Full Paper

CHEMICAL EMULSIFICATION OF NIGERIAN HEAVY OIL

B. Ademodi
Department of Chemical Engineering
Faculty of Technology
Obafemi Awolowo University
Ile-Ife, Nigeria.

O.J. Oladeji
Department of Chemical and Polymer Engineering
Faculty of Engineering
Lagos State University
Lagos, Nigeria.
gazzo-17@yahoo.com

ABSTRACT

The inviability of the conventional methods of reducing the heavy oil viscosity by heating or dilution with organic solvent or light crude that is in short supply or uneconomic, and transporting the mixture over a long distance prompted this study. The work investigated the in situ enhanced recovery by modifying the intrinsic high viscosity of Agbabu Heavy Oil 2,166cP and specific gravity 1.0874 at ambient temperature through chemical means and the study of the stability of o/w emulsion produced. A 2^3 Yates' factorial experimental design analysis was carried out on the sample to determine the chemical formulation and combinations that will yield optimum o/w emulsion from NaOH concentration (0.1-1.0M), NaCl concentration (1-4wt %), and temperature (30-80°C). Optimum yield of Agbabu heavy oil in the form of concentrated oil-in-water emulsion was produced at 30°C temperature, 0.1M NaOH concentration, and 1wt% NaCl concentration indicating the effect of their levels on emulsion formation and viscosity. The investigated o/w emulsion behaved almost Newtonian with viscosity ranges from 31.07 to 372.86cP depending on the rate of shear.

1. INTRODUCTION

Heavy oil, a fossil fuel, which occurs naturally on its own or as a by-product of vacuum distillation of crude oil either as a viscous liquid, or a solid consists essentially of hydrocarbons and their derivatives, and is soluble in carbon disulphide, substantially non-volatile and softens gradually when heated. It is blackish or brownish in colour and possesses water proofing and adhesive properties (Hobson and Pohl, 1973). The Nigerian tar sand proven reserve has been conservatively estimated to be more than 505 million barrel. Based on average bitumen content of 15% by weight, the Nigerian rich tar sand deposits constitute a potential reserve of over 40 billion barrel of bitumen (Adegoke and Ibe, 1982), which is about 40% of Canada's deposit known to be one of world's richest deposits of tar sands (Bello *et al*., 2003).

A number of in situ recovery schemes principally steam injection and in situ combustion (ISC) processes that involve the introduction of heat into the reservoir to reduce the viscosity of bitumen have been proposed and tested in the field (Carrigy, 1983). Although the ISC process shows promise of higher thermal efficiency than steam injection, the latter is still the most widely used thermal recovery method, probably because it is less complicated than the combustion process. An unfortunate drawback of steam injection processes however, is where long distance is being considered for the transportation of extracted heavy oil coupled with the inability of the injected steam to break the capillary forces with the problem of steam override and channeling. The net effect is usually low sweep efficiency.

Capillary retention forces can be effectively broken by injecting chemicals such as surfactants and alkalis. Laboratory investigation of alkaline injection processes had begun as early as the 1950s, but tended to focus purely on the study of the reaction of caustic with organic (naphthenic) acid naturally present in the heavy oil to produce surfactant in situ. The use of alkaline chemicals as substitute for commercial surfactants is generally less expensive. Significantly also, the reaction of alkaline chemicals with the organic acid in heavy oil has the potential to reduce the severity of naphthenic acid corrosion in the refining operation (Adewusi, 1989). Reisberg and Dosher, (1956) measured interfacial tension (IFT) between a California crude and various sodium hydroxide solutions by the pendant-drop method. The interfacial tension was lowered by a factor of 1.0 with 0.5%NaOH solution but increased at higher and lower concentrations. Campbell (1977), in comparing sodium orthosilicate with sodium hydroxide for alkaline flooding, used the spinning-drop technique for up to 15 minutes and found an increase in IFT with time. A similar conclusion has also been made by Trujillo (1983).

Investigations of the chemical formulations that would produce optimum yield of o/w emulsion in this study were carried out on heavy oil sample obtained from a location namely: Agbabu site of the heavy oil deposit in Ondo State, Nigeria. Agbabu is located in latitude 60 351.448N and longitude 40 491.883E in the southwestern part of Nigeria (Adebiyi *et al*., 2005). Emulsions are colloidal dispersions in which a liquid is dispersed in a continuous liquid phase of different composition with the aid of sufficient mechanical energy (Narsimhan and Goel, 2001; Delmar, 2006).

Experimental design techniques were employed to determine the optimal setting of each factor viz; temperature; NaOH concentration; and NaCl concentration to maximize or minimize yield of the projected objective of the combination of factors that would produce optimum yield of o/w emulsion and this was achieved with the use of a 2-level factorial experimental design and analysis. Montgomery (2001) gives an account of detailed description of factorial design and analysis.

The present work reports the effect of various phenomena such as alkalinity level, caustic concentration, temperature, and their treatment combinations, (Table 1) that affect o/w emulsion yield, recovery and transportation as earlier done on extracted Agbabu heavy oil by Adewusi, (1989) but in this case with the use of Yates' factorial design and analysis to determine the effect on both the solvent extracted and raw Agbabu heavy crude. This study is part of a research program oriented to develop an effective alkaline slug system for in situ exploitation of heavy oil, bitumen, and tar sands.

2. METHODOLOGY

2.1 Factorial Experimental Design: first level screening

2.1.1 Temperature:

i. the low level was chosen as room temperature (30°C); and
ii. high level, 80°C, represented by t and (+); was based on the context of reservoir temperature conditions where variations of up to 70-80°C are expected, (Trujillo, 1983).

2.1.2 Caustic concentration:

i. low level taken as 0.1M, corresponds to a pH of 13 and
ii. high level taken as 1.0M represented by c and (+); having a pH level of 14, (Adewusi, 1989).

2.1.3 Brine salinity:

i. low level - 1 wt%; and
ii. high level - 4 wt%, represented by s and (+), (Adewusi, 1989).

Meanwhile, each of the low levels of factors was represented by negative arithmetic sign (-).

Studies and analysis of Yates' factorial design method using the levels of factors above on the raw Agbabu heavy oil sample and the solvent extracted Agbabu heavy oil from soxhlet extraction revealed that heavy oil emulsification was being favoured more at the low levels of the design method and this prompted further experiment being carried out on same heavy oil sample at same temperature levels but different caustic and brine concentration, representing a second level screening;

i. caustic concentration:

low level taken as 0.5M and high level taken as 0.8M.

ii. brine salinity:

low level taken as 0.5wt and high level as 1.0wt%.

The order of arrangement of the arithmetic signs in the each of the Factorial design treatment combination (Table 1) below are; temperature; NaOH concentration; and NaCl concentration respectively.

2.2 Procedure

Emulsification tests were performed to serve two purposes. Firstly, to reveal the various phases that might form in the reservoir as heavy oil is mobilized and displaced under specific operating conditions. The second purpose was to identify and assess the individual contribution of factors that promote the formation of a (stable) middle-phase emulsion in the caustic-heavy oil systems set-up.

Table 1: Factorial design treatment combination values

Combination	Value
- - - (1)	30°C; 0.1M; 1wt%
+ - - (t)	80°C; 0.1M; 1wt%
- + - (c)	30°C; 1.0M; 1wt%
+ + - (tc)	80°C; 1.0M; 1wt%
- - + (s)	30°C; 0.1M; 4wt%
+ - + (ts)	80°C; 0.1M; 4wt%
- + + (cs)	30°C; 1,0M; 4wt%
+ + + (tcs)	80°C; 1.0M; 4wt%

Here, the experiment was carried out using pilot scale on both solvent extracted and raw Agbabu heavy oil to generate data on the behaviour of the system for use in design of larger facilities and to reduce the risk associated with the running of a large process plants since it is substantially less expensive than running a full-scale plants.

Equal volumes (20cm^3 each for pilot scale and more for large production) of the heavy oil sample, the caustic concentration, and the brine were mixed together using a stirrer, some were considered at ambient temperature of 30°C and some heated to 80°C, depending on combination in Yates' factorial design analysis. Likewise, the concentration of caustic and brine were also varied. The mixture for each combination was then transferred into a burette to occupy 48cm^3 height of the burette. Each system was observed for the degree of emulsification and of formation of the various phases as measured by the relative height of the phases in the burettes. The course of emulsification was monitored by change in phase height with time until phase stabilization occurred over a period of 20 and 10 days for solvent extracted and raw Agbabu heavy oil respectively. The results of emulsification process and the corresponding phase yield of o/w emulsion after stability on solvent extracted and raw Agbabu heavy oil sample were recorded.

3. RESULTS AND DISCUSSION

Effects of combinations tc, ts, and cs are the interacting effects of two of the factors in a combination. Meanwhile, the effect of increasing the levels of both temperature of mixing and caustic concentration at the same time also lead to a higher yield of emulsion, as indicated by the value of +10.0, corresponding to a combination of tc. ts also gave a +ve value of +0.75, but far less than tc meaning that no significant increase in emulsion yield was observed when the mixing temperature and salinity level were increased simultaneously. Valuable result (+7.0), obtained for cs implied that an increase in caustic concentration and salinity favour emulsion yield.

It can also be seen that increasing the three variables will not be significant. The most significant of the effects is that corresponding to: "- - -" with the highest yield which implies that factors at their low-levels favours the maximum yield of the middle-phase emulsion. This agrees with an earlier work on Nigeria heavy oil with the conclusion that at low mixing temperature of 30°C, a pH of 13 (0.1M NaOH concentration), and 1wt% NaCl, an optimum o/w emulsion yield was obtained (Adewusi, 1989). When the three factors are at their high levels, it was observed that the effect is –ve. This is given by the value of -6.5 corresponding to the combination "+ + +".

Plotting of phase yield size against time for each of the treatment combinations helps to indicate the trend towards stabilization of yield. As shown in Fig. 1, "30°C; 0.1M; 1wt%", showed an increase in middle emulsion phase with time. This may be attributed to the caustic interfacial tension (IFT) which reduces with time as the emulsion phase capacity to solubilize the upper oil phase increases due to the continuous migration of surfactant molecules across the interface. This combination is favourable to the emulsification process because the caustic concentration for emulsification is at its maximum value (pH of 13); and the salinity level (1wt %), combined with the caustic solution drives some of the surfactants initially residing in the phase toward the oil-water interface to cause a further decrease in the IFTs (Adewusi, 1989). This combination gives the highest yield of the emulsion phase.

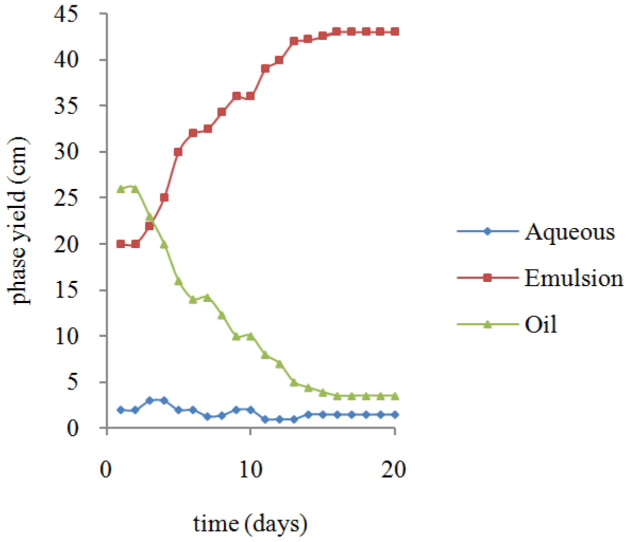

Figure 1: Phase yield-time on extracted Agbabu heavy oil for 30°C, 0.1M, 1wt%, (- - -) factorial combination.

Fig. 2 describes the emulsion phase yield for the combination "80°C; 1.0M; 1wt%" rises with time within 5 days of set-up during which, the top oil phase also reduced proportionally. Afterwards, there was a slight increase before phase stabilization about the 18th day of the set-up. The emulsion yield for this combination is not as favourable as that of '- - -'. This could be due to the mixing temperature of 80°C, which has a reducing effect on the yield because of its increasing the rate of surfactant desorption. Meanwhile, caustic concentration of 1.0M also increases the ionic conductivity of the bitumen-caustic system to a level that there is an increase in IFTs. But, the seemingly high yield effect recorded was brought about by the 1wt% NaCl concentration presence which depresses the surfactant desorption rate (Adewusi, 1989).

Little emulsion phase was recorded for the combination "30°C; 1.0M; 4wt%" (Fig. 3) and this is attributed to high levels of caustic concentration and brine salinity. The trend is the same for the combinations:- + -: 30°C;1.0M;1wt% (Fig. 4), where the caustic level of 1.0M was beyond the peak value of 0.1M. It brings about an increase in IFT at the oil-water interface hence, a reduced emulsion yield; + - +: 80°C;0.1M;4wt% (Fig. 5); and + + +: 80°C;1.0M,4wt% (Fig. 6).

Combination of factors at 80°C; 0.1M; 4wt%, revealed that the emulsion formation capacity of the bitumen-caustic system was reduced by the mixing temperature of 80°C which has a reducing effect on the size of the middle phase. This could be attributed to the effect of the temperature on mass transfer of surfactants across the interfaces, (desorption process), which increases with temperature

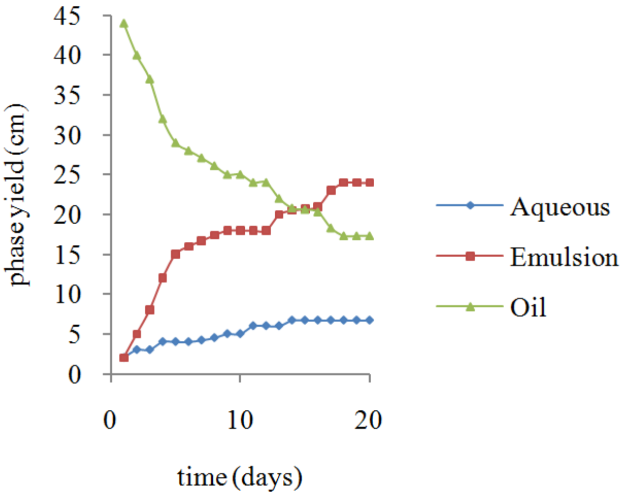

Figure 2: Phase yield-time on extracted Agbabu heavy oil for 80°C, 1.0M, 1wt%, (+ + -) factorial combination.

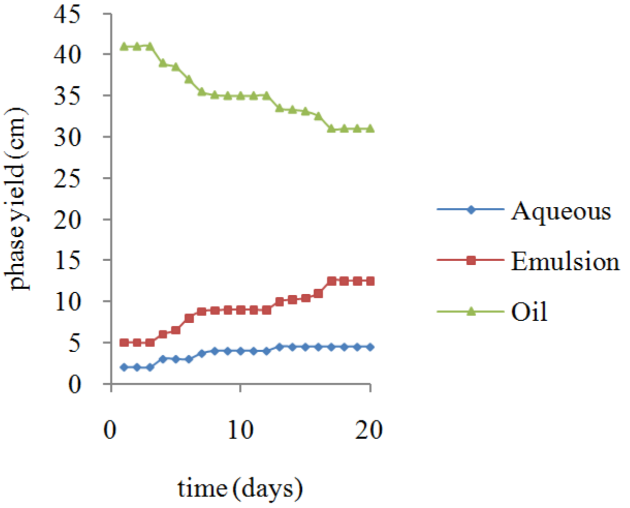

Figure 3: Phase yield-time on extracted Agbabu heavy oil for 30°C, 1.0M, 4wt%, (- + +) factorial combination.

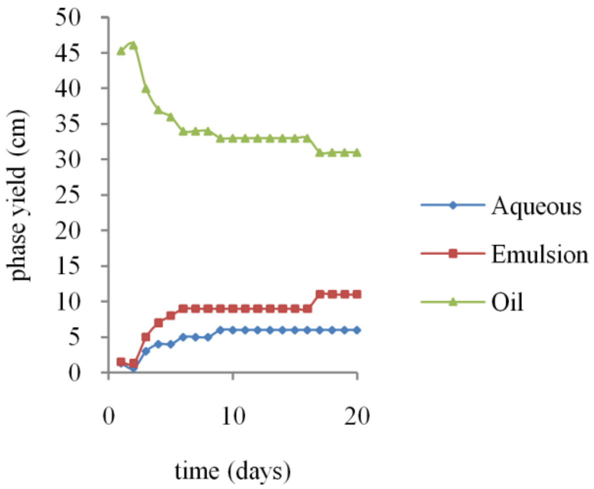

Figure 4: Phase yield-time on extracted Agbabu heavy oil for 30°C, 1.0M, 1wt%, (- + -) factorial combination.

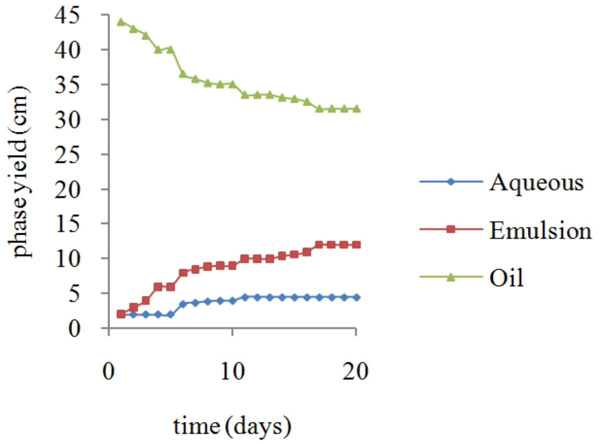

Figure 5: Phase yield-time on extracted Agbabu heavy oil for 80°C, 0.1M, 4wt%, (+ - +) factorial combination.

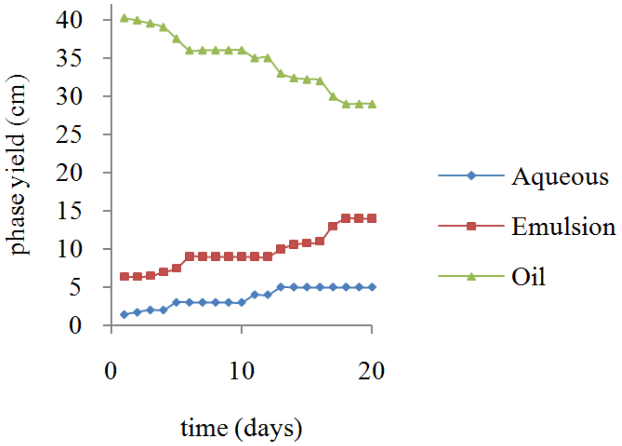

Figure 6: Phase yield-time on extracted Agbabu heavy oil for 80°C, 1.0M, 4wt%, (+ + +) factorial combination.

rise, and leads to a high Interfacial Tension (Adewusi, 1989). Thus, the potential benefit of increased emulsification achievable at the optimum caustic concentration (0.1M) is eliminated by the prevailing high temperature. The unfavourable yield is as a result of the levels of each of the variables being at the high side for combination of factors at 80°C; 1.0M, 4wt%. As said earlier, the mixing temperature 80°C has a reducing effect on emulsion formation. The caustic acts as a salt at the concentration 1.0M, thus the 4wt% salinity level affects the emulsion yield.

The high mixing temperature increased the interfacial tension across the interface initially as seen in combination at 80°C; 0.1M, 1wt%. But, when the bitumen-caustic was cooled, there was a gradual increase in the emulsion phase up to an 'average', as compared to the combination 30°C; 0.1M; 1wt%. (Fig. 7). As shown in heating (Fig. 8) the combination 30°C; 0.1M; 4wt% gave a favourable yield of emulsion. This may be due to the favourable mixing temperature and caustic concentration. However, this 'perfect' combination was affected by the 4wt% salinity level, which was in excess, thus acting as a salt and increased the ionic conductivity of the system to a point where surfactant desorption took place at the interface, and leading to high IFTs.

The emulsification time and emulsion phase stabilization of the combination "30°C; 0.1M; 1wt%" was favourable compared to that of "80°C; 1.0M; 1wt%", as the latter took longer period of time. This could be as a result of the addition of the 1wt% NaCl to the 0.1M caustic solution in the former (which was at its peak value that favoured maximum yield), as this assist to produce effective surfactant migration across the o/w interface and also accelerated the rate of achieving phase stabilization. But, this was not so in the "80°C; 1.0M; 1wt%", as the salinity level which should be helpful in emulsion yield and phase stabilization was affected by the caustic concentration, which at 1.0M acted as a salt, thus producing little or no surfactant being at the oil-water interface, leading to high IFTs, forming little middle-emulsion phase.

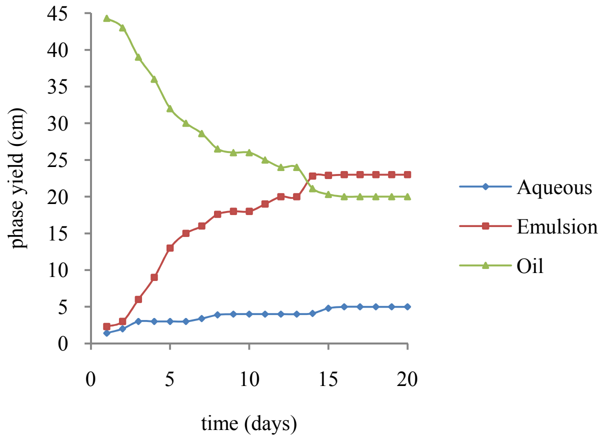

Figure 7: Phase yield-time on extracted Agbabu heavy oil for 80°C, 0.1M, 1wt%, (+ - -) factorial combination.

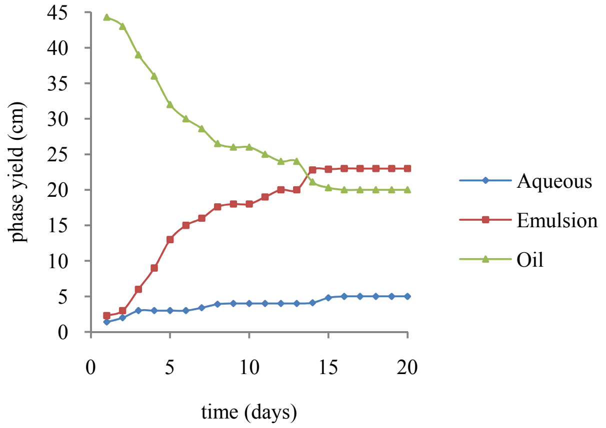

Figure 8: Phase yield-time on extracted Agbabu heavy oil for 80°C, 0.1M, 1wt%, (+ - -) factorial combination.

Similar procedures carried out on the raw Agbabu heavy oil sample showed same trend of the effect for combination of factors at levels: 30°C, 0.1M, 1wt% and 80°C, 0.1M, 1wt% respectively. Though in terms of volume yield they were not the same, with the o/w emulsion produced from solvent extracted Agbabu oil covering about 89.58% by volume of the mixture whereas, o/w emulsion from raw sample covers about 72.92% by volume of the mixture. Meanwhile, no emulsions were produced with the combination of factors at levels 30°C, 1.0M, 1wt%; 80°C, 1.0M, 1wt%; 30°C, 0.1M, 4wt%; 80°C, 0.1M, 4wt%; 30°C, 1.0M, 4wt%; and 80°C, 1.0M, 4wt% on the raw sample. This could be confirmed from the effect on yield from factorial design analysis which is +19.63 (positive mean) on the extracted sample, meaning that the levels of either of the factors can be increased in their interactions and still have o/w emulsion produced, though may not be to the satisfactory volume, this can be confirmed with the emulsion phase yield results of t, c, tc, s, ts, cs and tcs on the analysis. Moreover, the mean value for the raw heavy oil was -6.25 (negative), indicating that lowering the levels of either of the factors (specifically NaOH and NaCl concentrations) below 0.1M and 1wt% could favour emulsification process while increasing may not. This was confirmed when no o/w emulsion were produced from another experimental runs conducted on the raw Agbabu heavy oil using factorial design at NaOH concentration (0.5M to 0.8M) and NaCl concentration (0.5wt% to 1wt%).

The yield of a maximum emulsion phase is not all-in-all because if the emulsion is too loaded with too much oil, a condition of high viscosity arises in which the original problem trying to be solved is generated i.e. poor transportability. Hence, one may have to sacrifice emulsion oil content and/or emulsion phase volume for lower phase viscosity.

NaOH concentration, NaCl concentrations and temperature also play further important role in the production and stability of o/w emulsion, with emulsion produced being homogeneous in nature.

Examining the effect of drop electrostatic charge on coalescence in oil-water emulsion showed that surface charge on drops can substantially inhibit the drop coalescence process (Tobin and Ramkrishna, 1992). The factorial design experimental studies on Agbabu heavy oil showed that electrostatic effects due to hydroxide ion (OH^-) adsorption at the oil-water interface significantly stabilize dispersions with stabilization against coalescence occurring even in the absence of proprietary surfactants. Oil droplets then acquired charge due to the large differences in the dielectric constants of the two immiscible phases even when there are no ionic additives present. Oil drops in water experience electrostatic repulsion due to the overlapping of the double layer forces and thus have low coalescence efficiencies whereas water drops in oil have high coalescence efficiencies because no such repulsion exists. Oil-in-water emulsions do not have sufficiently high coalescence efficiencies to give simultaneous multi-body collisions. In addition, the water droplets engulfed in the oil drops have high coalescence efficiencies thus escaping the oil drop rapidly (Yeo *et al*., 2000).

All the three phases were observed in the emulsification processes using Yates' factorial design and analysis on solvent extracted Agbabu heavy oil with 1.0M (pH = 14) and 0.1M (pH = 13) NaOH concentrations whereas no emulsion was produced with raw Agbabu heavy oil using 1.0M NaOH concentration. The use of 0.1M NaOH concentration with raw Agbabu heavy oil produced all the three phases. Meanwhile, no emulsion was noticed in Adewusi (1989) with 1.0M NaOH concentration under stirred and unstirred conditions with Agbabu heavy oil. Only emulsion phase was noticed with 0.1M NaOH concentration under stirred condition with no aqueous phase under both stirred and unstirred conditions.

When role of surface charges on drops was investigated by dissolving sodium chloride (NaCl) into the aqueous phase (Norato *et al*., 1998). It was observed that adding NaCl to the system reduced the separation time of the phases. Norato *et al.* (1998) suggest that the presence of ions compresses the electric double layer to an extent that the Van der Waals forces overlap the repulsive forces. This implies that the addition of NaCl at high concentrations negates any effect of surface charges, thereby increasing the drop coalescence rate. Hence low concentration of NaCl (1wt %) used enhanced the stability of emulsion.

In crude oil production, the problems related to naphthenic acids arise from the processing conditions. As the pressure drops during production and carbon dioxide is lost from solution, the pH of the brine increases, which in turn leads to dissociation of the naphthenic acid ($RCOOH \rightarrow RCOO^- + H^+$). As a result deposition of naphthenates (Rousseau *et al*., 2000) in oil/water pipelines following complication of naphthenic acids with metal cations present in the aqueous phase may occur.

An increase in temperature favours the formation of water-in-oil emulsions and vice versa (Mao and Marsden, 1977). Temperature affects most of the physical properties of the oil and its effects are important in oil-water systems where surfactants are present. There exists a temperature at which the hydrophilic and oleophilic natures of the surfactant are not balance which is known as the phase inversion temperature. As temperature is increased through the phase inversion temperature, a surfactant alters from stabilizing a certain emulsion configuration (e.g. oil-in-water emulsion) to the other (e.g. water-in-oil emulsion). The existence of a phase inversion temperature is indicative that phase inversion is a spontaneous process and has a strong relationship with thermodynamic behaviour since interaction of the polar group of a non-ionic surfactant with the surrounding water molecules is temperature dependent (Dickinson, 1982). Application of heat will reduce the emulsion viscosity, promote coalescence through increased droplets' motion, alter relative fluid densities and reduce boundary layer strength around the emulsified droplets (Burris, 1977).

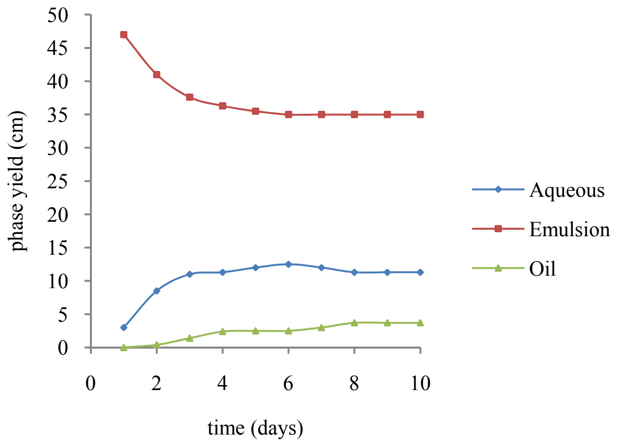

Figure 9: Phase yield-time on raw Agbabu heavy oil for 30°C, 0.1M, 1wt%, (- - -) factorial combination.

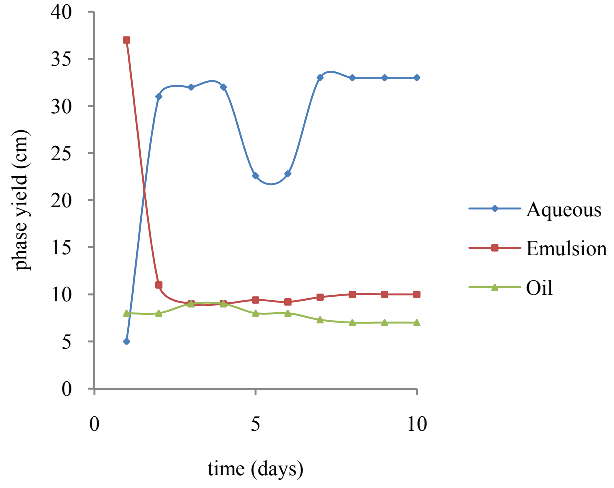

Figure 10: Phase yield-time on raw Agbabu heavy oil for 80°C, 0.1M, 1wt%, (+ - -) factorial combination.

Using Yates' factorial design and analysis for emulsification processes on both solvent extracted and raw Agbabu heavy oil at 80°C and 30°C respectively yielded all the three phases. Meanwhile, in Adewusi (1989) under stirred and unstirred conditions with Agbabu heavy oil at 80°C, all the three phases were observed and same with unstirred condition at 30°C but only emulsion phase was noticed at 30°C under stirred condition.

4. CONCLUSIONS

Transportation of heavy crude oil by conventional methods of heating (Sloan *et al.*, 1979) or mixing with light petroleum fractions (McClaflin, 1982) primarily to reduce their intrinsic high viscosities and hence pressure drops in pipelines has been a problem since the inception of their exploitation this problem is produced especially where long distances are involved, where diluents are in short supply, or where their use for dilution purpose is uneconomic. In such cases, application of other methods in which Agbabu heavy crude of viscosity 2,166cP at ambient temperature could be transported in the form of oil-in-water emulsions (Kessick and Dennis, 1982; Simon and Poynter, 1968) of viscosity range 31.07 to 372.86cP depending on the rate of shear at ambient temperature becomes a more economic option since the viscosity range is less than classical maximum desired pipeline viscosity of 400cP (Urquhart, 1986).

Based on the results obtained in this study, it can be concluded that; the best chemical combination of factors from Yates' factorial design analysis that produced the optimum yield of oil-in-water emulsion is the combination of all the factors at their low levels of 30°C temperature, 0.1M NaOH (pH of 13) concentration, and 1wt % NaCl concentration respectively.

The use of sodium hydroxide for promoting the formation of crude oil-in-water emulsions appears to be a very promising alternative to that of aqueous surfactants because alkalis are generally less expensive than surfactants and at the same time, the reaction of alkalis with organic acids in heavy crude oil or bitumen has the potential to reduce the severity of naphthenic acid corrosion in the refining operation (Adewusi, 1989).

Consequently, no middle-phase o/w emulsion was produced with raw Agbabu heavy oil at any treatment combination involving either optimum level of NaOH concentration (pH of 14) or 4wt % NaCl concentration.

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