بسم الله الرحمن الرحيم

Sudan University of Science and Technology

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The effect of extraction methods, and deep-frying process on the Physico-chemical Properties of (shea oil) *vitellaria paradoxa ssp.*

تأثير طرق الإستخلاص وعمليات التحمير المستمر على الخصائص الفيزيوكيميائية لزبدة الشيا

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الآيـة

قال تعالي :

(فَتَعَالَى اللَّهُ الْمَلِكُ الْحَقُّ وَلَا تَعْجَلْ بِالْقُرْآنِ مِنْ قَبْلِ أَنْ يُقْضَى إِلَيْكَ وَحْيُهُ وَقُلْ رَبِّ زِدْنِي عِلْمًا).

صدق الله العظيم

سورة طه الآية (114)

Dedication

To My Father

To my mother

T0 my wife

To my children

To my Teachers,

And my all Friends

ACKNOWLEDGEMENTS

First, I would like to thank almighty God who gave me his blessings, good health and support to accomplish this study.

Sincere thanks are given to my Supervisor: **Dr. Mahdi Abass Saad Shakak** for his kind help and support.

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Abstract

The main goals of this research were to study the effect of extraction methods, and deep-frying process on the Physico-chemical Properties of *vitellaria paradoxa* ssp. *nilotica*, beside the chemical composition of shea nuts, the sensory evaluation of potato chips fried in shea butter, groundnut, and their blend oil (1:1) and finally the fatty acid profile of shea butter.

The samples were collected from Alradoum area Southern Darfour State – Sudan. Samples were well prepared and kept in deep freezer for further analysis.

This study was evaluated the effect of both extraction methods (solvent and boiling) on the physical properties of shea butter concerning, their specific gravity, viscosity, refractive index and color intensity. The reported results for solvent extraction method were, (0.9410 at 30˚c (g/cm^3), 1.1(CP), 1.47 and 3.57) were the obtained results for boiling extraction method were, (0.8901 at 30˚c (g/cm^3), 3.6 (CP), 1.48 and 10.37) respectively. These findings indicated that, there was no significant difference ($P \leq 0.05$) between the two methods in their specific gravity and refractive index, where the viscosity and color intensity were completely different ($P \geq 0.05$).

On the other hand, the obtained results for the effect of the solvent extraction method on the chemical properties of shea butter that concerning (moisture content, fat content, free fatty, Peroxide, Saponification and iodine value). The results were, (0.05%, 45.80%, 1.50%, 1.07 (meq/Kg), 181.23 (mg KOH/g oil), and 70.63 (I_2/100 g oil) respectively. Beside the stated results for boiling method which were, (0.07%, 28.60%, 1.90%, 3.83 (meq/Kg), 178.07 (mg KOH/g oil), and 65.90 (I_2/100 g oil) respectively. These results showed a significant difference ($P \geq 0.05$) among the two methods except for their moisture content and free fatty acid % which were not indicating any significant difference between them ($P \leq 0.05$) .

More over this study also stated the influence of deep–frying process on the physico-

chemical properties of shea butter, groundnut, and their blend oil (1:1) that subjected to different thermal treatments (first, second and third frying), beside their control samples, the obtained results showed a significant difference between the samples ($P \leq 0.05$).

The obtained results of the chemical analysis of shea nuts for moisture content, fat, protein, ash, fiber and CHO were, (8.00%, 45.00%, 7.07%, 2.47%, 6.47% and 31.07%) respectively.

Also a sensory evaluation was carried out on potato chips fried in groundnut, blended and shea oil to evaluate their color, flavor, texture and overall acceptability. The best ranking score was obtained by groundnut oil followed by blended oil and lastly shea oil. This might be due to the fact that, the groundnut oil was already refined oil (where refining process is usually improving the physical properties of oils), where the Shea butter and their blend oil (1:1), were not refined oils. This confirmed the Sensory analysis conducted by Akingbala *et al*., (2006).

Finally the fatty acid profile of shea butter, showed that, the five major fatty acids, oleic, stearic, linoleic, palmitic, and arachidic, their values in different extraction methods, were all within values reported by Adikini (2002) and Maranz et al., (2004).

بسم الله الرحمن الرحيم

ملخص البحث

الهدف الأساسي من هذا البحث هو دراسة التحليل الكيميائي لبذرة الشيا، وتأثير طرق الاستخلاص وعملية التحمير العميق (المتكرر) على الخصائص الفيزيوكيميائية لزبدة الشيا إلى جانب التقييم الحسي لشرائح البطاطس المحمرة بزبدة الشيا وزيت الفول السوداني والزيت المخلوط منهما بنسبة (1:1). واخيرا محتوى الاحماض الدهنية الاساسية الموجودة بزبد الشيا.

فالنتائج المتحصل عليها من التحليل الكيميائي لبذرة الشيا كانت كالاتى: المحتوى الرطوبة 8.00، الدهن 45.00، البروتين 7.07، الرماد 2.47، الألياف 6.47 والكربوهيدرات 31.07%.

أيضا هذه الدراسة قيِّمت تأثير طرق الاستخلاص على الخصائص الفيزيائية لزبدة الشيا والتي تضمنت الكثافة النوعية، اللزوجة، معامل الانكسار والكثافة الضوئية. النتائج المتحصل عليها من طريقة الاستخلاص بالمذيب العضوي كانت 0.9410عند حرارة 40^{0}م (جرام /سم^3)، 1.1(سنتبويز)، 1.47و 3.57 على التوالى. نما النتائج الفيزيائية المتحصل عليها من طريقة الاستخلاص بالغليان كانت على النحو التالي: الكثافة النوعية 0.8901 عند حرارة 40^{0}م (جرام /سم^3)، اللزوجة 3.6 (سنتبويز)، معامل الانكسار 1.48 والكثافة ال ضوئية 37.10.حيث كانت هنالك فروق معنوية واضحة. اما النتائج الكيميائية المتحصل عليها عن طريقة الاستخلاص بالمذيب والتي تضمنت (محتوى الرطوبة، الدهن، الأحماض الدهنية الحرة، قيمة البيروكسيد، قيمة التصبن وقيمة الأيودين)، كانت نتائجها كالتالي 0.05%، 45.80%، 1.50%، 1.07(مليمكافى/كيلوجرام)، 181.23(مليجرام هيدروكسيد بوتاسيوم/جرام زيت) و70.63يود/ 100جرام زيت) على التوالي. بينما النتائج المتحصل عليها عن طريقة الاستخلاص بالغليان كانت:07.%، 28.60%، 1.90% ،3.83(مليمكافى/كيلوجرام) ,178.07(مليجرام هيدروكسيد بوتاسيوم/جرام زيت) و65.90(يود/ 100جرام زيت) على التوالي. حيث كانت هنالك ايضا فروق معنوية واضحة.

وعلاوة على ذلك فإن هذه الدراسة أيضا هدفت لتحديد الاثر المباشر للتحمير العميق (ثلاث قليات متكررة + عينة الضابطة) على الخواص الفيزيائية المتمثلة في: (الكثافة النوعية ، اللزوجة ، معامل الانكسار و الكثافة الضوئية) لكل من زبدة الشيا ، زيت الفول السوداني و الزيت المخلوط منهما بنسبة (1:1) بالاضافة الى الخواص الكيمائية المتمثلة في: (المحتوى الرطوبى، نسبة الدهن ، الاحماض الدهنية الحرة , رقم البيروكسيد , رقم التصبن و الرقم اليودى) للزيوت الثلاث, لقد اوضحت النتائج ان هنالك فروق معنوية واضحة فيما بينها.

هذا بالاضافة الى الاختبارات الحسية التى اجريت لتقييم شرائح البطاطس المحمرة فى زيت الشيا ، زيت الفول السودانى والزيت الخليط بينهما (1:1) من حيث اللون والطعم والنكهة والقوام والقبول العام. فنال زيت الفول السودانى اعلى

درجات التقييم الحسى تلاه الزيت الخليط ثم اخيرا زيت الشيا.

وختاما تم تقييم زيت زبد الشيا المستخلص بالطريقة التقليدية من حيت محتواه من الاحمض الدهنية الموجودة به ، فثبت انه يحتوى الاحماض الدهنية المهمة (الاوليك ،الاستيارك ، الينوليك ، البا لمتك والارشيدك) وتتوافق النسب المئوية الموجودة به مع النسب التى تم تحديدها سابقا بواسطة - اداكينى(2002) ومارينز(2004).

CHAPTER ONE

INTRODUCTION

Shea tree (*Vitellaria paradoxa* ssp. *nilotica*) is among fruit bearing indigenous tree which is found in, Alradom region - Southern Dar-fur state. Sudan has abundant and unexplored indigenous wild plants with great socio-cultural and commercial potential. In most case, the forest wild fruits are excellent sources of food. Fruits are especially good sources of minerals and vitamins. The nuts from fruits yield oil/butter that provide energy and fat-soluble vitamins such as A, D, E and K (FAO, 2011).

Fruits and nuts, which are rich of vitamins and minerals, are used especially for growing children and child raising mothers. In addition, they are marketable commodities in the local markets often contribute considerable income for household economy (Moore, 2008). Worldwide, natural vegetable oil and fats are increasingly become center of attention in view of their nutritional, cosmetic and pharmaceutical application in chemical industries. Vegetable oils account for 80% of the world's natural oils and fat supply (FAO, 1999). With increasing awareness on importance of vegetable oils in food, pharmaceutical and cosmetic industries, there is a need to increase the amount of oil produced in order to meet the increasing demand. Like Asia and the Americas, the continent of Africa is blessed with a rich tropical flora. Hence, there is good reason to promote the utilization of Africa's fruits that have not been brought up to their potential in terms of quality, production and availability to the international market (NRC, 2008). Utilization of Shea tree will not only reduce the expenditure of the country through import substitution, but also improve the livelihood of the rural community through marketing of (Shea butter) as one of the pragmatic interventions to alleviate the

incidence of poverty problem is use of indigenous fruit trees, which are known to play an important role in food and nutritional supplement, especially during droughts times (Parnwell, 2005).

There for the objectives of this study are to:

1- Determine the chemical composition of shea butter nuts *(vitellaria paradoxa ssp. nilotica*).

2- Study the effect of extraction methods on the physico -chemical properties of shea oil *(vitellaria paradoxa ssp. nilotica*).

3- Estimate the influence of deep-frying process on the physico -chemical properties of shea butter *(vitellaria paradoxa ssp. nilotica*).

CHAPTER TWO
LITERATURE REVIEW

2.1. *Vitellaria paradoxa* plant species

Vitellaria paradoxa also called the shea tree belongs to the family sapotaceae and is one of the most dominant and abundant species found in semi-arid areas of at least 19 Sub Saharan African countries (Hall *et al.,* 1996). This tree grows to a height that ranges from 8 to 23 meters and has many spreading branches in the areas with temperatures range of 24 – 32ºC and rainfall between 800 - 1400 mm per annum (FAO, 2001). There are two sub species of the shea tree. The sub species *Vitellaria paradoxa* is the one growing in West Africa and sub species *Vitellaria nilotica* southern Sudan, Ethiopia and Uganda (Hall *et al.,* 1996). In Uganda, *Vitellaria sub species nilotica* is abundant in the northern parklands covering the areas of West Nile, Acholi, Abim (Acholi jabwor), Lango and Teso (Okullo *et al.,* 2004).

2.1.1. Lulu Tree: Sudan's and other parts of Africa's vital natural resource.

The nilotica variety of Shea nut tree (called lulu in Arabic) grows mainly in a narrow band of savannah extending from Senegal to Ethiopia. Lulu trees grow abundantly on the ironstone plateau and the alluvial plains of Southern Sudan and other parts of Africa. It is a medium sized deciduous tree rarely exceeds 15m, and is estimated to live between 200 to 300 years, with 15 to 20 years of growth required before fruiting. Because the tree's rich bounty of nutritious lulu nuts yields at the exact time of the seasonal hunger, the lulu tree is greatly celebrated in Southern

Africa as a vital natural resource. The economic value of lulu nuts is also extremely high, providing women guardians with income and household food security.

2.1.2 Vitlleria paradoxa distribution

Vitlleria paradoxa is an indigenous fruit tree in the Sudano-Sahelian Africa. There are two subspecies of the tree, one of which subsp. Paradoxa which extends from Senegal eastwards to the Central African Republic whilst the other subsp. nilotica occurs in southern Sudan and Ethiopia, Uganda and northeast Zaire (Hall et al., 1996). As shown in Fig 1. it is found in Sahelian Africa belt, that extended for approximately 5,000 km long and 500 km wide which started from Senegal to Uganda and Ethiopia in areas receiving 600 – 1400 mm of rainfall (Deribe, 2005; Maranz et al., 2003)

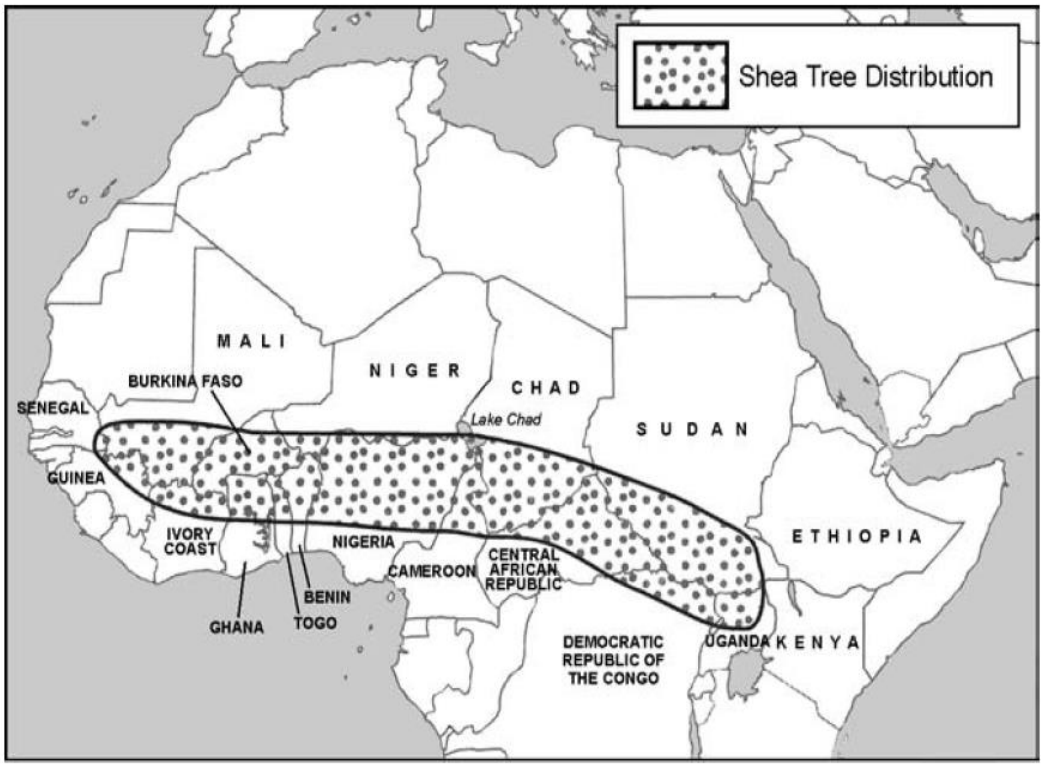


Figure 1: Shea tree distribution in Africa (Elias & Carney, 2004)

2.1.3. Nilotica vs Paradoxa: The Advantage of Purity.

The *vitellaria nilotica* variety of Shea nut varies from the better-known *paradoxa* variety that has been commercially exploited in West Africa for decades. The oil from darker *paradoxa* nuts has to be bleached and deodorized before processing into skin care products, so the resulting butters lack the untouched purity of butters crafted from *nilotica* oil. Since *nilotica* oil has a naturally pure and mild flavor that requires little processing, it has become a highly coveted additive for natural skin and hair care products. Rich in olein, *nilotica* oil from these nuts is superior in cosmetics and is known in Africa and around the world for its positive effects on the digestive system, hair, and skin. (Maranz *et al* 2004).

2.2. *Vitellaria paradoxa* shea tree products.

The shea tree does not only provides shea fruits, kernels and butter but it is also a source of fuel (charcoal), shade, medicine, traditional apiculture for placing hives and traditional cultural ceremonies (Maranz *et al* 2004). Other parts of the tree such as sap, leaves and roots have industrial potential application (USAID, 2007). Although this tree is very difficult to domesticate, it's being threatened by the charcoal industry in South Sudan and Uganda (Master and Puga, 1994, Okullot *et al.,* 2004).

2.2.1. Shea fruits:

Fruiting of the tree commences only after 15-20 years (Karin, 2004) and reaches maximum productivity after 45 years (FAO, 2001). The trees Fruit at the end of the dry season and are harvested during rainy season between the months of May and August (Okullo *et al.,* 2004). The fruit takes 4-6 months to develop and each tree produces 15-20 kg of fruits (Karin, 2004).

The ripening improves the taste to the sweet pear taste and the colour is greenish-yellowish with ellipsoidal shape (10–15 cm) or spherical berry with two to three grains per fruit. According to Maranz *et al.*, (2004), FAO, (2007) and Kapseu, *et al.*, (2007). the pulp of the fruit is edible with sweet and spice able flavor, play an important role in the local diet, can also be sold in the local markets and can be used as a source of food for other animals such as elephants, sheep, pigs, bats and birds.

2.2.2. Shea nuts:

Each fruit contains a kernel with oval or round hard red brown or dark brown seed referred to as a "shea nut". The fresh nut size and shape of particular trees are distinctive (Boffa *et al.,* 1990) and contains 41% water, 18% residue, 21% oil and 20% husk (USAID, 2004). The shiny, smooth and fragile shea nut shell is always processed into the "shea kernel" whereby each tree can yield 3-6 kg (Leakey, 1999; FAO, 2007). The shea kernels are the main source of shea butter, sold for income and used as medicine (USAID, 2004; FAO, 2007).

2.2.3 Shea oil:

After shelling, the shea kernels are processed into shea oil or butter which is tallow like substances that solidifies at room temperature to form a yellow, cream or grey colour (Adikini, 2002). The shea oil is used as food sauce, cosmetic, soap making and traditional medicine and is also traded in the international markets as a valuable raw material in the cosmetic, chocolate and pharmaceutical industry (USAID, 2004). It has been reported by many authors including Tallantire & Goode (1975); FAO (1998); Puganosa and Amuah (1991) that shea oil comprises fatty acids and vitamins which are essential in the human diet. As a result, Africans have mostly depended on it as their substitute for the valuable dairy butter and natural source of vitamins.

2.3. Uses of *Vitellaria* species.

The kernel of the seed (often incorrectly called ‘nut’) contains a vegetable fat known as shea butter. High quality shea butter is consumed throughout West and east Africa as a cooking fat. Refined fat has been marketed as margarine and baking fat. It is also used for pastries and confectionery because it makes the dough pliable. It is a substitute for cocoa butter, which has similar properties. Many cosmetic products, because its high unsaponifiable matter content imparts excellent moisturizing characteristics. Where Low-quality shea butter, often mixed with other oils, is a base material for soap. It is also very suitable for making candles because of its high melting point. Shea butter is a suitable base for topical medicines. Its application relieves rheumatic and joint pains and heals wounds, swellings, dermatitis, bruises and other skin problems. It is used traditionally to relieve inflammation of the nostrils. Shea butter is given externally and internally to horses to treat sores and galls. As a water proofing agent, shea butter is used as daubing for earthen walls, doors and windows. The black sticky residue, left after oil extraction, is used to fill cracks in walls and also as water proofing materials. Waste water from shea butter production has pesticidal properties and has been mixed with stored cowpea seeds in Burkina Faso to protect them from being eaten by the weevil *Callosobruchus maculatus*. The press cake is unsuitable as livestock feed because it contains anti-nutritional compounds. However, detoxified meal can be given as feed in low proportions. In Europe the cake is utilized as a non-nutritional bulk for compound cakes. The press cake and the husks are also potential fertilizer and fuel. The flowers and fruits are important foods. The flowers are sometimes made into fritters. In spite of their slightly laxative properties, mature fresh fruits are commonly eaten in savanna regions as they ripen during the land preparation and planting season.

The sweet pulp of fallen ripe fruits can also be fed to livestock. The leaves are used to treat stomach-ache. They are also added to vapour baths to treat headache and as an eye-bath. Leaves soaked in water produce a good lather for washing. Ground roots and bark are used to treat diarrhoea, jaundice and stomach-ache. Roots are used as veterinary medicines for horses. Bark infusions have medicinal and antimicrobial properties, e.g. against dysentery. They are applied as eyewash to counteract spitting-cobra venom. A bark decoction has been used in baths to facilitate child birth and stimulate lactation among feeding mothers. The reddish latex (gutta shea or red kano rubber) which exudes from deep cuts in the bark is made into glue, chewing gum and balls for children's games. Musicians use it to repair drums. Only unproductive and unhealthy trees are cut for timber. The wood is used for poles, house posts, rafters, flooring, domestic utensils, and furniture. It is an excellent fuel wood, burning with great heat, and a source of charcoal. Shea butter tree is an important source of honey. Bee hives placed in its branches are assured a good supply of nectar and pollen. The widely collected edible and protein- rich caterpillar of *Cirina butyrospermi* feeds solely on its leaves. The tree is considered sacred by many tribes. The oil is placed in ritual shrines and used for anointing. In some areas, leaves are hung in doorways to protect new born babies, and are also used in making masks. (Maranz *et al* 2004).

2.4. Other properties of shea butter

Shea butter from fresh seeds is white, odourless and of high quality, while that from stale seeds is dark, and tastes bitter. The approximate chemical composition of the kernel per 100 g dry matter is: fat 31–62 g, protein 7–9 g, carbohydrate 31–38 g, unsaponifiable matter 2.5–12 g.

The fatty acid composition of shea butter is approximately: lauric acid trace, myristic acid trace, palmitic acid 4–8%, stearic acid 31–45%, oleic acid 43–56%, linoleic acid 4–8%, linolenic acid trace and arachidic acid 1–2%. The chemical properties of shea butter vary across its distribution range, Burkina Faso and Uganda representing the two extremes. The highest oleic acid content was found in Uganda (57%), the lowest in Burkina Faso (45%), while shea butter from the Mossi plateau in Burkina Faso has the highest proportion of stearic acid (45%) and that from Uganda the lowest (31%). Shea butter is a useful cocoa butter substitute because it has a similar melting point (32–45°C) and high amounts of di-stearin (30%) and some stearo-palmitine (6.5%) which make it blend with cocoa butter without altering flow properties. The high proportion of unsaponifiable matter, consisting of 60–70% triterpene alcohols, gives shea butter creams good penetrative properties that are particularly useful in cosmetics. Allantoin, another unsaponifiable compound, is responsible for the anti-inflammatory and healing effect on the skin. It is used in toothpastes and other oral hygiene products, in shampoos, lipsticks, cosmetic lotions and creams, and other cosmetic and Pharmaceutical products. Clinical tests with patients suffering from rhinitis, and having moderate to severe nasal congestion, showed that shea butter may relieve nasal congestion better than conventional nasal drops. The seed cake is a potential source of feed for livestock. Per 100 g dry matter it contains: protein 8–25 g, fat 2–20 g, carbohydrate 48–67.5 g, fiber 5–12 g. However, it has low digestibility and toxic properties attributed to saponins or tannins. Mouldy seeds contain relatively low quantities of aflatoxin, while commercial samples have a maximum of 20 μg aflatoxin B1 per kg.

The fruit pulp contains per 100 g: glucose 1–2 g, fructose 1–2 g, sucrose 1–2 g, ascorbic acid 200 mg, Ca 36 mg, Mg 26 mg, Fe 2 mg, and trace amounts of Zn, Mn and Cu. Sweetness of the pulp is the main quality criterion.

The wood of *Vitellaria paradoxa* is moderately heavy (density about 720 kg/m³ at 12% moisture content) and hard. It is liable to crack on drying and needs to be seasoned slowly. It is difficult to work and tends to split on sawing, but it polishes well. It glues, nails and screws well, but pre-boring is recommended to avoid splitting. It is durable and resistant to termites. Both sap wood and heart wood are resistant to impregnation with preservative. (PROTA, 2007) (Plant resources of tropical Africa).

2.4.1. Harvesting

Fruits are gathered in the rainy season, usually in June–August depending on latitude. Harvesting continues for about 2.5 months and is done mostly by women and children. Fallen fruits are collected from the ground because it is difficult to distinguish between ripening and fully mature fruit. Harvesting rights depend on tenure. A woman collects 20–45 kg of fruits per day, depending on ethnic group, proximity of trees to the village, and distance between trees. Fruits are brought back to the village in head-loads of about 25 kg. (PROTA, 2007) (Plant resources of tropical Africa).

2.4.2. Yield

Productivity of shea butter trees is variable. In a sample taken in Burkina Faso, the best 25% of the trees produced 60% of the yield, while the poorest 30% of trees produced little fruit. A good tree can bear on average 15–30 kg fruits per year. In a good year this may be as much as 50 kg, but then only about 15 kg in the next two years.

Although a clear production cycle is not confirmed, observations show a tendency for *Vitellaria paradoxa* to give only 1 good harvest per 3–4 years. (PORTA, 2007)

2.4.3. Handling after harvest

In rural areas, the fruit pulp is first removed for food, or by fermentation or boiling. Seeds are traditionally processed by hot water for oil extraction; usually this job is done by women. The seeds are then boiled and later sun- or kiln-dried. Sun-drying may take 5–10 days. Seeds are cracked using mortar and pestle, or stones; the kernels are removed by trampling and re-dried before being crushed, ground and needed to form a paste; the paste is put in water, heated or boiled and the boiling mass is churned until a grey, oily fat separates from the emulsion. The fat is skimmed off from the surface and washed to remove impurities. The congealed fat may subsequently undergo further refining before being moulded in to various forms. This traditional method of processing is inefficient and labour intensive. According to USAID (2004) and Karin (2004), the traditional preparation or processing of shea oil in East Africa differs from West Africa. Mechanization of the various operations, in particular the use of hydraulic or continuous screw expellers or application of solvent extraction, will improve oil extraction efficiency considerably. Pretreatment of the kernel paste with enzymes (e.g. proteases and cellulases) may also result in higher extraction rates.

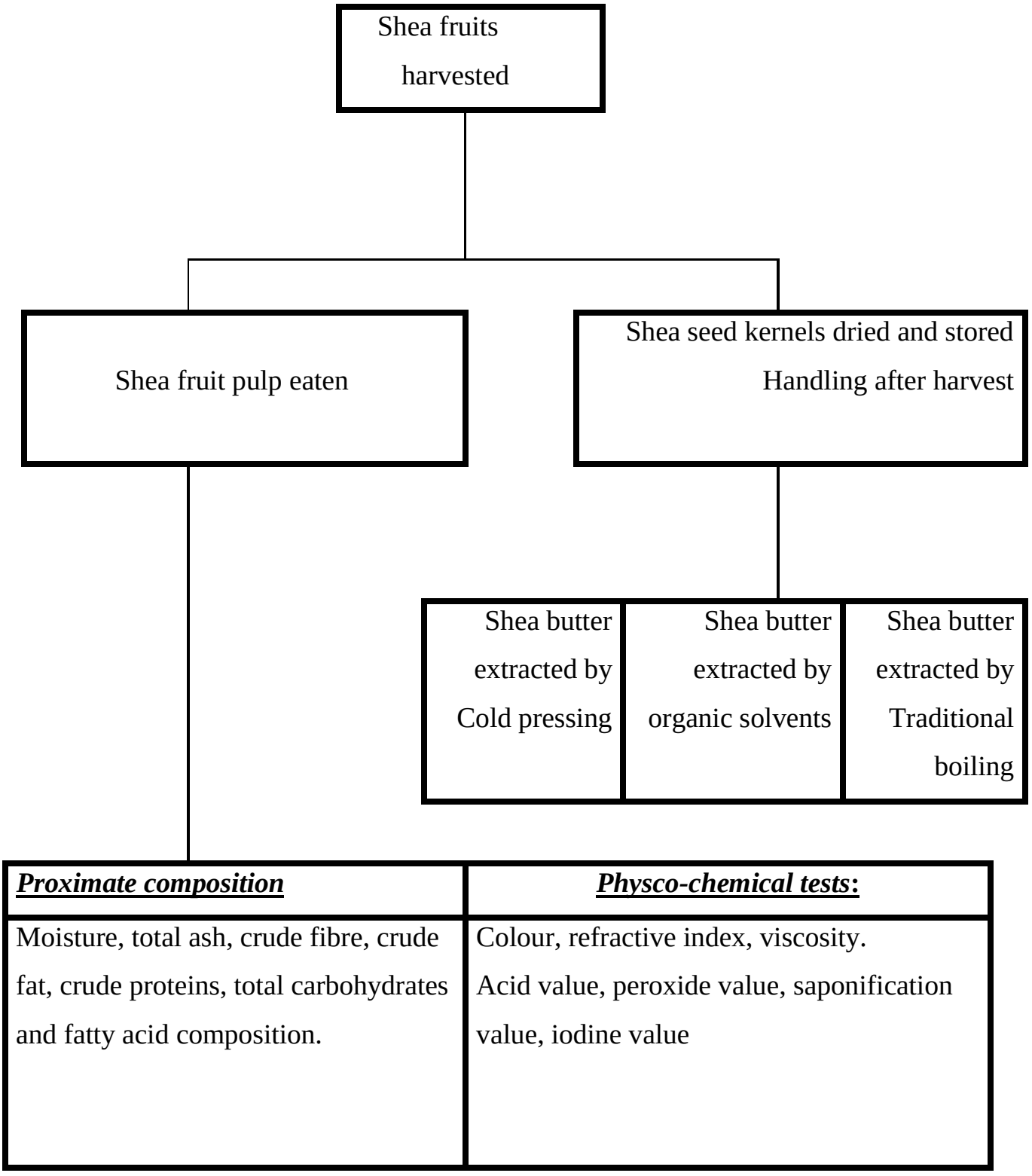


Figure 2: Theoretical frame work for the study

2.5. Prospects

Shea butter tree is of great economic importance in Sudan savanna zones. It grows over a wide area, regenerates well, it is traditionally managed and protected by farmers. However, natural regeneration and sustainability of seed production are threatened by Agricultural intensification in the area. Progress made on grafting techniques suggest that selected vegetative material with specific fruit or butter qualities can be multiplied for small scale clonal plantations to meet market demand for high quality fruit or butter production. *Vitellaria paradoxa* has a niche in the international markets as a cocoa butter substitute in the food, cosmetic and pharmaceutical industries. Recent studies on the variation in fat composition across the species distribution range indicate that the soft shea butter from Sudan and Uganda is preferred for cosmetic purposes, while shea butter with higher stearic acid content as found in Burkina Faso is more suitable for the chocolate industry. Shea butter is increasingly popular as an ingredient in cosmetics and soaps, especially in European countries and the United States. Now that the European Union allows the use of 5% cocoa butter substitutes in chocolate, chocolate and confectionery products account for 95% of the shea butter demand, with only 5 percent currently used for cosmetic and pharmaceutical products. It is likely that the overall dem in better knowledge of its various properties (PROTA, 2007) (Plant resources of tropical Africa).

2.6. Post-harvest handling practices of shea butter.

2.6.1. Shea fruit harvesting.

Once fruits are ripe, they fall down by themselves beneath the mother tree and it is left to become over ripe (Karin, 2004). During harvesting, the shea fruits are mainly collected from the ground by village women and children who move long distances from home to pick and gather them under the trees.

The children and women eat the pulp and remove the seed kernel. The shea fruits can be collected from as near as the home stead and as far as 10km or so. The collected fruits can then be transported in lots of 20kg to the village where processing takes place

(Karin, 2004). In addition to eating, the pulp can also be removed by scraping, boiling, sun drying, fermentation and boiling (FAO, 2007). The removed pulp can be dried and also processed into shea cakes (Master and Puga, 1994).

2.6.2 Shea nut drying

Shea nuts are usually sun dried for 1-2 weeks and dehusked to obtain the shea kernel which is further sun dried for another two weeks. Although the shea kernels can sometimes be baked (roasted) to concentrate the oil in the kernel and lengthen the storage period, this has been discouraged because it is a limiting factor to quality of shea butter. Methods of solar drying on polythene sheeting have been developed in some African countries, but they have limited durability (FAO and CFC, 2004). According to USAID (2004), the shea kernels can be stored for several years without spoilage by maintaining its moisture content between 6% and 7%. This is so because the drying process inactivates enzymes responsible for the build-up of fatty acids in the seed kernel (USAID, 2004).

2.6.3 Shea kernel storage

Shea kernels are stored in sacks, woven baskets and plastic buckets that are stored either in house, granary or kitchen floors. Sometimes the kernels are hanged in houses or kitchens instead of floors. In West Africa Jute bags from cocoa industry are widely applicable. In recent decades, polythene bags or sacks have come into wide use for storage of shea kernels. However, this has been reported to stimulate fungal growth important for quality because they do not allow air circulation (FAO and CFC, 2004).

Moreover, because of the recalcitrant properties of shea nuts, it storage is very difficult (Karin, 2004).

2.7. Shea butter extraction and processing

A report by USAID (2004) indicates that technologies that have been used for extracting vegetable oil including shea butter are traditional boiling, mechanical pressing and solvent extraction. However, for centuries, shea butter has been processed by indigenous traditional boiling method which has been described as labour intensive (Masters and Puga, 1994). This has made the quality of indigenous traditionally extracted shea butter variable (FAO and CFC, 2004). Due to this, improved methods such as mechanical pressing (both manual an (Hydraulic) are being adopted in many African countries. Even then conventional motorized oil expellers are not recommended for shea butter extraction due to high latex content of the shea kernel apart from solvent extraction method which is mainly used for small laboratory extractions (FAO and CFC, 2004).

2.7.1. Traditional boiling extraction

This method involves roasting of the kernels with sand and ash before crushing in a local wooden mortar and there after milled on grinding stone. The paste is boiled in water until the fat begins to float on surface. After extraction, the butter or oil is transferred into storage plastic or glass containers. According to USAID (2004) and Karin (2004), the traditional preparation or processing of shea oil in East Africa differs from West Africa.

2.7.2. Mechanical pressing extraction

As has been reported by FAO and CFC (2004), this processing technique mainly involves the crushing of the shea kernel into coarse particle sizes. The paste is soaked in little hot water before pressing to release the shea butter. The method was developed to improve the efficiency of production of shea butter since

traditional boiling method was found to be labour intensive and wasteful (Masters and Puga, 1994).

2.7.3. Solvent extraction

Solvent system is mainly used in laboratory experiment although it is sometimes used for commercial extraction in developed countries. According to FAO and CFC (2004), this method is not usually used in domestic and commercial shea butter extraction due to the high costs involved, environmental problems and lack of technical skills in developing countries. Post shea butter extraction process by traditional and mechanical pressing methods.

Following extraction by traditional boiling and mechanical pressing methods, shea butter is clarified by wet boiling with water for about 20 minutes at 2:1 ratio of oil to water, cooled and decanted into dry vessel where it is boiled again to remove any water residues (FAO and CFC, 2004).

2.8 Chemical Composition and Nutritional value of shea fruits and shea butter.

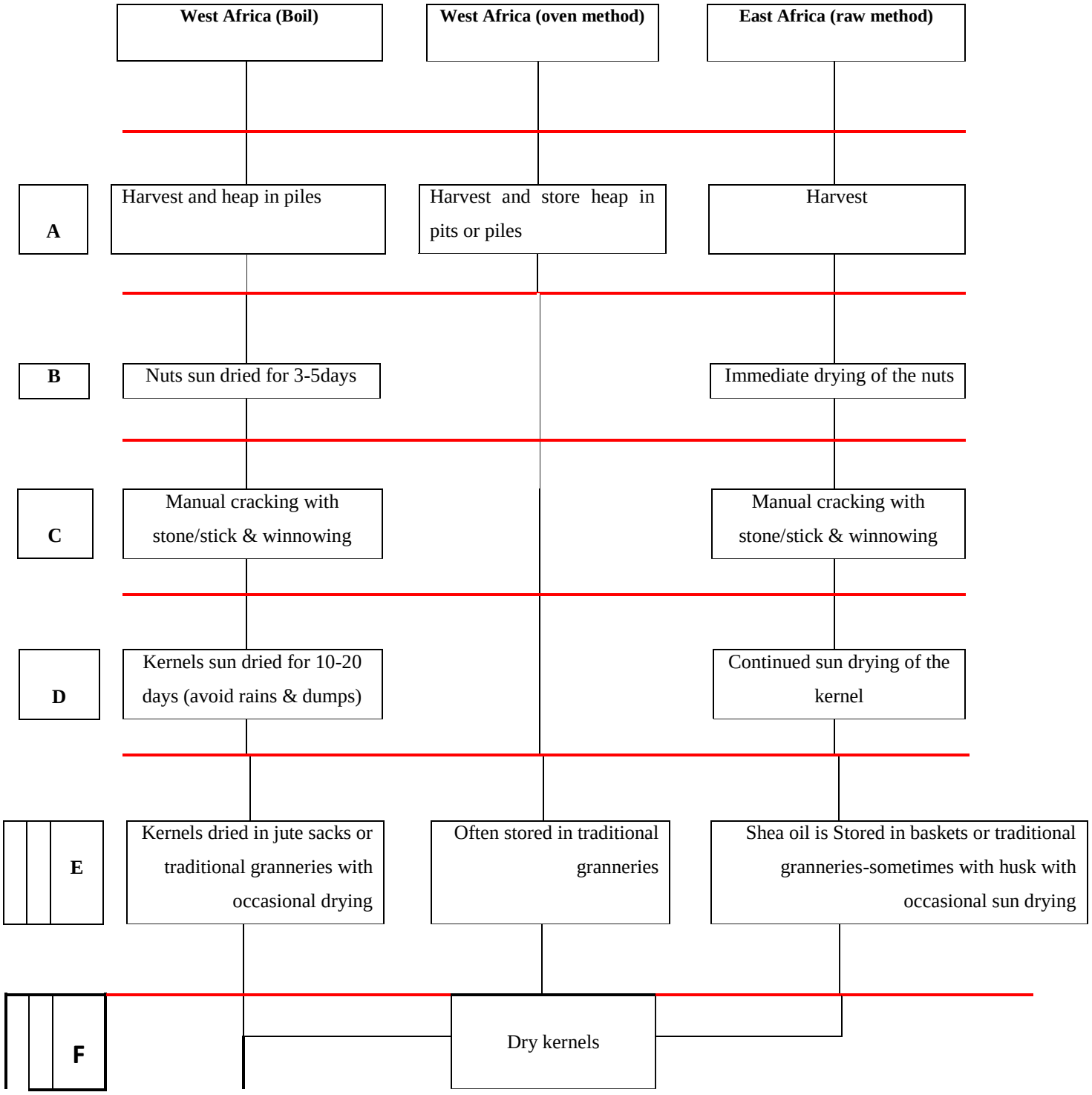
2.8.1 Proximate composition of shea fruit pulp.

Although fruits are generally poor sources of proteins and oil, they contain reasonable amounts of carbohydrates, fiber, minerals and vitamins C (Pearson, 1991). According to (CRIG, 2002). The shea fruit is a seasonal source of calories in the sub Saharan Africa with edible pulp containing 41.2g of carbohydrates, 0.7-1.3% proteins and 196mg/100g vitamin C (CRIG, 2002). A report by Cheman *et al.,* (1999) indicates that while carbohydrates are good sources of energy, proteins can catalyze, regulate, protect and provide energy. Vitamin C on the other hand is essential for normal growth and development of human body.

2.8.2 Mineral composition of shea fruit pulp.

Minerals can be classified as either macro or micro. The macro minerals consist of calcium (Ca), sodium (Na), potassium (K) and magnesium (Mg) while micro mineral is iron (Fe), zinc (Zn), selenium (Se), copper (Cu) and many others. In Ghana, the shea fruit pulp has been reported to contain Ca (36mg/100g), Mg (26mg/100g) and iron (1-3g/100g) and others in small quantities (CRIG, 2002, FAO, 2007). According to Maranz *et al.*, (2004) the most abundant mineral in shea fruit is K with values as high as 1300mg/100g in West African countries and 400mg/100g in Uganda. Even other minerals were reported by Maranz *et al.*, (2004) to be higher than those reported by CRIG (2002) and FAO (2007). These mineral compositions across Africa were found to vary by environmental conditions.

The above finding shows that shea fruits are very important in nutrition because of multiple functions of minerals in the human body. According to Hegarty (1988), Na and K are very important in water balance, normal functioning of the nerves and muscles, absorption of glucose and glycogen while Ca plays a very important function in bones, nervous system, stimulates some hormonal secretions, and activates some enzymes and blood coagulation. Magnesium on the other hand can assist enzymes involved in the synthesis and breakdown of carbohydrates, fats, proteins and synthesis of DNA and RNA while Fe is a constituent of hemoglobin, myoglobin and a number of many enzymes.



A- Accumulate, **B-** Heating, **C**-Sun drying, **D**- dehusking. **E**- Final drying**. F**- Storage.

Figure 3: Traditional methods of shea kernel processing diagram (USAID, 2004).

2.8.3 Physico-chemical composition of shea oil

The physico-chemical composition of shea oil can be broken into physical parameters and chemical parameters.

2.8.4 Physical parameters

According to Lewis (1990), colour, refractive index and viscosity play an important role in determining the quality of any oil because they give physical specification for description of the oil. While the colour of oil comes from natural colouring matters from α- and β-carotene, xanthophyll and chlorophyll, the refractive index is ratio of light in vacuum to speed of light in the oil under examination and the viscosity is material friction acting within the oil. These are quick parameter for characterization of oils and assessment of the level of purity. Shea butter has been reported to have a pale yellow, cream or grey colour solid at room temperature and refractive index of 1.467 (Adikini, 2002). However, a report by Stryer (1988) indicates that variation in the physical parameters of vegetable oils might be associated with changes in the un-saturated fatty acids due to oxidation, polymerization and isomerisation.

2.8.5 Chemical parameters

Acid value, peroxide value, iodine value and saponification value are also important in assessing the quality of natural oils (Choe and Min, 2006). While acid value is a measure of acid hydrolysis that has occurred in the oil or fat, peroxide value is used to quantify the primary oxidative products in the oil due to substantial amount of unsaturated fatty acids (Pearson, 1991). The saponification value on the other hand is weight in milligrams of potassium hydroxide required to hydrolyze one gram of fat or oil while iodine value is an indication of unsaturation in the natural oils. According to Uganda National Bureau of Standards (UNBS ,2004).

Draft standard for shea butter, the shea butter acid valueis less than 6.0 mg/KOH/kg, peroxide value is less than 10 mEq/kg, saponification value is between 170-190 mgKOH/g and iodine value, 40I2/100g. Although Adikini (2002) reported values within the range of UNBS (2004) for shea butter samples from Uganda, higher values were reported by Kapseu *et al.*, 2007 and Tano-debrah and Yoshiyki (1994) for West African shea butter samples. These variations in chemical parameters were attributed to processing, fruit harvesting and kernel storage methods (USAID, 2004).

According to Maranz and Weisman (2004), tocopherols are also chemical parameters that are important in nutrition and cosmetic properties of oils because they act as good anti-oxidants. Their presence also causes oxidative stability of the oil. Although shea butter has been reported to have over 800mg/100g of α-tocopherol in West African shea butter, the shea butter from Uganda has been 29mg/100gm, the lowest in Africa (Maranz and Weisman, 2004). This variation in the α-tocopherol values was associated with environmental conditions. It should be noted, however, that low level of tocopherols can result in a serious decrease in the protective power of any oil against auto-oxidation (Choe and Min, 2006).

2.9 Fatty acid profile of shea oil

The five principal fatty acids in shea butter oil (Table 1) are palmitic acid (C16), stearic acid (C18), oleic acid (C18: 1) linoleic acid (C18: 2) and arachidic acid (C20: 0) (Table 1). The shea oil fatty acid composition is dominated by stearic acid and oleic acid, which together account for 85-90% of the fatty acids (Maranz *et al*. 2004). Adikini (2002) also found that shea butter was dominated with oleic and stearic acids with values of 57% and 30% respectively. The other fatty acids in shea oil include linoleic (4.3-6.3%), palmitic (3.0-4.4%) cis-vaccenic (0.5-.0.8%) and gadoleic (0.2-.3%). The relative proportion of these oleic and stearic fatty \

(Table 1): **Reported shea butter fatty acid composition**

Parameter / ***Fatty acid*** **(%)**	**Sources**		
	FAO (2007)	**PROKARITE (2007)**	**Leakey (1999)**
Palmitic acid	5-9	4.7	4.0
Stearic acid	30-41	31.2	46.0
Oleic acid	49-50	56.5	41.0
Linoleic acid	4-5	5.8	7.0
Linolenic acid	_	_	1.0
Arachidic acid	_	1.0	_

acids are responsible for the differences in shea butter consistency. According to Maranz *et al* (2004), Uganda shea butter have high oleic acid content (50-60%) producing the most liquid shea butter on the African continent. Although a small amount of linoleic acid is in shea butter, it is critical to the stability and flavor of oil.

2.9.1 Variation of physico-chemical characterics of shea butter

Since there is large dietary consumption of shea butter, variation in physico-chemical parameters becomes a major concern for nutrition and public health experts (Stryer, 1988). A number of steps in post handling processes such as harvesting, drying, and storage and oil extraction are responsible in determining the physico-chemical characteristics of shea butter (USAID, 2004). According to FAO and CFC (2005), reducing the moisture content of shea kernel to lower than 8% improves the quality of butter. The low moisture maintains the acid value of shea butter within the edible range in addition to reducing fungal infections on the kernels. To ensure that kernels have low acid value, the West African countries have adopted a method of parboiling of the fruits prior to depulping as opposed to East Africa where the kernels are directly sun dried after de-pulping without any boiling (USAID, 2004). In addition to improper drying, poor storage of the kernel can also cause increase in free fatty acid due to moisture uptake resulting in hydrolysis of the fatty acids. A report by USAID (2004) indicates that shea kernels stored in the open or under humid areas can bring changes in the oxidative parameters of shea butter. During oil extraction, the exposure of oil with unsaturated fatty acids to oxygen may also increase the deterioration rate due to formation of polycyclic aromatic compounds and polar compounds such as triacylglycerol dimers and triacylglycerols (Kapseu *et al.*, 2007). This may result in release of low molecular volatile compounds such as

Aldehydes, ketones, carboxylic acids, and short chain alkanes and alkenes which are responsible for changes in the physico-chemical characteristics of the oil. According to Boffa, *et al*, (1999) after extraction, exposure of oil to fluorescence light and dark condition during Storage increases peroxide value and free fatty acid. A study done by Kapseu, *et al* (2007) found that acid value of shea nuts stored in a refrigerator increased after three months due to enzyme action leading to high peroxide value. Post-harvest handling practices may also have effect on fatty acids. According to plant resources of tropical Africa (PROTA, 2007), linoleic acid and palmitic acid ratio has `been used as indicators for measurement of the extent of fat deterioration. When oils are exposed to oxygen at high temperatures, they tend to undergo oxidation (Choe and Min, 2006). The fishy flavor of oil formed during heating is due to oxidation of linolenic acid in the oil. Other volatile compounds such as aldehydes, ketones, benzene which produce un-desirable flavour resulting in the development of rancidity may also be released. With high level of un-saturation in the oil chances of oxidation may be high (Ferris *et al.,* 2004). Although tocopherols have been used as natural antioxidants, purification and bleaching during processing of the shea oil can lead to losses of alpha-tocopherol. The degradation rate of tocopherol rapidly increases in the presence of molecular oxygen and free radicals (Player *et al.,* 2006). Even if α-tocopherols degrade, high levels can show exceptional storage stability of the oil or fat (King, 1980).

2.9.2 Shea tree as foood product

[Shea butter extracted from the nuts is one of the most affordable and widely used vegetable fats in the Sahel. Today, shea nuts are important internationally and are sold to European and Japanese food industries.

The refined fat is sold as baking fat, margarine and other fatty spreads under various trade names and finds increasing use in various foodstuffs.

Shea butter has a fatty composition similar to that of cocoa butter, so is often used as a substitute for cocoa and in pastry because it makes highly pliable dough. Traditionally prepared unpurified, shea butter is sold in 'loaves' in markets and, if properly prepared and wrapped in leaves, is resistant to oxidative rancidity and will keep for years if not exposed to air and heat. Nuts that have been cleaned and lightly sun dried without previous maceration yield a tasteless, odourless fat. Traditionally prepared shea butter, after refining, is also tasteless and odourless. The edible fruit pulp constitutes 50-80% of the whole fruit. It is allowed to become slightly overripe before being eaten raw; it can also be eaten lightly cooked. Children of some ethnic groups eat the nuts raw, while the flowers are made into fritters. Caterpillars of Cirina butyrospermii A. Vuilet, which feed exclusively on the leaves of the shea-butter tree, are dried and sold in markets in Nigeria and Senegal. They are rich in protein and sometimes eaten in a sauce.

CHAPTER THREE

Materials and Methods

3.1 Materials

3.1.2 Sample collection

The sample of Shea seeds were collected from alradoum locality - southern Darfur state – Sudan, which an extended region through Bahr algazal state – South Sudan country. The seeds were collected from different trees in one Sub-county area, stored in a plastic container and transported to chemical laboratory for analysis. The methods of butter extraction were documented and samples were transported to chemistry laboratory where they were maintained at 4ºC in the refrigerator for further analysis.

Potato pieces were collected from shambat central vegetables market and also were kept in the refrigerator for further analysis.

3.1.3 Preparation of Shea raw materials

The seeds were sorted, washed with cold tap water, dried and crushed in an electric grinder into course powder.

3.2 Methods

3.2.1 Traditional extraction method

the technologies that have been used for extracting vegetable oil including shea butter are traditional boiling as report by USAID (2004) However, shea butter has been processed by indigenous traditional boiling method was described as labour intensive (Masters and Puga, 1994). This which made the quality of indigenous traditionally extracted shea butter variable (FAO & CFC, 2005).

Procedur:

This involves roasting of the kernels with sand and ash before crushing in a local wooden mortar and there after milled on grinding stone. The paste is then boiled in water until the fat begins to float on surface. After extraction, the butter or oil is transferred (skimed) into storage plastic or glass containers. According to USAID (2004) and Karin (2004), the traditional preparation or processing of shea oil in East Africa differs from West Africa.

3.2.2 Solvent Extraction method:

The collected seed were then grinded into equal sizes; the extractor used was soxhlet apparatus with n-hexane as solvent. After extraction, the mixture of the solvent and extract was allowed to cool and then filtered to remove solid particles. The filtrate was concentrated under vacuum in a rotary evaporator (Akpan et al., 2005). The results obtained were noted. The extracted oil was analyzed for the physical and chemical properties. All reagents used were of analytical grade.

3.2.3 Deep-frying process method

The deep fat frying process was carried out in the similar way as reported by Sharma et al. (2006). The frying of a known weight (100 g) of potato chips was carried out by drawing in a 500ml of oil sample from control sample (groundnut oil) as well as blended oil (1:1) beside shea oil separately in a frying domestic frier (diameter 28 cm, depth 6 cm) at a deep fat frying temperature between 180 - 200 °C for 8 –10 minutes After frying, the oil samples from control, blended (1:1) and shea oil were cooled to room temperature and stored separately in plastic bottles for further two frying cycles. After each deep frying cycle, a 70 ml of oil sample was taken from control (groundnut oil), blended and shea oil for analyzing the physico-chemical parameters and lipid profile.

Procedure:

After each frying cycle, the oil samples were analyzed for Free fatty acids (FFA) by titrating the free fatty acid with alkali in presence of ethyl alcohol as solvent, Peroxide value (PV) was estimated by using sodium thiosulfate solution as titrating agent against the evolved iodine in the sample, after reacting the peroxides present in the sample with salt of iodine (KI), Iodine value (IV) was determined by treating the sample with an excess of solutions of iodine monochloride (ICl) in glacial acetic acid. Unreacted iodine monochloride reacted with potassium iodide, converting it to iodine, whose concentration was determined by titration with sodium thiosulphate, Saponification value (SV) was determined by treating the sample with alkali and the unreacted parafins were then titrated against 0.5 N hydrochloric acid and Refractive index (RI) was determined by using refractrometer with temperature adjusted to 37 °C. Specific gravity was determined by using standard methods (AOCS 2004). Color of the oil was measured by using Lovibond tintometer (Model F, Effem Technologies Pvt. Ltd., New Delhi, India).

3.2.4 Fatty acid profile method

To the sample in the vial, 1 mL hexane was added to the sample then vortexed for 30 seconds, followed by dryingthe sample by passing through anhydrous sodium sulfate (Na2SO4) placed in a glass Pasteur pipette with a glass wool. The dried sample was transferred into Teflon caped vial from which 10 µL was diluted by adding 990 µL hexane then vortexed for 30 seconds. Two hundred µL of the mixture was pipetted into a clean Teflon caped sample vial with an insert. The vial was placed into an auto sampler injector where 1 µL was injected into GCMS.

3.2.5 Sensory evaluation method

In this study, the sensory parameters of potato-fried chips were assessed for all samples, to evaluate their colour, flavour, taste, texture, and overall Acceptablity. **[**

3.3 Shea seeds proximate analysis

Moisture, ash and crude fiber contents were analyzed according to standard methods described in AOAC (1997). Nitrogen was assayed using Kjeldahl method and the nitrogen content was converted to protein by a multiplication factor of 6.25 (AOAC, 1997) Total Carbohydrates were determined by difference using a standard method of AOAC (1997). All the proximate analyses were carried out in triplicate and the results expressed as percentage of the sample analyzed.

3.3.1 Seeds moisture content

The dry seeds powder (2-5g) was weighed into a clean dry aluminum dish with a known weight. The sample was dried in vacuum oven at a temperature of $105C^0$ for 6 hours, cooled in a desiccator and weighed. Weighing was repeated twice until there was no difference in the two successive weights.

The moisture content was calculated following the method of AOAC, (1997).

$$\textit{Moisture content \%} = \frac{\textit{(weight of wet sample} - \textit{weight of dry sample}}{\textit{Weight of sample}} \times 100$$

3.3.2 Crude oil content

The dry seeds powder (5g) was extracted with n-hexane solvent using soxhlet apparatus for 6 hours. The crude oil extracted was concentrated in a rotary evaporator and dried by heating in a vacuum oven at 50C for one hour. The Percentage crude oil content was then determined gravimetrically (AOAC, 1997).

$$\textit{Crude oil content \%} = \frac{\textit{Weight of extracted oil}}{\textit{Weight of dry sample}} \times 100$$

3.3.3 Total ash

The dry seed powder sample (2-3g) was placed in a dry-clean porcelain dish and heated progressively for 6 hours at 550ºC until, grey-reddish ash was obtained according to (AOAC, 1997)**.** The sample was cooled in a desiccator, weighed and total ash calculated using the following formula:

$$\text{Total ash \%} = \frac{(\text{weight of dish + ash}) - (\text{weight of dish}) \times 100}{\text{Weight of sample}}$$

3.3.4 Crude fiber

About 2-3g of shea seeds powder was transferred into a 200 ml Labeled beaker after which 50 ml of sulphuric acid (1.25 %) and 150 ml of distilled water were added. The sample mixture was then boiled for 30 minutes under reflux flask and later treated with 50 ml of potassium hydroxide (1.33%) and 150 ml of distilled water, and then the solution was re-boiled again for 30 minutes and re-filtered using vacuum crucible filtrate on system. The sample in the crucible was rinsed with distilled water followed by acetone. The samples were put into a pre-weighed crucible and transferred to the oven to dry for 4 hours, cooled in dissector and weighed. The weighed sample was ashed in a muffle furnace set at 660 ºC for 5 hours until it became grey ash which was cooled in the desecrator and weighed (AOAC, 1997). The weight of ash was then calculated as follows:

$$\text{Crude fiber \%} = \frac{\text{weight of fiber Original}}{\text{Weight of sample}}$$

3.3.5 Crude protein (%)

Protein can be determined through the following stages:-

(a) Digestion stage:

The dry shea seeds powder (2g) was placed in kijeldahl tube and a 4g mixture (catalyst; sodium sulphate and copper sulphate) was added the mixture was digested with concentrated sulphuric acid (25ml) for 2 hours in fume hood until the solution became clear to light green.

(b) Distillation stage:

120 ml of distilled water was added to the solution and allowed to cool. Sodium hydroxide (45%) was also added without agitation. The flask was then connected to the distillation bulb with the terminal tip of the condenser immersed in a standard acid solution of boric acid (2%) containing 5 drops of mixed indicator (pink colour). The flask was then heated to release ammonia into the indicator solution which changed from pink to blue colour, a volume of 100 – 125 ml an indication of the end point of distillation stage.[

(c) Titration stage:

The excess standard acid in the distillate was titrated with 0.1N standard HCL The conversion factor of 6.25 was used (AOAC, 1997) and % of Nitrogen calculated as Below:

$$Nitrogen\% = \frac{(ml\ of\ acid \times N\ of\ acid) \times 14 \times 100}{Weight\ of\ sample \times 1000}$$

$$Protein\ \% = N\% \times protein\ factor\ (6.25).$$

3.3.6 Total and available carbohydrates

Total carbohydrates were determined by difference using the method in (AOAC, (1997).

Total carbohydrates = [100 - (Moisture content- Crude lipids- Crude proteins and ash %)

3.4 Oil extraction

3.4.1 Preparation of the Seeds for oil Extraction:

The collected shea butter seeds (nuts) were dehulled, cleaned and dried under the sun light for a day and later dried in the normal oven for 3 hours at 50 °C to ensure that moisture content was reduced as minimum as possible.

3.5 Physico-Chemical Characteristics of the extracted shea butter

3.5.1 pH Determination

Two grams (2 g) of the butter sample was poured into a clean dry 25 ml beaker and 13 ml of hot distilled water was added to the sample in the beaker and stirred slowly. It was then cooled in a cold-water bath to 25 °C. The pH electrode was standardized with buffer solution and the electrode immersed into the sample. The pH value was read and recorded (Akpan et al., 2005).

3.5.2 Moisture Content Determination

Five grams (5 g) of shea butter sample was weighed and dried in an oven at 80 °C. After every 2 hours, the sample was removed from the oven and placed in the desiccator for 30 minutes to cool. It was then removed and weighed (Akpan et al., 2005). The percentage moisture in the seed was then calculated from:

$$Moisture\ content = \frac{(W1 - W2) \times 100}{W1}$$

Where:

W1 = Original weight of sample before drying (g),
W2 = Weight of sample after drying (g)

3.5.3 Specific Gravity Determination

The specific gravity bottle was cleaned with acetone, ether and dried in an oven at 60 ºC. The weight of the empty bottle was taken, after which the bottle was filled with the oil sample and properly covered. The weight was then recorded using a sensitive balance, after which the Sample was removed from the bottle. The bottle was properly washed, dried and filled with distilled water, after which the weight was taken and finally, the specific gravity was computed using the relationship below (Akpan *et al.*, 2005).

$$\textit{Specific Gravity} = \frac{(Wo-W)}{W1-W}$$

Where:

W = Weight of empty bottle (g),

Wo = Weight of the bottle and oil content (g).

W1 = Weight of bottle and water content (g).

3.5.4 Refractive Index Determination

The refractive index was determined using Abbey refractometers. The glass prism of the refractometer was thoroughly cleaned with alcohol to ensure that it is free from dust, a drop of oil sample was placed on the lower prism and smeared, then closed with the other covering prism and the light source of the refractometers was switched on, while viewing through the telescope. The coarse adjustment knob was rotated until the black shadow appears central in the cross-wire indicator and while still viewing through the telescope, the fine knob adjustment was made until the rainbow-coloured fringe which appeared on the black dividing line disappeared, the coarse knob was rotated to give fine adjustment and make the black shadow

appear exactly central in the cross-wire indicator. The reading under the telescope and that of the fine adjustment knob were noted and divided by 10,000, this value was then added to the value obtained through the telescope to give the value of the refractive index of the oil at room temperature (Akpan *et al.*, 2005).

3.5.5 Acid Value Determination

Two grams of the sample was dissolved in 50 ml of mixed neutral solvent (25 ml diethyl ether with 25 ml ethanol carefully neutralized with 0.1M NaOH using 1% phenolphthalein solution). The mixture was titrated with 0.1M KOH aqueous solution with constant shaking to faint pink colour (Akpan *et al*., 2005).

$$\textit{Acid value\%} = \frac{\textit{Titer volume} \times 5.61 \times 0.00282\ (\textit{mgKOH/g})}{\textit{Weight of sample (g)}}$$

3.5.6 Free Fatty Acid Determination

The amount of free fatty acid (FFA) was calculated as being equivalent to half the value of acid value (Akpan *et al*., 2005), that is,

$$\textit{FFA \%} = \frac{\textit{Acid value}\ (\textit{mgKOH/g})}{2}$$

3.5.7 Saponification Value Determination

5g of oil sample was weighed and 50 ml of alcoholic KOH 0.5M was added, 50 ml of the blank solution (alcoholic KOH 0.5M) was also measured into a conical flask. The two samples were then connected to a reflux apparatus and allowed to boil for an hour until the reflux is completed, 1 ml of phenolphthalein was added to

the mixture and the resulting mixture was titrated while hot against 0.5 M HCL acid solution. The volume of the acid used to attain the end point was recorded, the blank determination was carried out using the same procedure described above until the colour changes from blue to transparent white, then the volume of acid used was noted, the Saponification value was determined using the relationship below (Akpan *et al*., 2005).

$$\textit{Saponification value (S.V)} = \frac{56.1 \times T\,(V0-V1)}{M}$$

Where,

T = Molarity of the standard KOH solution used (M),

VO = Volume of acid used for the first titration with oil sample (ml),

V1 = Volume of acid used for the second titration blank solution (ml),

M = Mass of the oil sample used (g).

3.5.8 Peroxide Value Determination

A known weight (2 g) of sample was weighed into clean dried boiling tube; 1 gram of potassium iodine (KI) powder was added to the oil and 20 ml of the solvent mixture (glacial acetic acid and chloroform in the ratio 2:1). Then the boiling tube was placed in boiling water bath so that the liquid mixture boils within 30 seconds and allowed to boil vigorously for not more than 30 seconds, the content after boiling was quickly poured into a flask containing 20 ml of 5 % potassium iodine (KI) solution and the tube was washed out twice with 25 ml of water. Then the mixture was titrated with 0.002 M sodium thiosulphate using fresh 1 % starch solution, a blank titration was carried out at the sample time, the peroxide value was calculated using the relationship below **(**Akpan *et al*., 2005**).**

$$\textit{Peroxide value} = \frac{T \times M \times 1000}{\textit{Weight of sample / (g)}}$$

Where:

T = titter value of $Na_2S_2O_3$ = (Sample titer – Blank titer)

M = Molarity of $Na_2S_2O_3$

3.5.9 Iodine value (Hanus method)

The hanus iodine reagent was prepared by dissolving of (13.2 gm) iodine in glacial acetic acid (1litre) with the help of heat. The solution was cooled and 3 ml of bromine added. The hanus iodine reagent was then kept in a brown bottle until the analysis was complete. Shea butter (2g) was weighed into a 500 ml conical flask and 10 ml of chloroform added. By use of a pipette 25 ml of hanus iodine was added and left to stand in the dark for 30 minutes with occasional shaking. After this 10 ml of potassium iodine (15%) was added, shaken thoroughly and distilled water (100ml) added to rinse down any iodine on the stopper. The solution was then titrated with 0.1N sodium thiosulphate until a yellow solution turned almost colorless (titration = S ml). Three drops of starch indicator (1%) was added towards the end point and titration was continued until the blue colour turned colourless. A blank determination was done and results recorded (titration = B ml). All the analysis was done in duplicate (AOAC, 1997) using the formula.

$$\textit{Iodine value} = \frac{(B - S) \times 0.1 \times 12.69 \times 100}{\textit{Weight of sample (g)}}$$

3.6 Statistical analysis

The results were subjected to Statistical Analysis System (SAS) by using One-Factor Analysis of Variance (ANOVA). The Mean values were also tested and separated by using Duncan's Multiple Range Test (DMRT) as described by Steel, *et. a1.,* (1997).

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Chemical analysis of Shea butter kernel (nuts)

Table (2) shows the chemical analysis of Shea butter kernel (nuts) *Vittrellia paradoxa- sub sp.nilotica* that including, moisture , fats, protein ,ash, fiber and carbohydrate which were, (8.00, 45.00, 7.07 , 2.47, 6.47 and 31.07%) respectively. In this study the moisture content was 8% which was similar with that reported by Mbaiguinam *et al.*, (2007). Fats content was noticed to be 45% which was within the range of (17.4 to 59.1%) that reported by Nkouam *et. al.,* (2007) and Akihisa *et. al.,* (2010). On the other hand, the stated protein content was found to be 7.07% which was similar with that reported by Tano-Debrah, Ohta, (1994). More over the fiber content was 6.47%, which was similar with that reported by Tano-Debrah and Ohta, (1994). Ash content was noticed to be 2.47%, which was within the range of (1.8 to 3%) that reported by Tano-Debrah, Ohta, (1994). Finally, Total carbohydrate content was recorded to be 31.07 % which was within the range of (25.0 to 34.8%) That mentioned by Tano-Debrah, Ohta, (1994).

Table: (2) the chemical composition of shea butter nut

Parameters	Shea butter nut
Moisture content (%)	8.00± 0.1
Fat (%)	45.00± 0.1
Protein (%)	7.07± 0.15
Ash (%)	2.47± 0.05
Fiber (%)	6.47± 0.05
CHO (%)	31.07± 0.23

Values are mean ± SD, n =3

4.2 Shea butter extraction

4.2.1 The effect of two different methods of extraction on the physical proprieties shea butter

The physical properties of natural fats and oils vary widely because of two main reasons: the proportions of fatty acids and the triacylglycerol structures. Where the later also affected by chain length, number and position of the double bonds and position of the fatty acids within the glyceride molecule (O'Brien, 2009). Specific gravity, viscosity, refractive index, and color intensity are some physical characteristics that play important role in determining oil quality, as they give physical specification for a particular product (Francis, 2009).

The physical properties of Shea butter extracted by Soxhlet method (solvent extraction) and traditional method (boiling extraction), which were including the specific gravity, viscosity, refractive index and colour intensity, showed the following results.

4.2.2 Specific gravity

Specific gravity also referred to as relative density which considered as an important physical character that can give information on the identity of the sample as well as detect adulteration of Shea butter in which density may increase or decrease. It can also provide information for the shippers on the weight of the Shea butter from the given volume while exporting it in large volumes (Hamilton *et al*., 1986).

Table (3) shows the reported results of Specific gravity for both methods of extraction (solvent and boiling), these results were (0. 9410 and 0.8901 g/cm^3) which indicating that, there was no significant difference between them ($p \leq 0.05$). These findings were similar with that reported by Akingbala *et al.*, (2006) and Olaniyan *et al.*, (2007).

4.2.3 Viscosity

The viscosity is the other vital physical characteristics to quantify the body/texture of butter in terms of relative thickness or resistance to flow (Bockisch, 1998) and (Gunstone, 2004).

Table (3) indicates the viscosity for both extraction methods (solvent and boiling) which were noticed to be (1.1 and 3.6 cp), these results showed a significant difference between each other ($P \geq 0.05$). The increasing of the viscosity value exhibited in this study by boiling extraction (3.6 cp) as compared to solvent extraction (1.1cp), attributed to oxidation and polymerisation in the Shea oil because the methods of extraction were completely different. On the other hand, these results were not matching the viscosity values obtained for Shea oil that extracted by solvent and boiling methods (2.6 and 14.4 cp) as reported by Dhellot *et al.* (2006) and confirm to the work of Adikini, (2002). These variations might be due to, genotype, storage condition, method of extraction, and agricultural practices, as reported by Dhellot *et al.* (2006).

4.2.4 Refractive index

Refractive index is the ratio of the speed of light in a vacuum to that in the oil cis*/trans* double bonds and can provide hints on the oxidative damage (Hamilton *et al.*, 1986). Refractive index can be used for rapid grading of fats and oils which are suspected to be adulterated (Olaniyan *et al*, 2007).

Table (3) shows the refractive index results for both method of extraction (solvent and boiling), which were (1,4780 and 1.4681) respectively, these results indicating that, there was no significant difference between them ($p \leq 0.05$) , there for the obtained results were also not differ from (1.468 - 1.469) that reported by Zeb and Ahmad (2004).

4.2.5 Colour intensity

Colour of the Shea butter samples ranged from whitish yellow-to-yellow colour, which is consistent with the typical Shea butter colour (Goreja, 2004) and (Moharram et *al*., 2006). The colour of Shea butter varies depending on the processing technique, particularly the temperature used during processing. Some roots or bark of *Cochlospermum tinctoriumare* often used to improve Shea butter colour as reported by Chukwu and Adgidzi (2008). The change in the colour of vegetable oil is mainly attributed to peroxidation, pigmentation or contamination (Lewis, 1990).

Table (3) indicates the results of Color intensity for both method of extraction (solvent and boiling) which were (3.57 and 10.37) respectively, these results in compression between each other's, indicating that, there was a significant changes ($P \geq 0.05$) in the color of both oil samples obtained by the two different methods of extraction. The colour of Shea oil obtained in this study was similar to those colors found by Tano-Debrah and Yoshiyuki (1994). Generally, the yellow-orange colour of Shea oil samples might be an indication of the presence of ß-carotene pigments in Shea oil, which is nutritionally important (Adikini, 2002).

Table (3) Effect of extraction methods on the physical proprieties of shea butter

Parameters	Methods of extraction		LSD	CV %
	Solvent	boiling		
Specific gravity at 40°c (g/cm^3)	$0.9410^a \pm 0.00$	$0.8901^a \pm 0.00$	0.03	0.00
Viscosity (CP)	$1.10^b \pm 0.022$	$3.60^a \pm 0.026$	0.050	0.001
Refractive index	$1.47^a \pm 0.00$	$1.48^a \pm 0.00$	0.00	0.01
Color intensity	$3.57^b \pm 3.47$	$10.37^a \pm 1.01$	28.40	11.60

Values are mean ± SD, n =3

4.3 The effect of two different methods of extraction on the chemical proprieties of Shea butter

Unlike physical properties, chemical properties have much importance toindicate the butter stability than the appropriate use of butter. Free fatty acid, peroxide value, saponification fraction, and iodine value are some of Shea butter chemical properties.

Table (4) shows the chemical properties of the Shea butter extracted by two different methods (solvent and boiling extraction), which including, the moisture content (%), fats (%), free fatty acid (%), peroxide (Meq. O_2/Kg), saponfication (mg KOH/g oil) and iodine value (I_2/100 g oil). Moisture content, free fatty acid, and iodine value, showed no significant difference between the two extraction methods. But there was a significant difference in, fat content, peroxide and saponification values of the two different methods of extraction. The reported values for (solvent extraction), were found to be (0.05 %, 45.8 %, 1.6 %, 1.07 Meq/Kg, 181.23 mg KOH/g oil and 70.6 I_2/100 g oil) respectively. Where the obtained results for the (boiling extraction) were, (0.07 %, 38.02 %, 1.90 %, 3.83 Meq. /Kg, 178.07 mg KOH/g oil and 65.90 I_2/100 g oil) respectively.

4.3.1 Moisture content

Moisture and volatile matter, which could be removed by oven drying, were determined according to MOHFW (2005). The high moisture content in plant fats and oils usually leads to increasing the microbial load as well as lipid oxidation which resulting in oil rancidity. Moisture contents of Shea butter were quite low, ranging from 0.01 to 0.20 % in all the Shea varieties.

Table (4) shows the reported moisture content of the Shea butter extracted by (Solvent and boiling) methods which were, (0.05 and 0.07 %) these results indicating that, there was no significant difference ($p \leq 0.05$) in moisture content among the two methods. These results were similar to that reported by GRET (2007) and Mbaiguinam *et al.*, (2007). The moisture content decreased as the processing temperature increased, that which observed by Olaniyan and Oje, (2007).

4.3.2 Fat content

Fat content is the most important characteristic should be considered. The Shea butter yield ranging from 20% to 60% have been reported by Tano-Debra *et al.* (1995); Boffa *et al.* *(*1999); Maranz *et al, (2003*); Maranz *et al. (*2004); Di-Vincenzo *et al*. (2005) and Kapseu *et al.* (2007).

Table (4) shows the oil content of shea butter obtained by the two different methods of extraction (solvent and boiling) which were, (45.8 and 29.6%) respectively. These results showed a significant difference between the two methods of extraction ($P \geq 0.05$), this variation might be due to processing method, especially the traditional method that mainly depends on water extraction where water extraction is so far not highly efficient to extract the whole oil content from the nuts. On the other hand, these results were matching the obtained results that ranging between 30 and 35% for the traditional method (boiling extraction), as compared to the solvent extraction method which gives over 50% of the oil from shea kernel (Maranz *et al.*, 2004).

4.3.3 Free fatty acids

The free fatty acid was heavily dependent on the processing practices that carried on Shea seeds.

Table (4) indicates the obtained results of the free fatty acid for the two different extraction methods (solvent and boiling) which were (1.50 and 1.90 (%). These results showed no significant difference among each other's ($p \leq 0.05$). Although the two methods of extraction were completely different, but the exhibited values in this study were within the range of (1 – 5.7(%) that reported by Olaniyan and Oje, (2007).

4.3.4 Peroxide value

Kirk and Sawyer (1991) described peroxide as a first product of oxidation of unsaturated fats and oils, with an average of 7.6 meq O_2/kg, the reported peroxide value ranges from 0.5 to 29.5 meq O_2/kg Njoku, *et al*. (2000) and Dandjouma *et al*. (2009). Most of the authors found peroxide values below the reported average values, but the high value (29.5 meq O_2/kg) reported by Dandjouma, *et al*. (2009).

Table (4) indicates the obtained results of the peroxide value of shea butter extracted by the two methods (solvent and boiling) which were (1.07 and 3.83 meq O_2/kg) respectively. The peroxide value of shea butter extracted by solvent method was significantly lower than that extracted by traditional method ($P \geq 0.05$), this might be due to the already fermented kernels (nuts) used for the butter extraction. Generally, these results were within the range 0f (0.5 – 29.5 meq O2/kg) that obtained by Okullo et al., (2010) and Honfo et al., (2011).

The required peroxide value of Shea butter utilizations in food industries should be (less than 10) and for cosmatics was (1meq O_2/kg) (Kassamba, 1997). In fact, unlike acid value, peroxide value depends on the oxidation rate of unsaturated fatty acids to form peroxides and hydroperoxide other than the hydrolysis of triacylglycerols to from free fatty acids (O‟Brien, 2009).

4.3.5 Saponification value

Saponification is the process of breaking down or degrading a neutral fat into Glycerol and fatty acids by treating fat with alkali. Saponification is an index of the mean molecular weight of the triacylglycerols in the sample. The smaller the saponification value, the longer the average fatty acid chain length (Nielsen, 2010). Table (4) shows the results of saponification values of shea butter for both methods of extraction, (solvent and boiling) (181.07 and 178.07 mg KOH/g oil) respectively. The saponification value of the shea butter obtained by boiling method (178.07 mg KOH/g oil) was significantly lower than that obtained by solvent extraction (181.07 mg KOH/g oil) ($P \geq 0.05$), that variation might be due to the impact of the thermal traditional method. On the other hand, these results agreed with the range of the various works that were reported on saponification values which between 132 and 261.3 mg KOH/g oil (Ezema and Ogujiofor, 1992) and (Olaniyan and Oje, 2007). Where the average value reported as 180.9 mg KOH/g oil was stated by Honfo, *et al*. (2013).

4.3.6 Iodine value

Oils and fats contain saturated and unsaturated fatty acids, and many of their properties depend on the ratio of these two types of fatty acids. The iodine value (iodine number) is a measure of degree of unsaturation, which is the number of carbon–carbon double bonds in relation to the amount of fat or oil. The higher the amount of unsaturation, the more iodine is absorbed and the higher the iodine value (Gunstone, 2004) and (Nielsen, 2010).

Table (4) indicates the results of iodine values of shea butter extracted by both methods of extraction (solvent and boiling) (70.63 and 65.90 I_2/100 g oil). There was no significant difference ($p \leq 0.05$) between iodine values of shea butter

Exhibited by the two different methods of extraction, there for; the applied mode of extraction by the two methods, did not affect the iodine values of the extracted oil. More over these results were within the range of iodine values (21.68 – 89.5 I_2/100 g oil) as reported by Okullo et al., (2010) and Honfo et al (2011)

Table (4): the effect of two different methods of extraction on the Chemical proprieties of shea butter.

Parameters	Methods of extraction		LSD	CV %
	Solvent	Boiling		
Moisture content (%)	$0.05^{a} \pm 0.01$	$0.07^{a} \pm 0.01$	0.50	23.57
Fat content (%)	$45.80^{a} \pm 0.23$	$29.60^{b} \pm 0.00$	1.00	1.24
Free fatty acid (%)	$1.50^{a} \pm 0.06$	$1.90^{a} \pm 0.12$	0.43	7.20
Peroxide value (Meq/Kg)	$1.07^{b} \pm 0.03$	$3.83^{a} \pm 0.09$	0.38	4.41
Saponiflcation value (mgKOH/g oil)	$181.23^{a} \pm 1.18$	$178.07^{b} \pm 0.84$	1.86	0.30
Iodine value (I_2/100 g oil)	$70.63^{a} \pm 3.60$	$65.90^{a} \pm 0.92$	11.91	4.96

Values are mean ± SD, n =3

4.4 Stability of Shea butter, groundnut and their blend oil (1:1) when subjected to deep-frying process

Deep-frying is one of the most common methods used for the preparation of food. Repeated frying causes several oxidative and thermal reactions, which results in changing the physicochemical, nutritional, and sensory properties of the oil (Che Man and Jasvir, 2000). During deep-frying, due to hydrolysis, oxidation and polymerization processes the composition of oil changes, which in turn changes the flavor, and stability of its compounds (Gloria and Aguilera, 1998). During deep frying different reactions, depend on some factors such as replenishment of fresh oil, frying condition, original quality of frying oil and decrease in their oxidative stability **(**Choe and Min, 2007). Atmospheric oxygen reacts instantly with lipid and other organic compounds of the oil to cause structural degradation in the oil which leads to loss of quality of food and may be harmful to human health (Bhattacharya *et al.*, 2008).

4.4.1 Effect of deep-frying on the physical properties of Shea butter, groundnut and their blend oil (1:1).

4.4.2 Specific gravity

Table (5) shows the results of Specific gravity of Shea butter, groundnut oil and their blend (1:1), for (control samples) the obtained results were (0.9091, 0.9390 and 0.9104g/cm^3) respectively. Where the specific gravity results for the first , second, and third deep-frying process for all types of oil were, (0.9050, 0.9351, 0.9100, g/cm^3) - (09030, 0.8352, 0.9081 g/cm^3) and (0.8980, 0.8161, 0.9061g/cm^3) respectively. These results showed a significant decrease ($P \geq 0.05$) in specific gravities of shea butter, groundnut oil and their blend (1:1) throughout deep-frying process when compared to their controls, these variations might be due to the impact of repeated heat treatment, methods of extraction, storage conditions and refining process.

The specific gravities obtained were in accordance with the specific gravity ranged between (0.8710 and 0.9102 g/cm^3) that reported by Akingbala *et al*. (2006) and Olaniyan *et al*. (2007) but lower than the value (0.9710 g/cm^3) obtained by (Njoku *et al*., 2000).

4.4.3 The viscosity

The viscosity is the other vital physical characteristics to quantify the body/texture of butter in terms of relative thickness or resistance to flow, Bockisch, (1998) and Gunstone, (2002).

Table (5) shows the results of viscosity of Shea butter, groundnut oil and blend (1:1) for their (control samples) which were (3.65, 3.02, 3.24, (cp) Where the viscosities of shea butter, groundnut and their blend oil (1:1) throughout deep-frying process(first, second and third frying) were 3.70, 3.14, 3. 35cp) - 3 .81, 3.31, 3.54 (cp) and 3.95, 3.50, 3.72(cp) respectively, these results when compared to their control samples showed a significant decreasing ($P \geq 0.05$) in their values, this variation might be due to oxidation and polymerisation in the Shea oil. The viscocisites obtained were in accordance with the viscosity observed by Olaniyan and Oje, (2007).

4.4.4 Refractive index

Changes in the refractive index of fat may reflect changes in the crystallinity of the fat. The greater increase in the refractive index of the bleached Shea butter thus indicates greater instability of the fat to storage. (Arys *et al*, 1969).

Table (5) shows the results of refractive index of Shea butter, groundnut and their blend oil, for (control samples) which were (1.478, 1.463, 1.472) Where the results of the other different thermal treatments (the first, second and the third deep-frying process) for Shea butter, groundnut oil, and their blend were (1.478, 1.464 and 1.475) – (1.484, 1.465, 1.478) and (1.489, 1.469, 1.488) respectively.

In these findings the significant difference (P≥0.05) in refractive index was observed among the heat-treated samples in comparison to their control samples. So the greatest refractive index was reported for Shea butter (1.489) followed by the blended oil (1.488), where the lesser one's was reported for groundnut oil (1.469). These variations might be due to impact of repeated heat treatment (where refractive index increased as temperature increased), methods of extraction and storage conditions. Yen, Gow –Chin, (1991) used refractive index to compare the thermal stability of soybean oil with the stability of oils from roasted and unroasted sesame seeds.

4 .4.5 Colour intensity

Colour on the other hand can be used as a good indicator of vegetable oil quality.

Table (5) revealed the results of colour intensity of Shea butter, groundnut oil and their blend for (control samples) which were, (30.57, 21.20 and23.20). Where the obtained results for the other different thermal treatments (the first, second, and the third deep-frying stages) for Shea butter, groundnut and their blend oil were (31.57, 21.63, 25.57) - (31.87, 22.60, 25.60) and (32.64, 23.90, 25.77,) respectively. These results indicating that, there were a significant increase (P≥0.05) in the color intensity values of heat treated samples when compared to their control samples. The change in the colour of vegetable oil is mainly attributed to peroxidation, pigmentation or contamination.On the other hand, according to Maritnez *et al*, (2008), differences in colour could be due to either peroxidation or polymerisation of triglycerides in the Shea oil.

Table (5) the effect of deep-frying process on the physical properties of Shea butter, groundnut and their blend oil (1:1).

Physical Properties	Treatment / Samples	Control	First frying	Second frying	Third frying
Specific gravity at 40°c (g/cm^3)	Shea butter	$0.9091^{c} \pm 0.001$	$0.9050^{ef} \pm 0.00$	$0.9030^{f} \pm 0.001$	$0.8980^{g} \pm 0.001$
	Groundnut oil	$0.9390^{a} \pm 0.001$	0.9351 ± 0.001	$0.8352^{h} \pm 0.001$	$0.8161^{i} \pm 0.00$
	Blend oil (1:1)	$0.9104^{c} \pm 0.001$	$0.9100^{cn} \pm 0.001$	$0.9081^{cd} \pm 0.001$	$0.9061^{de} \pm 0.00$
Viscosity (CP)	Shea butter	$3.65^{C} \pm 0.001$	$3.70^{C} \pm 0.008$	$3.81^{b} \pm 0.001$	$3.95^{a} \pm 0.002$
	Groundnut oil	$3.02^{h} \pm 0.002$	$3.14^{g} \pm 0.001$	$3.31^{g} \pm 0.002$	$3.50^{d} \pm 0.005$
	Blend oil (1:1)	$3.24^{fg} \pm 0.00$	$3.35^{ef} \pm 0.00$	$3.54^{de} \pm 0.003$	$3.72^{bc} \pm 0.13$
Refractive index	Shea butter	$1.478^{c} \pm 0.00$	$1.478^{c} \pm 0.00$	$1.484^{b} \pm 0.00$	$1.489^{a} \pm 0.00$
	Groundnut oil	$1.463^{h} \pm 0.00$	$1.464^{gh} \pm 0.001$	$1.465^{g} \pm 0.00$	$1.469^{f} \pm 0.00$
	Blend oil (1:1)	$1.472^{e} \pm 0.00$	$1.475^{d} \pm 0.00$	$1.472^{e} \pm 0.00$	$1.488^{a} \pm 0.00$
Colour Intensity	Shea butter	$30.57^{c} \pm 0.18$	$31.57^{b} \pm 0.15$	$31.87^{b} \pm 0.15$	$32.64^{a} \pm 0.19$
	Groundnut oil	$21.20^{f} \pm 0.06$	$21.63^{g} \pm 0.22$	$22.60^{d} \pm 0.12$	$23.90^{d} \pm 0.06$
	Blend oil (1:1)	$23.20^{h} \pm 0.00$	$25.57^{d} \pm 0.15$	$25.60^{g} \pm 0.12Q$	$25.77^{e} \pm 0.09$
LSD		2.474	1.13	1.18	0.39
CV%		0.16	1.91	0.06	0.87

Values are mean ± SD, n =3

4.5 Effect of deep-frying on the chemical properties of Shea butter, groundnut and their blend oil (1:1).

4.5.1Moisturecontent.

Analytical value of moisture can be used to indicate food liability for spoilage, quality, convenience for packaging/shipping and many others parameters, Nielsen, (2010). Concerning Shea butter high moisture content is susceptible to recontaminations or rancidity Olaniyan and Oje, (2007). The reported moisture contents of Shea butter vary from 0.1% Olaniyan and Oje, (2007) to 4.9% Honfo et al., (2013).

Table **(**6**)** shows the results of moisture content of Shea butter, groundnut and their blend oil (1:1) for their (control samples), which were (0.07, 0.08 and 0.07%). Where the recorded moisture content for the other different thermal treatments (the first, second, and the third deep-frying stages) for Shea butter, groundnut and their blend (1:1) oil, were (0.06, 0.06, 0.07, %) - (0.05, 0.05, 0.05%) and (0.05, 0.05, 0.05%) respectively. In this study, the obtained results of the (control samples) showed no significant difference ($p \leq 0.05$) in their moisture contents, As well as the ststed results for the different thermal treatments (the first, second, and the third deep-frying stages) for Shea butter, groundnut and their blend (1:1) oil, also showed no any significant difference btween them ($p \leq 0.05$)

Moreover, it was noticed that, the moisture content of all samples were decreased, this might be due to successive heat treatment which led to the loss of water through evaporation during the deep-frying process. Generally, these results were in accordance with the reported range of 0.1 to 4.9% by Olaniyan and Oje, (2007) and Honfo *et al*., (2013). However, the required moisture contents of Shea butter destined for food industries should be less than 0.2%, and 0.05 for cosmetic industry. (Kassamba, 1997).

4.5.2 Free fatty acid (FFA).

Free fatty acid content increased after the deep-frying cycles and a significant difference was observed in the (FFA) content among the oil samples between consecutive frying cycles, 1st to 3rd for the moistened potato chips. Several studies conducted free fatty acids analysis on commercial vegetable oil samples and the values were in a range of 0.02-1.38 % Osawa *et al.*, (2007), which were much lower than the FFA of the crude Shea butter samples used in this study.

Table (6) Indicates the results of free fatty acid of the Shea butter, groundnut and their blend oil for (control samples), which were, (1.87, 0.60 and 2.00%). Where the recorded one's for different thermal treatments (the first, second, and the third deep-frying stages) for Shea butter, blended, and groundnut oil, were (2.50, 0.90, 2.50 %) - (2.67, 1.40, 3.10 %) and (3.20, 1.50, 3.67%) respectively. In this finding, the Shea butter and blended oil, showed no significant difference ($p \leq 0.05$) among each other, in comparison with groundnut oil in their control samples. Moreover, the results of the second and the third frying process for all types of oil showed a significant difference ($P \geq 0.05$) between each other's accept the shea oil and the blend oil(1:1) during the firs frying process,were showed the same results ($p \leq 0.05$). That variation might be due to the impact of heat treatment, storage and femented kernels.The (FFA) values for all samples were increased, and this increasing might be due to the already fermented kernels, storage condition, auto-oxidation and decomposition of triglycerides. Finally, the obtained results were in accordance with the reported range of 1 to 10.7% with an average of 5.3% as stated by Badifu, (1989). The acid value and (FFA) of Shea butter increase with the duration of the storage of the Shea fruits, this increasing was explained by the physiological activity of fruits during storage, the fruit fatty acids are degraded to

Produce some energy and precursors for the synthesis of new molecules as reported by (Kapseu *et al.*, 2001).

4.5.3 Peroxide value

The oxidation of oils is a major cause of their deterioration. Hydroperoxides formed by the reaction between oxygen and unsaturated fatty acids are the primary products of this reaction. Hydroperoxides themselves have no flavour or odour but break down rapidly to form aldehydes, many of which have a disagreeable odor (Gunstone, 2002).

Table (6) Indicates the results of peroxide value of the Shea butter, groundnut and their blend oil (1:|1) for their (control samples), the obtained results were (4.00, 1.10 and 2.70 meq O2/kg) Where the recorded one's for different thermal treatments (the first, second, and the third deep-frying stages) for Shea butter, blended, and groundnut oil, were (6.70, 3.10 , 4.90 meq O2/kg)) - (8.50, 5.20 , 6.53, meq O2/kg) and (9.80, 7.00, 8.40 meq O2/kg)) respectively. In these findings, the obtained values of peroxide for Shea butter, groundnut and their blend oil, are completely different ($P \geq 0.05$). These variations attributed to the effect of successive heat treatment, extraction methods and storage conditions. It was also noticed that, the peroxide values of all samples were increased, this increasing might be due to the already fermented kernels, auto-oxidation, storage condition or hydrolysis of triglycerides. According to Kaul *et al.,* (*2008*) the increase in peroxide value could be due to oxidative hydrolysis reaction during extraction process. The boiling with water that is undertaken in traditional method could have caused oxidative hydrolysis leading to formation of peroxides. As reported by Maritnez *et al.,* (*2008*), water is generally increased the hydrolysis of triglycerides in the oil. Anhwange *et al.,* (2004), also stated that, high peroxide value for example, could be responsible for the development of rancidity in the oil and fats**.**

4.5.4 Saponification value

Saponification is the process of breaking down or degrading a neutral fat into glycerol and fatty acids by treating the fat with alkali.

Table (6) Indicates the results of saponification value of the Shea butter, groundnut and their blend oil (1:1) for their (control samples), which were (181.80, 186.77 and 191.87 mg KOH/g). Where the recorded one's for different thermal treatments (the first, second, and the third deep-frying stages) for Shea butter, groundnut and their blend oil (1:1) were (175.57, 179.47, 184.57 mg KOH/g) - (163.80, 171.63, 175.33 mg KOH/g) and (150. 33,161.93, 164.30 mg KOH/g) respectively. In this study, the obtained results of saponification value for Shea butter, groundnut and their blend oil (1:1) showed a significant difference ($P \geq 0.05$), except in the third frying processs the saponification values of groundnut and blend oil revealed the same result. These variations attributed to the effect of successive heat treatment, extraction methods, and storage conditions. In this study the obtained sapnification values were in accordance with the range that is between (132 mg KOH/g) that stated by Ezema., Ogujiofor, (1992) and (261.3 mg KOH/g) mentioned by Olaniyan., Oje, (2007), The average value was reported as (180.9 mg KOH/g) by (Honfo *et al*., 2013).

4.5.5 Iodine value

Oils and fats contain saturated and unsaturated fatty acids, and many of their properties depend on the ratio of these two acid types. The iodine value (iodine number) is a measure of degree of un-saturation, which is the number of carbon–carbon double bonds in relation to the amount of fat or oil. The higher the amount of un-saturation, the more iodine is absorbed and the higher the iodine value Gunstone, (2004) and Nielsen, (2010).

Table (6) Indicates the results of iodine value of the Shea butter, groundnut and their blend oil (1:1) for (control samples), which were (64.30, 123.70 and 124.80 I_2/100g oil). Where the recorded ones for different thermal treatments (the first, second, and the third deep-frying processes) for Shea butter, groundnut and their blend oil (1:1), were (61.27, 117.77, 118.70 I_2/100g oil) - (55.10, 100.20, 111.00 I_2/100g oil) and (41.27, 93.80, 92.60 I_2/100g oil) respectively. In this finding, the reported results for control and different thermal treatments indicated that, there was a significant difference ($P \geq 0.05$) among them and also showed a gradual decreasing in iodine values. This decreasing might be due to the oxidative and thermal degradation reactions that occurred during the deep-frying processes Sharma *et al.* (1976). The decrease in iodine number was also more prominent in case of groundnut oil as compare to Shea butter and blended oil. The tested oil samples, showed a significant change in their iodine values especially after the third repeated deep-frying process when used to fry the moistened potato chips. The data indicating that, the quality deterioration of oils was started earlier when the potato chips were containing high moisture content. Generally, the obtained iodine values under different thermal conditions for Shea butter were in accordance with that ranged from (21.7 mg I_2/100g) as stated by Nkouam *et al.*, (2007) and to (89.5 mg I_2/100 g) as reported by (Womeni *et al.*, 2004)

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Table (6): the effect of deep-frying process on the chemical properties of Shea butter, groundnut and their blend oil (1:1)

Chemical properties	Treatment / samples	Control samples	First frying	Second frying	Third frying	LSD	CV%
Moisture content (%)	Shea butter	$0.07^{ab} \pm 0.01$	$0.06^{abc} \pm 0.00$	$0.05^{bc} \pm 0.01$	$0.05^{bc} \pm 0.01$	0.03	2.82
	Groundnut oil	$0.08^{ab} \pm 0.02$	$0.06^{abc} \pm 0.00$	$0.05^{bc} \pm 0.01$	$0.05^{bc} \pm 0.01$		
	Blend oil (1:1)	$0.07^{ab} \pm 0.01$	$0.07^{abc} \pm 0.01$	$0.05^{bc} \pm 0.01$	$0.05^{bc} \pm 0.01$		
Free fatty acids (%)	Shea butter	$1.87^{d} \pm 0.15$	$2.50^{c} \pm 0.06$	$2.67^{c} \pm 0.09$	$3.20^{b} \pm 0.12$	0.29	7.93
	Groundnut oil	$.60^{g} \pm 0.120$	$0.90^{f} \pm 0.12$	$1.40^{e} \pm 0.12$	$1.50^{e} \pm 0.12$		
	Blend oil (1:1)	$2.00^{d} \pm 0.12$	$2.50^{c} \pm 0.060$	$3.10^{b} \pm 0.12$	$3.67^{a} \pm 0.20$		
Peroxide value (meq o_2/kg)	Shea butter	$4.00^{ef} \pm 0.06$	$4.90^{de} \pm 0.12$	$6.53^{c} \pm 0.1$	$8.40^{b} \pm 0.17$	1.03	10.79
	Groundnut oil	$1.10^{h} \pm 0.06$	$3.10^{fg} \pm 0.12$	$5.20^{d} \pm 0.12$	$7.00^{c} \pm 1.15$		
	Blend oil (1:1)	$2.70^{g} \pm 0.12$	$6.70^{c} \pm 0.17$	$8.30^{b} \pm 0.06$	$9.80^{a} \pm 0.17$		
Saponification value (mg KOH/g)	Shea butter	$181.80^{cd} \pm 1.21$	$175.57^{e} \pm 1.13$	$164.30^{g} \pm 1.79$	$163.80^{g} \pm 1.21$	3.78	1.28
	Groundnut oil	$191.87^{a} \pm 1.76$	$184.57^{bc} \pm 1.24$	$175.33^{ef} \pm 0.77$	$161.93^{g} \pm 0.93$		
	Blend oil (1:1)	$186.77^{b} \pm 0.66$	$179.47^{d} \pm 1.30$	$171.63^{f} \pm 0.66$	$150.33^{h} \pm 1.47$		
Iodine value (I_2g/100g)	Shea butter	$64.30^{g} \pm 1.21$	$61.27^{fg} \pm 2.20$	$55.10^{fg} \pm 1.50$	$41.27^{g} \pm 0.93$	22.00	13.52
	Groundnut oil	$124.80^{bc} \pm 1.21$	$118.70^{cd} \pm 1.79$	$111.00^{cde} \pm 2.08$	$92.60^{e} \pm 1.44$		
	Blend oil (1:1)	$123.70^{bc} \pm 1.21$	$117.77^{a} \pm 1.73$	$100.20^{de} \pm 1.39$	$93.80^{e} \pm 1.56$		

[Values are mean ± SD, n =3

4.6 Sensory evaluation of potato chips fried in Shea butter, groundnut and their blend oil (1:1).

In this study, the sensory parameters of potato-fried chips were assessed for all samples, to evaluate their colour, flavour, taste, texture, and overall Acceptablity.

Table (7) indicates the Sensory evaluation of potato-chips fried in Shea butter, groundnut and their blend oil (1:1). This evaluation was including, the colour, flavour, taste, texture and overall Acceptablity , the obtained scores for the Shea butter were, (1.44, 1.81, 1.94, 1.81, 1.69) respectively. Where the obtained scores for groundnut and blend oil (1:1) were, (4.88, 4.31, 450, 4.00. 4.69) and (2.88, 3.44, 3.50, 3.25, 3.37) respectively. From the above-mentioned scores, the potato-chips fried in ground nut oil showed the best results for the sensory evaluation ranking score, where the evaluation of the potato- chips fried in blended oil and Shea butter, gained the second and the third score respectively. This might be due to the fact that, the groundnut oil was already refined oil (where refining process is usually improving the physical properties of oils), where the Shea butter and their blend oil (1:1), were not refined oils. This confirmed the Sensory analysis conducted by Akingbala *et al*., (2006) which indicating that, the unrefined shea butter extracted by traditional method always gained lower scores than refined butters regarding to their colour. Although, refining procedure also causes the loss of some minor substances, but a valuable component such as un-saponifiable fraction with some medicinal properties were existed as reported by Tasan *et al*., (2005); Moharram *et al*., (2006) and Van Hoed *et al*., (2006).

Table (7): The sensory evaluation of potato chips fried in shea butter, groundnut, and their blend oil (1:1).

Parameters	Colour	Flavour	Taste	Texture	Overall acceptability
Shea butter	1.44	1.81	1.94	1.81	1.69
Ground nut	4.88	4.31	4.50	4.*00*	4.69
Blended oil (1:1)	2.88	3.44	3.50	3.25	3.37
G. mean	3.06	3.19	3.31	3.02	3.25

5 = excellent, 4 = very good, 3 = good, 2 = acceptable, 1 = unacceptable

4.7 Shea butter fatty acid profile

Table: (8) which expressing the fatty acid profile of shea butter that contains, oleic, stearic, linoleic, palmitic and archidic acid, the obtained results were (51.1, 40.5, 4.6, 3.4, and 0.40) respectively. These results were within the limits obtained by FAO, (2007), porkarite, (2007) and Leakey, (1999).

According to FAO and CFC (2005), the fatty acid profile is also important in determining the nutritional and physico-chemical characteristics of shea oil. It was noticed that. the five major fatty acids, oleic, stearic, linoleic, palmitic, and arachidic, their values in different extraction methods, were all within values reported by Adikini (2002) and Maranz et al., (2004).

Table (8) the fatty acid profile of shea butter nuts extracted by boiling method

Fatty acid	Concentration mg/100 mg
Oleic	51.10
Searic	40.50
linoleic	04.60
palmitic	03.40
Arachidic	00.40

CHAPTER FIVE

Conclusion and Recommendations

5.1 Conclusion

From the obtained results of this study the following points can be concluded: -

1- The chemical composition of shea oil as potential raw material indicates that, it can be used in food processing as edible vegetable oil especaily after refining.

2- The two different methods of extraction (solvent and boiling) showed a significant difference in their physico-chemical proprieties,

3- The influence of heat treatment (the deep-frying processes) on physico-chemical proprieties of shea butter, groundnut and their blend oil (1:1), showed a significant difference btween each others.

4- The sensory evaluation of potato chips fried in shea butter, groundnut and their blend oil, indicating that, the shea butter and its blend oil were showed less acceptance in their sensory parameters in comperssion with that groundnut oil.

5- The fatty acid profile for shea butter extracted by traditional extraction methods, showed no significant changes in their fatty acid profile, but the increase in oxidative parameters of the traditional boiling method may demonstrate reduction in the quality of shea butter.

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5.2 Recommendations

1- Shea butter seeds contain high yield of oil, there for it is recommended to be used as vegetable edible oil.

2- Refining process is recomended to improve the characteristics of the oil extracted by traditional (boilling method).

3- The deep-frying process has showed a remarkable negative effect on the physico-chemical properties of oils, there for it is recommended\ to minimize deep-frying cyciles to one or two times to save human health.

4- To improve the sensory characteristics of the fried products in shea oil, it is recommended to use refined shea oil.

5- There is a need to investigate cause of changes in physicochemical characteristic of shea butter in traditional boiling methods, as well as their shelf life as a way of promoting the trade of shea butter in Sudan.

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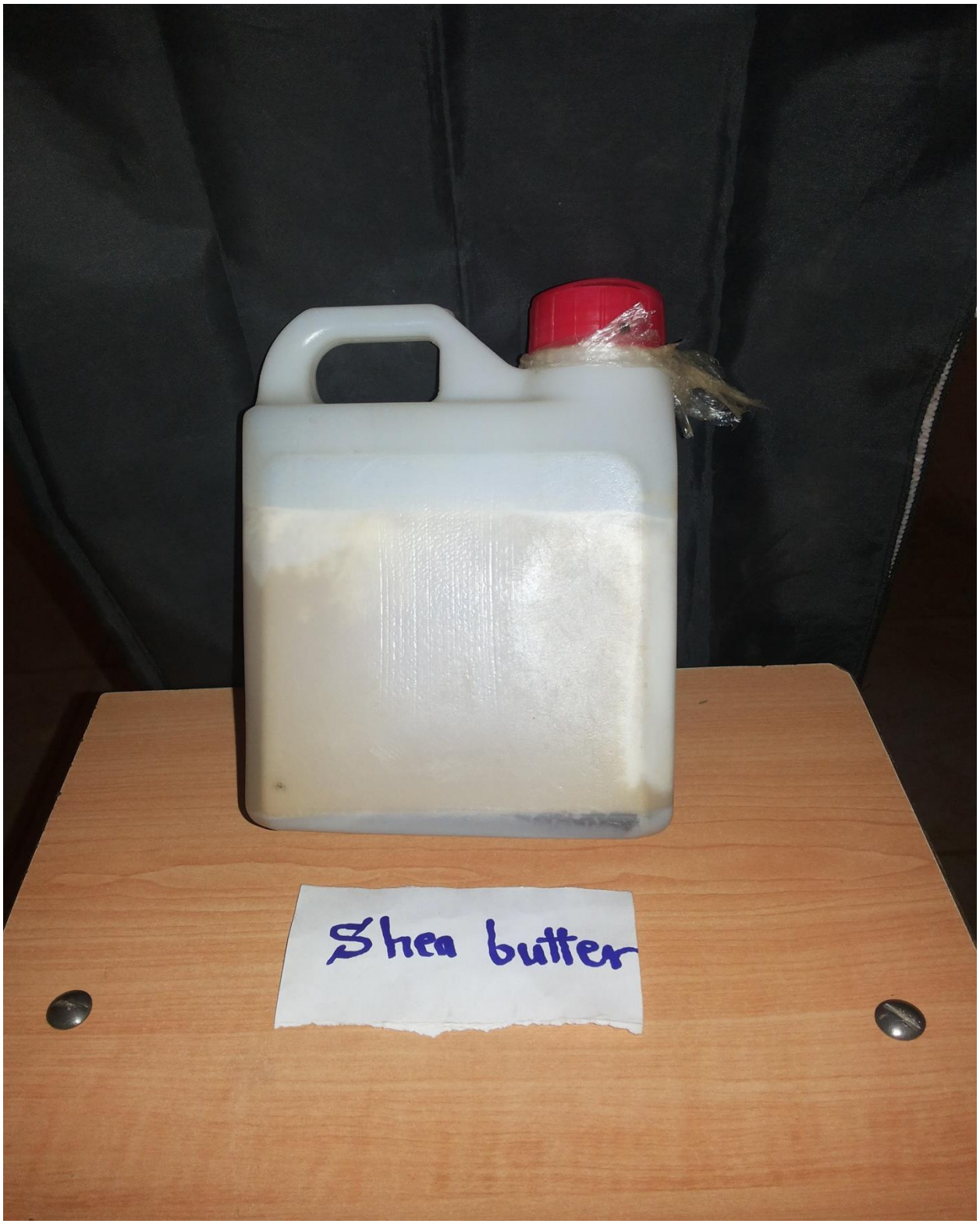
Appendex

Shea seeds

Shea butter



Fatty acid profile -- Shea butter