

## Extraction of Bio-Base Surfactant(SAPONIN) from *Cissuspopulnea* plantleaves from Gurhengwal town, Askira/Uba local government of Borno state Nigeria.

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### Abstract

*Cissuspopulnea* plant shows higher surface tension and sorption behaviour property as well as foam formation/capacity far better especially leaves and roots than some commercial/synthetic surfactants (OMO) that were used in the study. This saponin- based surfactant is significantly be as an eco-friendly and may show better biodegradability than the commercial surfactants. The natural surfactant from *Cissuspopulnea* plants leave and roots will contribute to efforts in diversify the Nigerians economy, a way to solve the problem of non-biodegradable, high price and non-eco-friendly phenomenon that exists in the production of commercial/synthetic surfactant and add value to green chemistry and sustainable development and exploitation of the cleansing properties of saponin present in *Cissuspopulnea* plants.

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Date of Submission: 11-08-2020

Date of Acceptance: 27-08-2020

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### I. Introduction

Surfactants are usually organic compounds that are amphiphilic, meaning they contain both hydrophobic groups and hydrophilic groups. Surfactants carry polar and non-polar groups. They therefore, are soluble in both organic solvents and water, surfactants possess surface-active properties which functions as wetting, cleaning, rinsing and /or fabric softening, hence their typical inclusion in the formulation recipe of detergents and soaps in the field of pharmaceutical sciences, surfactants have also been reported to belong to a group of chemicals of high environmental relevance due to their large production volume. The dangers posed by the synthetic surfactants to the environments therefore suggest the search for safer products (Kimeet *et al.*, 2016). The wide application of surfactant has opened room for accelerated revolution in surfactant making with the introduction of new methods and ingredients as raw materials, such as the use of natural Bio-base surfactants to make soaps, dish washing liquids and shampoo, foaming, emulsifying, in many practical applications and products processes (Azab, 2001).

Surfactants are routinely deposited in numerous ways on land and into water systems, whether as part of an intended process or as industrial and house hold waste. Some of them are known to be toxic to animals, ecosystems, and humans and can increase the diffusion of other environmental contaminants (Azab, 2001).

### Origin and Distribution of *Cissuspopulnea*.

*Cissuspopulnea* belongs to the family of *amplidacea (vitacea)* as confirmed by (Okafor, 2009), which are woody climbers, sometimes vines, rarely small succulent's trees, and hermaphrodite or *polygamoneaciousto polygamodroecions*. *Cissuspopulnea* grows in divers' ecozones of Nigeria, Uganda, Niger Republic, Cameroon and Coted'voire (Geldam, 2004). The plant is climbing stem, widely distributed in many parts of Nigeria, especially within the guinea savannah region of Anambara, Kogi and Benue States (Alfaetal., 2003). The Igala and Idoma ethnic group refers to this plant as Okoho, Yorubas as Ogholo, and Hausas as dafaaraa. It is used extensively in medicinal preparation in West Africa. Thickening agents from plants such as melon, beniseed, Ogbono, Okoro and Okoho (*Cissuspopulnea*) are used to thicken soups in different parts of Nigeria. These thickening agent contain nutrients that are beneficial to health.

All part of *Cissuspopulnea* exudes, a mucilaginous materials are traditionally utilized as food raw materials (Ojেকেleetal., 2004). Gum exudates are mainly obtained from the stem and root of the plant. (Garcia *et al.*, 1997). The gum obtained from *Cissuspopulnea* has been evaluated for its potential use as a dispersants in pharmaceuticals liquid system.



**Figure 1:** Tree of *Cissuspopulnea*

### **The leaves**

Nigeria is enriched with different types of useful plants whose fruits, seeds, stems, roots and leaves serve various important role in medicine and nutrition. Among the numerous varieties of plants is *Cissuspopulnea* (Inavova et al., 2005). Bioactive ingredient made from the plant has proven medicinal properties. Experts in the evaluations of *Cissuspopulnea*, reported that they have antimicrobial activities which cure many sexually transmitted diseases infections that could be responsible for male infertility (Ojekale et al., 2006). Other studies have also shown that the essential oil from the stem powder of the plant inhibits the growth of several germs of bacteria origin and as such may correct male infertility arise from the bacterial infections. In Niger, Kogi, Plateau, Kwara, Borno and Benue States of Nigeria, the plant is used for making vegetable soup for post-natal stoppage of blood flow (Soladoye and Chukwuma, 2012) the presence of alkaloid, tannins, saponins, anthraquinone and flavonoids in the root, leaves and stem of *Cissuspopulnea* in different locations in Benue State Nigeria where the plant is in high demand as a delicacy.

### **Structure and basic properties of saponins**

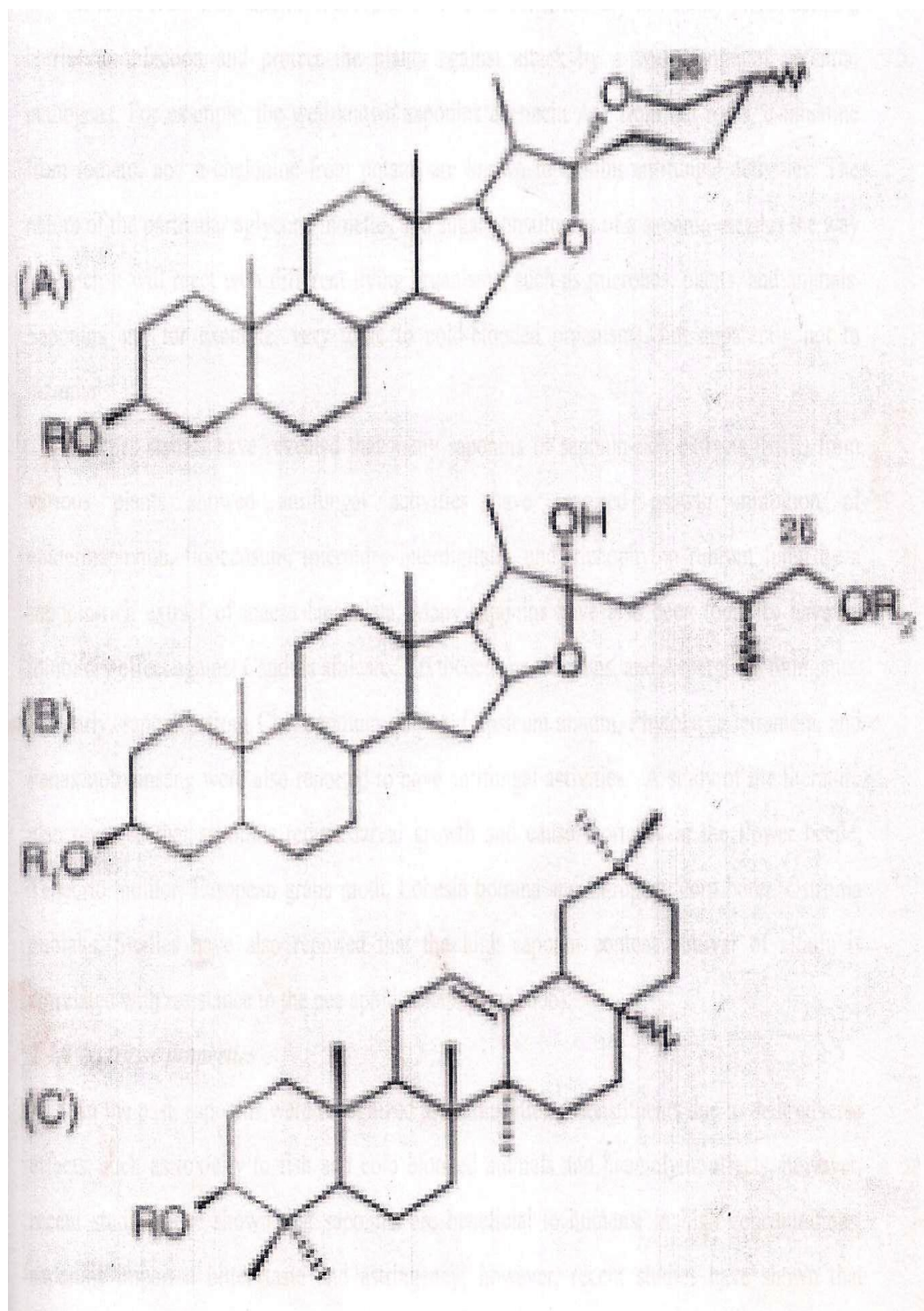
Saponin is a class of chemical compound found in particular abundance in various plant species. More specifically, they are amphipathic glycosides grouped phenomenological by the soap-like foaming they produce when shaken in aqueous solutions and structurally by having one or more hydrophilic glycoside moieties combined with a lipophilic triterpene derivative. Saponins are naturally occurring in a variety of higher plants and a few marine species. These *amphiphilic* compounds consist of a hydrophobic aglycone linked to a hydrophilic sugar moiety. According to Chapagain, (2006), Saponins are basically classified as triterpenoids, steroids, or steroid alkaloids. According to the structure of the aglycone (non sugar part) and *monodesmosidic*, *bidesmosidic*, *ortridesmosidic* (Greek *desmos*=chain) is according to the number of the sugar moieties attached to the aglycone. The steroid saponins comprise two categories, based on the structure of the aglycone ring. They are said to be *furostane* when the E ring of the aglycone is opened and *spirostane* when it is closed. All classes of aglycones may have a number of functional groups causing big natural diversity. This diversity can be further expanded by the composition of sugar chain, sugar numbers, branching patterns, and type of substitution. Hexoses (glucose, galactose), 6-dehydroxyhexoses (*rhamnose*, *furanose*), *pentoses* (xylose, arabinose), and

uronic acids (*glucuronic, galacturonic*) are the most common sugar residues in the saponin molecules that are attached at the C-3 position 9 *monodesmosidic*, and C-26 or C-28 (*bidesmosidic*). Tri-*desmosidic*saponin(saponins having three sugar chains) are seldom found in nature. The sugar moiety linked to the glycone through an ether or ester glycosidic linkage at one or two site. The configuration of inter-glycosidic is either  $\alpha$  or  $\beta$  and the monosaccharide can be in the *pyranose* or *furano*se forms (Chapagain, 2006).

Since the *aglycone* is very hydrophobic and the sugar chains are very hydrophilic, these characteristics provide saponin molecules with very excellent foaming and emulsifying properties. The presence of both polar (sugar) and non-polar (steroid or triterpene) groups provide saponins with strong surface-active properties that possess a strong foaming capacity in aqueous solutions. In aqueous solution they form small micelles individually, and these hydrophobic micelles of triterpene or steroid groups stack together like small piles of coins. Saponin molecules also form micelles with sterols, such as cholesterol and bile acids. The hydrophobic portion of the saponin (*aglycone* or *sapogenin*) associates (*lipophilic* bonding) with the hydrophobic sterol molecules, in stacked micelle aggregation. Indeed, it should be no surprise that the name 'saponin' comes from the Latin word *sapo* meaning soap (Chapagain, 2006). Figure 2. below shows the aglycone skeletons of the saponins.

### **Application of saponin**

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 chain. Saponins belong to a complex and chemically diverse group of compounds which derive their name from their ability to form stable, soap-like foam in aqueous solutions. In plants, saponins play a role as secondary metabolites and are assigned for defence mechanism. Consumer demand for natural products coupled with their physicochemical (surfactants) properties and mounting evidence on their biological activity such as anticancer and anticholesterol activity has led to the emergence of saponins as commercially significant compounds with expanding applications in food, cosmetics, and Pharmaceutical sectors. The realization of their full commercial potential requires development of new Processes/processing strategies to address the processing challenges posed by their complex nature. Their adverse effects are mainly reflected in depressed feed intake that causes growth inhibition to animals, monogastrics in particular, and reduced animal reproduction (Chapagain, 2006).



**Figure 2:** Aglycone Skeletons of (A) Steroidal Spirostane, (B) Steroidal Furostane, and (C) Triterpenoidsaponins. R = Sugar moiety (According to Chapagain, *et al.*, 2006).

### Surfactant technology

Application of surfactant in many technological developments has not only ensured costeffectiveness. It has helped many industries or countries to diversify their economy: - Alkaline surfactant polymer technologies: surfactant flooding is a well-known concept that has been practiced in the field for many decades. Current technology is a progressive and gradual development of technologies and ideas that have existed for a long time. The key difference is the amount of surfactant used in projects today is much lower due to high purity. Surfactant flooding is usually carried out after a water flood. There are two main types of surfactant flooding-alkaline surfactants polymer (ASP) or surfactant polymer (SP) flooding. It helps reduce the slope of oil

recovery decline and helps extend the production for a longer period of time. It serves as wettability of the reservoir rock, oil wet can, be altered to become water wet, which releases the oil from the rock (Pope, 2008). Foam for mobility control: in situations where polymer usage is not suitable, foam can also be used as a mobility control agent to replace polymer in chemical EOR projects. This would typically be considered in low permeability reservoirs or in offshore areas where polymers are considered to be environmentally unfriendly.

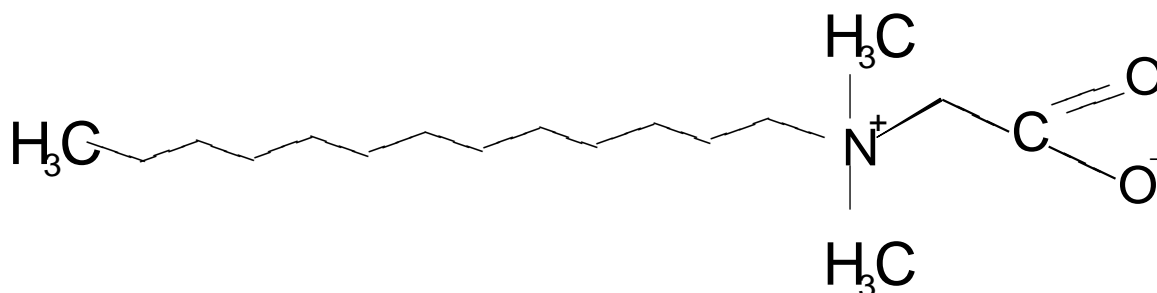
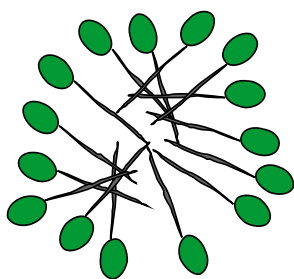


Figure 3: Alkyl betaine (Muthuprassanna *et al.*, 2009).

### Micelles

Surfactants have low solubility in water. A typical surfactant has a large lipophile(hydrophobe) which restricts its aqueous solubility. For example, sodium dodecylbenzenesulphonate has a solubility maximum of 0.04.mol I-I. Beyond this concentration the molecules associate to form colloidal aggregates known as micelles. This concentration is the critical micelles concentration (CMC). Different surfactants have different CMC values. In a micelle the surfactant orients itself with its lipophiles towards the interior, thus presenting a hydrophilic surface to the water. The simplest micelles are spheres but as surfactant concentration increases the micelles grow and form rods. At high surfactant concentrations the rods form larger structures such as hexagonally packed rods and palisade arrangements. As these structures increase in size they take on a greater degree of order until, for the biggest structures, they occur as liquid crystals. These structural changes are reflected in the viscosity the surfactant 'solution' (Royal society, 2014).

### Micelle structure



### Micelle structure

Non polar molecular in its interior

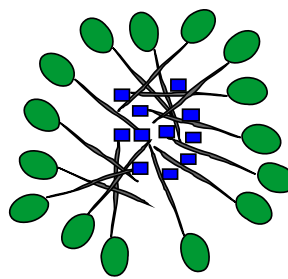


Figure 4: Micelle structure (Keddington, 1999).

## II. Materials and Methods

### Apparatus/instruments

Heating mantle, desiccators, glass funnel, Soxhlet extractor apparatus, round bottom flasks, burette (100 cm<sup>3</sup>), beakers (100 cm<sup>3</sup>), stop watch, glass syringe (100 ml), graduated cylinder (25 ml), pestle and mortar, sieve, test tube, oven, precision weighing balance, Fourier Transform Infrared Spectrometer (FTIR), with an attenuated total reflectance (ATR) attachment, measuring cylinder (10 cm<sup>3</sup>), distillation apparatus, pH meter, leaves of *Cissuspopulnea* plant and general cleaning agents (Good Mama).

### Reagents

Acetone, distilled water, Methanol, acetic acid, Good Mama and paraffin oil.

### Collection of Plant Materials

*Cissuspopulnea* plants obtained from Gurhngwal town of Askira/Uba Local Government Area of Borno State, North East Nigeria was used in this study. Fresh leaves of matured plants of *Cissuspopulnea* grown in

Gurhengwal town of Askira/Uba were collected. The general available cleansing agents, synthetic/commercial detergents (a detergent manufactured by lever brothers Nigeria with trade name GOOD MAMA) were purchased from the market in Maiduguri Borno State.

**Plant Sampling and Pre-treatment**

The random sampling method used by (Ponaruselvam, *et al.* 2012), Nagati (2012) and (Ninfaa, *et al.*, 2014) was adopted. The fresh plant leaves samples were collected and the voucher specimen was numbered and kept in Chemistry Research Laboratory of the University of Maiduguri, Borno state for further reference. The plant sample collected was freed from twigs and extraneous matter by sifting the Soil, grit, sand and dirt was removed. The sample was rapidly and thoroughly washed under tap water, rinsed with distilled water and then air dried at room temperature for 15 days.

**Preparation of Sample**

The method of Agu (2010) was adopted; the sample (leave) were crushed to powder form using iron pestle and mortar. The Powdered samples were sieved by using sieve to obtain powdered samples to be used as raw surfactant.

**Extraction of Crude Saponins-base from Cissuspopulnea Leave.**

The procedure outlined by Agu, (2010) which described the extraction of saponins by the gravimetric method of AOAC (1984) was followed in this work, using Soxhlet extractor and two different organic solvents. 5g of the sample dried grounded powder was weighed into a thimble and transferred into a Soxhlet extractor chamber fitted with a condenser and a round bottom flask containing 200 cm<sup>3</sup> of acetone. Heat was applied using a heating mantle and exhaustively extract lipid and interfering pigments for 3 hrs and thereafter, distilled off the solvent. The defatted sample was further transferred into another Soxhlet extractor fitted to both a condenser and a dried weighed round bottom flask containing 200 cm<sup>3</sup> of methanol with applied heat using a heating mantle and exhaustively extract for another 3 h. The methanol was recovered by distillation at the end of the extraction.

The flask with its extracts was transferred to the oven when evaporation to dryness occurred before cooling in desiccators. The flask with its contents was reweighed and the percentage of saponin-based surfactants was calculated as shown below:

$$\% \text{ of saponin} = \frac{\text{weight of saponin}}{\text{weight of sample}} \times 100$$

**Determination of the presence of saponins in crude and extracts of the sample (leaves)**

The method described by Odebiyi and Sofowora (1978) and Abulude, *et al.* (2009) was used to test for the presence of saponins in the test samples. 5 g of each of the crude surfactants and the saponin extracts was shaken vigorously with 10 cm<sup>3</sup> of water in the test tube, frothing which persisted was taken as an evidence for the presence of saponins.

**III. Result And Discussion**

**Tests for Saponins**

Odebiyi and Sofowora (1978); Abulude *et al.*, (2009) method were used to test the presence of saponin in crude surfactants and extracted saponin of leaves of *Cissuspopulnea*. The entire sample shows abundant and persistent foam on addition of distil water to the samples with vigorous shaking. The foam formed by leaves appear to have more bubbles, confirming the presence of saponin with the clear formation of foam at different concentrations of leave. The capacity to provide lather is one of the first measures of performance of surfactant and based on Kime *et al.*, (2016) reports that, most widely appreciated properties of surface active substance when in aqueous solution is their ability to promote the formation of foam and persistence bubble.

**Table 1: FT – IR Spectra of Crude and Extracted Saponin from Leaves**

Functional group	Bond	Wave number range (cm <sup>-1</sup> )	Mode of vibration	Intensity
Alcohol	O – H	2255.43	Stretching H – Bonded	Broad peak
Phenol	R – OH	3840.19		
Alkanes	C – H	2364.04 2922.49	Stretching	Distinct peak
Aliphatic amino	C – N	1020.48 1272.58	Stretching	Strong peak
Alkanes	C – H	1369.67	Bending	Medium

Amines	N – H	1449.10 1507.70 1594.21 1613.68	Bending	Medium
Esther, saturated Aliphatic	C=O	1702.97 1732.14	Stretching	Strong peak

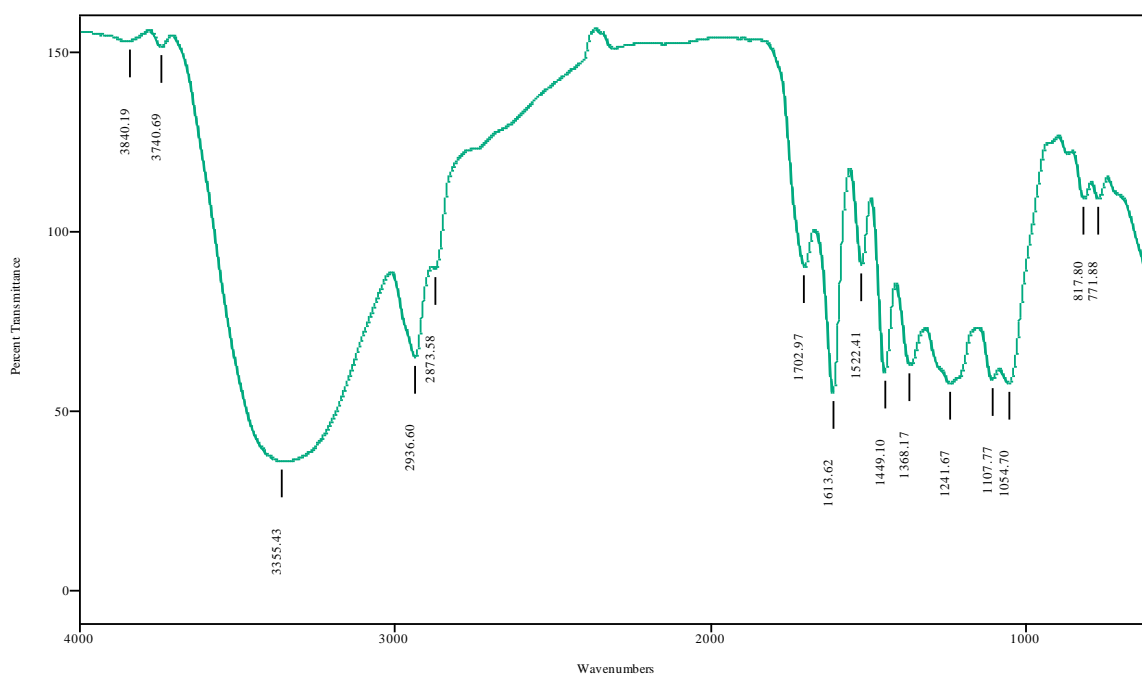
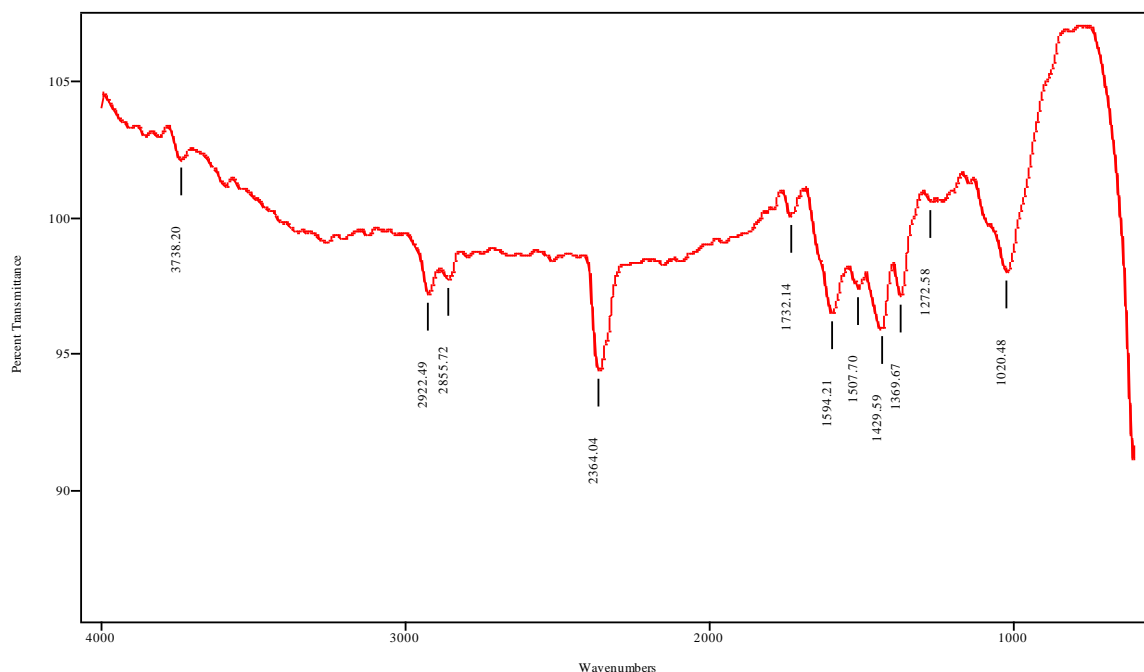



Figure 5: FT-IR Spectra of Extracted Saponin of Leaves

Manual Peak Pick Results

ample Filename	Leaf.spc
File Title	02/17/2020 9:41:33
Date	Thu Feb. 17 9:44:16 2020



**Figure 6:** FT-IR Spectra of Crude Surfactant of *Cissuspopulnea* Leaves.

**Table 2.** Leaf.spc: 02/17/20209:41:33 : FT-IR Spectra of Crude Surfactant of Leaves.

Filename	Peak At	Peak Height
Leaf.spc [0]	1020.48	98.00
Leaf.spc [0]	1272.58	100.59
Leaf.spc [0]	1369.67	97.16
Leaf.spc [0]	1429.59	95.94
Leaf.spc [0]	1507.70	97.40
Leaf.spc [0]	1594.21	96.48
Leaf.spc [0]	1732.14	100.07
Leaf.spc [0]	2364.04	94.43
Leaf.spc [0]	2855.72	97.76
Leaf.spc [0]	2922.49	97.21
Leaf.spc [0]	3738.20	102.15

**Table 3.** Determination of Surface Tension of Natural Saponin Extracted from Leaves

Mass of saponin extracted from leave in 50ml distil water	No. of drops of sample	Time of drops (Sec)	Volume of sample (cm <sup>3</sup> )	Weight solution.	Density of sample	Surface tension (N/m)
0.01	32	0:00:54	2	1.33	0.67	75.09
0.03	33	0:00:46	2	1.35	0.68	69.28
0.06	35	0:05:55	2	1.37	0.69	66.28
0.10	38	0:00:49	2	1.40	0.7	61.93
0.30	43	0:06:53	2	1.44	0.72	56.30
0.60	46	0:01:03	2	1.48	0.74	54.09
0.80	48	0:00:14	2	1.50	0.75	52.53

**Table 4:** Sorption Behaviour of Extracted Saponin from Leaves at Different Concentrations in Grams

Mass of extracted saponin from leaves in 50ml distil water	No of drops of sample solution	Times of drops (Sec)	Volume of Sample solution (cm <sup>3</sup> )	Weight of solution	Destiny of sample	Surface tension (N/m)
1	35	0:01:38	2	1.37	0.69	66.2
2	38	0:01:59	2	1.40	0.70	61.9
3	43	0:02:28	2	1.44	0.72	56.3
4	47	0:2:43	2	1.46	1.73	52.2
5	49	0:03:12	2	1.48	1.74	50.8

#### IV. Conclusion

This study has showed that *Cissuspopulnea* plants can serve as bio-base surfactant and saponin as its surface active agents. It is however satisfactory to observe that all saponin extracted from *Cissuspopulnea* exhibit similar surfactant properties compared to *Balaniteaegyptica* plant surfactant. Its good emulsion capacity



and stability implied the usefulness of the plant as natural surfactants. It is also as a better emulsion as a result of its high stabilization of oil-in-water (o/w) emulsion. *Cissuspopulnea* plant shows higher surface tension and sorption behaviour property as well as foam formation/capacity far better especially leaves and roots than some commercial/synthetic surfactants (OMO) that were used in the study. This saponin- based surfactant is significantly be as an eco-friendly and may show better biodegradability than the commercial surfactants. The natural surfactant from *Cissuspopulnea* plants leave and roots will contribute to efforts in diversify the Nigerians economy, a way to solve the problem of non-biodegradable, high price and non-eco-friendly phenomenon that exists in the production of commercial/synthetic surfactant and add value to green chemistry and sustainable development and exploitation of the cleansing properties of saponin present in *Cissuspopulnea* plants.

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Edmond Moses. I, et. al. "Extraction of Bio-Base Surfactant(SAPONIN) from *Cissuspopulnea* plantleaves from Gurhengwal town, Askira/Uba local government of Borno state Nigeria." *IOSR Journal of Applied Chemistry (IOSR-JAC)*, 13(8), (2020): pp 55-63.