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Fluid emulsion base potential of shea butter

Ayobami O. Oyedele*

Department of Pharmaceutics, Faculty of Pharmacy, Obafemi Awolowo University, Ile-Ife. Nigeria.

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Abstract

The potential of shea butter (SB) as base for fluid, soap-stabilized emulsion and the stability of such formulations have been studied. Stability features (homogeneity, viscosity) of SB/Liquid paraffin (LP) blends containing 1–50 %w/w of SB at ambient temperature were determined to locate the threshold concentration range for fluid outcome. SB emulsion formulations were prepared from the fluid SB/LP blends by homogenizing each at 1:9 ratio (v/v) with potassium hydroxide (KOH) aqueous solution (0.1, 0.2, 0.4, or 0.6 M concentration, respectively), and with 0.2 M KOH at different mixing ratios (2:8, 3:7, 4:6, or 5:5 v/v), respectively. The physical consistency and stability characteristics (redispersibility, creaming or breaking) of the formulations were studied at room temperature over 12 weeks. The mean disperse-phase globule size and viscosity of formulation samples stable for ≥8 weeks were determined. SB/LP blends containing 1–20 %w/w SB were pourable liquids; SB separated out as sediment from the fluid blends left to stand for 48 h. Higher concentrations solidified. Fluid emulsions were produced at 0.5–18.0 %w/w SB concentrations, having mean globule size of 4.4–43.1 μm and viscosity of 153–1863 cP. The graded KOH concentrations gave emulsion products with different levels of stability. While some were unstable (broken, immobile) in storage, those formulated with 0.1 M KOH (153–354 cP viscosity) remained stable liquids throughout the 12 weeks of study. The best (most stable) formulation contained 4.5 %w/w SB emulsified with 0.2 M KOH.

Keywords: Shea butter; Emulsion base; Fluid; Formulation

INTRODUCTION

Shea butter is the natural fat obtained by extraction from seeds of the tree, *Vitellaria paradoxa* C.F. Gaertner (synonym, *Butyrospermum parkii* [G. Don.] Kotschy), Family Sapotaceae, which is indigenous to the East, West and Central African sub-regions (Djekota *et al*., 2014) and thrives in Southwest Nigeria (Irvine, 1963). The constituents (Krist *et al*., 2006; Maranz and Wiesman, 2004) and physicochemical properties of the shea seed butter from various regions have been published (Chukwu and Adgidzi, 2008; Vincenzo *et al*., 2005).

Shea butter has several important domestic and industrial uses, such as an edible cooking oil (Okafor, 1980; Okullo *et al*., 2010), for preparation of cosmetics and skin care products (Athar and Nasir, 2005; Glew and Lovett, 2014), in soap making (Warra *et al*., 2009; Warra, 2013) and several pharmaceutical applications. The pharmaceutical uses of shea butter have, however, been limited mainly to semisolid formulations such as ointments, creams and suppositories. For instance, whereas shea butter has been severally studied as promising excipient (base) in the formulation of creams (Mital and Dove, 1973), suppositories (Ezema

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[*] Corresponding author. *E-mail*: ayo_oyedele@yahoo.com *Tel*: +234 (0) 803 370 3029

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and Ogujiofor, 1992; Taylor *et al*., 1993) and ointments (Odusote and Ifudu, 1987; Thioune *et al*., 2003; Oyedele, 2012), studies are scarce and no literature was found reporting the use of shea butter as a base for liquid pharmaceutical products such as fluid emulsions. This potential of shea butter appears so far unexplored.

However, there are several benefits for the production of stable, pharmaceutical liquid shea butter emulsions, which facilitate and are intended for the use of such products on skin as a lotion. First, the fact is generally acknowledged that the use of fluid topical drug delivery base makes a drug more accessible to liquid absorption medium and enhances a more rapid exchange of the medicament between the formulation and site of action (Anschel and Lieberman (1965). Also, drug release rate from a mobile (fluid) base is known to increase with decrease in base viscosity, particularly with drugs soluble in the base (Kingman et al., 1979; Senior, 1974). Furthermore, the fluidity of lotions permits rapid and uniform application over a wide surface area.

Many properties of shea butter favour its topical use on human skin: — It is a natural, innocuous and emollient substance of low softening/melting temperature (≈37 °C; near the human body temperature), and contains only about 2 %w/w of fully saturated glycerides (Hilditch and Saletore, 1931). Furthermore, it has been reported to demonstrate analgesic, anti-inflammatory (Verma *et al*., 2012; Arendt-Nielsen *et al*., 2009) and wound healing effects on the skin (Odukoya *et al*., 2005; Fowler and Silverberg, 2008).

The study of shea butter's potential as a base for liquid pharmaceutical products (lotions, emulsions) is important in that it would give understanding of the performance of shea butter in such fluid formulations that guarantee absence of an occlusive effect on skin. An emulsion base (whether the oil-in-water (o/w) or water-in-oil (w/o) type) has the capacity to accommodate both water-soluble and oil-soluble active ingredients. Moreover, an emulsion base for topical use may be unmedicated, as also for cream bases. Unmedicated topical bases are useful to provide an emollient effect or for treating certain skin conditions (Searles, 2009).

The aim of this study is to determine shea butter's ability to serve as a base for liquid, unmedicated soap-stabilized emulsion formulations, and elucidate the stability requirements and characteristics for such preparations where the emulgent is produced in situ by reaction of the constituent fatty acids of shea butter with an alkaline aqueous phase. The formulation objective would be to produce a stable fluid system; one having a desirable dispersed phase distribution status and consistency, which remain unchanged throughout an expected shelf life of the preparation.

EXPERIMENTAL

Shea butter processed from shea nuts was obtained from the National Center for Agricultural Mechanization, Ilorin Nigeria, and purified by hot filtration: The butter was melted and filtered hot through coarse filter paper (No. 100, 24cm diameter, Rundfilter MN713 Macherey-Nagel D-5160 Duren, Germany) in a heated funnel in an oven (60°C). The filtrate was placed in clean glass container and left for seven days at room temperature (29 ± 3 °C) to solidify and become stable.

Determination of specific gravity of shea butter and liquid paraffin. The specific gravity of shea butter and of liquid paraffin used for the study was determined according to the British Pharmacopoeia (BP, 2009) pycnometer method. This method determines the ratio of the weight of a unit volume of the sample at a specified temperature to the weight of a unit volume of water at the same temperature. A previously weighed 25 ml-capacity specific gravity bottle (pycnometer) was used, well-filled with each sample (shea butter, liquid paraffin, or freshly distilled water [standard sample]), respectively; stoppered and placed in a water-bath maintained at 25 °C for 2 h prior to the determination, to allow equilibration of the sample with the temperature. Then the weight of sample in bottle was determined, in triplicate tests. The specific gravity of each sample at the specified temperature (25 °C) was determined by the ratio:

(Weight of bottle and sample – Weight of bottle) ÷ Weight of water at same temperature

In order to enable transfer and filling of shea butter into the pycnometer for the determination, the shea butter was heated to melt and flow (45-50 °C); and this sample in the pycnometer was left undisturbed for 4 days at ambient temperature prior to weighing for the determination (in view of polymorphic character of shea butter), to permit resumption of its stable physicochemical quality (Oyedele, 2007, Mital and Dove, 1971). Although shea butter melts (glides, softens) at 37–38 °C, the melted fat required higher temperature (45-50 °C) to be sufficiently liquefied to be sufficiently pourable for this determination.

Determination of maximum shea butter proportion in oil blends to produce liquid formulation. Shea butter was melted at 45-50 °C and mixed with liquid paraffin (BP) at same temperature in different proportions to yield 1, 5, 10, 15, 20, 25, 30, 40, and 50 % w/w concentrations of the butter in the blend (50 g samples, triplicates), and stirred gently until cold (room temperature: 29±2 °C). The viscosity of oil blends (one set of samples) that remained pourable after 48-h room temperature storage was determined using the Brookfield synchrolectric viscometer (LVT model), as described subsequently for emulsion samples. Duplicate samples of a 20 ml oil blend were placed in calibrated test tubes and observed for fluidity, homogeneity and phase separation over 6-week storage at the ambient temperature.

Preparation of shea butter emulsion formulations for study. Duplicate samples (100 g each) of shea butter/liquid paraffin blend, containing 1, 5, 10, 15, or 20 %w/w of shea butter in the blend, were prepared by mixing both liquids at same temperature (45-50 °C), and then mixed with 0.1, 0.2, 0.4, or 0.6 M aqueous potassium hydroxide (KOH) solution in a 1:9 (v/v) ratio, and homogenized for 5 min with the Silverson mixer (L5M-A model, Silverson Machines Inc., East Longmeadow USA) to produce a series of o/w emulsion formulations according to the composition scheme listed in Table 1. Another set of emulsion formulations were prepared by mixing and homogenizing fresh, duplicate samples (100 g each) of shea butter/liquid paraffin blend (containing 1, 5, 10, or 15 %w/w of shea butter in the blend) with 0.2 M KOH solution in four different ratios, namely: 2:8, 3:7, 4:6, and 5:5 (v/v), respectively, according to the composition scheme listed in Table 2.

Stability study of shea butter emulsion formulations. The creaming profile, redispersibility and physical consistency of the emulsion formulations were determined daily for 7 days, then weekly for 12 weeks at the ambient storage-temperature condition (29±2 °C). The creaming (upward emulsion-phase separation leaving behind a translucent or clear lower aqueous phase) was determined on duplicate 20 ml emulsion samples placed in stoppered calibrated test tubes. The cream-phase volume ratio (CVR, Vu/Vo) of the samples was calculated, where Vu and Vo were the volume of the separated emulsion phase and total volume of the preparation, respectively.

Redispersibility of the emulsion formulations (i.e. whether or not they remained fluid and apparently homogenous on gentle mixing during storage) was determined on duplicate 20 ml samples placed in 25 ml-capacity colourless glass bottles each with a screw cap. The physical consistency of the emulsion formulations (i.e. whether fluid [pourable], cracked [broken] emulsion, or semisolid/solid consistency during storage) was determined on other 20 ml samples in glass bottles, and from the general characteristics observed while handling the various samples earlier designated for studying the creaming profile, redispersibility and (later) viscosity of the different formulations.

Freshly prepared, duplicate 100 g samples of the emulsion formulations that remained stable liquids for at least 8 weeks were made, and their viscosity and globule size distribution were determined after 48-h room-temperature storage.

Dynamic viscosity of the formulations (in centipoises, cP) was determined on 50 ml samples using the Brookfield synchrolectric viscometer (8-gear LVT model; Brookfield Engineering Laboratories Inc., Middleboro USA). The shear stress (torque) values obtained for each sample in duplicate experiments were plotted against the respective rates of shear (0.3, 0.6, 1.5, 3, 6, 12, and 30 revolutions per min.) that corresponded to the torque values, to obtain a linear (Newtonian) graph, the slope of which gave the viscosity value of the sample. The viscosity value was based on using the Brookfield viscometer Spindle no. 4 and Brookfield Standard-viscosity Fluid no. 500 that were employed to establish the equipment setup constant at 29±2 °C. Correlation coefficient (R^2) values of best-fit (regression) lines through the plotted viscosity derivation data (i.e. of the overlapping up- and down-curves) were determined as a measure of the linear perfection of the Newtonian plots.

The globule diameters of the samples of those emulsion formulations that remained stable liquids for at least 8 weeks were determined using the microscopic method described by Levius and Drommond (1953). One or two drops from the emulsion sample under test (gently mixed to obtain a uniform dispersion) were placed on a slide, stained with methylene blue dye, cover-slip overlaid and examined at ×1000 magnification under a graticuled eye-piece lens of the microscope (Binocular, Leica, China) previously calibrated against a stage micrometer. The number of the dispersed droplets of different size ranges from three representative fields of view of each sample was counted in triplicate experiments. The mean globule diameter (overall) was determined for each formulation from the globule size distribution results obtained for the respective samples. The diameters of a total of 1000 dispersed droplets (minimum) were determined for each formulation.

RESULTS

Specific gravity of shea butter, liquid paraffin; and consistency of shea butter/ liquid paraffin blends. The specific gravity values determined for the shea butter and liquid paraffin used in the study were 0.932±0.004 and 0.840±0.004, respectively. The shea butter/liquid paraffin blends containing 1 – 20 %w/w shea butter remained fluid (pourable) preparations, while those containing higher shea butter concentrations (25 – 50 %w/w) were solid or semisolid within 48 h after preparation (Table 3). The latter (solidified) blends exhibited no separation of their component oils, but the fluid mixtures showed different levels of separation. The weightier of the two oils (shea butter) separated out in the liquid blends by sedimentation. Table 3 shows the viscosity of the blends and their sedimentation volume ratio, F = Vu/Vo; where Vu and Vo are the volume of sediment at 48th h and total volume of the preparation, respectively. The sedimentation volume ratio (0.14 – 0.86) and viscosity (84 – 259 cP) increased as the shea butter content of the oil blends increased from

1 % through 20 %w/w. Duplicate experiments consistently gave identical results. The separation of the oil layers did not proceed further after 48 h.

Consistency and Stability of Shea butter Emulsion Formulations. The consistency of 15 shea butter emulsion formulations (out of the 36 studied) met the desirability criteria, namely: to be fluid (pourable) and redispersible (homogenous for longer than a brief [e.g. 5 min] period after mixing) throughout initial 8 weeks of room-temperature storage. The 15 formulations remained stable throughout the 12 weeks of study (Tables 1 and 2).

Table 4 shows the stability results (viscosity and overall mean droplet diameters) of the stable formulations, listed in ascending order of the mean droplet sizes, which ranged from 4.4 to 43.1 μm. Table 4 also presents again, for comparison, the concentrations of shea butter and KOH solution (aqueous phase) of the respective formulations for the purpose of elucidating any effect or trend of these concentrations on product stability. The highest and lowest shea butter contents of the stable fluid emulsion formulations were 18 and 0.6 %w/w (Formulation codes: A17 and B3), respectively (Table 4). Lower shea butter content (0.5 %w/w) gave fluid but unstable product (Formulation code B4; Table 2). The overall most stable formulation was A6, which exhibited the smallest overall mean droplet diameter, 4.4 μm (Table 4). It contained 4.5 %w/w shea butter emulsified with 0.2 M KOH solution.

Other formulations with comparable superior stability (showing the bare-minimum mean droplet diameters of ≈4–6 μm) were: A10, A7, A8, and A12 (formulation codes), which all contained either 4.5 or 9.0 %w/w shea butter, emulsified with soap resulting from reaction with 0.2, 0.4, or 0.6 M KOH solution (Table 4). The trend (0.2, 0.4, 0.6 M) of increase of the KOH concentration of the aqueous phase of this 'most stable group' of five formulations (A6, A10, A7, A8, and A12) correlated with the sequential increase of their mean droplet diameters (Table 4), implying that the relative stability of the formulations decreased as the KOH concentration increased from 0.2 to 0.6 M. However, the stability profile of formulations containing shea butter outside the 4.5 – 9.0 % range did not comply with such alkali-concentration trend.

On the other hand, the 'least stable group' of the shea butter emulsion formulations that also met the inclusion criteria, namely: A1, A5, A9, A17, and A13 (formulation codes; Table 4), showing the largest overall mean droplet diameters (38.1 – 43.1 μm), were produced with 0.1 M KOH solution and contained a wide range of shea butter content (0.9 – 18.0 %w/w). This showed that the stabilizing effect of the interfacial film of the soap emulgent formed by reaction of the 0.1 M KOH concentration (aqueous phase) component of the formulations with the shea butter fatty acids was not optimal, regardless of the concentration of shea butter in the formulations.

The viscosity of the 'most stable group' of the shea butter emulsion formulations ranged from 236 cP (for A7) to 651 cP (for A12); and for the 'least stable group' of formulations ranged from 153 cP (for A1) to 354 cP (for A17) (Table 4). No correlation was apparent between the viscosity values of all the formulations and their stability based on the overall mean droplet sizes.

DISCUSSION

This study undertook to tackle the challenge of using shea butter as base for the development of stable, soap-stabilized liquid emulsion formulations for promising topical use, and with a view to further expanding the beneficial exploitation of shea butter's many potentials. Neat shea butter melts at 37.8 °C (Mital and Dove, 1971) and therefore exists as a semisolid (soft solid) substance at the ambient temperatures (≈20 – 35 °C) of habitable regions of the world.

Table 1: Composition, consistency and cream-volume ratio of 1st series shea butter emulsion formulations

Composition Parameters				Consistency	Length of Storage/ Cream-Volume Ratio (CVR)					
I	II	III	IV		1 Day	2 Day	3 Day	1 Wk	8 Wk	12 Wk
A1	1	0.1	0.9	F	0.20	0.20	0.20	0.20	0.20	0.20
A2	1	0.2	0.9	F, CK	1.0	0.97	CK	CK	CK	CK
A3	1	0.4	0.9	F, CK	1.0	CK	CK	CK	CK	CK
A4	1	0.6	0.9	F	1.0	0.97	0.97	0.67	0.43	0.43
A5	5	0.1	4.5	F	0.25	0.25	0.25	0.25	0.25	0.25
A6	5	0.2	4.5	F	1.0	0.85	0.74	0.44	0.36	0.36
A7	5	0.4	4.5	F	1.0	0.71	0.67	0.52	0.32	0.32
A8	5	0.6	4.5	F	1.0	0.99	0.97	0.71	0.39	0.39
A9	10	0.1	9	F	0.33	0.33	0.33	0.33	0.33	0.33
A10	10	0.2	9	F	1.0	1.0	0.71	0.46	0.49	0.49
A11	10	0.4	9	F	1.0	1.0	0.97	0.89	0.51	0.51
A12	10	0.6	9	F	1.0	1.0	0.96	0.86	0.66	0.66
A13	15	0.1	13.5	F	0.56	0.56	0.56	0.56	0.56	0.56
A14	15	0.2	13.5	F, CK	1.0	0.98	0.93	0.83	0.66	0.66
A15	15	0.4	13.5	F, CK	1.0	1.0	1.0	1.0	1.0	1.0
A16	15	0.6	13.5	F, CK	CK	CK	CK	CK	CK	CK
A17	20	0.1	18	F	0.56	0.56	0.56	0.56	0.56	0.56
A18	20	0.2	18	F, CK	1.0	1.0	1.0	1.0	1.0	1.0
A19	20	0.4	18	F, CK	1.0	1.0	1.0	1.0	1.0	1.0
A20	20	0.6	18	F, CK	1.0	1.0	1.0	1.0	1.0	1.0

I = Formulation Identity Code II = Shea butter content of oil phase* (%w/w) III = Molar concentration of aqueous KOH solution used IV = Overall (resultant) shea butter content in final formulation** (%w/w)

* Oil phase of emulsion formulations consisted of shea butter/liquid paraffin blend

****** Formulations contained the oil blend with KOH solution in 1:9 (v/v) ratio.

F: Fluid (pourable) CK: Cracked (broken) emulsion F, CK: Fluid product occasionally cracked when redispersed.

Table 2: Composition, consistency and cream-volume ratio of 2nd series shea butter emulsion formulations

Composition Parameters				Consistency	Length of Storage/ Cream-Volume Ratio (CVR)					
I	II	III	IV		1 Day	2 Day	3 Day	1 Wk	8 Wk	12 Wk
B1	1	2:8	0.8	F	1.0	1.0	0.97	0.97	0.82	0.82
B2	1	3:7	0.7	F	0.97	0.97	0.93	0.8	0.63	0.63
B3	1	4:6	0.6	F	1.0	1.0	0.93	0.93	0.8	0.8
B4	1	5:5	0.5	F, CK	0.5	0.5	0.5	0.5	CK	CK
B5	5	2:8	4	F, S, CK	0.33	0.33	0.33	0.33	CK	CK
B6	5	3:7	3.5	F, S, CK	1.0	1.0	1.0	1.0	1.0	1.0
B7	5	4:6	3	F, S, CK	1.0	1.0	1.0	1.0	1.0	1.0
B8	5	5:5	2.5	F, S, CK	1.0	1.0	S	S	S	S
B9	10	2:8	8	S, CK	1.0	1.0	S	S	CK	CK
B10	10	3:7	7	S	1.0	1.0	S	S	S	S
B11	10	4:6	6	S	1.0	1.0	1.0	S	S	S
B12	10	5:5	5	SP	S	S	S	S	S	S
B13	15	2:8	12	S	1.0	1.0	S	S	S	S
B14	15	3:7	10.5	S, CK	0.33	0.33	0.33	0.33	CK	CK
B15	15	4:6	9	S, CK	1.0	1.0	S	S	CK	CK
B16	15	5:5	7.5	SP	S	S	S	S	S	S

I = Formulation Identity Code II = Shea butter content of oil phase* (%w/w) III = The KOH (0.2 M) solution/ Oil phase blend ratio (v/v) used** IV = Overall (resultant) shea butter content in final formulation (%w/w)

* Oil phase of emulsion formulations consisted of shea butter/liquid paraffin blend

****** Formulations contained the oil blend with KOH (0.2 M) solution at ratios indicated.

F: Fluid (pourable) S: Semisolid/solid SP: Product solidified while preparation (stirring until cold) was in progress
CK: Cracked (broken) emulsion
F, CK: Fluid product occasionally cracked when redispersed S, CK: Semisolid product occasionally cracked when mixed
F, S, CK: Consistency of replicated products varied between indicated features over the storage period

Table 3: Consistency and stability characteristics of shea butter/liquid paraffin blends

Shea butter content of blend preparation (%w/w)	Consistency	Stability Indicators	
		Sedimentation volume ratio (Vu/Vo)*	Viscosity (cP)**
1	F	0.14	84 (0.98)
5	F	0.26	140 (0.96)
10	F	0.48	167 (0.98)
15	F	0.76	231 (0.97)
20	F	0.86	259 (0.97)
25	S	1.0	N
30	S	1.0	N
40	S	1.0	N
50	S	1.0	N

* Replicated experiments gave consistent, identical results
** Newtonian graph correlation coefficient (R^2) values in parenthesis
F = Fluid (pourable) S = Semisolid/solid N = Not determined

Table 4: Overall mean droplet diameter and viscosity of stable shea butter emulsion formulations

Formulation identity code	Overall mean droplet diameter (µm)*	Overall shea butter content of formulation (%w/w)	Molar concentration of aqueous KOH solution used in formulation	Viscosity of formulation (cP)**
A6	4.4 (0.34)	4.5	0.2	250 (0.99)
A10	4.7 (0.32)	9.0	0.2	541 (0.98)
A7	4.7 (0.21)	4.5	0.4	236 (0.99)
A8	5.6 (0.19)	4.5	0.6	283 (0.99)
A12	6.2 (0.18)	9.0	0.6	651 (0.98)
A4	6.3 (0.18)	0.9	0.6	462 (0.97)
A11	7.9 (0.16)	9.0	0.4	567 (0.98)
B3	11.3 (0.12)	0.6	0.2	1863 (0.96)
B1	15.6 (0.14)	0.8	0.2	398 (0.96)
B2	17.9 (0.15)	0.7	0.2	597 (0.98)
A1	38.1 (0.13)	0.9	0.1	153 (0.96)
A5	39.1 (0.14)	4.5	0.1	205 (0.97)
A9	39.1 (0.14)	9.0	0.1	268 (0.96)
A17	42.9 (0.14)	18	0.1	354 (0.97)
A13	43.1 (0.14)	13.5	0.1	317 (0.98)

* Standard deviation values in parenthesis ** Newtonian graph correlation coefficient (R^2) values in parenthesis

In accordance with its normal solid/semisolid state at the ambient temperatures, shea butter is commonly and conveniently studied and employed as a base for semisolid pharmaceutical products (Mital and Dove, 1973; Ezema and Ogujiofor, 1992); and several attempts to formulate liquid emulsions of shea butter are unsuccessful (Tables 1 and 2). This study of using shea butter as a fluid emulsion base, therefore, represents a new way of using shea butter pharmaceutically.

An objective of this study was to determine the proportion limit(s) of shea butter/paraffin oil blends required for successful stabilization of shea butter-based emulsion formulations; exploiting, as emulgent for the stabilization, the alkali soap produced in situ from reaction of KOH with fatty acids naturally resident in shea butter. Fluid-consistency shea butter/paraffin oil blend proportions were investigated and used in the study on proposition that the oil admixture(s) exhibiting stable fluid consistency at ambient temperature would remain so even after being formulated as emulsion system, and this could lead to availability of a whole new design of stable, fluid shea butter-based emulsion formulations that were hitherto unexplored and difficult to produce. This proposition was validated by the results of successful liquid shea butter formulations that remained stable for 12 weeks (Table 4). The results showed that the highest shea butter concentration permitting liquid-consistency blend with paraffin oil and its potassium soap-stabilized liquid emulsion is 20 and 18 %w/w, respectively (Tables 3 and 4).

Stable emulsions are a system in which the dispersed globules retain initial distribution profile within the continuous phase for a considerable storage period (Zografi *et al*., 1990). The shea butter-based emulsion formulations that remained stable liquids for at least 8 weeks (Table 4) were therefore preferred, considered as meeting desirability criteria in line with the study's formulation objective. The emulsifier component (emulsifying agent) of an emulsion system performs the function of stabilizing the interfacial film it produces around the dispersed globules. The consistency (physical nature) and strength (firmness) of the interfacial film (barrier) between the dispersed phase and continuous medium determines and controls emulsion stability (Kayes, 1988). Homogenizers (e.g. the Silverson mixer) are commonly used for emulsion preparation to obtain micronized globule sizes, because the quality and stability of the product is improved by decreasing globule size and reducing the size range of globules of the dispersed phase (Collett, 1990). Thus the fine globule size (unit-digit overall mean globule diameter) of the stable formulations: A6, A10, A7, A8, A12, A4, and A11 (Table 4) was an indication of their superior stability compared to the other formulations. The fine, mean globule size and distribution ensured slow or minimal rate of creaming.

Creaming (phase separation) and cracking (or breaking) of an emulsion are the two common deviations from ideal stable emulsion status. Creaming is separation of the emulsion into two regions one of which is richer in the dispersed phase than the other, and is caused by density difference between the phases constituting the emulsion and also caused by influence of gravity on the dispersed globules, resulting in upward movement of a lower-density disperse phase (Billany, 1988). For instance, the upward creaming of the dispersed oil blend of the shea butter-based emulsion systems of this study, was partly due to a lower specific gravity (0.840, 0.932) of the components of the dispersed phase relative to that of the continuous phase (water, the standard fluid employed for specific gravity determination, which is conventionally accorded the specific gravity value of 1.0) (BP, 2009). Creaming is mild instance of emulsion instability, especially when gentle shaking restores homogeneity of the dispersion. Thus, the emulsion formulations of this study that creamed but were uniformly redispersible throughout the 12 weeks of study were considered acceptable (Tables 1, 2 and 4).

Breaking of an emulsion, on the other hand, involves coalescence of the dispersed globules with complete severance of the dispersed phase into an immiscible, separated layer, making redistribution by shaking impossible. In this case of emulsion instability, the product in question may no longer be rightly referred to as an emulsion because the interfacial film barrier consummated by aid of the emulgent during the emulsification process (e.g. homogenization) has completely broken down. Emulsion breaking may be caused by any chemical, physical or biological factors that change the nature of the interfacial film or that tend to make the barrier-film less stable (Collett, 1990). The integrity of the interfacial film barrier is what necessarily determines whether the emulsion globules will remain uniformly dispersed (when the barrier is firm), or whether the globules will coalesce, aggregate and/or coagulate. Aggregation and subsequent (or eventual) coagulation of dispersed oil phase (shea butter/liquid paraffin blend) globules occurred in this study for some of those formulations that cracked (Tables 1 and 2). The aggregated and coagulated fatty particles of the cracked shea butter emulsion formulations caused the emulsion products to be no longer redispersible and become unsightly.

The KOH used (in graded molar concentrations) in this study to react in situ with shea butter's fatty acids in order to create the emulsifier barrier-film to stabilize the emulsion formulations was a chemical factor that invariably determined the tenacity of the interfacial film barrier against coalescence formed within the emulsion systems. The strength of barrier, in turn, directly determined the capacity of the respective emulsion formulations to resist normal thermodynamic instability stress and tendency toward globule coalescence and cracking.

Physical factors also, which prospectively challenged the stability of the multifarious shea butter formulations studied (Carter, 1975) were present as circumstantial and compositional conditions of different levels of severity, such as: (1) the polymorphic character of shea butter; (2) the ambient storage temperature condition that remained below shea butter's melting point, and so could cause disperse-phase congealing in some of the formulations; and/or (3) the presence (or incorporation) of an excess disperse phase component (by default of the experimental design) – i.e. the presence of disperse-phase excess beyond the limit that the continuous phase (water) could accommodate within a given microenvironment of the emulsion system during or after processing (homogenizing and stirring till cold) of the preparations. Consequently, products that were only minimally affected by these factors remained stable (Table 4). But the majority of the formulations (in Tables 1 and 2), which could not withstand the untoward chemical and/or physical destabilizing factors, cracked during or after preparation of the products.

Although all the emulsions formulated using 0.1 M KOH remained stable liquids throughout the study, they exhibited larger values of overall mean droplet diameter (indicating relatively inferior physical stability) than the formulations produced with higher KOH concentrations (Table 4). The superior stability (on basis of the globule size data) of the formulations produced from using 0.2, 0.4, and 0.6 M KOH suggests that the interfacial barrier-film of the products was better, due to greater (than 0.1 M) KOH concentrations used.

Given adequate presence of shea butter fatty acids for reaction with the alkali at the interface during emulsification, it is evident from the results that tenacity of the interfacial film should improve with increasing concentration of KOH used. Thus the limiting factor for strengthening the barrier-film with KOH-concentration increase would be the availability of shea butter acids and, by extension, the concentration of shea butter used in the formulation. The film-strengthening power of increasing concentration of KOH would expectedly decline on saturation of available free acids of shea butter for formation of the film.

Moreover, analyzing the results from the study of the characteristics versus composition of the best (most stable group of) shea butter based emulsion formulations (A6, A10, A7, A8, and A12), it is apparent that the respective concentrations of alkaline aqueous solution (KOH) used (0.2, 0.4, and 0.6 M) complemented particular concentrations (4.5, 9.0 %w/w) of shea butter in the emulsion formulations to give enhanced product stability (Table 4). This result suggests optimal complement of the physicochemical reaction occurring between shea butter's fatty acid moieties at 4.5 and 9.0 %w/w shea-butter concentrations and the alkaline aqueous solution (KOH) used at those concentrations (particularly the 0.2 M concentration) for production of excellent (optimally strong and durable) interfacial barrier-film of stable emulsion systems having very fine mean globule size and size-distribution under normal room temperature (29°±2°C) storage condition.

REFERENCES

Anschel, J. and Lieberman, H.A. (1965); Suppositories II: Formulation problems; *Drug Cosmet. Ind.* 97(4), 507-510.

Arendt-Nielsen, L., Rosetzsky, A. and Weidner, M.S. (2009); A double-blind randomized placebo controlled parallel group study demonstrates analgesic effects of sheanut oil extract in exercise induced muscle tenderness; *J. Musculoskelet. Pain* 17(1), 8-14.

Athar, M. and Nasir, S.M. (2005); Taxonomy perspective of plant species yielding vegetable oils used in cosmetics and skin care products; *Afr. J. Biotech.* 4(1), 36-44.

Billany, M.R. (1988); Emulsions, In: Pharmaceutics—the science of dosage form design, Aulton, M.E. ed. English Language Book Soc. Churchill Livingstone, p 294.

British Pharmacopoeia (BP) (2009); British Pharmacopoeia Commission Office, London.

Carter, S.J. (1975); Cooper and Gunn's Dispensing for pharmaceutical students, 12th ed. Pitman Medical Pub. Co. Ltd. London, p 122.

Chukwu, O. and Adgidzi, P.P. (2008); Evaluation of some physicochemical properties of shea butter (*Butyrospermum paradoxum*) related to its value for food and industrial utilization; *Int. J. Postharv. Tech. Innov.* 1(3), 320 – 326.

Collett, D.M. (1990); Emulsions and creams, In: Pharmaceutical practice. Collett, D.M. and Aulton, M.E. eds. Longman Group UK Ltd, Singapore, p 115.

Djekota, C., Diouf, D., Sane, S., Mbaye, M.S. and Noba, K. (2014); Morphological characterization of shea tree (*Vitellaria paradoxa* subsp. *paradoxa*) populations in the region of Mandoul in Chad; *Int. J. Biodivers. Conserv.* 6(2), 184-193.

Ezema, D.O. and Ogujiofor, K.O. (1992); The Evaluation of *Butyrospermum paradoxum* as a Suppository Base; *Pharm. Biol.* 30(4), 275-280.

Fowler, J. and Silverberg, N. (2008); Active naturals have a key role in atopic dermatitis, *Pediatric News (Supplement),* Clinical Update, International Medical News Group, 60B Columbia Road, Morristown, NJ 07960; Elsevier. www.pediatricnews.com

Glew, D. and Lovett, P.N. (2014); Life cycle analysis of shea butter use in cosmetics: from parklands to product, low carbon opportunities; *J. Clean. Prod.* 68, 73-80.

Hilditch, T.P. and Saletore, S.A. (1931); Fatty acids and glycerides of solid seed fats. 1. Composition of the seed fats of *Allanblackia stuhlmannii*, *Pentadesma butyracea*, *Butyrospermum parkii* (Shea) and *Valeria indica* (Dhupa); *J. Soc. Chem. Ind.* 50, 468T-472T.

Irvine, F.R. (1963); A textbook of West African Agriculture – Soil and Crops, 2nd ed. Oxford University Press London, pp. 200-205.

Kayes, J.B. (1988); Disperse systems, In: Pharmaceutics—the science of dosage form design, Aulton, M.E. ed. English Language Book Soc. Churchill Livingstone, p 114.

Kingman, J.J.R., Polderman, J. and de Blaey, C.I. (1979); Biopharmaceutical studies of fatty suspension suppositories, I: Spreading in situ; *Int. J. Pharm.* 3, 179-186.

Krist, S., Bail, S., Unterweger, H., Ngassoum, M.B., Mohagir, A.M. and Buchbauer, G. (2006); Volatile compounds of original African black and white shea butter from Tchad and Cameroon; *Eur. J. Lipid Sci. Tech.* 108(7), 583-588.

Levius, H.P. and Drommond, F.G. (1953); Elevated temperature as an artificial breakdown stress in evaluation of emulsion stability; *J. Pharm. Pharmacol.* 5, 743-756.

Maranz, S. and Wiesman, Z. (2004); Influence of climate on the tocopherol content of shea butter; *J. Agric. Food Chem.* 52(10), 2934-2937.

Mital, H.C. and Dove, F.R. (1971); The study of shea butter 1. Physico-chemical properties; *Planta Med.* 20, 283-288.

Mital, H.C. and Dove, F.R. (1973); Shea butter as a base for ointments and creams; *Drug Cosmet. Ind.* 113(4), 46-47, 139-142.

Odukoya, A.O., Jenkins, M.O., Ilori, O.O. and Sofidiya, M.O. (2005); The use of selected Nigerian natural products in management of environmentally induced free radical skin damage; *Pakistan J. Biol. Sci.* 8(8), 1074-1077.

Odusote, M.O. and Ifudu, N.D. (1987); Nigerian shea butter as an ointment base; *The Nigerian J. Pharm.* 18(6), 31-34.

Okafor, J.C. (1980); Trees for food and fodder in the Savanna areas of Nigeria; *Int. Tree Crops J.* 1(2-3), 131-141.

Okullo, J.B.L., Omujal, F., Agea, J.G., Vuzi, P.C., Namutebi, A., Okello, J.B.A. and Nyanzi, S.A. (2010); Physico-Chemical characteristics of shea butter (Vitellaria paradoxa C.F. Gaertn.) oil from the Shea district of Uganda; *Afr. J. Food Agric. Nut. Dev.* 10(1), 2070-2084.

Oyedele, A.O. (2007); Investigation of ointment bleeding in some absorption and water-miscible base preparations; *Nig. J. Pharm. Res.* 6(1), 94-100.

Oyedele, A.O. (2012); Delivery of metronidazole from purified Nigerian sheabutter in comparison to standard and modified ointment bases; *Ife J. Sci.* 14, 253-257.

Searles, G.E. (2009); Update on topical approaches for managing scalp psoriasis; *Skin Ther. Lett.* 5(1), 1-7.

Senior, N. (1974); Rectal administration of drugs; *Adv. Pharm. Sci.* 4, 363-435.

Taylor, O., Igwilo, C.I., Adeoye, D.I., Adeoye, O. and Awosika, O.A. (1993); In-vitro release of paracetamol from various suppository formulations of purified sheabutter; *J. Pharm. Sci. Pharm. Prac.* 1(2), 93-99.

Thioune, O., Khouma, B., Diarra, M., Diop, A.B. and Lo, I. (2003); The excipient properties of shea butter compared with vaseline and lanolin; *J. Pharm. Belg.* 58(3), 81-84.

Verma, N., Chakrabarti, R., Das, R.H. and Gautam, H.K. (2012); Anti-Inflammatory effects of shea butter through Inhibition of Inos, Cox-2, and cytokines via the Nf-Kb Pathway in Lps-activated J774 macrophage cells; *J. Complement. Integr. Med.* 9(1) Article 4, 1-13.

Vincenzo, D.D., Maranz, S., Serraiocco, A., Vito, R., Wiesman, Z. and Bianchi, G. (2005); Regional variation in shea butter lipid and triterpene composition in four African countries; *J. Agric. Food Chem.* 53(19), 7473–7479.

Warra, A.A. (2013); A report on soap making in Nigeria using indigenous technology and raw materials; *Afr. J. Pure Appl. Chem.* 7(4), 139-145.

Warra, A.A., Gunu, S.Y., Jega, S.A., Aisha, J.A. (2009); Soap production from shea nut butter; *Int. J. Nat Appl. Sci.* 5(4), 410-412.

Zografi, G., Scott, H. and Swarbrick, J. (1990); Disperse systems, In: Remington's pharmaceutical sciences, 18th ed. Gennaro, A.R. ed. Mack Pub. Co. Easton, Pennsylvania, p 307.