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Chemistry Research and Practice

Research Article

Investigation of the Emulsification and Demulsification Abilities of Gmelina Arborea Leaves Extract in Oil-In-Water Emulsions

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Received: 2024 Aug 02 **Accepted:** 2024 Aug 22 **Published:** 2024 Nov 06

Abstract

The emulsifying and demulsifying abilities of ethanol, n-hexane and distilled water extracts of Gmelina arborea leaves in oil-in-water phase was investigated. Quantitative and qualitative phytochemical analyses of the extracts were done by GC-MS method. The results obtained showed the presence of many bioactive compounds like saponins, alkaloids, flavonoids, tanins etc. The emulsification and demulsification tests were carried out at different volume concentrations of the extract and different time, using total volume of water separated as the yard stick. The results obtained revealed that the ethanol and distilled water extracts emulsified the liquid mixture while the n-hexane extract demulsified the solution formed. This is attributed to the surfactant molecules found in the extracts which reduced the interfacial tension between the two liquids. The surfactant molecules (saponins) were more in the ethanol extract than the distilled water extract. Only the n-hexane extracts demulsified the mixture. This attributed to the presence of dehydrating compounds in the extract (Terpenoids). Synthetic demulsifier was used for comparison sake and it was able to the break the oil-in-water emulsion easily. This is attributed to the concentration of the constituents compared to the natural demulsifier.

Keywords: Gmelina Arborea, Extracts, Oil-in-Water, Phase, Ethanol, Hexane, Distilled Water, Synthetic and Emulsifier

1. Introduction

Emulsion is a mixture of two or more liquids that are immiscible, where one of the liquids is finely dispersed in the other. In oil in water emulsion, the continuous phase is water and dispersed phase is oil, while in a water in oil emulsion, oil is the continuous phase. Emulsions belong to a class of two-phase systems of matter called colloids, [1].

Emulsification refers to a process of dispersing two or more immiscible liquids to form a semi-stable mixture, by the use of emulsifiers. Emulsifiers are surface active agents that stabilize immiscible dispersions. They permit the mixing of two or more dissimilar liquids and prevent individual separation of the liquids. Emulsifiers are also known as surfactants [2]. Stability of emulsions are affected by four main factors: age of emulsion, viscosity, specific gravity and water percentage.

Surfactants are surface active agents with hydrophilic head (polar, water loving head), lipophilic (fat loving) and hydrophobic tail (water resistant or non-polar tail). They lower the surface tension between fluids, act as wetting agent, detergents, emulsifying agents, foaming agents, demulsifying agents or dispersants. Surfactants migrate to the surface between phases.

Abubakar and Aliyu also reported that chemicals are unable to resolve an emulsion when they have no dehydrating component which could result in water separation [3]. Ojinnaka et al. (2016) categorized demulsifiers and emulsifiers as ionic and non-ionic surfactants which may either break an emulsion or prevent emulsion from resolving. Petroleum industries employ surface-active agents for application in several fields related to crude oil extraction, recovery, transportation, and cleaning of oil carrying tanks [4]. Synthetic chemicals are expensive, hazardous and toxic as they can cause serious health and environmental challenges [3]. Biosurfactants are promising alternative molecules to synthetic emulsifiers and demulsifiers for industrial and domestic applications due to their high biodegradability, low toxicity, multi-functionality, environmental capability, and availability [4].

Demulsification is a process of breaking of an emulsion due to the presence of surfactants, esters and alcohol found in the extracts and their interactions with the emulsion which leads to weakening of the interfacial films that is holding the emulsion. This is in accordance with the study of Falode and Aduroja which stated that demulsifiers are surface-active agents so when mixed with two immiscible liquids

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(example, oil and water), they migrate to the oil-in-water interface, weaken and break the rigid films and improve water droplet coalescence. Esters and alcohol have good ability in promoting water separation and the hydroxyl groups are better flocculent.

This study utilized Gmelina arborea (of the family, Verbenaceal). It is a tree of the genus Gmelina and specific epithet arborea. It is native to tropical moist forest from india, Burma, Southern China, Gmelina is widely introduced in Brazil, Gambia, Ivory Coast, Nigeria, Malaysia, Honduras, Panama, Philippines and Sierra Leone, etc. [5]. The main chemical constituents of Gmelina arborea include lignans, iridoid glycoside, flavonoids, flavons, flavone glycoside and sterols.

2. Materials and Methods

Gmelina Arborea leaves were collected from the premises of NDDC hostel in Rivers State University, Port Harcourt, Rivers State, Crude oil sample was gotten from Ahoada Local government Area, Rivers State, dichloromethane (DCM), n-hexane, methanol, and distilled water were obtained from the Department of Chemistry, Rivers State University laboratory. All the reagents used were of analytical grade. The leaves were identified by a taxonomist. They were plucked and washed with distilled water so as to reduce impurities and contamination. air dried for approximately fifteen (15) days, ground into fine powder using a mini hand grinding machine. Extraction was done using distilled water, ethanol and n-hexane.

2.1. Formulation of Green Emulsifiers

10g of each of the extract was weighed into a beaker; 20ml of the DCM was added to the beaker containing the extracts and was stirred continuously until a homogeneous mixture was formed.

2.2. Emulsification Test

500ml of crude oil and 250ml of distilled water were mixed together to form a homogeneous solution. 10ml of the mixture was transferred into six centrifuge tubes calibrated 15ml each. Into the 10ml of the oil in water formulated emulsion, 1 ml of the ethanol, hexane and distilled water prepared, and synthetic emulsifiers were added respectively and placed a tube rack for 60mins, at 10mins intervals, readings were taken for each solvent extract respectively until 60mins.

The synthetic emulsifier used was glycerine emulsifier.

3. Results

Table 1: Phytochemical Components of the Distilled Water Extract

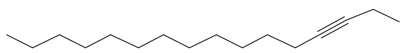
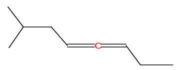
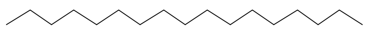
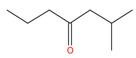
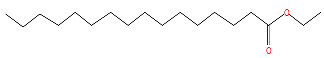
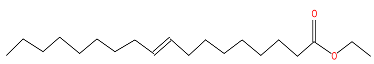
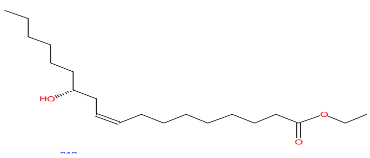
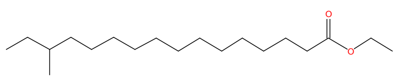
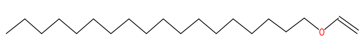
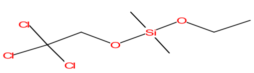
s/n	RT	Compound Name	MW	Structure	%Comp
1	6.469	3-Hexadecyne	222		2.107%
2	8.305	3,4-Octadiene, 7-methyl- (C_9H_{16})	124		3.279%
3	8.918	Heptadecane ($C_{17}H_{36}$)	240		8.076%
4	9.988	4-Heptanone, 2-methyl- ($C_8H_{16}O$)	128		3.911%
5	11.847	Hexadecanoic acid, ethyl ester ($C_{18}H_{36}O_2$)	284		16.156%
6	13.432	9-Octadecenoic acid ethyl ester ($C_{20}H_{38}O_2$)	310		28.376%
7	13.489	9-Octadecenoic acid, 12-hydroxy-,ethyl ester, [R-(Z)]- ($C_{20}H_{38}O_3$)	326		22.064%
8	13.650	Ethyl 14-methyl-hexadecanoate ($C_{19}H_{38}O_2$)	298		10.901%
9	18.942	Octadecane, 1-(ethenyloxy)- ($C_{20}H_{40}$)	296		2.618%
10	20.058	Silane, dimethyl(2,2,2-trichloroet hoxy)nonyloxy- ($C_6H_{13}C_{13}O_2Si$)	250		2.512%

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Table 2: Phytochemical Components of the Hexane Extract

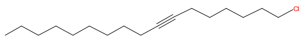
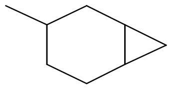
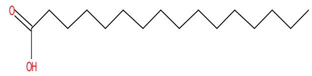
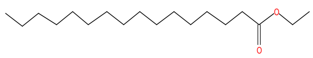
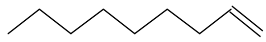
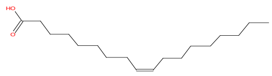
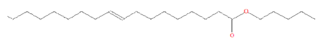
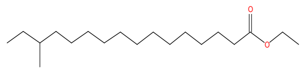
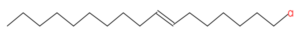
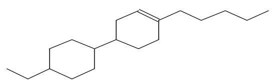
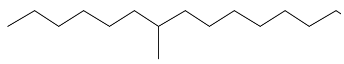
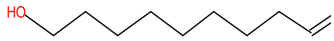
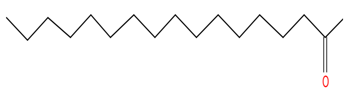
S/n	RT	Compound Name	MW	Structure	%Comp
1	10.995	7-Heptadecyne, 1-chloro- ($C_{17}H_{31}Cl$)	270		5.769%
2	16.305	Bicyclo[4.1.0]heptane, 3-methyl- (C_8H_{14})	110		8.027%
3	17.592	n-Hexadecanoic acid ($C_{16}H_{32}O_2$)	256		7.212%
4	17.838	Hexadecanoic acid, ethyl ester ($C_{18}H_{36}O_2$)	284		17.060%
5	19.246	1-Nonene (C_9H_{18})	126		20.163%
6	19.435	Oleic Acid ($C_{18}H_{34}O_2$)	282		11.821%
7	19.492	trans-9-Octadecenoic acid, pentyl ester ($C_{23}H_{44}O_2$	352		8.878
8	19.652	Ethyl 14-methyl-hexadecanoate ($C_{19}H_{38}O_2$)	298		6.292
9	26.776	7-Heptadecene, 1-chloro- ($C_{17}H_{33}Cl$)	272		7.110%
10	26.856	Cyclohexene, 4-(4-ethylcyclohexyl) -1-pentyl- ($C_{19}H_{34}$)	262		7.669%

Table 3: Phytochemical Components of the Ethanol Extract

s/n	RT	Compound Name	MW	Structure	%Comp
1	8.917	Heptadecane, 7-methyl- ($C_{18}H_{38}$)	254		5.589%
2	10.319	9-Decen-1-ol ($C_{10}H_{20}O$)	156		4.213%
3	10.760	1-Octadecyne ($C_{18}H_{34}$)	250		3.266%
4	11.847	Hexadecanoic acid, ethyl ester ($C_{18}H_{36}O_2$)	284		15.208 %

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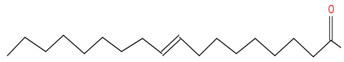
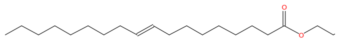
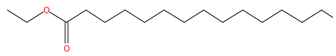
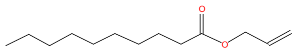
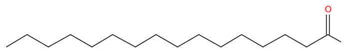
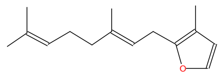
5	13.432	(E)-9-Octadecenoic acid ethyl ester ($C_{20}H_{38}O_2$)	310		28.572 %
6	13.489	trans-9-Octadecenoic acid, pentyl ($C_{23}H_{44}O_2$)	352		21.275 %
7	13.650	Octadecanoic acid, ethyl ester ($C_{20}H_{40}O_2$)	312		11.307 %
8	18.965	Decanoic acid, 2-propenyl ester ($C_{13}H_{24}O_2$)	212		3.094%
9	20.256	Palmitic acid vinyl ester ($C_{18}H_{34}O_2$)	282		4.575%
10	22.885	Sesquirosefuran ($C_{15}H_{22}O$)	218		2.985%

Table 4: Phytochemicals present in the Different Solvent Extracts

Phytochemical	Hexane extract	Ethanol extract	Distilled water extract
Tannins	+	+	+
Saponnins	-	++	+
Terpenoids	+	-	-
Flavonoids	-	+	+
Alkaloids	-	+	+
Cardiac Glycosides	-	-	-
Antraquinone	-	+	-

Keys: ++ absolutely present, + moderately present, - Below detectable limit

Table 5: Emulsification Test Result of 1 ml Concentration of the Green and Synthetic Emulsifiers

Emulsifiers	10mins/ml	20mins/ml	30mins/ml	40mins/ml	50mins/ml	60mins/ml
Hexane emulsifiers	**1.0**	**1.5**	**1.8**	**1.7**	**1.9**	**2.0**
Ethanol emulsifiers	**0**	**0**	**0**	**0**	**0**	**0**
Distilled water emulsifiers	**0**	**0**	**2.5**	**6.0**	**6.5**	**6.5**
Synthetic emulsifier	**0**	**0**	**0**	**0**	**0**	**0**
Blank	**2.8**	**3.7**	**5.5**	**5.5**	**5.6**	**5.6**

Table 6: Emulsification Test Result of 2 ml Concentration of the Green and Synthetic Emulsifiers

Emulsifiers	10mins/ml	20mins/ml	30mins/ml	40mins/ml	50mins/ml	60mins/ml
Hexane	0	0	7.0	7.5	7.5	7.5
Ethanol	0	3.5	4.5	5.5	6.5	7.2
Distilled water Emulsifier	0	0	0	0	0	0
Synthetic emulsifier	0	0	0	0	0	0
Blank	2.8	3.7	5.5	5.5	5.6	5.6

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Table 7: Demulsification Test Result of 1 ml of Green and Synthetic Demulsifier

Extracts	10 mins/ml	20 mins/ml	30 mins/ml	40 mins/ml	50 mins/ml	60 mins/ml
Synthetic demulsifier	3.4	4.2	4.5	5.6	5.6	5.6
N-hexane	1.0	1.5	1.8	1.9	1.9	2.0
Ethanol	0	0	0	0	0	0
Distilled water	0	0	5.5	6.0	6.5	6.5
Blank	2.8	3.7	5.5	5.5	5.6	5.6

Table 8: Demulsification Test Result of 2 ml of Green and Synthetic Demulsifier

Extracts	10 mins/ml	20	30	40	50	60
		mins/ml	mins/ml	mins/ml	mins/ml	mins/ml
Synthetic demulsifier	5.6	5.6	6.0	6.5	6.5	6.5
N-hexane	0	0	7.0	7.5	7.5	7.5
Ethanol	0	3.5	4.5	5.5	6.5	7.2
Distilled water	0	0	0	0	0	0
Blank	2.8	3.7	5.5	5.5	5.6	5.6

4. Discussion

4.1. Phytochemicals

Results of the phytochemical analyses of the different solvent extracts presented in Tables 1 to 4, showed the presence of Alkaloids, Saponins, Terpernoids, Tannins, Antraquinones and Flavonoids. Ethanol extract yielded absolute amount of saponin while other solvent extract yielded moderate to little amount of saponin.

4.2. Emulsification

The emulsification test carried out with the different solvent extracts at same time interval and different volumes concentrations (1 ml and 2 ml concentration), showed a trend in the emulsifying ability of the distilled water and ethanol extract emulsifiers. The ethanol extract emulsifier emulsified the liquids homogeneously at 1 ml concentration while distilled water extract emulsifier did not completely emulsify the liquids. This is because there were traces of emulsion breakage in the solution. This may be as a result of the quantity of saponin and the surfactant likely compounds present in the ethanol extract. Even at low concentration, it was able to reduce the interfacial tension of the liquids thereby increasing the solubility of the hydrophobic molecule of the oil in water interfaces. The inability of the distilled water extract emulsifier to emulsify completely may be because of insufficient amount of saponin and other surfactant likely compound in it. With increase in concentration (2 ml), the distilled water extract emulsifier was enriched thereby gaining ability to reduce the interfacial tension of the liquids and increasing the emulsifying ability.

For an emulsion to occur successfully the solution system must be in equilibrium with equal amount of polar molecule from the extract and the water, and equal amount non-polar molecule from the extract and the oil. This is in accordance to the study of Yaqoob, et. a., l (which revealed that the dispersed oil droplets inside the water field emulsion has films that surround them which help stabilize the emulsion and the films are believed to be formed by the adsorption of high molecular polar molecules [1].

The hexane extract emulsifier did not emulsify the solution both at 1 ml and 2 ml volume concentration. The may be because hexane may not have extracted sufficient amount of surfactant like compounds reduce the interfacial tension between the oil and water interfaces and aid the emulsification process. This report is in agreement with the study of Rosen, & Kunjappu which stated that the efficiency of a surfactant is determined by its ability to reduce surface/ interfacial tensions which is a mechanical energy [2].

The synthetic emulsifier employed in this research emulsified the solution. The blank was used as a control to certify the fact that water and oil are two immiscible liquids and will spontaneously separate on its own.

4.3. Demulsification

At 1 ml concentration of the extracts, the n-hexane solvent extract and distilled water solvent extract were able to demulsify the water-in-oil emulsion, this may be as a result of the presence of surfactant likely compounds in the extracts. Whereas at the same 1 ml concentration the ethanol

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solvent extract was unable to demulsify rather it gave a clear emulsion without any breakage or interface, although surfactant likely compounds were also found in the ethanol solvent extract, it's inability to demulsify was attributed to the concentration of the extracts that was added to the oil-in-water mixture.

At 2 ml concentration of the extracts, the ethanol solvent extract and the hexane solvent extract were able to demulsify while the distilled water solvent extract was unable to demulsify. This may be because surfactants can as well act as demulsifying agents. When there is an unequal amount of polar and non-polar components in the solution it makes the system unstable. the reason can also be likened to what happened at the 1 ml concentration, surfactant likely compounds were found in all the extracts used for the analyses but the ability to break an emulsion (demulsify) was likened to the volume of the concentration added to the oil-in-water mixture.

This study shows that the ability of an extract to demulsify is affected by the volume of extract. The hexane solvent extract was able to demulsify at all concentration, this may be attributed to the percentage composition of the surfactant like molecules in the extract which helped to resolve the emulsion. The ability of water molecules to break up solute molecules into a solution explains this. The polar water molecules are attracted to the hydroxyl group by hydrogen bonding which occurs between the hydrogen of water molecules and the oxygen in the alcohol. Emulsion demulsification starts within 10 minutes of injecting demulsifier. Abubakar & Aliyu and Boisa et al, made similar observations [3, 6]. Falode and Aduroja reported that a large dose of demulsifier may be detrimental because demulsifiers are surface-active agents like the emulsifiers.

Extracts that have the ability to demulsify have been likened to have lower turbidity. The interface quality was sharp as a result of good dehydrating ability of the extract.

The ethanol extraction solvent demulsifier gave a similar result with that of n-hexane extraction solvent demulsifier. Reduction in the volume of water separated at higher concentration may be due to the high dosage of the demulsifier injected. A comparable observation was made by Abubakar and Aliyu [3]. In terms of water clarity ethanol extract demulsifier was clearer.

The ethanol extraction solvent demulsifier was able to break the emulsion. This shows little percentage of dehydrating properties in the extract therefore, it was unable to separate large volume of water compared to n-hexane extract demulsifier. Chikezie and Adewale got a similar result in their research work when they used mangrove plant extract to break oil-in-water emulsion.

The result of synthetic commercial demulsifier used in this study shows that the synthetic demulsifier are able to break oil-in-water emulsion easily. This may be because commercial synthetic demulsifiers are more concentrated, hence, separates more volume of water compared to natural demulsifier from plant [7-34].

5. Conclusion

Gmelina arborea leaves contain phytochemicals that are surface active agents. Distilled water and ethanol extracts have the ability to emulsify oil in water. Ethanol extract gave a better emulsion compared to distilled water extract. Hexane extract of Gmelina arborea leaves is not a good emulsifier of oil in water phase. The synthetic emulsifier gave better result than the leaves extracts.

Acknowledgement: The contributions of Mrs Harmony Nwosu of Petroleum Engineering Department, Rivers State University, and all the technical staff of the Department of Chemistry, Rivers State University, Port Harcourt are highly appreciated.

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