelor's degree in Petroleum Engineering from Federal University of Technology Owerri, a master's degree in petroleum engineering from University of Port Harcourt, and a PhD degree in Natural Gas Engineering from Federal University of Technology Owerri.

Anthony Kerunwa is a Senior Lecturer in the Department of Petroleum Engineering, Federal University of Technology Owerri with Research Interest in drilling, production, reservoir engineering and petroleum economics. Kerunwa holds a bachelor's degree in Petroleum Engineering from Federal University of Technology Owerri, a Master's degree in Petroleum Engineering from Federal University of Technology Owerri, and a PhD degree in Petroleum Engineering from Centre for Oilfield Chemicals Research, IPS, University of Port Harcourt.

Nzenwa Dan Enyioko is a Research Technologist in the Department of Petroleum Engineering, Federal University of Technology Owerri with research interest in drilling fluids and enhanced oil recovery. Enyioko holds a bachelor's degree in Geology from Federal University of Technology Owerri.

Lilian Ndoma-Egba is a Master's Candidate in the Department of Petroleum Engineering, Federal University of Technology Owerri with research interest in drilling fluids.

Chukwuebuka Gabriel Mbah is B.Eng Graduate of Petroleum Engineering with research interest in enhanced oil recovery.

Ogonnaya Michael Ogbuatu is a Master's Candidate int the Department of Chemistry with research interest in Petroleum Chemistry.