

Formulation, Characterization and Application of Green Demulsifier for Water-in-oil Emulsion Treatment

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Abstract: This research investigates the challenges associated with conventional chemical demulsifiers in the petroleum industry, including inadequate performance, poor biodegradability, and toxic effects. To tackle these issues, this study introduces a novel, eco-friendly demulsifier made from a natural, sustainable material (blend of Citrus Limon concentrate and glycerol). The synthesized demulsifier was characterized using particle size analysis, transmission electron microscope (TEM), Fourier transform infrared (FTIR) and thermogravimetric analysis (TGA). The synthesized green demulsifier was tested using the standard bottle test method, which involved breaking down a specially formulated water-in-oil emulsion. The findings indicate that the synthesized demulsifier achieves an outstanding rate of demulsification and the maximum efficiency achieved was 82 % within 180 minutes at a concentration of 7% v/v at 80 °C. While for the commercial demulsifier, the maximum efficiency obtained is 94 % at similar conditions. Consequently, the synthetic demulsifier outperforms the green demulsifier. But the green demulsifier have some advantages which includes; easy formulation, low cost, highly biodegradable. The work also explores the impact of various parameters, including settling time, temperature, and demulsifier concentration, on the demulsification process, providing a statistical analysis of their effects. The outcome of this study provides a viable and eco-friendly demulsifier solution for effective demulsification of field emulsions.

Keywords— Citrus Limon, glycerol, green demulsifier, demulsification, emulsion.

1. Introduction

Emulsification is the mechanism that generates emulsions [1]. Emulsions has been present in oil and gas production since its inception [2]. It is a significant issue that typically affects the oil and gas sector. Emulsion typically forms as soon as the well begins to produce. Emulsions are generated when formation water mingles and mixes with the oil stream. The presence of emulsifying agents and the agitation energy from the well stream flow contribute to emulsion formation. Emulsions are a common occurrence throughout many phases of oil and gas activities. Three kinds of emulsion exist: water-in-oil, oil-in-water, and multiple emulsions [3]. Tertiary oil recovery processes involving chemicals that increase the likelihood and stability of emulsion formation [4]. Zolfaghari et al. [5] states that, the presence of an aqueous phase in crude oil emulsions can cause corrosion and erosion in pipelines, as well as impair the performance of facilities such as pumps, storage tanks, and more. Additionally, the presence of emulsions can change the physical characteristics of crude oil, such as its density and flow behavior [6]. When crude oil characteristics change, it can lead to the formation of hard, solid sludge as dense asphaltenes and fine components precipitate out of solution. The coexistence of sludge and emulsion can result in blockages and impediments in facilities used for oil production, transportation, and storage.

Demulsification is the process of resolving emulsions into their distinct components [5]. By disrupting the surface film around water droplets, demulsifiers enable them to combine and separate from the oil, allowing for water removal through settling. The success of demulsification is determined by two primary indicators: the rate of separation and the volume of separated aqueous phase [7]. The evolution of technology has allowed for the integration of different demulsification strategies. There are three primary demulsification approaches: chemical (using synthetic demulsifiers), physical (encompassing mechanical, thermal, and electrical methods), and natural [5]. Mechanical approaches to demulsification, such as desalters, separators, heater treaters, settling tanks and separators, face significant cost challenges due to high operational and maintenance expenses. Chemical techniques are commonly used in tandem with physical methods to improve separation effectiveness, and natural methods employing surfactants and microorganisms provide another option. Chemical demulsifiers are preferred by operating companies due to their efficiency in breaking down oilfield emulsions within a short period [8].

The reduction of interfacial tension by commercial demulsifiers allows for easier disruption of interfaces and faster coalescence of water globules. Chemical demulsifiers are generally grouped into ionic and non-ionic types based on their chemical properties [3]. According to Wong et al. [3], a chemical demulsifier has an advanced molecular weight than a natural surfactant and is made up of two major constituents

a hydrophobic and hydrophilic. Demulsifiers work by attaching to the interface, reducing the thickness of the intermediate phase film, and ultimately causing demulsification. The use of chemical demulsifiers is restricted by several factors, including high costs, poor demulsification performance, limited demulsifier supply, potential negative impacts on crude oil quality, and environmental concerns related to wastewater disposal [9]. These limitations led to the exploration and adoption of green demulsifiers as a viable alternative.

Unlike traditional chemical demulsifiers, green demulsifiers are a more environmentally friendly option, as conventional demulsifiers contain toxic compounds like aromatic groups and phenol [10]. The discharge of wastewater containing residual toxic demulsifiers can have negative environmental impacts, including increased water toxicity and harm to aquatic life, requiring additional treatment and increasing costs. Yaakob and Suleiman [10], propose the use of natural demulsifiers, which are less toxic and biodegradable, as a viable alternative to mitigate the adverse effects of chemical demulsifiers. Studies have examined the potential of natural demulsifiers as an eco-friendly alternative to conventional chemical demulsifiers. The constituents of biological demulsifiers comprise natural compounds like enzymes, plant-derived extracts, cellulose, vegetable oils and microbial cells. These demulsifiers can be separated and naturally broken down after demulsification, reducing their environmental impact and promoting sustainability. Octadecenoic acid, obtained from vegetable oil by-products, has been found to have promising demulsification properties, enabling the aggregation and coalescence of water droplets [10]. The fatty acid-rich *Jatropha* oil by-products have been identified as a potential feedstock for the synthesis of demulsifiers [11].

Similarly, Saat et al. [12] showed that coco betaine and coconut oil can be used as effective natural demulsifiers to separate water from crude oil mixtures. Based on their experimental results, Saat et al. [12] concluded that coco betaine was more effective than coconut oil in separating water. The key to coconut oil's functionality lies in its oil solubility and its derivative's water solubility [12]. The optimal water separation (33%) was obtained with 3 mL of coco betaine in 10 mL of crude oil emulsion, with no notable improvements at higher concentrations.

Erfando et al. [4] explored the demulsifying properties of Citrus Hysteria and Citrus Limon, mixed with liquid soap at a 2:1 ratio, resulting in two innovative formulations. At 70°C, the demulsification efficiency of the organic demulsifier from citrus formulations was 92% at 5 ml demulsifier. Also, Erfando et al. [9] carried out research on corn oil-based demulsifier formulation was explored as an alternative solution for water-oil emulsion challenges. From the study, the results obtained at concentrations of 1ml, 3ml and 5ml that were tested at varying temperatures ranging from 40°C, 50°C,

60°C 70°C and 80°C, shows that, the separation efficiencies of the demulsifier increases as the concentration of the demulsifier increases. At temperature of 80°C, the commercial demulsifier and the green demulsifier has no difference in separation efficiency at concentration of 5ml, and 50°C, 60°C, 70° and 80°C with their respective separation performance of 26ml, 30ml, 32ml and 39ml respectively.

Additionally, researchers have explored demulsifying properties in extracts from, *Jatropha curcas* [13], *Thevetia perfoliata* [14], and *Citrus limonium* [15]. From all this natural or green demulsifiers, are environmentally conducive, economical and have high efficiency of water separation. Despite these studies, more research is still required to improve the performance of this natural demulsifiers deployable in field application instead of the commercial counterparts. The aim of the study is to carry out a laboratory formulation, characterization and demulsification of water-in-oil emulsion with a green demulsifier. The objectives of the study comprise of; to extract, formulate and characterize green demulsifier, to assess demulsifier performance using a bottle test technique and to statistically analyze the laboratory obtained data with ANOVA.

2. Materials and Methods

Lemon (*Citrus Limon*) was bought from a vendor in Swaili-market in Yenagoa Bayelsa state. As well the distilled water, industrial salt (sodium chloride) and an analytical grade non-anionic surfactant (glycerol). A commercial demulsifier, that is widely used in the petroleum industry was used as a standard to compare the green demulsifier efficiency. The synthetic demulsifier used is a PO/EO block copolymer with an average molecular weight of around 2,500 Da [16]. The crude oil samples were obtained from XY field in the Niger Delta region, Nigeria. The equipment used in carrying out the experimental operations include; squeezer, stirring rod, knife, filter paper, static bottles of 15 mL, beakers (50 mL, 250 mL, 300 mL), measuring cup, measuring cylinders, heater, stop watch, digital balance, digital thermometer and water bath

2.1 Demulsifier Formulation

Citrus extract was obtained from the citrus fruit using a juice extractor. The extract was then transferred to a borosilicate glass beaker, where water was gradually evaporated at approximately 70°C to concentrate the solution. The concentration was carried out using a BLS-260A Dean Stark Equipment for a time interval of 90 minutes as depicted in Figure 3.2. The concentration process yielded a viscous liquid, which was then stored in covered plastic sample bottles. The concentrated citrus extract was combined with glycerol, a non-ionic surfactant, at a constant 2:1 ratio. this ratio was in lined with recent study carried out by Erfando et al. [9]. The formulated demulsifier was characterised using FTIR analysis, Thermographic analysis, TEM, and particle size analysis.

2.2 Emulsion Formulation and Demulsification

Firstly, the crude physical properties such as density and API gravity was determined. The density was determined using 50 mL density bottles. The API of the crude was calculated from the obtained density using equation 1 below. The emulsion was formulated with a water cut of 20%. The brine was synthesised using an industrial sodium chloride. The salt concentration was 500 ppm marching the range of salt concentration of the Niger Delta formation. Thorough mixing of the brine solution and the crude oil was achieved using electronic magnetic stirrer.

$$API = \frac{141.5}{\text{Specific gravity}} - 131.5 \dots \dots \dots (1)$$

The bottle test is a laboratory method used to evaluate the demulsifying performance of various chemicals by adding them in different concentrations to emulsion samples in tubes or bottles. Following a predetermined time period, the degree of phase separation and the clarity of the interface between the phases are observed and recorded [17]. In accordance with the water-to-oil ratio, the prepared formation water and the oil samples will be added to the bottle. The water-oil mixture is raised to the test temperature by heating it in a controlled water bath environment. The temperatures values are 30 °C, 55 °C and 80 °C. The highest percentage of water separation will be used to determine the ideal temperature. By increasing the demulsifier volume, the impact of the concentration of the demulsifier will be investigated. Equation 2 shows the equation used in the calculation of the percentage separated water or the efficiency. This laboratory experiment was carried out according to ASTM D4007-02 procedures and standards [18].

$$\% \text{ of water separated} = \frac{\text{Volume of water separated}}{\text{Total water content}} \times 100 \dots \dots \dots (2)$$

3. RESULTS AND DISCUSSION

This chapter discusses the results obtained from the laboratory experimental work. The analysed data will be presented and discussed in the subsequent sections. As the study mainly focused on formulation, characterization and demulsification. Table 1 shows the concentrated yield of *Citrus Limon*. The concentration process yields a mean percentage of 39.8 % at 70 °C for 90 min. The physical properties of the crude oil are shown in Table 2.

Table 1: *Citrus Limon* extraction and concentration parameters

Mean water separated	174 ml
Mean sample yield	115 ml
Total yield volume	289 ml
Percentage yield	39.8 %
Concentration time	90 min
temperature	70 °C

Table 2: Properties of crude oil

Crude oil density	0.9312 g/ml or 58.1329 ib/ft
Specific gravity	0.9316
API °	17.92

3.1 FTIR Analysis

FTIR spectroscopy has become a widely applicable and versatile tool for concurrently analysing organic compounds, encompassing chemical bonding and organic constituents such as proteins, carbohydrates, and lipids. Researchers place significant reliance on FTIR spectroscopy for analysis. FTIR analysis can process a wide range of sample formats, from liquids and solutions to fibres and powders demonstrating its adaptability. The technique also enables the analysis of surface-bound materials on various substrates [19]. FTIR has gained widespread acceptance and is now a favoured characterization technique among researchers and analysts. The characterization analysis is notable for its rapidity, high accuracy, and impressive sensitivity [20].

Figure 1 show FTIR spectra of the formulated green demulsifier and the water-in-oil emulsion. The broad peak at 3270.45 cm⁻¹ corresponds to O-H stretching vibrations in secondary aliphatic alcohols. Additionally, the sharp peak at 3006.19 cm⁻¹ is attributed to symmetrical stretching of alkane (-CH₃) groups within aliphatic hydrocarbons. The peaks at 1742 cm⁻¹ and 1164 cm⁻¹ are characteristic of fatty acids, specifically indicating the stretching vibrations of C=O bonds in ester groups [21]. The presence of a primary aliphatic alcoholic C-OH group is confirmed by the band at 1047 cm⁻¹.

The FTIR analysis revealed the existence of various functional groups within the formulation, specifically fatty, carboxyl, hydroxyl, alkane, and aliphatic alcoholic groups, providing valuable insights into its composition. The detected functional groups are polar and reactive in nature conforming to their significance in demulsification process.

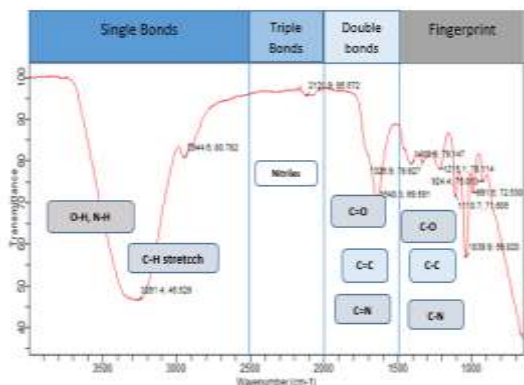


Figure 1: FTIR spectrum of green demulsifier.

3.2 Transmission Electron Microscopy (TEM Analysis)

The TEM analysis revealed varied sizes of formulated green demulsifier. Sizes of the demulsifier ranged from 5.22nm to 15.62nm, with notable sizes being 5.52nm, 10.32 nm, and 13.72 nm, indicating poly-dispersity. Notable sizes (5.54nm, 7.88nm, 10.80nm) indicated poly-dispersity, corroborating particle size analysis revealing a relatively homogeneous distribution. This smaller particle sizes will penetrate into the water-oil interface for easy dissociation of emulsion.

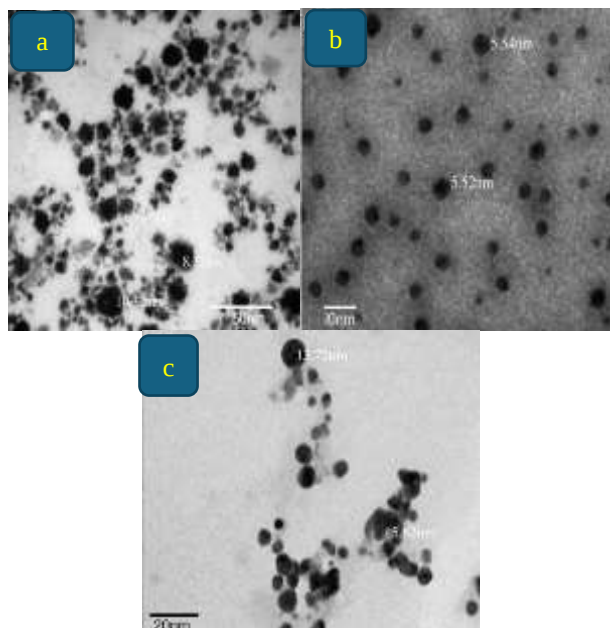


Figure 2: TEM analysis of green demulsifier (a, b, c)

Figure 2 TEM images of prepared demulsifier (a, b and c), revealed that citrus juice concentrate blend with glycerol had spherical shapes with slightly rough edges, probably due to aggregation and flocculation. Nanoscale dimensions enable the demulsifier to interact at the water-oil interface, enhancing water recovery from the emulsion according to Mirhaj *et al.* [22].

3.3 Particle Size Analysis

The particle size distribution analysis of the formulated green demulsifier and the water-in-oil emulsion yields valuable insights (Figure 3). A prominent peak emerges within 7-35 nm for the green demulsifier indicating optimal particle size distribution, suggesting uniform nanoparticle dimensions. The dominant peak signifies relatively homogeneous particle sizes. A higher frequency of particles converges within this range, underscoring uniformity [23]. Table 3 show percentage distribution of peaks of green demulsifier.

Table 3: percentage distribution of peaks of green demulsifier

Peaks	Size (d.nm)	%Intensity
1	15.36	75.7
2	368.4	17.6
3	4364	4.1

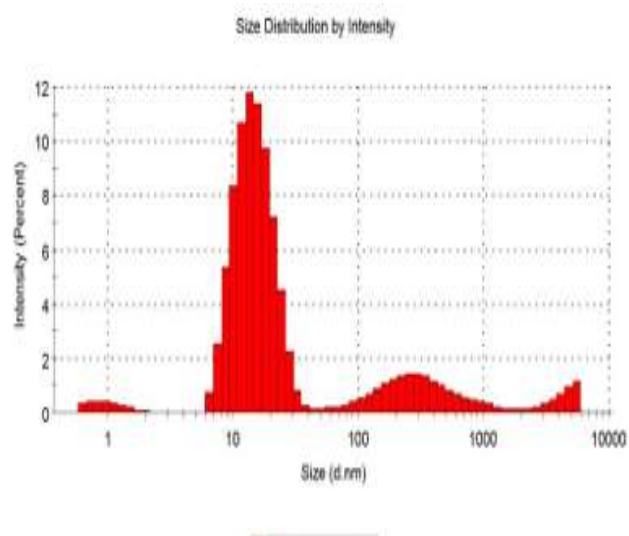


Figure 3: The obtained results graph for green demulsifier.

3.4 Thermographic Analysis

Thermographic analysis is majorly applied to assess the sample's resistance to thermal degradation. The results of the TGA of the citrus blended demulsifier are depicted in Figure 4. The mass loss occurred in three distinct phases. Mass decreased by 4.415% from temperature of 110 °C in the first zone, which is associated with the loss of weakly connected water molecules. A substantial weight reduction of 68.37% occurred in the temperature range of 110-460 °C. Similarly, the temperature ranges of 460-800 °C exhibited a considerable mass loss of 25.91%. The molecular breakdown of the demulsifier was observed at temperatures over 110 °C. However, the formulation retains up to 99.2% of its original mass and structure for laboratory and field application temperature range.

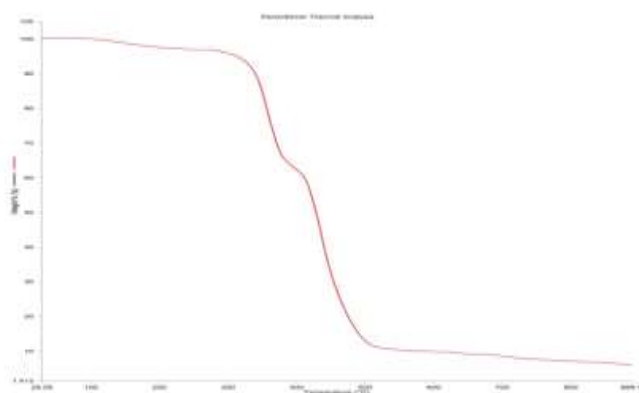


Figure 4: TGA of formulated green demulsifier

3.5 Demulsifier Application

The performance of a demulsifier is significantly influenced by concentration, and the ideal concentration can be identified by the peak demulsification efficiency obtained for each formulation. This study investigates the effects of three different concentrations: 1 % v/v, 4 % v/v, and 7 % v/v. Figure 5a-c provides a graphical representation of how the concentration of the green demulsifier influences the results. Optimal separation was observed with the green demulsifier at a concentration of 7 % v/v, yielding 82 % water separation under conditions of 80 °C and 180 minutes. While for the minimum separation efficiency obtained from the green demulsifier is 20 % at room temperature, 1 %v/v and 30 minutes. For the same operating conditions, at 7% v/v concentration and 80°C temperature, the commercial demulsifier achieved its maximum demulsification efficiency, reaching 94%. As shown in Figures 6a-c, the commercial demulsifier demonstrated its poorest performance, with a demulsification efficiency of 48%, at 30 minutes, 1% v/v, and 30°C. For this current study the

performance of the green demulsifier is less than the commercial counterpart. A direct correlation between concentration and demulsification efficiency is observed, with efficiency increasing steadily as concentration rises from 1% to 7% v/v. This outcome of the study aligns with recent study by Erfando *et al.* [4]. Among the tested concentrations, 7 %v/v emerged as the optimal level for maximizing demulsification efficiency, surpassing the 1 %v/v and 4 %v/v concentrations. As concentrations rise, the components' ability to accelerate the demulsification process also increases. For every concentration value, time is also a crucial factor in the response. The performance efficiencies increase as the time increases from 30 min to 180 min for both green and the commercial demulsifier which aligns with study carried out by Saad *et al.* [24]. But, the commercial demulsifier separated the emulsion more rapidly than the formulated green demulsifier at similar conditions. The formulated demulsifier requires much time to achieve its optimal performance which is depicted in figure 5c.

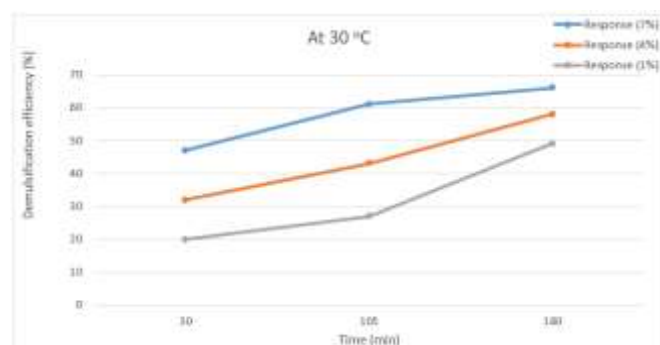
As examined in this study, temperature is a key factor that can significantly alter the characteristics of an emulsion. The effect of temperature on water separation efficiency was studied. 30 °C, 55 °C, and 80 °C were used for the tests as shown in figure 5a-c and 6a-c for both the formulated and the commercial demulsifier. The results indicated that increasing temperatures were associated with higher efficiency values. For both the formulated green demulsifier and the commercial one exhibited optimal performance at all tested temperatures when applied at a dosage of 7 %v/v. At 30 °C, 55 °C, and 80 °C. The corresponding water separation values were 74 %, 81 %, and 82 % for the green demulsifier, and 77 %, 92% and 94 % for the commercial one respectively.

Table 4: Analysis of variance (ANOVA) - green demulsifier

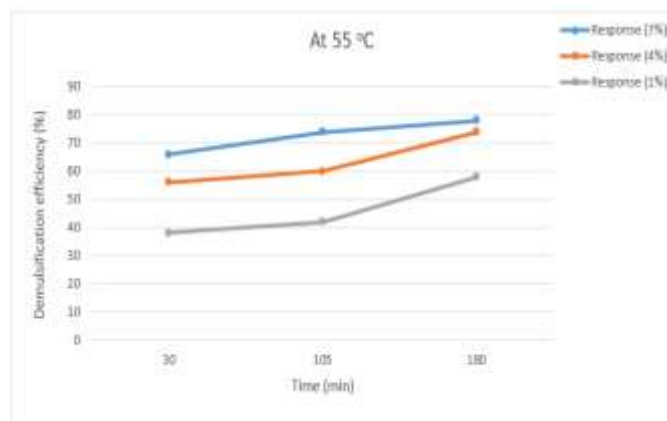
Source	Sum Sq.	d.f.	Mean Sq.	F	Prob> F
Conc.	3008.96	2	1504.48	302.01	0
Temp.	2619.19	2	1309.59	262.89	0
Time	1461.63	2	730.81	146.71	0
Conc.*Temp.	39.48	4	9.87	1.98	0.1904
Conc.*Time	107.04	4	26.76	5.37	0.0212
Temp.*Time	142.81	4	35.7	7.17	0.0094
Error	39.85	8	4.98		
Total	7418.96	26			

The increase in temperature improved the efficacy of water removal. This is because an increase in temperature decreases

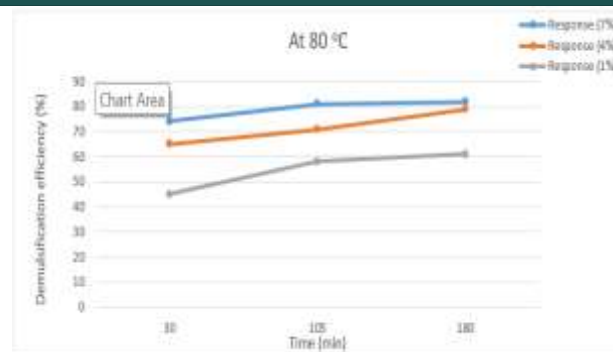
the viscosity of the continuous phase, thereby accelerating the sedimentation of water particles [25]. In addition, heating reduced the interfacial viscosity between the water and oil phases, which destabilized the existing film [9]. In addition, the increase in particulate thermal energy accelerated coalescence in the dispersed phase. Furthermore, applying heat enhanced the Brownian motion occurring within emulsions, facilitating the rapid displacement of demulsifier molecules towards the emulsion interface and accelerating the demulsification phenomenon [26]. These results demonstrated the significance of temperature, time and demulsifier concentration for optimal water separation efficiency as confirmed from the analysis of variance. The p-values for temperature, time and concentration for the green and commercial demulsifiers are less than the significance level as shown in table 4.



(a)

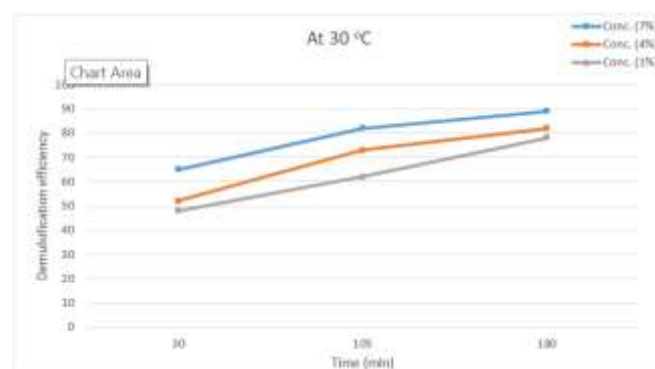


(b)

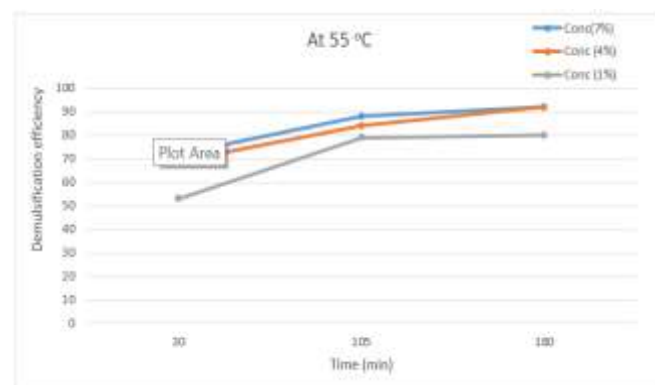


(c)

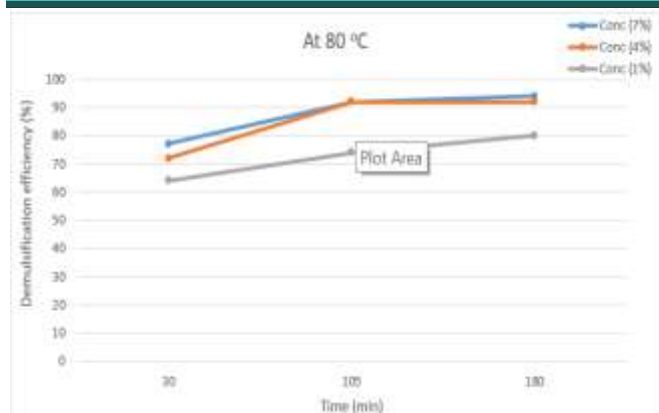
Figure 5a-c: Green demulsifier water separation efficiencies at; 30 °C (a), 55 °C (b) and 80 °C (c).



(a)



(b)



(c)

Figure 6a-c: DML demulsification efficiencies at; 30 °C (a), 55 °C (b) and 80 °C (c).

4. Conclusion

In this work, environmentally friendly green demulsifier were prepared for water-in-oil emulsion separation. FTIR, TGA, TEM and particle size analysis were carried out on the formulated green demulsifier. Static bottle test was conducted to study the performance. Parameters investigated includes concentration, temperature and settling time on synthesized green demulsifier efficiency. The results proved that this green demulsifier was able to separate water-in-oil emulsion. Improving the performance and fully utilizing the formulated demulsifier in field scale application will definitely reduce the environmental impact of conventional demulsifiers. In comparison, the highest demulsification percentage obtained from the green demulsifier was 82% at 7 % v/v 180 minutes of settling time, 80 °C temperature and 94 % efficiency was achieved with the commercial one. Considering the major limitations imposed by applying the commercial demulsifier, the local formulated green demulsifier at its optimal operating conditions can be employed instead. The operational parameters were observed to be highly affecting the performance of the green demulsifier as inferred from the statistical ANOVA analysis. Additional research and development are needed to enhance the locally synthesized green demulsifier performance.

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Conflict of Interest: The authors declare no conflict of interest

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