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Effect of some citrus juice concentrates on crude oil emulsion

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Crude oil emulsions constitute a major challenge in oil field operations. To resolve this challenge, many researchers have studied crude oil emulsion demulsification patterns in the presence of various demulsifiers. Research is ongoing for discovery of locally sourced biodegradable and inexpensive demulsifiers. The aim of this research is to demusify heavy crude oil emulsion, using locally sourced biodegradable citrus concentrates. The effects of juice concentrates have therefore been studied from three citrus fruit varieties lime, grape and orange on emulsion produced from Basra heavy crude oil. Oil-in-water emulsions were produced from a mixture of heavy crude oil and deionized water in a ratio of 1:100. The mixture was mixed at 13,000 rpm for 5 min. The emulsion formed was mixed with various volumes of juice concentrate (0.05 to 2.0 mL) at 13,000 rpm for 1 min. Demulsification patterns were monitored for a period of 60 min at 5 min interval using the bottle test method. Results show that lime concentrate used at the experimental volumes proved to be an effective demulsifier. Grape concentrate stabilized the emulsion at lower concentrate volumes, but higher volumes resulted in the production of two different emulsions with the less dense emulsion having chocolate colour, showing a typical case of emulsion inversion to water-in-oil (W/O). The orange concentrate caused water in oil emulsion whose stability increased with increasing concentrate volume to be formed. In conclusion, the lime concentrate could be used in breaking crude oil emulsions while the grape and the orange concentrates may be used as emulsifiers in oil spill clean-up or in food and cosmetic Industries.

Key words: Demulsifier, emulsion, emulsifier, grape, lime, orange.

INTRODUCTION

Emulsions are the colloidal dispersion of one liquid in another liquid known as continuous phase (Kokal and Al-Dokhi, 2008). They are also a system in which one liquid is relatively distributed, in the form of droplets, in another substantially immiscible liquid (Salam et al., 2013; Tadros, 2013). Emulsions can be found as an undesired by-product of crude oil production (Sjöblom et al., 2003; Kokal, 2005) and water-in-oil emulsions are sometimes formed after oil products are spilled (Fingas, 2014). According to Alwadani (2009), emulsions can be found in almost every part of the petroleum production and recovery process and can be encountered at many stages during drilling, producing, transporting and processing of crude oil. Crude oil is seldom produced

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alone since it is generally co- mingled with water, which creates a number of problems during oil production (Kokal and Al-Dokhi, 2008). However, produced water occurs in two ways; some of the water may be produced as free water (that is water that will settle out fairly rapidly), and some of the water may be produced in form of emulsions. Emulsions are difficult to treat because of a number of operational problems, such as tripping of separation equipment in gas oil separation plants (GOSPs), production off specification crude-oil and creating high pressure drops in flow lines (Al Ghamdi et al., 2007). Emulsion stabilizers like silt, shale particles, iron and zinc, exist in nature and possess peculiar characteristics, and therefore require different treatments (Abdulkadir, 2010). Emulsions have to be treated to remove the dispersed water and associated inorganic salts to meet crude-oil specification for transportation, storage and exports, and to reduce corrosion and catalyst poisoning in downstream-processing facilities. It is therefore of paramount importance to determine the best treatment method that can be employed to demulsify them to result in enhanced oil recovery.

Demulsification has been defined by Fan et al. (2009) as a process of breaking emulsions in order to separate water from oil, which is also one of the first steps in the processing of crude oil. It is the separation of the dispersed liquid from the liquid in which it is suspended (Salam et al., 2013). Demulsification can be achieved by heating or by mechanical, electrical or chemical means. The traditional methods of eliminating emulsions include high heat and utilization of chemicals. The addition of chemical demulsifiers is the most widely used method (Nour et al., 2007; Atta, 2013). Usually these methods are expensive and the chemicals are carried into the waste water streams or follow the hydrocarbon into the refining process (Nour et al., 2010). The objective of demulsification is to destroy the interface and drive the surfactant to either the oil or the water side (Efeovbokhan and Hymore, 2010) allowing the oil particles and sediments to coalesce and rise to the surface as in creaming. According to Nuraini et al. (2011), chemical destabilisation is caused by three main factors: first, the displacement of the asphaltenic film from the water-oil interface, next, flocculation and finally coalescence of water droplets. Demulsification can be enhanced by decreasing water-phase viscosity or increasing oil viscosity (Souleyman, 2007; Matt et al., 2005).

Demulsifiers used in during oil exploration and exploitation are majorly synthetic in nature and many of them are neither biodegradable nor renewable and they are also expensive. It is therefore necessary to search for methods of demulsification which are cheap, from renewable source and also environmentally friendly and thus do not add to the pollution burden of the ecosystem.

There are natural substances that have been used to break emulsions that occur in day to day living processes. For example, lime juice is used locally in home kitchens to break creams and mucilage. This is the basis for our decision to examine the effect of citrus fruit juice concentrates on crude oil emulsions. In this study, concentrates have been added from three different citrus fruit juices at varying volume concentrations to prepare crude oil in water emulsions to determine their demulsification efficiency. The fruit juice concentrates used is obtained from lime, grape and orange.

MATERIALS AND METHODS

Citrus fruits

Citrus aurantifolia (lime), *Citrus paradisii* (grape) and *Citrus sinensis* Linn. *(Orange),* were purchased at Bodija Market, Ibadan, Nigeria. The citrus fruits were authenticated at Department of Botany, University of Ibadan, Nigeria.

Heavy crude oil

Basra heavy crude oil from Iraq (API 24.7) was obtained from Kaduna Refinery and Petrochemical Company (KRPC), Kaduna, Nigeria on the 6th of December, 2013.

Juicing and concentration process

Juice was removed from the citrus fruit with a plastic cup juicer. The juice was then poured into a borosilicate glass beaker and concentrated by evaporating water gradually at about 70°C on a regulated heating mantle until a viscous liquid was obtained. The concentrate obtained was then stored in plastic sample bottles with cover.

Emulsion production

Oil-in-water emulsions were prepared by dispensing 1 mL of Basra heavy crude oil into 100 mL of deionized water. The mixture was blended with a Qlink electronic blender at 13000 rpm for 5 min. The crude oil emulsion formed was poured into a measuring cylinder and monitored for a period of 24 h and then left on the shelf for a week. Emulsion was produced fresh for each determination.

Demulsification of emulsion by citrus juice concentrate

Known aliquots of citrus juice concentrate, was added to 100 mL of the crude oil emulsion in a blender. The mixture was blended at 13 000 rpm for 1 min and 100 mL of the emulsion poured into a graduated measuring cylinder. The emulsion was monitored for 60 min at 5 min interval at ambient conditions to determine volume of water separated. Volume of water separated at 60 min was used to calculate demulsification efficiency. The procedure was repeated for varying volumes (0.05 to 2 mL) of the concentrate. The same procedure was used for the different citrus fruits:

% of water separated = Volume of water separated / Total water content × 100

Demulsification efficiency = Volume of water separated out after 60 min / Total water content × 100

% Emulsion resolution = Volume of water separated / Total volume of emulsion × 100

Table 1. Rate of demulsification of crude oil emulsion by varying volumes of lime concentrate.

Time (min)	Volume of lime concentrate (mL)											
	0.05	**0.10**	**0.20**	**0.40**	**0.60**	**0.80**	**1.00**	**1.2**	**1.4**	**1.6**	**1.8**	**2.0**	
t_o	0	0	0	0	0	0	0	0	0	0	0	0	
t_5	99	99	97	97	98	96	90	87	87	81	84	88	
t_{10}	99	99	97	97	98	95	91	87	87	83	86	89	
t_{15}	99	99	97	97	98	95	92	89	87	83	86	90	
t_{20}	99	99	97	97	98	95	92	89	87	83	86	90	
t_{25}	99	99	97	97	98	95	92	89	87	83	86	90	
t_{30}	99	99	97	97	98	95	92	89	87	83	87	90	% of water separated
t_{35}	99	99	97	97	97	95	92	89	87	83	87	90	
t_{40}	99	99	97	97	97	95	92	89	87	83	87	90	
t_{45}	99	99	97	97	97	95	92	89	87	83	87	90	
t_{50}	99	99	97	97	97	95	92	89	87	83	87	90	
t_{55}	99	99	97	97	95	95	92	89	87	84	87	90	
t_{60}	99	99	97	97	97	95	92	89	87	84	87	90	

RESULTS AND DISCUSSION

Volume of concentrate obtained from citrus fruit juice

Lime fruits (363), 72 grape fruits and 96 orange fruits produced 463.1, 502 and 365.4 mL of concentrates, respectively. This represents an average of 1.3, 7.0 and 3.3 mL per lime, grape and orange fruit, respectively.

Emulsion production

The emulsion produced was off white in colour, free flowing, and of the oil in water type. The emulsion showed no sign of breaking for 24 h and was stable at ambient conditions (27 to 28°C) for 7 days.

The type of emulsion formed normally depends on volume fraction (Sjöblom et al., 2003). Usually if the volume fraction of one phase is very small as compared to the other, then the phase with the smaller fraction becomes the disperse phase while the other phase becomes the continuous phase. In this study the continuous phase is water. This is because the volume of crude oil (1 mL) used in the formation of the emulsion was very small as compared to the water (100 mL). The oil in water emulsion produced was a tight emulsion as it did not separate for over 24 h (Auflem, 2002). The stability of the emulsion may have been as a result of stabilising agents like asphaltenes, resins, naphthenics and carboxylic acids which are naturally occurring in the crude oil as no stabilisers were added during the production. These stabilisers suppress processes like sedimentation, creaming, flocculation, aggregation, phase inversion and coalescence which are responsible for emulsion destabilisation (Efeovbokhan and Hymore, 2010). This may be as a result of films created around the disperse phase droplets at the oil-water interface by high molecular weight polar molecules which reduce interfacial tension and increase interfacial viscosity.

Effect of lime fruit juice concentrate on heavy crude oil emulsion

Water separation (81-99%) occurred by the 5th minute (Table 1) in all cases within the experimental volume (0.05 to 2.0 mL) of the concentrate used. De-emulsification efficiency remained about the same in all cases. 0.05 mL of the concentrate caused the greatest de-emulsification as it caused 98% of the water to separate out from 100 mL of the emulsion. The amount of water separated decreased gradually with increase in volume of the concentrate until it reached a minimum after which it started to rise again (Figure1). As from 1.8 mL of concentrate, the resolution of the emulsion resulted in the separation of the emulsion into three phases, a crude oil phase at the top, followed by an emulsion phase and the water phase at the bottom of the test container.

Maximum de-emulsification efficiency was 98% and this value is greater than that obtained using microwave irradiation for 180 s as reported by Abdurahman et al. (2006) and was achieved by the 5th minute. The fast de-emulsification process observed with this concentrate may be because the lime concentrate contains surfactants that modify the rigidity of the interfacial film as emulsion stability can be correlated with the mobility of the interfacial films (Guo et al., 2006). This de-emulsification may be as a result of displacement of asphaltenic film at the interface between oil and water followed by flocculation and coalescence (Nuraini et al., 2011). With increase in the concentrate volume, this percent de-emulsification was reduced with an

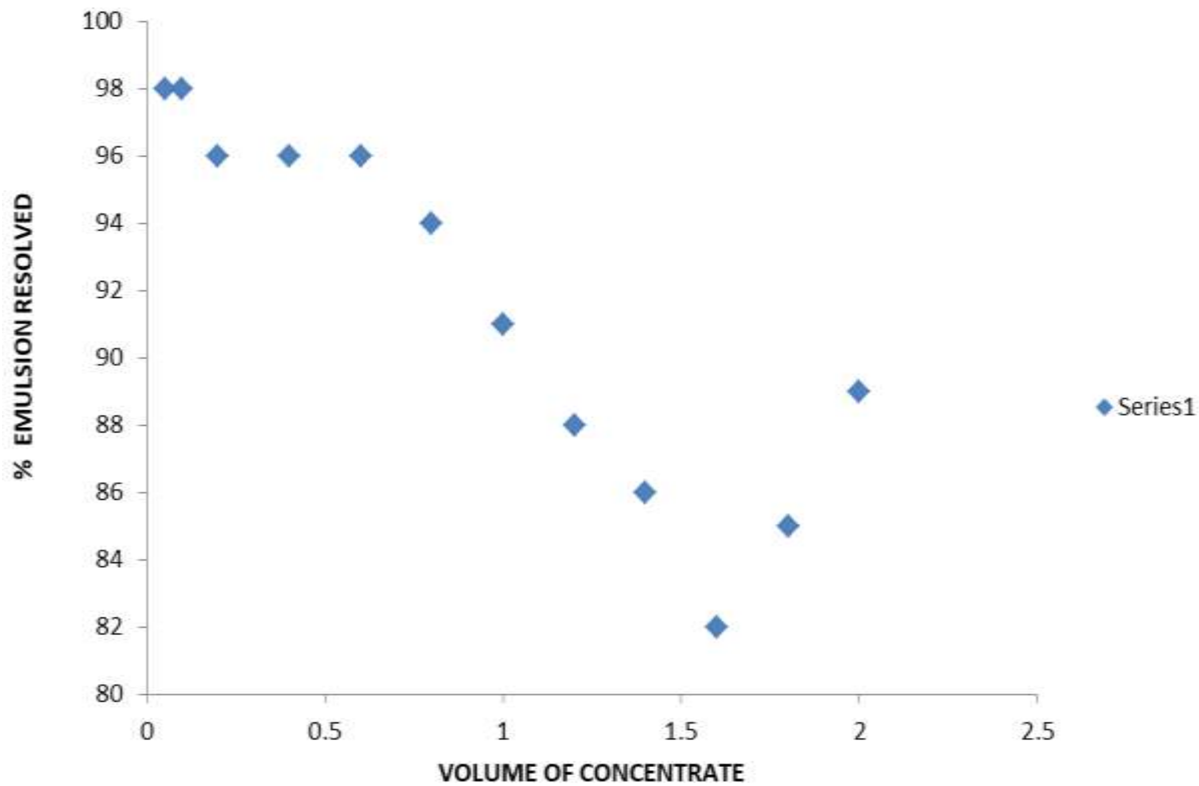

Figure 1. Effect of lime fruit juice concentrate on heavy crude oil emulsion.

accompanying formation of a chocolate coloured emulsion, probably due to increased adsorption of other types of surface active molecules from concentrate at the interface resulting in the inversion of the original emulsion at the right volume, fraction as the fraction of the water left unresolved approached that of the crude oil. Volume of water in crude oil emulsion formed kept on increasing up to the addition of 1.8 mL of the concentrate. Addition of this concentrate volume appears to destabilise the emulsion causing more water to separate out. However, the volume of emulsion resolved was not greater than 98%. This is in agreement with Matt et al. (2005), who reported that no further increase in resolved water occurred once the effective demulsifier concentration was exceeded. The trend observed here could also be as a result of formation of highly viscous interfacial film which is incompressible as concentrate volume increased causing retardation in the rate of oil-films drainage thereby providing a mechanical barrier to coalescence leading to a reduction in the rate of de-emulsification and resolution (Abdulkadir, 2010).

Effect of grape fruit juice concentrate on heavy crude oil emulsion

The addition of 0.05 to 0.6 ml of the concentrate did not cause any separation of water to occur, hence node-emulsification and resolution of the emulsion (Table 2). Instead a very thin layer of a darker coloured emulsion formed on top of the original emulsion. On addition of 0.8 ml of the concentrate, at the 45 to 60th minute, 1% of the emulsion existed as separate layer of the dark coloured emulsion. This value increased to 3% when 1.0 ml of the concentrate was added by the 15 to 60th minute of addition. On addition of 1.2 ml of the concentrate to the original emulsion, the emulsion became completely dark coloured and was like a chocolate mousse for the total experimental period. When from 1.4 to 2.0 ml of the concentrate was added, once again the double layer of emulsion was observed. The darker coloured emulsion was between 90 and 99% of the total emulsion, this value reducing with increasing volume of concentrate.

The thin layer of darker emulsion observed may be as a result of the bulky groups (asphaltenes and resins) from the crude oil which stabilized the original emulsion being displaced by the surfactants present in the grape concentrate which are of higher percent even though with smaller sizes and more polar. The properties of the produced emulsion suggest a situation in which the mixture contains constituents that have surface activities but with wide variation in their molecular sizes. These characteristics cause these surfactants to continually compete for space on the interfacial film surface. At the

Table 2. Effect of grape concentrate on crude oil emulsion.

Time (min)	Volume of lime concentrate (mL)												
	0.05	**0.10**	**0.20**	**0.40**	**0.60**	**0.80**	**1.00**	**1.2**	**1.4**	**1.6**	**1.8**	**2.0**	
t_o	100	100	100	100	100	100	100	100	0	0	0	0	
t_5	100	100	100	100	100	100	100	0	0	0	0	0	
t_{10}	100	100	100	100	100	100	100	0	0	0	0	0	
t_{15}	100	100	100	100	100	100	97	0	0	0	0	4	
t_{20}	100	100	100	100	100	100	97	0	1	1	0	4	
t_{25}	100	100	100	100	100	100	97	0	1	3	3	4	
t_{30}	100	100	100	100	100	100	97	0	1	3	3	4	% of original light brown emulsion
t_{35}	100	100	100	100	100	100	97	0	1	3	3	4	
t_{40}	100	100	100	100	100	99	97	0	1	3	3	4	
t_{45}	100	100	100	100	100	99	97	0	1	3	3	4	
t_{50}	100	100	100	100	100	99	97	0	1	2	2	10	
t_{55}	100	100	100	100	100	99	97	0	1	3	3	10	
t_{60}	100	100	100	100	100	100	98	0	1	3	3	10	

beginning of the study, the low volume of the concentrates may have ensured that mainly the bulky groups align at the interface. As higher volumes of concentrate are added the interface becomes eventually crowded and the bulky groups could no more be adsorbed in the interface, the smaller groups then try to align in available spaces. As voids spaces are filled up, the emulsion rheology changes and a double layer emulsion is observed on account of crowding effects known as structural viscosity (Wang et al., 2013). At certain volume fractions of the water phase when the volume of water left approaches that of crude oil, a different kind of emulsion is formed which is probably water in crude oil emulsion. The newly formed emulsion had a lower density than the former emulsion. The emulsion was also mousse like and thus may have some air incorporated in it. There was gradual increase in volume of the emulsion as the quantity of the concentrate increased. The volume varied slightly with time, showing that the observed trend could be the result of fluctuations in the interfacial film strength, which on account of the presence of multiple surface active agents in the medium may have resulted in formation of mixed emulsions.

Effect of orange concentrate on heavy crude oil emulsion

This concentrate (0.05 ml) produced no resolution but from the 10th minute two layers of emulsion existed with the darker coloured layer being about 1% and increased to 2% from the 15th minute onwards (Table 3). When 0.10 to 0.60 mL of the concentrate was added to the produced emulsion, the darker colored emulsion was produced and was stable throughout the experimental period and the emulsion became increasingly more viscous. Upon addition of 0.8 ml of concentrate, however the emulsion became once again two layered by the 15th minute of addition with the darker and more viscous emulsion being about 99%. Addition of 1.0 to 1.4 ml of the concentrate caused the two layers to disappear, with the emulsion becoming double layered again when 1.6 ml of the concentrate was added. The emulsion then remained stable increasing in stability while the colour became darker when 1.8 and 2.0 ml of the concentrate was added to it. The double layered emulsion was more readily formed. Formation was observed as early as the 10th minute after the addition of 0.05 ml of concentrate. This volume of the concentrate appear to be enough to cause the original emulsion to invert suggesting that the surfactants responsible for causing the formation of the darker coloured emulsion was present at higher concentration in the orange concentrate than in the grape concentrate and also that most of them probably contained short alkyl groups as to preferentially stabilize water in oil emulsions. It also suggests that these surfactants were more soluble in crude oil than water ensuring that crude oil became the continuous phase and that the viscosity increases. There may also have been highly viscous interfacial films which retarded the rate of oil films drainage during the flocculation of water droplets by providing mechanical barrier to separation of the phases (Kilpatrick, 2012; Abdulkadir, 2010). The appearance of the double emulsions when 8 ml of the concentrate was added to the original emulsion and again suggest a case of competitive stabilization by the various surfactants present in the emulsion as the experiment (Table 3) proceeded and increased quantity of the concentrate became present in the mixture suggesting that the emulsion was slightly destabilised at those concentrations and thus about 1% of the emulsion inverted.

Table 3. Effect of orange concentrate on crude oil emulsion.

Time (min)	Volume of lime concentrate in mL												
	0.05	**0.10**	**0.20**	**0.40**	**0.60**	**0.80**	**1.00**	**1.2**	**1.4**	**1.6**	**1.8**	**2.0**	
t_o	100	0	0	0	0	0	0	0	0	1	0	0	
t_5	100	0	0	0	0	0	0	0	0	1	0	0	
t_{10}	99	0	0	0	0	0	0	0	0	1	0	0	
t_{15}	98	0	0	0	0	1	0	0	0	1	0	0	
t_{20}	98	0	0	0	0	1	0	0	0	1	0	0	
t_{25}	98	0	0	0	0	1	0	0	0	1	0	0	% of original light brown emulsion
t_{30}	98	0	0	0	0	1	0	0	0	1	0	0	
t_{35}	98	0	0	0	0	1	0	0	0	1	0	0	
t_{40}	98	0	0	0	0	1	0	0	0	1	0	0	
t_{45}	98	0	0	0	0	1	0	0	0	1	0	0	
t_{50}	98	0	0	0	0	1	0	0	0	1	0	0	
t_{55}	98	0	0	0	0	1	0	0	0	3	0	0	
t_{60}	98	0	0	0	0	1	0	0	0	3	0	0	

Conclusion

Conclusively, the lime fruit concentrate was shown to be a very good demulsifier for crude oil in water emulsion, while the grape and orange concentrates are not. 0.05 ml of the lime concentrate or less is all that is required to effect demulsification of a crude oil in water emulsions produced from a mixture containing about 1% crude oil in water. However, the grape and orange concentrates further stabilised the emulsion by causing it to invert to water in crude oil emulsion and therefore could be used to mop off crude oil spills. The orange concentrate is a better stabiliser than the grape concentrate.

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CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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