

Bintu Kime¹, P. M. Dass¹, O. N. Maitera¹, C. S. Archibong^{2*}, Ayodele Akinterinwa¹ and B. A. Thliza³¹Department of Chemistry, Modibbo Adama University of Technology, Yola, Adamawa State, Nigeria²Department of Chemical Sciences, Federal University Wukari, Taraba State, Nigeria³Department of Chemistry, University of Maiduguri, PMB 1069, Borno State, Nigeria*Corresponding author: archibong@fuwukari.edu.ng**Received:** March 23, 2021 **Accepted:** June 16, 2021

Abstract: To create a way for green chemistry and sustainable development using bio-based surfactant as the potential for treatment of leather, which lead to saponin extraction from fruit of *Balanites aegyptiaca* plant. The total percentage of crude saponin extract obtained was 84.9%. The most important properties are the foam production and stability of extract and OMO at 5 min; it drops from 4.5 to 4.3 cm and 5.5 to 5.2 cm, respectively at the concentration of 0.1 g/ml of the surfactant solutions. The emulsion formation/capacity of the extract was good, drops from 98.3 to 55.0% and 92.5 to 35.2%, which is correspondingly higher than that of OMO drops from 98.0 to 40.0% and 92.0 to 34.3% for 2 and 5 g, respectively. The experiment shows that extract has higher and better capacity when compared to OMO (98.0 to 40%) within the hours of evaluation from 1 to 48 h, respectively. FTIR and GC-MS analysis shows promising results. The crude extracts at different concentration and temperature gave a better result to synthetic surfactant (OMO) both on the tensile strength and elongation at break.

Keywords: *Balanites aegyptiaca*, crude saponin extract, male goatskin, leather processing

Introduction

Saponins are the most widely distributed group of natural secondary metabolite compounds found in plants and have numerous uses, traditional as well as modern (Chapagain, 2006). Guclu-Ustundag & Mazza (2007) explained that saponins are a diverse group of compounds widely distributed in the plant kingdom, which are characterized by their structures containing a triterpene or steroid aglycone and one natural sugar chains.

Generally, surfactants are a large group of surface active substances with a great number of applications. The surfactant exhibits surface activity by lowering the surface tension of liquids (Muthuprasanna *et al.*, 2009), so it can wet the fibers and surfaces, they loosen and encapsulate the dirt and, in that way, ensure that the soiling will not re-deposit on the surfaces. Surfactants are important in the manufacture of leather, right from liming the skin and to finished product called leather. Surfactant prevent the leather fibers becoming stiff and inflexible, thereby keeps the fiber network flexible and increasing the tensile strength of the final products.

Desert date with the botanical name *Balanites aegyptiaca* belongs to the family Balanitaceae. About twenty-five known species of the plant are widely distributed through tropical Africa (Manji *et al.*, 2013). *Balanites aegyptiaca* even though has the history of traditional use as a surfactant, has not been

efficiently investigated to obtain data and information that will present it as raw material with potentials in surfactant applications. *Balanites aegyptiaca* plant is available in abundance in the north eastern part of Nigeria (Manji *et al.*, 2013). In most African countries it is used in the treatment of constipation and eye irritation, liver disease and as a purgative and sucked by school children as a confectionary in some countries (Chevalier *et al.*, 2004).

The aim of the research is to extract, characterize saponin-based surfactant from fruit of *Balanites aegyptiaca* plant for the treatment of leather from male goatskin.

Material and Methods

Sample collection and preparations

Sampling was done randomly. *Balanites aegyptiaca* plant in Demsa Local Government Areas of Adamawa state, Nigeria were used in this study. Fresh fruit of matured plant were collected. The outer cover (the epicarp) was removed by using sterile sharp surgical blade and the fruit pulps/mesocarps were scraped manually then air dried under shade for about four months, then the dried specimens were manually ground into powder using pestle and mortar (Molla *et al.*, 2013; Chapagain, 2006).



Plate 1: Mature fruit (A) and mature fruit pulp (B) of *Balanites aegyptiaca* plant

Saponin test in the fruit of *Balanites aegyptiaca* plant

With 10 gram of fresh samples were dissolved in 100 ml distilled water (1:10), blend, and filtered. The filtrate inside a test tube was warmed to obtain a stable persistent froth; this was mixed with 3 drops of olive oil and shaked vigorously to form emulsion, which indicated the presence of saponins (Edeoga *et al.*, 2005 and Mimi, 2011).

Method of Extraction**Extraction and quantitative determination of saponin in *Balanites aegyptiaca* fruit**

The saponin content in the sample was determined by double extraction gravimetric method described by Ezeabara *et al.* (2014). Powdered sample (5 g) was mixed with 50 ml of 20% aqueous ethanol solution in a flask. The mixture heated with periodic agitation in water bath for 90 minutes at 55 °C then filter through what-man filter paper (No 42). The residue was extracted with 50 ml of 20% ethanol and both extract was pour together and the combine extract was reduced to about 40 ml at 90°C. This was transferred to a separating funnel where 40 ml of diethyl ether was added and vigorously agitated. Re-extraction by partitioning was carried out until the aqueous layer become clear. The saponin were extracted with 60 ml of normal butanol. The combine extract was washed with 5% aqueous sodium chloride solution and evaporated to dryness in a pre-weigh evaporation dish. It was dried at 60 °C in the oven and reweigh after cooling in desiccators. The process was repeated two more times to get an average. Saponin content was determined by difference and calculated as a percentage of the original sample thus:

$$\% \text{ of saponin} = \frac{W_2 - W_1}{\text{Weight of Sample}} \times \frac{100}{1}$$

Where: W_1 = Weight of evaporating dish; W_2 = Weight of evaporating dish + sample

Characterization of crude saponin extract in fruit of *Balanites aegyptiaca* plant

The sample (crude saponin extract) was characterized using Fourier Transform Infrared Spectroscopy (PerkinElmer Spectrum Vision 13. 24. 19, and Gas chromatography- mass spectroscopy. The sample (extract) was packed in a sample bottle and the analysis was done at American University of Nigeria using Model GC-MS 7890A, Agilent Technologist Inert MSD-597CM. The NIST Version 11.L library database of National Institute Standard and Technology more than 62,000 patterns were used for identifying the chemical components.

Performance tests of the crude saponin extract of *Balanites aegyptiaca* fruit

Foaming studies on the crude saponin extract

It was determined by the Ross-Mile method as described by Azab (2001) using foam accumulate measuring systems. The method of foam production was measured by the initially height of the foam produced. The foaming stability was measured by the height after 10 minutes. The surfactant solutions were prepared at different concentrations between 1.0-5.0 g/dm³. Then 100 cm³ surfactant solution was taken in the burette and allowed to run out into the receiver from a height of 10 to 12 cm³. For each surfactant at different concentrations, the heights of the foam formed in the receiver was measured immediately and between time interval 1 minute to 9 hours.

Emulsion formation capacity of the crude saponin extract

The procedure of Kime *et al.* (2015) was adopted with little modification. An emulsion was prepared by mixing 50 cm³ paraffin oil and 50 cm³ sample solutions which were mixed in a beaker. The mixture allowed to homogenized using a 100 cm³ glass syringe. This improved homogenization process was required repeating cycles of sucking and rapid expulsion of emulsion from the syringe to ensure proper droplet break-

up until a creamy homogenous emulsion is obtained. Emulsion capacity was expressed as the amount of oil emulsified and held per gram of sample as given by Padmeshree *et al.* (1987);

$$\text{Emulsion Capacity} = X/Y \times 100/1$$

Where X = Height of emulsified layer; Y = Height of whole solution in the syringe

Skin treatment (leather)

The procedure described by RamÓn (2016) was used by weighing skin and recorded, before and after salting. The leather was measured by using the methods introduced by the International Organization for Standardization (2002a, b, c; 2006). Where the crude saponins extract obtained from the fruit of *Balanites aegyptiaca* plant were used as bio-based surfactant and compared with synthetic surfactant (OMO) using the same concentrations. The applied sequence of processes steps and techniques on goatskin storage and beam house operations were followed by these steps (Table 1). The hand flaying method was used by hanging, pulling a skin by hand and carefully used special flaying knife.

Table 1: Goatskin storage and leather operations (Hauber and Knodler, 2008)

Process unit	Inputs
Curing and storing (1 week) Soaking (24 h)	salt, cooling and drying water, crude saponin extract, alkali
Flashing (1 h)	Cold water
Liming and unhairing (24 h)	water, surfactant (fruit extract), lime and alkali sulphides
Washing after unhairing (1 h)	Water
Deliming and bathing (12 h)	ammonium salts, water, gabaruwaa plant
Rinsing (1 h)	Water and gabaruwaa plant (<i>Acacia nilotica</i>)
Treatment/degreasing (10, 15, 20, 25 and 30 g)	surfactant (fruit extract) and water
Pickling before tanning	Water, salt and fungicides

The tensile strength measurement of the leather

For measuring the tensile strength, the leather samples were cut into pieces by applying a press knife capable of cutting a test piece with standard dimension of 110 mm to the grain surface, parallel to the backbone and the other one with the longer side, perpendicular to the backbone.

a) Standard Test Method for Tensile Strength of Leather, ASTM D2209-00 and D4704 (2015), was adopted. The tensile strength is the force (Kg) per unit area of cross section (sq. Cm) required to cause a rapture of the test specimen. The tensile strength of the specimen was calculated using the following formula;

$$\text{Tensile strength} = \frac{\text{Breaking load (Kg)}}{\text{Thickness (cm)} \times \text{Width}}$$

b) Percentage elongation at break for specimen was calculated from the distance of the jaws after breaking had occurred;

$$\% \text{ Elongation} \quad \text{at} \quad \text{break} = \frac{\text{distance increase by breaking}}{\text{distance of the two jaws in normal}} \times \frac{100}{1}$$

The hardness test

The standard test methods for hardness resistance to scratching and pressure was adopted, used Shore Durometer NOVOTEST TS-A complies to: ISO-7619 and ISO-868, DIN53505, ASTM D2240 and JISK7215 (2018). Resistance to scratching and pressure.

Results and Discussion

Saponin test with fresh sample in fruit of *Balanites aegyptiaca* plant

The result showed that saponin was present in the fruit *Balanites aegyptiaca* investigated; a stable and persistent froth was obtained. There was an appearance of foam that persisted for at least 17 min. An emulsion was formed after three drops of olive oil was added which confirmed the presence of saponins in the fruit of *Balanites aegyptiaca* plant. Saponin have been reported in the fruit of *B. aegyptiaca* plant (Chapagain, 2006). In addition, steroid saponin are common in plants used as herbs and promoting for their health-promoting properties (Osbourne, 2003).

Extraction, determination and the quantification of saponin in the crude sample of *Balanites aegyptiaca* plant fruit

However, pure saponin for 180 g of *Balanites aegyptiaca* plant fruit through Soxhlet extraction method was obtained as 84.9%. The high amount of saponin content of *Balanites aegyptiaca* in methanol extract agreed with (Chapagain 2006 and Kime et al., 2015), who used methanol for extraction and reported a high percentage of saponin obtained as suitable solvent. The most fundamental parameters influencing the high production of a good quality are the types of solvent used, ratio of the solvents, the methods of extraction, how many times and the duration of extractions or depending on the method used (Handa, 2008).



Plate 2: Crude saponin extract in fruit of *Balanites aegyptiaca* plant

Characterization of crude saponin extract in fruit of *Balanites aegyptiaca* plant

Fourier transform Infra-Red (FT-IR) measurement

Figure 1 present FT-IR spectra of crude saponin extract in fruit of *Balanites aegyptiaca* plant. The broad band between 3780.79 and 3409.53 cm⁻¹ in the spectra is due to the O-H stretching band. Peaks at 3959.35 cm⁻¹ and 2932.55 cm⁻¹ and the small peak at 1726.15 cm⁻¹ on the fruit extract can be respectively attributed to C-H overtone, and C=O stretching bands. The presence of saponin in the crude extract showed absorbance between 1054 to 1261.08 cm⁻¹, that is C-O-C which indicated oligosaccharide linkage absorption to sapogenins. The functional groups identified in these spectra infer the presence of saponin in the crude extract (Mozghan et al., 2014) and this can be held responsible for the foaming properties of the extract (Chapagain, 2006). The spectra obtained in this study are similar, and consistent with the one obtained in our previous work (Barminas et al., 2016). The significance of this analysis is that saponin is present in the crude extract of fruit using FTIR spectroscopy.

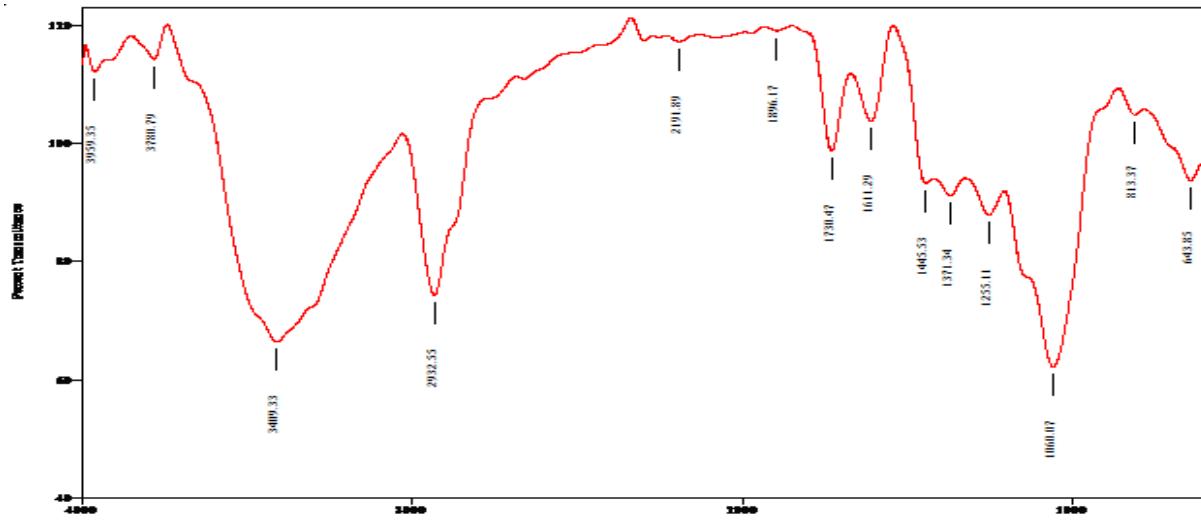


Fig. 1: FTIR spectra of *B. aegyptiaca* plant fruit extract

Table 2: Result of the GC-MS of *B. aegyptiaca* plant fruit

Peak	RT	Area Sum (%)	Name (C:\Database\NIST14.L)	Area
1	6.745	0.05	N-Acetyl-D-glucosamine	84586 007512-17-6 46
2	6.798	0.02	Heneicosanoic acid, isopropyl ester	219435 1000405-13-6 56
			E-11-Hexadecenoic acid, ethyl ester	142114 1000245-71-9 43
			trans-4-Aminocyclohexanol, N, O-dia	64118 1000384-20-8 38
3	7.177	1.52	4-(2-Acetyl-5,5-dimethylcyclopent-2-enylidene)butan-2-one	66042 1000195-40-7 47
4	7.267	2.72	Phenol, 2,5-bis(1,1-dimethylethyl)	66111 005875-45-6 53
5	7.351	0.69	Phenol, 2-methoxy-4-(1-propenyl)-Z	33412 000097-54-1 49
6	7.389	0.48	Eugenol	33239 000097-53-0 52
7	7.525	0.06	2-(2,5-Dimethoxyphenyl) ethylamine,PFP	166387 1000071-83-5 6
8	7.570	0.14	Pentanoic acid, 4-methyl-2-(2,5-di methyloxazolo[5,4-d]pyrimidin-7-yl)amino-	137755 1000294-63-5 25
9	8.531	0.24	cis-Vaccenic acid	142073 000506-17-2 38
			cis-5-Dodecanoic acid	63236 002430-94-6 38
10	8.577	0.08	Ethyl 9-deenoate	59502 067233-91-4 53
11	8.637	6.17	2-Dodecen-1-yl(-)succinic anhydride	115650 019780-11-1 90
			trans-13-Octadecenoic acid	129357 000693-71-0 35
			7-Octadecenoic acid, methyl ester	141274 057396-98-2 35
12	8.804	28.5	Oleic Acid	129336 000112-80-1 59
13	8.872	0.13	11-Octadecenoic acid, methyl ester	155737 052380-33-3 3
14	8.925	0.24	15-Hydroxypentadecanoic acid	109130 004617-33-8 60
15	9.273	0.57	Erucic acid	175491 000112-86-7 53
			Cholestan, 4,5-epoxy-, (4.alpha., 5.alpha.)-	205901 006079-19-2 40
16	9.341	0.69	7-Hydroxy-3-(1,1-dimethylprop-2-enyl) coumarin	86082 056881-08-4 56
17	9.425	0.22	2-Octen-1-ol, 3,7-dimethyl-	28296 040607-48-5 55
18	10.212	4.67	9-Hexadecenoic acid, methyl ester, (Z)-	117511 001120-25-8 50
19	10.431	6.38	Tetradecanoic acid, 5,9,13-trimethyl-, methyl ester	131323 056196-55-5 87
			Tricosanoic acid, methyl ester	195799 002433-97-8 87
			Hexadecanoic acid, methyl ester	119405 000112-39-0 78
20	10.825	2.31	cis-11-Eicosenoic acid	153110 005561-99-9 55
			cis-10-Nonadecenoic acid	141266 073033-09-7 55
21	10.893	1.34	Hexacosanoic acid	236701 000506-46-7 47
22	10.961	0.44	Cis-Vaccenic ester	186923 002433-96-7 64
23	10.999	1.69	7,11-Hexadecadienal	90897 1000130-85-7 59
24	11.127	29.07	Docosanoic acid	197561 000112-85-6 47
25	11.173	1.13	12-Octadecenoic acid, methyl ester	141284 056554-46-2 55
			6-Octadecenoic acid, methyl ester, (Z)-	141307 002777-58-4 35
26	11.279	0.89	7-Methyl-Z-tetradecen-1-ol acetate	117486 1000130-99-6 62
27	11.483	10.94	cis-13-Octadecenoic acid, methyl ester	141299 1000333-58-3 99
			11-Octadecenoic acid, methyl ester	141290 052380-33-3 98
28	11.771	13.03	n-Hexadecanoic acid	107548 000057-10-3 91
29	11.862	6.87	9-Octadecenoic acid, (E)-	129349 000112-79-8 90
30	12.081	4.66	Methyl trans-9-(2-butylcyclopentyl) nonanoate	1141321 108708-61-8 81
31	12.490	7.32	Oxiraneundecanoic acid, 3-pentyl-, methyl ester, trans-	171287 038520-31-9 45
32	12.876	37.89	Tridecanoic acid, 4,8,12-trimethyl-, methyl ester	119429 010339-74-9 35
33	12.952	22.83	Card-20(22)-enolide, 3-[(2,6-dideoxy-4-O-.beta.-D-ribo-hexopyranosyl)oxy]-5,14-dihydroxy-19-oxo-, (3. beta.,5. beta.)-	275242 000560-53-2 30
34	14.526	0.46	3-Octyne	5999 015232-76-5 42
35	14.912	0.90	Eicosanoic acid	154909 000506-30-9
36	15.147	0.58	4a.alpha.,4b.beta.-Gibbane-1.alpha.10.beta.-dicarboxylic acid, 4a-f	207548 006980-45-6 53
			ormyl-7-hydroxy-1-methyl-8-methylene-, dimethyl ester	
37	15.396	0.45	cis-11,12-Epoxytetradecen-1-ol	119231 1000130-82-5 55
38	15.661	0.42	1,4-Dihydro-pyridine-3-carboxylic acid	130876 1000259-17-855
39			1,3-Benzenediol, 5-methyl-2-(3,7,11-trimethyl-2,6,10-dodecatrienyl)-, (E,E)-	186735 006903-07-7 45
40	17.160	0.16	22,23-Bisnor-5-cholenic acid, 3.beta.-hydroxy, acetate(ester)	232291 1000127-30-8 30
41	17.273	0.38	Squalene	

Gas chromatograph-mass spectrometry analysis of the crude saponin extract in fruit of *Balanites aegyptiaca* (L) plant

The GC-MS analysis of the crude extract of *Balanites aegyptiaca* fruit revealed the presence of a total of 55 compounds. The major compounds were 9-Octadecenoic acid (Z)-, methyl ester and Tridecanoic acid, 4,8,12-trimethyl-,

methyl ester (37.89%), Docosanoic acid (29.07%), unsaturated fatty acid: Oleic Acid (28.5%), Card-20(22)-enolide, 3-[(2,6-dideoxy-4-O-.beta.-D glucopyranosyl-3-O-methyl-.beta.-D-ribo-hexopyranosyl)oxy]-5, 14-dihydroxy-19-oxo-, (3. beta., 5. beta.)- (22.83 %), n-hexadecanoic (13.03 %), saturated fatty acid namely: Tetradecanoic acid, 5,9,13-trimethyl-, methyl ester, Tricosanoic acid, methyl ester,

Hexadecanoic acid, methyl ester (6.38 %), 9-Hexadecenoic acid, methyl ester, (Z)- (4.67%), cis-13-Octadecenoic acid, methyl ester 11-Octadecenoic acid, methyl ester (10.94 %) and 2-Dodecen-1-yl (-) succinic anhydrid (6.17%) (Table 2).

The results of this study indicated that GC-MS profile of the phytochemical constituents of the extract of *Balanites aegyptiaca* fruit revealed the presence of saturated and unsaturated fatty acid. The 2-Dodecen-1-yl (-) succinic anhydride and Erucic acid are the carboxylic acid anhydrides, which are used as the synthesis of amphiphilic-N-[C₂-(dodec-2-en-10-yl) succinoyl] (Tikhonov *et al.*, 2008) and chemical modification of sago starch, via esterification (Abdul *et al.*, 2001). The 9-Tetradecen-1-ol, acetate, (Z)-, the main pathway leading to both types of saponins is similar and involves the head-to-tail coupling of acetate units (Van *et al.*, 2013). One common feature shared by all saponin is the presence of a sugar chain attached to the aglycon (Faizal, 2013), this is supported by the result obtained revealed the presence of sugar compounds namely: Card-20(22)-enolide, 3-[(2,6-dideoxy-4-O-β-D-glucopyranosyl-3-O-methyl-β-D-ribo-hexopyranosyl)oxy]-5,14-dihydroxy-19-oxo-, (3. β-5. β-). The experiment showed that the unsaturated fatty acid named oleic acid is fatty acid used as emulsifying agent, as an emollient (Yakubu *et al.*, 2017). The cis-9-octadecanoic acid or cis-oleic acid that has double bond at C-9, is a principal acid obtained by saponification, like other fatty acid, it does not occur in the free state, but normally found as an ester of glycerol, which as a glyceride and long chain alcohol and carboxylic acid (Thakur *et al.*, 2011).

Performance tests of the crude saponin extract from *Balanites aegyptiaca* fruit

Foam production and stability over a period of time of the extract from the *B. aegyptiaca* fruit

Foam produced by agitation of surfactant is the metastable systems occurring at the liquid-air interface (Rouimi *et al.*, 2005). Fig. 2 present and inter-compares the foam capacity of the fruit extract and OMO. The extract exhibit higher foam capacity compared to OMO, saponin extract tends to be rising higher than the OMO as the concentration increases. The foam production and stability at 5 min for extract and OMO shows on the Table 3, drop from 4.5 to 4.3 cm and 5.5 to 5.2 cm respectively at the concentration of 0.1 g/ml. The data indicated that crude extract has the potential to be an effective surfactant property, also possessed moderate detergency (70%). Being that the important property of surfactant substances in aqueous solution is their ability to promote the formation of foams and bubbles. The study showed that, high quality thick foam was produced by crude extract solution as was observed during the experiment, this can be attributed to the saponin content of the extract and variation in such factors as the critical micelle concentration (CMC) and also specific molecular features of the, ionic strength and pH of the surfactant solutions (Tamura *et al.*, 2006).

Table 3: Present the foam production and stability of fruit extract and OMO at different concentration

Concentration (g/ml)	Initial foam height (cm)		Foam stability at 5 min	
	fruit extract (F extract)	OMO	Fruit extract	OMO
0.1	4.5	5.5	4.3	5.2
2.0	6.6	6.0	5.3	5.5
3.0	7.5	7.0	7.2	6.8
4.0	8.0	7.8	7.2	7.0
4.5	8.5	8.3	8.0	8.0

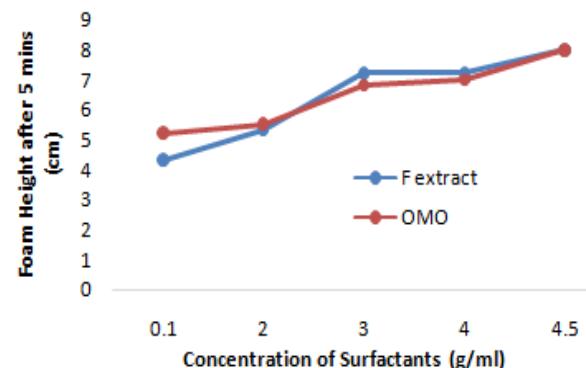


Fig. 2: Foam production and stability using different concentrations of the surfactant solutions at 5 min

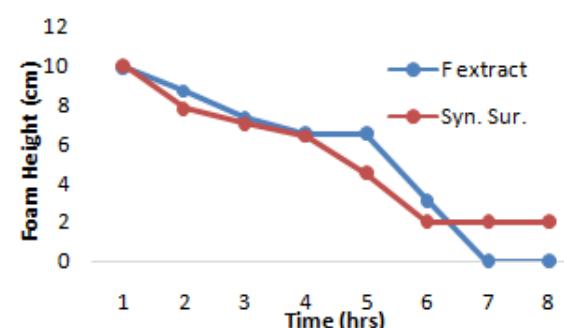


Fig. 3: Foam production and stability for 3 g of surfactants at 29°C with time (hrs)

From the study, Fig. 3 illustrated 3 g of surfactant solutions in 100 ml for fruit extract and synthetic surfactant (OMO), it was observed that at the hour of 7 and 8 the extract disappeared leaving the compare surfactant. This observation may be attributed to their physical and bubbled formation of the surfactants. Although the foam produced by the extract is much thicker and small bubbles than OMO. Agu (2013) reported that small bubbles will disappear with time, and the foam will tend towards a structure of nearly large bubbles. This may one of the major reason of crude extract disappearing earlier from the hrs of 7 and 8 than OMO. The production of thicker foam observed in extract during the experiment may due to its crude nature and absence of formulation like stabilizer, builder, etc. The foam stability of the synthetic surfactant (OMO) shows gradual decreased sharply from the first one hour and finally stable from 6, 7 and 8 hrs. The results obtained competes favorably with the (OMO) which shows good quality of foam production throughout the experiment,

Emulsion formation/capacity of the crude saponin extract from *B. aegyptiaca* fruit

Figure 4 present and compare the emulsion capacity of 2 g of *B. aegyptiaca* fruit extract and OMO as function of time. With this observation, the emulsion formation/capacity of the extract shows good emulsion (98.3 to 55.0%), which shows higher and better compared to OMO (98.0 to 40%) within the hrs of 1 to 48 h, respectively. This can be attributed to a quantitative presence of saponin in *B. aegyptiaca* fruit extract, as saponin has been reported by Barminas *et al.* (2016) to be a good emulsifying agent. Fig. 5 present and compare the emulsion capacity of 5 g of *B. aegyptiaca* fruit extract and OMO as function of time. The emulsion capacity of *B. aegyptiaca* fruit extract (92.5 to 35.2%), which is still correspondingly higher than that of OMO (92.0 to 34.3%) within the hrs of evaluation from 1 to 48 h, respectively. Although, the emulsion capacity at this higher concentration

(5 g) is higher compare to 2 g, but equate with OMO within 5th and 9th hrs. This concurs with the report by Kamba *et al.* (2013) that at low soap and detergent concentration, rapid coalescence among the inner and outer droplets to inner phase occurred, resulting in separation within a short period of time. This further indicates the diminishing emulsion capacity and stability with increase in the concentration of *B. aegyptiaca* fruit extract.

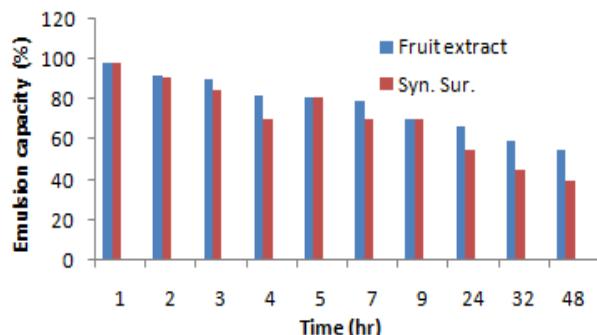


Fig. 4: Emulsion capacity (%) versus time (hr) for 2 g of crude saponin extract and OMO solutions

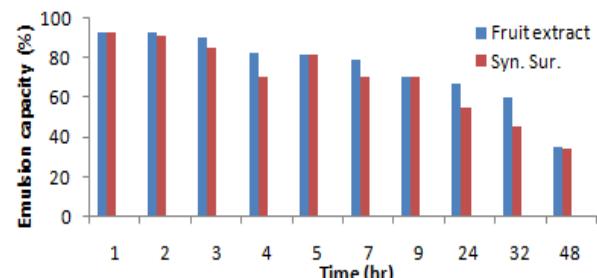


Fig. 5: Emulsion capacity (%) versus time (hr) for 5 g of crude saponin extract and OMO solutions

Skin treatment (Leather)

Flayed goatskin, salted and dry skin

Plates 2 presents flayed goatskin, salted and dry skin. Skin and leather characteristic of goat was study in relation to it age and sex. The random selected skin from goat reared in Geiri Adamawa state, Nigeria was weighed. The method of preservation was used based on varies, depending on the climate and environment ranging from simplest method of drying using salt instead of more expensive freezing method.



Plates 2: Flayed male goatskin, salted (A) and dry (B) ready for processing into parchment.



Plates 3: Parchment from male goatskin to remove excess fat (A), and finished leather (B)

Parchment from goatskin and finished leather

Plates 3 representing the areas of interest were the physical changes due to the manufacturing processes involved in converting the skin into parchment and leather. To obtain a useful material, the removal of hair, fats has been treated with crude extract. This research has used previous methods of leather production in the literature on the uneven thickness of goat skin to make it even. This is a dedication to the

improvement of leather quality using natural products. Plate 3 shows Parchment from goat skin and dry leather shows different physical characteristics and most notably the colour of wet leathers which changed upon drying.

Influence of the fruit extract at different concentrations on degreasing efficacy

As a preliminary trial for checking the possible efficiency of bio-surfactants we checked the emulsion of a leather grease

sample from male goatskin supervised in a household of a farmer in Girei Local Government Areas. The sample of saponin extract from the fruit of *B. aegyptiaca* was compared with synthetic washing agents (OMO), which are normally applied in tannery industry. The test was done with the same amounts of surfactant, using 10, 15, 20, 25 and 30 g/ml per 11.5 cm heights and 1.5 cm width of leather under of temperature of 25°C (77°F). The optimal pH emulsifying efficacy of the surfactants was usually range from 5 and 6. Plates 4 below shows physical appearance observed which was taken within 32 h. The extract samples were superior in emulsion and whiteness efficiency as compared with the OMO (synthetic product). The removal of dirt and fat is increased by increasing the surfactants concentration extract

and OMO. An approximate water content of 60-80% at the beginning of degreasing was used, the higher the concentration of the saponin (10, 15, 20, 25 and 30 g), the greater the efficacy of the leather which treated to preserve a quality and natural beauty. And also the higher the temperature the better the appearance and whiteness of the finished leather obtained which shows in Plate 5 below. *B. aegyptiaca* fruit used to treat leather differently were yellow-brown when wet but turned different colours after drying, the controlled sample showed a brown colour and synthetic surfactant became dark brown when dry (10 g and 30°C), respectively.



Plate 4: Treated goatskin (male) leathers using different concentration (g/ml) of fruit extract from *B. aegyptiaca* at 28°C, compared with OMO



Plate 5: Treated goatskin (male) leathers using different temperature (°C) of Fruit extract from *B. aegyptiaca* at optimum concentration of 30 g/ml, compared with OMO



Plate 6: Image representing the uses of male goatskin treated with crude saponin extract of *B. aegyptiaca* fruit

*Determination of tensile strength and percentage elongation The effect of extract from *Balanitis aegyptiaca* fruit on tensile strength and compared with OMO*

Tensile strength or fracture stress of tanned leather is the stress required to fracture a test specimen of specified thickness, fibre orientation and location on the skin (Dennis, 2016). Tensile strength is the ability of a material to withstand a longitudinal pulling force. The results of the measurement of tensile strength on the leathers treated with crude saponin extract are given at Figs. 6 and 7 below. Each figure represent different measurement based on the two parameters (concentrations and temperature). The machine used was Monsanto Tensometer type 'w', serial number 9875, made in UK.

Figure 6 show that as concentrations increases, the tensile strength decreases, because the removal of fat is increased by increasing the surfactant concentration, the higher the concentrations of the saponin, the greater the degreasing efficacy of the leather and lower the tensile strength. This agreed with RamÓn (2016), that tensile strength decreases with decreasing fat content of the leather. At concentration of 30 g/ml tensile strength decreases to 23 and 28 N/mm² for fruit extract and OMO, respectively. UNIDO standard 20 N/mm² for tensile strength (Mehmet *et al.*, 2016), the same results can meet or fail the quality standards depending on the parts of the plant and different locations. It was observed that tensile strength is higher when the treatment on samples are not properly effective on the leather because of the fats or protein based fibrillary network which consists mainly from collagen (Mehmet *et al.*, 2016) and decrease gradually as the concentrations and temperature increase (86.67 to 23 N/mm² and 86.98 to 28 N/mm²), at concentration of 10 to 30 g/ml, respectively. Figure 7 indicated that, as the temperature increase from 30 °C to 50 °C, the tensile strength of leather decreases from 86.67 to 21 N/mm² and 86.98 to 24 N/mm² for saponin extract and OMO, respectively. *Garad* tanned goat skins recorded a tensile strength of 23.5 N/mm² and Bureau of Indian Standards (BIS) sets the value at 19.6 N/mm² (Musa and Gasmelseed, 2013). Kenya Bureau of Standards (KEBS) has also documented a minimum standard of 15 and 6 N/mm² for leather uppers and linings, respectively (KEBS, 2012).

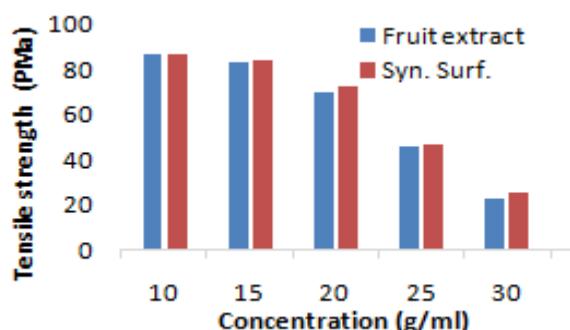


Fig. 6: Tensile strength at different concentration of sample in g/ml at a temperature of 28°C

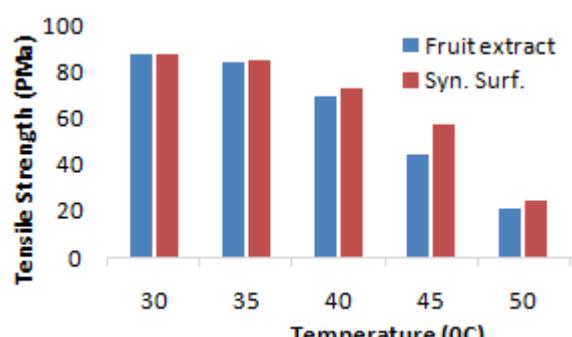


Fig. 7: Tensile strength at different temperature of sample in g/ml at optimum concentration of 30 g/ml

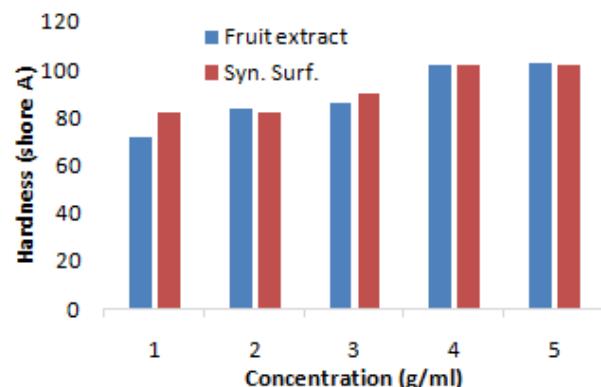


Fig. 8: Elongation (%) at different concentration of sample in g/ml at a temperature of 28°C

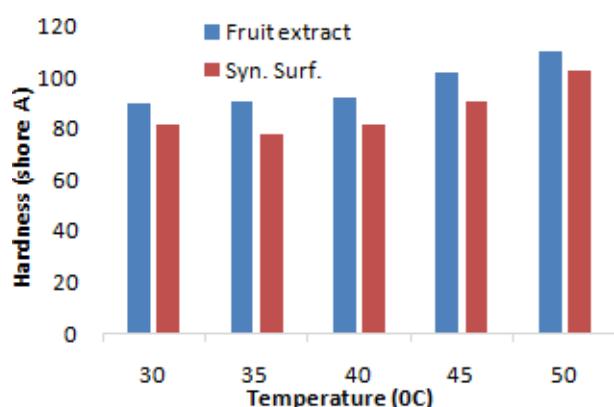


Fig. 9: Elongation (%) at different temperature of sample in g/ml at optimum concentration of 30 g/ml

*The effect of crude saponin extract of *B. aegyptiaca* fruit on the elongation at break (%) and compared with synthetic surfactant (OMO)*

Elongation at break known as fracture strain is the ration between change length and initial length after breakage of the test specimen (Petroudy, 2017). It expresses the capability of natural material to resist change of shape without crack formation. Fig. 8 show the elongation (%) at various concentrations (10, 15, 20, 25, 30 g) of fruit extract and OMO at temperature of 28°C. the results increases from 72, 84, 86, 102, 103% and 82, 84, 86, 102, 103%, respectively. Although in a tensile test, leather will generally extent to between 30 to 120% before rapture (Yu *et al.*, 2010) in which the results observed are fall within this range.

Figure 9 shows the percentage elongation at break values of the extract and compared with OMO. At optimum concentration of 30 g, a percentage elongation of 90, 90.5, 92.4, 102, 110.4% was obtained for crude saponin extract which is higher when compared with OMO has values of 81.5, 78, 81.6, 102, 103% at temperature of 30, 35, 40, 45, 50°C, respectively. This is in agreement with the KEBS minimum standard value at 30 % for leather intended for manufacture of linings and highest at 80% (KEBS, 2012). The relaxation time increase as the length of the time increased (Yu *et al.*, 2010).

*The effect of saponin crude extract of *B. aegyptiaca* fruit on the hardness of the leather and compared with synthetic surfactant (OMO)*

Leather can be classed by hardness, ranging from hard leather for shoe soles to soft leather for fashion accessories. From tanners' experiences it is believed that each precise step can influence the final texture of leather (Yu *et al.*, 2010).

Figure 10 present the values of crude saponin extract of *Balanites aegyptiaca* fruit on the effect of hardness of male goatskin leather. At the concentration of 10, 15, 20, 25 and 30 g the values decrease from 84.67, 84, 82.33, 78.67, 58.9 shore A and 90.67, 88, 81.67, 77.5, 56.8 shore A for fruit extract and OMO solutions, respectively. Generally, the essence of leather making is to remove substances which are not needed, but preserve and crosslink the collagen fibres within the main constituent of leather. The smaller the size of the dispersing particles in the emulsion, the softer is the leather (Yu *et al.*, 2010).

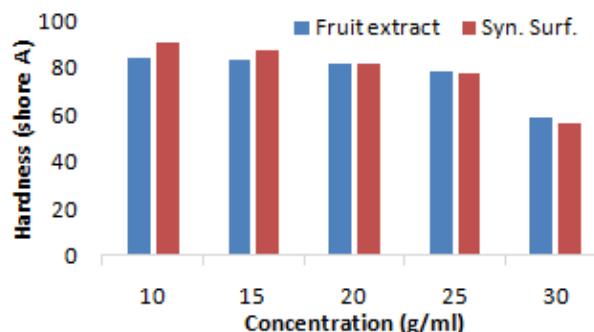


Fig. 10: Hardness test at different concentration (g/ml)

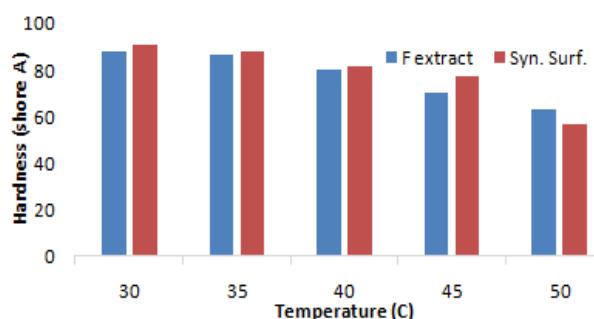


Fig. 11: Hardness test at different temperature (°C)

Figure 11 present the hardness test at different temperature (°C), likewise the values decrease as the temperature increase from 30 to 50°C. The extract was compared with synthetic detergent at optimum concentrations of 30 (g/ml), it decreases from 88, 86.33, 80.33 70.33, 63.33 shore A and 90.67, 83.33, 80.33, 70.33, 56.8, respectively. In both Figs. 10 and 11, the value of OMO tend to be higher than extract. Yu *et al.* (2010) reported that materials generally with less elongation tend to be stiffer than those with more elongation. Hardness may be defined as a material's resistance to permanent indentation.

Conclusion

Based on the above results, the *Balanites aegyptiaca* fruit grown in Demsa LGA may be a very useful source of surface-active agent compounds for treating leather from goatskin. Saponins contents in the fruit was high based on the extraction result, and saponins in *Balanites aegyptiaca* plant have been extensively used as detergents, foaming and surface-active agents. The foaming activities of the crude extract (crude aqueous) showed higher and better when compared with synthetic surfactant (OMO). The FT-IR revealed the presence of -OH, indicates the presence of phenolic that a class of chemical compounds consisting of a hydroxyl group bound directly to an aromatic hydrocarbon or may likely to be furostanol saponin which has been reported by Iorizzi *et al.* (2002) having monodesmosidic Sugar moites. The C-O-C absorption indicated glycoside linkage to the sapogenins.

Comparison at different concentration and temperature of crude extract and synthetic surfactant (OMO) on the tensile strength and elongation showed that, emulsifying properties of the crude extract improves with increased in concentration of the extract. Further studies are highly emphasized in order to isolate and identify the type of saponin present.

Conflict of Interest

The authors declare that there is no conflict of interest reported in this work.

References

- Abdul Khalil HPS *et al.* 2001. The Effect of Anhydride Modification of Sago Starch on the Tensile and Water Absorption Properties of Sago-filled linear Low-density polyethylene(LLDPE). *Polymer-plastics Technology and Engineering*, 40(3): 249-263.
- Agu Matthew Onyema, Jatau David Finchawa, Hotton Joseph Anthony (2013). Analytical Investigation of Foam Formation and Emulsifying Power of Sanya (*Securidaca Longepedunculata*) Roots and Comparism with Some Commercial/Synthetic Surfactants. *Int. J. Engr. and Sci.*, 2(9): 08-15.
- ASTM D2209-00 2015. Standard Test Method for Tensile Strength of Leather, ASTM international, West Conshohocken, PA, www.astm.org.
- Azab MM 2001. Preparation and evaluation of some water soluble polyester surfactants. *J. Appl. Polymer Sci.*, 81(14): 3313-3424.
- Barminas JT, Nkafamiya II, Kime B & Ayodele Akinterinwa 2016. Extraction and evaluation of foam formation and surface tension of saponin-base surfactants from *Balanites aegyptiaca* (L) plant. *Pharmac. and Chem. J.*, 3(1): 34-38.
- Belkhodja M & Benkablia M 2000. Proline response of Faba bean (*Vicia faba* L.) under salt stress. *Egypt. J. Agric. Res.*, 78 (1): 185-195.
- Chapagain PB 2006. Characterization of desert date (*Balanites aegyptiaca*) saponins and their biological activities. Ben-Gurion University of the Negev. Ph.D, Thesis, pp. 27-32.
- Chevallier MH, Vaillant A, Bensaid S, Sahki-Boutamine R, Diallo OB, Sanou J *et al.* 2004. Impact Des Pratiques Humaines Sur La Conservation Et La Gestion in Situ des Ressources Forestières: Cas d'Acacia Tortilis *Raddiana* Et De *Balanites Aegyptiaca*. CSFD, Rapport Du Projet N°57, p. 68.
- Chinelo A Ezeabara, Okeke CU, Bibian O Aziagba, Chinyere V Ilodibia & Adaeze N Emeka 2014. Determination of saponin content of various parts of six citrus species. *Int. Res. J. Pure and Appl. Chem.*, 4(1): 137-143.
- Danthu P, Soloviev P, Totte A, Tine E, Ayessou N, Gaye A *et al.* 2002. Caractères physico-chimiques et organoleptiques comparés de jujubes sauvages et des fruits de la variété gola introduite au sénégal. *Fruits*, 57(3): 173-182.
- Debela Hunde Feyssa TJ, Njoka Zemedie Asfaw & Nyangito MM 2015. Nutritional contents of *Balanites aegyptiaca* and its contribution to human diet. *Afri. J. Food Sci.* 9(4): 346-340.
- Dennis OK 2016. Evaluation of *Plectranthusbarbatus* a Potential Vegetable Tanning Agent in Nyamira County, Kenya. M.Sc. Thesis, Department of Public Health Pharmacology and Toxicology, University of Nairobi, Kenya.
- Djafari Petroudly SR 2017. In Advanced High Strength Natural Fiber Composites in Concentration. Trademark of Elsevier B.V. 1.

- Edeoga HO, Okwu DE & Mbaebie BO 2005. Phytochemical constituents of some Nigerian medicinal plants. *Afri. J. Biotechn.*, 4(7): 685-688.
- Faizal A 2013. Modulation of saponin production in Medicinal *Maesa* spp. Ph.D Thesis Ghent University, Ghent, Belgium.
- Guclu-Ustundag O & Mazza G 2007. Saponins: Properties, Applications and Processing. *Crit Rev Food Sci. Nutr.*, 47(3): 231-58.
- Handa SS 2008. An overview of extraction techniques for medicinal and aromatic plants. In: Extraction Technology for Medicinal and Aromatic Plants; Handa SS, Khanuja SPS, Longo G, Rakesh DD, Eds.; International Centre for Science and High Technology: AREA Science Park, Trieste, Italy, Chapter 1; Pp. 21-54.
- Hauber & Knodler 2008. Innovative Techniques: Available Techniques (BAT) in the Tanning Industry Pollution as a Contribution on Complying. Leather Institute-Reutlingen, Germany, Germany.
- Huda Farid, Ernst Haslinger, Olaf Kunert & Chritain Wegner 2002. New steroid glycosides from *Balanites Aegyptiaca* (L) plant. *Journal of Helvetica Chimica Acta*, 85(4): 1019-1026.
- International Organization for Standardization 2002a. Leather- Chemical, Physical, Mechanical and Fastness Tests: Sampling Location, ISO 2418, Geneva, Switzerland.
- International Organization for Standardization 2002b. Leather - Physical and Mechanical Tests: Determination of Thickness, ISO 2589, Geneva Switzerland.
- International Organization for Standardization 2002c. Leather-Physical and Mechanical Tests: Determination of Strength and Percentage Extension, ISO 3376, Geneva Switzerland.
- International Organization for Standardization 2006. Leather- Physical and Mechanical Tests: Sample Preparation and Conditioning, ISO 2419, Geneva, Switzerland.
- Iorizzi M, Lanzotti V, Ranalli G, De Marinno S & Zollo F 2002. Antimicrobial frustanol saponin from the seeds of *Capsicum annum*. L. Var. acuminatum. *J. Agri. Food Chem.*, 50: 5310-4316.
- Kenya Bureau of Standards 2012. KS 1732:2012: Footwear children's Shoes (above 2 to 6 years)- Specifications.
- Kime B, Barminas JT, Nkafamiya II & Akinterinwa A 2015. Extraction and evaluation of a saponin-based surfactant from *Balanites aegyptiaca* plant as an emulsifying agent. *Int. J. Innov. Sci., Engr. and Techn.*, 2(7): 4-6.
- Manisha M, Muthuprasanna P, Surya Prabha K, Sobhita Rani P, Satish Babu AI & Sarath Chandiran I 2009. Basics and Potential Applications of Surfactants – A. CODEN (USA): IJPRIF. *Int. J. Pharmtech Res.*, 1(4): 5-6.
- Manji AJ, Sarah EE & Modibbo UU 2013. Studies on the potentials of *Balanites aegyptiaca* seed oil as raw materials for the production of liquid cleansing agents. *Int. J. Physical Sci.*, 8(33): 1660.
- Mehmet Mete Mutlu, Nilay Ork, Birgul Kaya & Samet Gunesel 2016. Deparment and Mapping of Tear Load, Tensile Strength and Extension Values on Overall Area of Goatskin Leather. International Conference on Advanced Material and Systems.1
- Mimi Sakinah A. M., Sri Murmi Astuti, Retno Andayani BM & Awalludin Risch 2011. Determination of saponin compound from *Anredera cordifolia* (Ten) steenis plant (*Binahong*) to potential treatment for several disease. *J. Agric. Sci.*, 3(4): 224.
- Molla E, Giday M & Erko B 2013. Laboratory Assessment of the molluscicidal and cercariacidal activities of *Balanites aegyptiaca*. *Asian Pacific J. Trop. Biomed.*, 3(8): 657-662.
- Mozghan Soltani, Kazem Parivar, Javad Baharara, Mohammed Amin Kerachian & Javad Asili 2014. Hemolytic cyrotoxic properties of saponin purified from *Holothuria leucospilota* sea cucumber. *Biochemistry and Molecular Biology*, 3(1).
- Musa AE & Gasmelseed GA 2013. Application of *Acacia nilotica* spp nilotica (powder) as alternative vegetable re-tanning material. *J. Forest Products and Indu.*, 3(2): 112-117.
- Ndoye M, Diallo I & Gassama Dia YK 2004. Reproductive biology in *Balanites aegyptiaca* (L.) Del., A semi-arid forest tree. *Afr. J. Biotech.*, 3(1): 40-46.
- Ninfaa DA, Maalekuu BK & Akurugu GK 2014. Postharvest handling of the edible parts (leaves and fruits) of the desert date (*Balanites aegyptiaca*): A case study in the Jirapa and Nadowli Districts of the Upper West Region of Ghana. *Int. J. Plant, Animal and Envntal. Sci.*, 4(2): 1-11.
- Oleszek WA 2002. Chromatographic determination of plant saponins. *J. Chromatography A*, 967: 147-162.
- Osbourne AE 2003. Saponins in cereals. *Phytochemistry*, 62: 1-4.
- Padmashree TS, Vijayalakshmi L & Puttaraj SJ 1987. Food science technology, 24: 221-225.
- Palop R, Marsal M & Cot J 2000. *J. Soc. Leather Techn. Chem.*, 84: 170.
- RamÓn P. (2016). Sheepskin and Cattle Hide Degreasing. Barcelona, Cromogenia Limits, 10-20. cromogenia@cromogenia.com • www.cromogenia.com
- Rouimi S, Schorsch C, Valentini C & Vaslin S 2005. Foam stability and interfacial properties of milk protein-surfactant systems. *Food Hydrocolloids*, 19(3): 476-478.
- Sirelkhatim BE, Asha ME, Nourelhuda AK, Adil AG & Hayat OD 2014. A review on omega-3 and omega-6 essential fatty acids: Uses, benefits and their availability in pumpkins (*Cucurbita maxima*) seed and desert dates (*Balanites aegyptiaca*) seed kernel oils. *Pak. J. Biol. Sci.*, 17: 1195-1208.
- Tamura T, Taheuchi Y & Kaneko Y 2006. Influence of surfactant structure on the drainage of nonionic surfactant foam films. *Journal Colloid Interface Science*, 206(1): 112-121.
- Van P, Robert J, Van Der W, Jan C, De Jang Ed, Resrendra Johannes G 2013. *Hydroymethylfurfural*, A versatile platform chemical made, from Renewable Resources, 113(3): 1499-1597.
- Yakubu EO, Otitoju O & Onwuka J 2017. Gas chromatography-mass spectrometry (GC-MS) analysis of aqueous extract of *Daniellia oliveri* stem bark. *Pharm. Anal Acta*, 8: 568.
- Yu-Fench Chen Chao-Hsun Yang, Ming Shiang Chang, Yong-Ping Ciou & Yu-Chun Huang 2010. Foam properties and detergent abilities of the saponins from *Camellia oleifera*. *Int. J. Mol. Sci.*, 11(11): 4417-4425.