PHYSICOCHEMICAL CHARACTERISTICS OF CRUDE AND DEHYDRATED CASTOR OIL FROM LOCAL WILD PLANTS (*Ricinus communis*) FROM FIVE SELECTED REGIONS IN KENYA

By

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DECLARATION

This thesis is my own original work and has not been submitted for examination in any other University.

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DEDICATION

I dedicate this work to my beloved wife Njeri Mbitu and my sons Njoroge Mbitu and Ndacha Mbitu for their inspiration and enduring love during this study.

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ABSTRACT

Castor oil have a unique fatty acid i.e. C $_{18:1}$; 12-hydroxy-9-cis-octadecenoic acid which has influenced its applications. The oil has myriad uses in the manufacture of medicines, cosmetics, lubricants, paints and recently in bio fuels. In Kenya currently, no commercial cultivation and extraction of castor oil is being done to harness the economic potential in this plant. This necessitated analysis of the oil yields and the quality of the oil obtained from the local plants prior to promoting their cultivation. Castor oil seeds were collected from Rumuruti in Laikipia county, Naivasha in Nakuru county, Limuru in Kiambu county, Maragua in Murang'a county and Kibwezi in Makueni County. Solvent extraction and cold pressing was used to extract the oil. The percentage moisture content of the seeds ranged from 4.8 ± 0.2 to 6.0 ± 0.2. Percentage oil yields were found to range from 43.07 ± 1.58 to 51.44 ± 1.02. Other physicochemical parameters determined, averaged and compared for both solvent extracted and cold pressed oil were; specific gravity (0.9697 ± 0.0085 for both solvent extracted and cold pressed), refractive index (1.471 ± 0.0009 for solvent extracted and 1.472 for cold pressed), flash point in ^{0}C (260 ± 11 for solvent extracted and 272 ± 3 for cold pressed). Kinematic viscosity in cSt at 40 ^{0}C (I72.76 ± 5.99 for solvent extracted and 193.93 ± 10.01 for cold pressed), acid value (ranged from 5.35 ± 0.15 to 19.38 ± 0.37 for solvent extracted and 1.17 ± to 10.24 ± 0.09 for cold pressed), saponification value (180.60 ±1.12 mgKOH/g for solvent extracted and 182.21 ± 4.26 mgKOH/g for cold pressed). Iodine value (86.21 ± 0.92 for solvent extracted and 83.53 ± 0.69 for cold pressed), percentage unsaponifiable matter (0.78 ± 0.08 for solvent extracted and 0.14 ± 0.04 for cold pressed) and the Lovibond color number (0.5 ± 0.05 for solvent extracted and 0.2 for cold pressed).

All the parameters tested met the requirements of KS 03-799: 1989 'Specification for castor oil for cosmetic industry' except for specific gravity and acid values which had slight deviations.

Both the solvent and cold press crude castor oils were dehydrated after neutralization. The iodine value for the dehydrated oils was found to rise to 140.71 ± 0.92 for solvent extracted and 137.44 ± 1.67 for cold pressed while the kinematic viscosity decreased to 156.98 ± 1.10 for solvent extracted and 161.52 ± 0.58 for cold pressed. The set to touch time for both the solvent and cold extracted dehydrated oil was found to be 36 hours with a drying time of three days. The oils kinematic viscosity and iodine value were found to meet the ASTM D961-86 requirements for oil used in the coatings industry.

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ABBREVIATIONS AND ACRONYMS

ACP	Acyl carrier protein
ASTM	American Society for Testing and Materials
ATP	Adenosine triphosphate
β	Beta
Br_2	Bromine gas
BS	British Standard
C	Carbon
C-C	Carbon carbon bond
Cl_2	Chlorine gas
cSt	Centistoke
CO_2	Carbon (iv) Oxide
CoA	Co enzyme A
Enz	Enzyme
GC	Gas chromatography
H	Hydrogen
HOMO	Highest occupied molecular orbital
HSA	Hydro stearic acid
H_2S0_4	Sulphuric acid
I_2	Iodine
IUPAC	International union of pure and applied chemistry
I.V	Iodine value
KS	Kenya standard

KOH	Potassium Hydroxide
LUMO	Lowest unoccupied molecular orbital
ml	Milliliters
N	Normal
Na	Sodium
NADPH	Nicotine amide adenine dinucleotide phosphate hydrate
NaOH	Sodium hydroxide
^0C	Degrees centigrade
OH	Hydroxyl
PCl_5	Phosphorus pentachloride
$POCl_3$	Phosphorus trichlorate
RCA	Ricinus communis agglutinin
R.I	Refractive index
S	Sulphur
S.G	Specific gravity
R	Long organic group
%	Percentage

INTRODUCTION

1.1 The Plant

The castor oil plant, *Ricinus communis*, is a species of flowering plants in the purge family, Euphorbiaceae. It has green, reddish to purple stems and finger–like leaves (Figure 1.1). Some cases of greyish green and whitish stems have also been observed. In the wild, castor plant can reach up to 9 meters, but cultivated varieties generally grow to between 1-4 meters. Dwarf-hybrid varieties grow to an average height of between 0.9 to 1.5 m, compared to 1.8 to 3.7 m for normal varieties. The plant is a fast –growing, suckering perennial shrub. The plants are usually found in Kenya growing wildly with several varieties being observed.

The castor seed is the source of castor oil, which has a wide variety of uses. The seeds contain between 40 %-60 % oil that is rich in triglycerides, mainly ricinolein [Vashit and Ahmad, 2011].The seeds also contain toxins e.g. proteins called ricin. These toxins are also present in lower concentrations throughout the plant.

Figure 1.1: A farmer showing the castor oil plant *Ricinus cummunis* with mature seeds [World Agroforestry Research Report, 2009].

1.2 The castor oil

Castor oil is one of the few naturally occurring triglycerides that approach being a pure compound, since the fatty acid portion is nearly nine-tenths ricinoleic [Bagali *et al*, 2010].

Crude castor oil is pale yellow, viscous and generally odorless liquid. The oil is an ancient product that has been in use for thousands of years as lamp oil, unguents, medicines and more recently, for a long list of industrial applications [World Agroforestry Research Report, 2009]. Castor oil’s high molecular weight, low melting point, low solidification point and extremely high viscosity, makes it one of the most variable industrial oil. Relative to other vegetable oils, it has a good shelf life and does not turn rancid unless subjected to excessive heat [Ogunniyi, 2006]. Castor oil has over 700 uses, from medicines and cosmetics, to plastics and other applications to biofuel.

The average yield in the six largest castor producing countries in the world in 2007 was 401 kilograms per acre. Irrigated castor reported to yield between 800 to 1,600 Kilograms per acre [World Agroforestry Research Report, 2009]. The same research also reported oil content of the seeds ranging from 35 to 55 %.

1.3 The chemical structure of the castor oil

Like any other vegetable oil and animal fats, castor oil is a triglyceride, which chemically is a glycerol molecule with each of its three hydroxyl groups esterified with a long chain fatty acid (Figure1.2). Its major fatty acid is the unsaturated ricinoleic acid. Ricinoleic is an 18-carbon fatty acid with a single cis double bond between C-9 and C-10. The fatty acid has a hydroxyl (OH) group at carbon 12 (Figure1.3). It is among the few natural oils with this fatty acid structure [Bagali *et al*, 2010]. It is estimated that the fatty acid composition of a typical castor oil contain about 87 % of ricinoleic acid [Akpan *et al,* 2006].

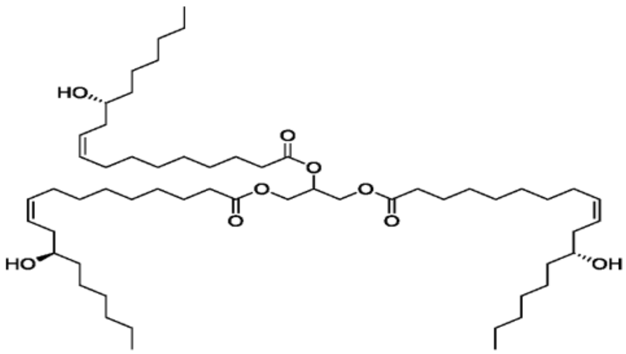


Figure 1.2: Chemical structure of castor oil.

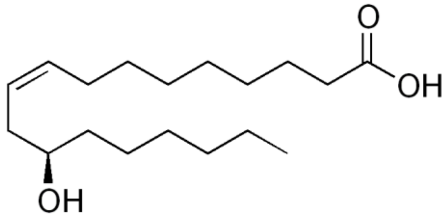


Figure 1.3: Ricinoleic acid (C $_{18:1}$; 12-hydroxy-9-cis-octadecenoic acid)

1.4 Commercial uses of castor oil

Castor oil is claimed to be one of the plant oil industry's most under appreciated asset [Shende *et al*, 2002]. It is one of the most versatile of plant oils, being used in over ten diverse industries. Owing to its unique chemical structure, castor oil can be used as the starting material for producing a wide range of end-products.

1.5 Kenya's castor oil industry

According to the ministry of agriculture's policy document on emerging crops, castor oil plants mostly grow wildly though they have been cultivated in small scale by farmers in some Arid and Semi Arid regions, e.g. in Laikipia County [National Emerging Crops Policy, 2010]. Some farmers use their stems for supporting banana stems. Most of the castor oil being produced is used in bio fuel pilot projects. Although indigenous to Eastern Africa and highly suitable for growing in many parts of Kenya, with an already substantial global markets for use in myriad industrial and pharmaceutical applications, castor has not been grown commercially in Kenya since the 1970s [World Agroforestry Research Report, 2009]. Data on total imports of castor oil during the period between year 2005 and 2011 is shown in Table 1.1.

Table 1.1: Kenya’s castor oil import between year 2005 and 2011

YEAR	**2005**	**2006**	**2007**	**2008**	**2009**	**2010**	**2011**
Total castor oil imports in Kg	1,055,136	428,311	587,149	250,572	28,956	66,566	57,944

Table courtesy of Kenya National Bureau of Statistics, 2013

1.6 Major world castor oil producers

India is by far the largest producer and exporter of castor oil, followed by China and Brazil. However, many other countries especially from South East Asia, Africa and South America are showing significant interest in being large scale producers of castor crop, castor oil and castor chemicals in future [Castor Oil Report Preview, 2010]. Table 1.2 shows a brief summary of the castor oil economies in the year 2007.

Table 1.2: World castor oil producing countries in the year 2007

Castor Yield, Production (2007) and Producer Price (2006) from six largest producing countries.

Country	Yield (Kgs / Acre)	Area Harvested (Acres)	Production (Tonnes)	Producer Price (US$ / tonne)
India	496	2,124,200	1,053,603	377
China	387	543,400	210,296	325
Brazil	280	403,929	113,100	207
Ethiopia	419	35,815	15,006	246
Paraguay	486	24,700	12,004	153
Thailand	338	32,446	10,968	234

[World Agroforestry Research Report, 2009]

1.7 Problem statement

The government of Kenya is committed to actions that will make the agricultural sector vibrant [National Emerging Crops Policy, 2010] as well as make Kenya a middle income country by the year 2030 [Kenya's Vision 2030]. One of the key opportunities is the cultivation of new and emerging crops. The government is in the process of putting measures to develop and promote these crops to address food insecurity and poverty in areas where there is potential for their production. One of the crops that have been identified is the *Ricinus communis* commonly called castor oil plant [National Emerging Crops Policy, 2010]. Due to the recent developments in the international and domestic markets, emerging crops have generated great interest among farmers and other stakeholders because of their enhanced economic value. The potential for growing these crops is high but still unexploited due to various challenges. These include uncertainty of market outlets, inadequate staff and low farmers' knowledge. In order to address uncertainty in market outlets, it is important to evaluate the quality of the castor oil produced in specific geographical areas against specifications of oil used in the manufacture of specific products and in this case for cosmetics and coatings such as paints. No comprehensive studies have been done to evaluate the quality of castor oil from Kenya for applications in these industries.

1.8 Overall objective

To determine the percentage yields and physicochemical characteristics of both crude and dehydrated castor oil from wild plants from selected five regions in Kenya for manufacture of cosmetics and paints.

1.9 Specific objectives

1. To determine the percentage yield of oil from castor seeds collected from selected five regions in Kenya.
2. To determine the physicochemical characteristics of crude castor oil and compare the results with those specified for castor oil used for manufacture of cosmetics.

3. To neutralize, bleach and dehydrate the extracted crude castor oil.
4. To determine the physicochemical characteristics of the neutralized, bleached and dehydrated castor oil and compare the results with those specified for castor oil used for coatings synthesis.
5. To compare the physicochemical characteristics of solvent and cold press extracted castor oil.

1.10 Justification.

Castor oil is one of the most versatile of plant oils, being used in over ten diverse industries [Castor Oil Review Report, 2010]. Owing to its unique chemical structure, castor oil has been used as the starting material for producing a wide range of end products including cosmetics and paints. The plant itself has been shown to require less fertilizers, pesticides, water and maintenance than most crops. The castor plants in Kenya grow wildly and can be observed thriving even in disturbed, marginal and wastelands [National Emerging Crop Policy, 2010]. However, its commercial cultivation and exploitation is still limited. Kenya imports a substantial amount of high-quality castor oil per year [World Agroforestry Research Report, 2009] not to mention the amounts of edible oils it would free from use in paint industry. It is therefore envisaged that through this final product focused research, the cultivation of the castor plant will be rejuvenated and promoted, hence complying with Kenya's Vision 2030. It is also expected that the results of this study will offer informed decisions to policy makers and private investors.

2.1 Hypothesis

The following were the hypotheses that were proposed before the start of this research.

1. The quality of castor oil depends on the geographical location where the castor plant was grown.
2. Castor oil obtained from wild castor plants growing in Kenya is unsuitable for use in cosmetic and coatings industries.
3. The quality of the oil extracted from castor seeds is not influenced by the method of extraction.

LITERATURE REVIEW

2.1 The castor plant and seed.

Ricinus communis is a species that belongs to the Euphorbiaceae family and it is commonly known as castor oil plant. This plant originates in Africa but it is found in both wild and cultivated state in all the tropical and subtropical countries of the world [Barajas, 2006].

Castor seeds are nearly flattened and oval, but differ in size and color. Seeds found in Kenya and Nigeria have been reported to weigh on the average 59.3 and 61.3 g per 100 seeds [Mensah and Ochran, 2005].

In wild condition, castor plant is well adapted to arid conditions and is able to stand long periods of drought [Barajas, 2006].Castor plant can present precocious, medium and delayed cycles. Precocious cycle is one where flowering occurs about 45 days after sowing. The medium cycle presents flowering at an intermediate time between the precocious and delayed cycles, which has a flowering time of 90 to 120 days after sowing.

The seed contains ricin, ricinine and certain allergens that are toxic [Ogunniyi, 2006]. Ricin is also present in lower concentrations throughout the plant. The toxins provide the castor oil plant with some degree of natural protection from pests, such as aphids [Vashit and Ahmad, 2011]. The castor plant is known to tolerate varying weather conditions. It requires a temperature of between 20 and 26 ^{0}C with low humidity throughout the growing season in order to obtain maximum yields. This condition favors most tropical regions of the developing countries.

2.2 Castor oil and physicochemical analysis data

Castor oil, also known as ricinus oil, is a pale-amber viscous liquid extracted from the seeds of castor plant. Castor oil is one of the few naturally occurring fatty oils that approach being a pure compound [Mensah and Ochran, 2005].

The fatty acid portion of the oil comprises about 80 to 90 per cent ricinoleic acid [Hui, 1996].The oil is characterized by a high viscosity owing to the hydrogen bonding of its hydroxyl group.

The chemistry of castor oil is centered on its high content of ricinoleic acid and the three points of functionality existing in the molecule. The carboxyl group, the unsaturated double bond and the hydroxyl group offer highest occupied molecular orbitals (HOMO) and lowest unoccupied molecular orbitals (LUMO) centers of reactions. Some of the reactions that castor oil can undergo are summarized in the Table 2.1.

Table 2.1: Various reactions of castor oil

	Location in molecule	**Nature of reaction**	**Added reactant**	**Type of product**
1.	Ester linkage	Hydrolysis	Acid, enzyme, or Twitchell reagent catalyst	Fatty acid and glycerol
		Esterifications	Monohydric alcohols	Esters
		Alcoholysis	Glycerol, glycols, penterythriotol and other compounds	Mono- and Diglycerides, Monoglycols
		Saponification	Alkalies, alkalies plus metallic salts	Soluble soaps, Insoluble soaps
		Reduction	Na reduction	Alcohols
		Amidation	Alkyl amine, alkanolamines, and other compounds	Amine salts, amides.
2	Double bond	Oxidation, Polymerization	Heat, oxygen, crosslink agent	Polymerized oils
		Hydrogenation	Hydrogen (moderate pressure)	Hydroxystearates
		Epoxidation	Hydrogen peroxides	Epoxidized oils
		Halogenation	Cl_2, Br_2, I_2	Halogenated oils

		Addition reactions	S, Maleic acid	Polymerized oils, factice
		Sulphonation	H_2SO_4	Sulphonated oils
3	Hydroxyl group	Dehydration, hydrolysis, distillation	Catalyst plus heat	Dehydrated castor oil, Octadecadienoic acid
		Caustic fusion	NaOH	Sebacic acid, capryl alcohol.
		Pyrolysis	High heat	Undecylenic acid, heptaldehyde
		Halogenation	PCl_5, $POCl_3$	Halogenated castor oils
		Alkoxylation	Ethylene and / or propylene oxide	Alkoxylated castor oils
		Esterification	Acetic-, phosphoric-, maleic-phthalic anhydrides	Alkyl and alkylaryl esters, phosphate esters.
		Urethane reactions	Isocyanates	Urethane polymers
		Sulphonation	H_2SO_4	Sulphated castor oil (Turkey red oil)

[Ogunniyi, 2006]

Castor oil can be extracted using a number of processes or a combination of processes. Hydrate presses and continuous screw presses are common. Heating the seeds before pressing, at about 100^0C helps to coagulate the proteins including the poisonous ricin. A number of solvents have been shown from previous studies to be good extractors of castor oil. These solvents include hexane, diethyl ether and petroleum ether. However, the most satisfactory approach is hot pressing using a hydraulic press followed by solvent extraction [Akpan *et al*, 2006].

There have been several studies into the physicochemical properties of castor seed oil from various countries. For example, Castor seeds obtained from Myammar were extracted and the crude oil was found to have free fatty acid value of 19, specific gravity of 0.9633 and an iodine value of 89.5 [Hlaing and Mya, 2008]. The dehydrated oil had an iodine value of 140, kinematic viscosity of 160 cSt, set to touch time of 4 hours and a drying time of 5 days. Castor seeds obtained from local wild plants in Ghana were cold pressed to extract the oil. The oils were found to have an iodine value of 82, saponification value of 177 mgKOH/g, acid value of 2.7, refractive index of 1.477 at 25^0C , a specific gravity of 0.962 and a hydroxyl value of 180 [Ochran and Mensah ,2005]. The physicochemical characteristics of castor oil from plants growing in Greece were analyzed with the intentions of formulating a methyl ester of the fatty acid for use as a bio fuel. Using Gas Chromatography (GC) it was observed that the major constituent of the fatty acids was ricinoleic acid (12-hydroxy cis octadec-9-enoic acid) at 89.3 %. The other fatty acids identified were as shown in Table 2.2.

Table 2.2: Fatty acid composition of castor seed oil

Fatty acid	**Chemical structure**	**Weight %**
Ricinoleic	$CH_3\ (CH_2)_5\ CH(OH)\ CH_2CHCH(CH_2)_7\ COOH$	89.3
Linoleic	$CH_3\ (CH_2)_3\ (CH_2CHCH)_2\ (\ CH_2)_7COOH$	4.20
Oleic	$CH_3\ (CH_2)_7CHCH(CH_2)_7COOH$	2.30
Stearic	$CH_3\ (CH_2)_{16}\ COOH$	1.00
Palmitic	$CH_3\ (CH_2)_{14}\ COOH$	1.00
Dihydroxystearic	$CH_3\ (CH_2)_7CH(OH)CH(OH)(CH_2)_7CO_2H$	0.70
Linolenic	$CH_3\ (CH_2CHCH)_3\ (CH_2)_7\ COOH$	0.30
Densipolic	$CH_3CH_2CHCH(CH_2)_2CH(OH)CH_2CHCH(CH_2)_7COOH$	0.93
Eicosanoic	$CH_3\ (CH_2)_{18}COOH$	0.30

[Deligiannis *et al*, 2009]

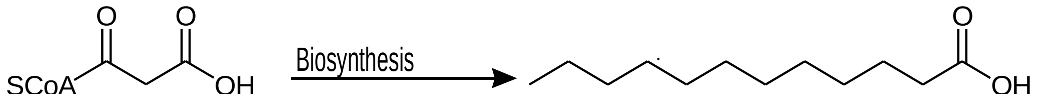
The castor oil also had a kinematic viscosity at 40 ^{0}C of 240.12 cSt, a specific gravity of 0.967 at 15 ^{0}C, flash point above 260 ^{0}C, acid value of 0.7, saponification value of 181.4 mg KOH/g and an iodine value of

84.4. Castor oil from castor seeds collected from the Esa Oke town in the western part of Osun State of Nigeria were extracted by the use of solvent and on analysis, the moisture content was found to be 0.30 ± 0.01, specific gravity of 0.948 ± 0.02, refractive index at 30 ^{0}C of 1.792 ±0.01, flash point of 225.0±2.1, saponification value of 178.0 ±0.42 mg KOH/g, iodine value of 58.39±0.71 and an average oil yield of 48.0 % [Abitogun *et al*, 2009].

2.3 Fatty Acids: biosynthesis.

Fatty acids, esterified with glycerol, are the main constituents of oils and fats. Storage fats (seed oils and animal adipose tissue) consist chiefly of about 98 % triglycerides. Animal fats contain a high proportion of glycerides of saturated fatty acids and tend to be solids, whilst those from plants and fish contain predominantly unsaturated fatty acid esters and tend to be liquids. Fatty acids are alkanoic acids and the majority occurring fatty acids have straight chains possessing an even number of carbon atoms. Biologically, fatty acids are a rich source of stored energy. When metabolized, they yield high amounts of ATP, water and carbon IV oxide. The process of fats breakdown is called β-oxidation. In this process the fatty acid is broken down, two carbon atoms at a time into acetyl coenzyme A.

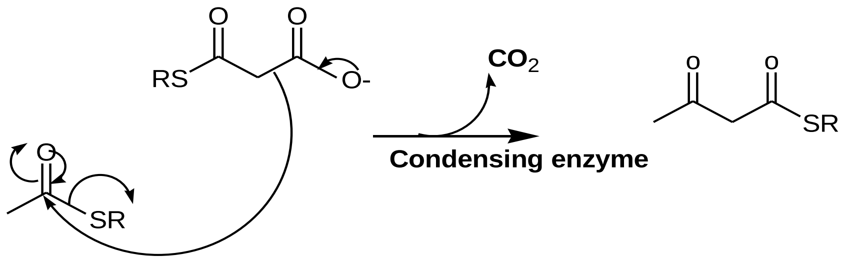
The prevalence of fatty acids with even numbers of carbon atoms suggests a two-carbon building block, the most obvious being acetate. If acetate is ‘fed’ to plants, the fatty acid emerges shown in reaction 1.



.........**Reaction 1**

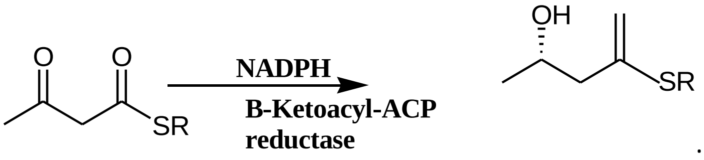
Acetyl CoA **Fatty acid**

However, the reaction is more complex than this since CO_2 is also needed and it turns out that only the first two carbon units is put in as acetyl CoA. The remainder is added as malonyl CoA. Malonyl CoA is made from acetyl CoA and CO_2 which is usually carried by the molecule biotin. The first stage in the fatty acid biosynthesis is a condensation between acetyl CoA (The starter unit) and malonyl CoA with the loss of CO_2. This is shown in reaction 2. One will note that CO_2 is lost as a new C-C bond is formed.

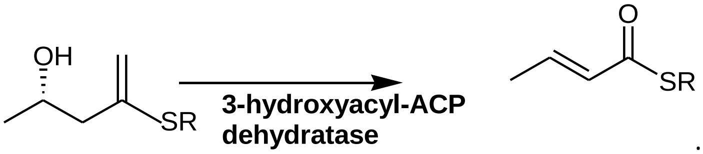


........................**Reaction 2**

The next step is reduction of the ketone group as shown in reaction 3. This NADPH catalysed reaction is stereo and chemoselective. Dehydration of the molecule also occurs through an enzyme catalyzed mechanism as shown in reaction 4 [Clayden *et al*, 2001].

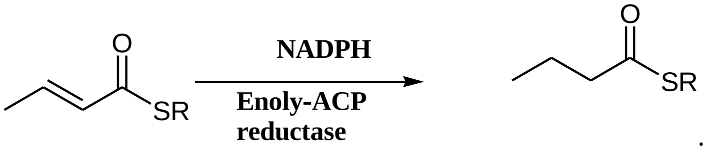


....**Reaction 3**



....**Reaction 4**

The elimination is known to be a cis removal of H and OH and the double bond is exclusively trans (E). Finally, in this cycle, the double bond is reduced using another molecule of NADPH to give the saturated side chain as shown in reaction 5.

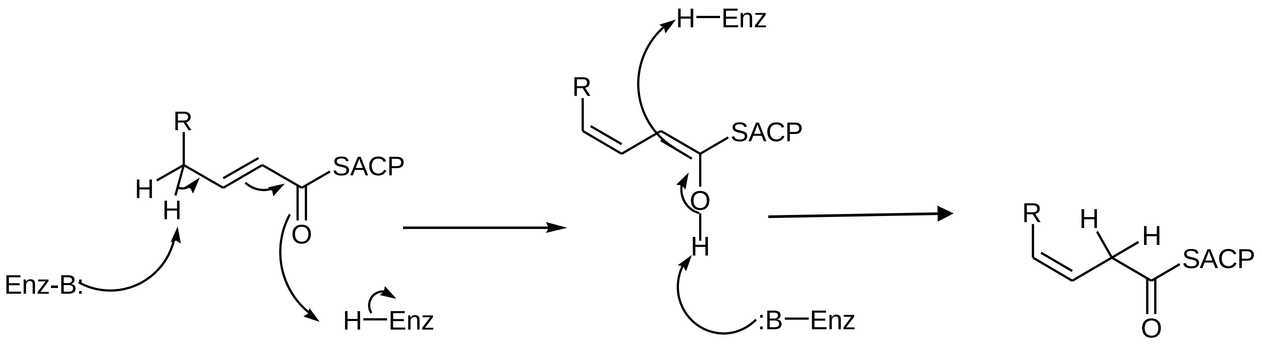


....**Reaction 5**

The whole cycle starts again using this newly made C4 fatty acid as the starter unit and building a C6 fatty acid by incorporating another malonyl CoA. Each time the cycle turn, two carbon atoms are added to the acyl end of the growing chain.

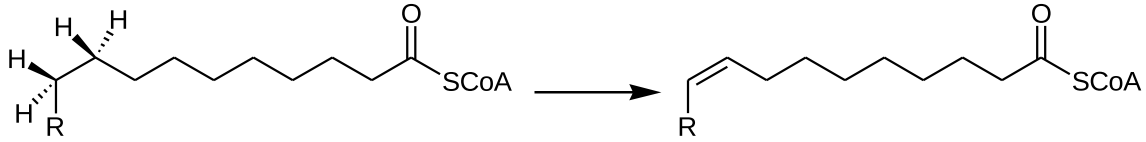
Conjugated unsaturated fatty acids are made by stopping the acylation cycle and hydrolyzing the thiol ester linkage between the unsaturated acyl chain and Acyl Carrier Protein (ACP). They always have the E (Trans) configuration. However, in most living things, unsaturated fatty acids have the Z (cis) configuration on the double bonds. This structure has biological importance since it hinders the molecules from ‘fitting’ into each other thus lowering their melting point. The formation of cis double bond in biological systems is postulated to happen using any of the following two mechanisms.

(i) Anaerobic route: The fact that unsaturated fatty acids are synthesized in the absence of oxygen is apparent from their wide spread occurrence in anaerobic bacteria. However, only monounsaturated fatty acids are synthesized [Clayden *et al*, 2001]. Dehydrogenation occurs during chain elongation. An example is shown in reaction 6



...**Reaction 6**

(ii) Aerobic route: This process involves a concerted stereo-specific removal of two adjacent hydrogen atoms from the chain of a fatty acid after synthesis. Oxygen is required in this process. Mammals e.g., man are believed to synthesize such fatty acids using this method.



.......**Reaction 7**

2.4 The industrial applications of castor oil and its derivatives

Castor oil has over 700 industrial uses and because of this, its demand keeps on increasing [Bagali *et al*, 2010]. Owing to the unique chemical structure of castor oil, it is used as a raw material in the production of many technical, medical and different industrial applications. Esterified castor fatty acids have been used as a biodiesel [Vashit and Ahmad, 2011] and its properties compared with the esterified oil obtained from Jatropha seeds.

Castor oil derivatives are used in the production of paints, varnishes, lacquers and other protective coatings, lubricants and grease, hydraulic fluids, soaps, printing ink, linoleum, oil cloth and as a raw material in the manufacturing of various chemicals e.g. sebacic acid and undecylenic acid which are used in the production of plasticizer and Nylon fabric [Bagali *et al*, 2010].

Some of the products and derivatives of castor oil are listed in Table 2.3.

Table 2.3: Products and derivatives of castor oil

a) Industrial castor oil
b) First special grade castor oil
c) Cold pressed castor oil
d) Pale pressed castor oil
e) Pharma grade castor oil
f) Hydrogenated castor oil
g) Sulfonated castor oil
h) Blown castor oil
i) Ricinoleic acid
j) 12-HSA
k) Methyl 12-HSA
l) Sebacic Acid
m) Undecylenic Acid
n) Methyl Ricinoleate
o) Methyl Undecylenate
p) 2-Octanol

It should be noted that one of the oils that has been earmarked for use as a biofuel is castor oil. A lot of research not only in Kenya but World over has been done on the use of castor oil as a bio fuel. Some of these researches include *Biodiesel from castor oil: A promising fuel for cold weather* [Barajas, 2006], *A comparative study of castor and Jatropha oil source and methyl ester test on the Diesel Engine* [Vashist and Ahmad, 2011] among others.

A number of companies are doing cutting edge research on the use of castor oil to produce bio plastics and biopolymers such as polyurethanes, nylon and more. With increasing use of bio lubricants and bio greases, castor oil is emerging as the preferred feedstock choice [Castor Oil Review Report, 2010]. Castor oil has an advantage over other mineral oils in that it is biodegradable, eco-friendly and a renewable resource [Belaid *et al*, 2000].

2.5 Castor oil uses in cosmetics industry

Castor oil is used extensively in the cosmetic industry. When castor oil is hydrogenated, hydrogenated castor oil is produced. This is a wax-like product with a melting point of 86^0C. Hydrogenated castor oil is used in cosmetics, hair dressings, ointments and in the preparation of Hydrostearic acid (HSA) and its derivatives [Ogunniyi, 2006].

Undecylenic acid, a castor oil derivative is an approved over the counter drug for skin disorders. Castor oil penetrates deep into the skin. Because of the OH, the crude castor oil is able to trap water thus preventing skin water loss. This makes castor oil a good emollient [Poucher, 1993]. Because of the non-drying nature of castor oil, it is widely used in lipstick formulations. The most characteristic single ingredient of lipstick base formulation is castor oil [Poucher, 1993]. In lipstick, it exhibits a number of desirable properties. These include, being soluble in alcohols, good wetting properties and its high viscosity makes it easy to apply.

According to data obtained from Multi Commodity Exchange of India Ltd, the main consuming segments of castor oil are paints 45%, soaps, detergents and cosmetics 30% while the others i.e. medicine, lubricants and polymer fibers consume the rest [MCX of India Report, 2008].

2.6 Castor oil uses in coatings synthesis

Castor oil and its derivatives had very limited outlet in the paints and varnish industry since it is not a drying oil [Radlove, 1948]. However in 1937, a synthetic drying oil derived from dehydrated castor oil made its appearance on the market. The growth and use of this new oil has been nothing short of phenomenal and today dehydrated castor oil ranks as one of the most important drying oils.

Castor oil has only one double bond in each fatty acid and so is classified as non-drying oil. However, it can be dehydrated to give drying oil used extensively in paints and vanishes [Castor Oil Report, 2010].
A drying oil is a vegetable oil which dries by oxidation. Linseed oils, palm oil and sunflower oils are mostly used in paints manufacture because they easily dry to form a durable film. However, these oils are also edible oils. This means that there is a competition for these oils as human food and as industrial raw materials. Castor oil has been used in paints, coatings and inks for many years and has quite a number of advantages as a raw material [Oldring and Hayward, 1993]. Castor oil is obtained from a naturally occurring renewable resource and non food source unlike palm and sunflower oils. As such, it is readily available, relatively inexpensive and environmentally friendly. Basco Paints Ltd manufacturers of Duracoat ^{TM} among other brands procure approximate 300 tonnes of sunflower oil per annum for paints manufacture. In Kenya, if one was to supply high quality dehydrated castor oil; the demand would be hard to meet [Obiero, 2013].

During the drying period, oxygen molecules react with the hydrocarbon chain to form free radicals which cause polymerization leading to ‘drying’ of the oil. Chemically, dying oils are water-insoluble, unsaturated glycerides of long chain fatty acids. Drying oils also contains saturated glycerides of fatty acids, but these do not participate in drying or polymerization of the oil.

Drying oils can be classified in many ways, but one principle way is to divide them into drying, semi drying and non drying [Koleske, 1972]. The property of drying is related to the level of unsaturation and thus related to the iodine value. Table 2.3 shows this relationship.

Table 2.4.Classification of dying oils by iodine values

OIL CLASS	IODINE VALUE
Drying oil	≥140
Semi drying oil	124-139
Non drying oil	≤125

[Koleske, 1972]

Similarly, the position of the double bond in the fatty acid has been shown to affect the drying of fatty acids. Conjugated double bonds are more susceptible to auto-oxidation and thus faster drying time.

Castor oil’s disadvantages include having only one double bond in each fatty acid moiety. Having only one double bond in each fatty acid chain mean that castor oil is appropriately classified as non-drying oil. As a result, coatings that incorporate castor oil alone will never achieve complete cure through cross linking as do coatings that contain oils with multiple bonds in each fatty acid moiety [Trevino and Trumbo, 2002]. This short-coming is often addressed by dehydrating the castor oil. Dehydration transforms the castor oil into a drying oil which improves the properties of films made with it. In 1914, Fokin made a systematic study of the dehydration of ricinoleic acid, recognizing the principle of dehydration to give both 9-11 and 9-12 linoleic acids [Radlove *et al*, 1948].

In the laboratory, before dehydration, the castor oil is first neutralized with the use of an alkali e.g. NaOH. Using a separating funnel and solution of sodium chloride (for salting out), the oil upper layer is separated from the soapy lower part. Various catalysts have been recommended for the dehydration of castor oil and a list of such catalysts is available [Sivasamban, 1954]. One of the common recommended catalysts is sodium bisulphate. However, previous studies done with this catalyst have shown many practical difficulties. The chief defects of the dehydrated castor obtained by the use of this catalyst are its dark color, high acid value and the after-tack of the film on drying.

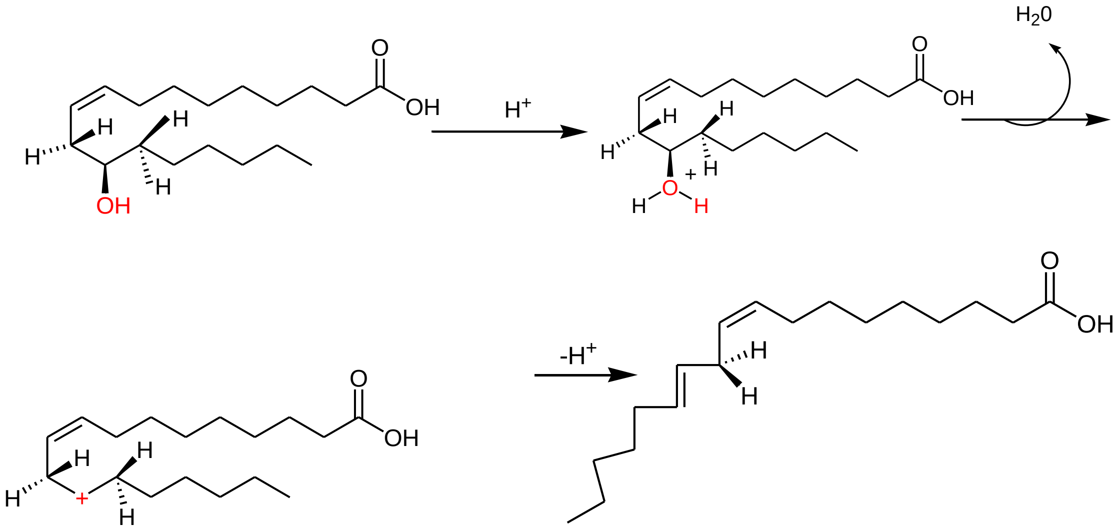
Polymerization is one of the most important industrial processes. Resins and emulsions are two main classes of polymers. Dehydrated castor oil is used in the manufacture of alkyd resins used in the coating's industry. Dehydrated castor oil is now recognized as an individual drying oil with its own properties and advantages. The drying oils owe their value as raw materials for decorative and protective coatings to their ability to polymerize or 'dry' after they have been applied to a surface to form tough, adherent, impervious and abrasion resistant films. The advantages claimed in a good surface coating application include excellent odor, heat stability, good drying properties, more uniform polymer structure and lack of "after-yellowing". The dehydrated castor oil is non-yellowing and so gives a better coating [Hlaing and Mya, 2008].

2.6.1 Alkyd Resins

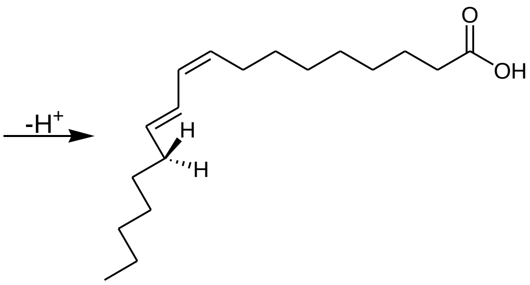
It is estimated that alkyd resins contribute about 70 % of the conventional binders used in surface coating today [Hlaing and Mya, 2008]. Alkyd resins are used in both clear and pigmented, industrial and trade coatings to protect and decorate a wide variety of substrates. The paint industry envisages a future expansion in view of development in automobile industry, utility in nuclear power stations, development in corrosion resistant coatings, expansion in housing activity and other industry uses. The demand for alkyd resins being an ingredient in paint, varnish and printing would be linked to these industries. An alkyd resin is a polyester modified by the addition of fatty acids and other components. The inclusion of fatty acids confers a tendency to form flexible coatings. Alkyds are used in paints and in moulds for castings. They are the dominant resin or binder in most commercial oil based coatings. The popularity of alkyd resins as vehicles for coatings is largely due to their properties such as film hardness, durability gloss and gloss retention and resistance to abrasion impacted on them through modification with drying oil [Hlaing and Mya, 2008]. Castor oil is also useful directly in protective coatings as a plasticizer in alkyd systems, and blown castor oil is an important nitrocellulose plasticizer. Crown Berger Ltd imports about 10 tonnes of blown castor oil per year [Obiero, 2013].

2.6.2 Dehydration reaction mechanism

During the dehydration process, the hydroxyl group of ricinoleic acid is replaced by a double bond. The acid catalyzed mechanism proceeds through the initial protonation of the hydroxyl group, resulting in a hydroxonium ion that subsequently loses water to form a carbonium ion. Owing to the availability of a proton from either side of the neighboring carbon atoms, different products are formed, these products are the nonconjugated linoleic acid and the conjugated linoleic acid. This is based on the position of the newly formed double bond in relation to the existing double bond. Conjugated linoleic acids have attracted much attention recently because of their proposed anticarcinogenic and anticholesterolemic activities [Ramamurthi, 1998]. The dehydration reaction mechanism is shown in reaction scheme 2.1.



Non conjugated or



Conjugated

Reaction scheme 2.1

The catalyst sodium hydrogen sulphate ionizes into Na^+ and $HSO_4{}^-$ and forms sulphuric acid and sodium hydroxide along with the liberated water molecule. This causes the lowering of effective concentration of the catalyst, sodium hydrogen sulphate [Nezihe *et al*, 2011]. The mechanism is shown in reaction scheme 2.2.

$$NaHSO_4 \longrightarrow Na^+ + HSO_4^-$$

$$2H_2O \longrightarrow H_3O^+ + OH^-$$

$$HSO_4^- + H_3O^+ \longrightarrow H_2SO_4 + H_2O$$

$$Na^+ + OH^- \longrightarrow NaOH$$

$$2NaOH + H_2SO_4 \longrightarrow Na_2SO_4 + 2H_20$$

$$NaOH + H_2S0_4 \longrightarrow NaHSO_4 + H_2O$$

Reaction scheme 2.2

2.7 Medicinal properties of castor oil

Castor oil has a long history of its use as a drug. The United States of America Food and Drug Administration (FDA) categorize castor oil as safe and effective for over the counter use as a laxative. Castor oil derivatives e.g., polyethoxylated castor oil called Kolliphor TM has been used as an additive in most drugs formulations.

2.8 Poisonous nature of castor seeds

The castor seeds are poisonous to people, animals and insects. One of the main toxic proteins is ricin. Ricin was named by Stillmark in 1888 when he tested the bean extracts on red blood cells and saw them agglutinate [Wiley and Oeltmann, 1991]. However, it was later realized that the agglutination was actually caused by another toxin called Ricinus communis agglutinin (RCA). Ricin is a potent cytotoxin but a weak hemagglutinin, whereas RCA is a weak cytotoxin and a powerful hemagglutinin.

Poisoning by ingestion of the castor bean is due to ricin, not RCA, this is because RCA does not penetrate the intestinal wall and does not affect red blood cells unless given intravenously. If RCA is injected into the blood, it will cause red blood cells to agglutinate and burst by haemolysis.

If the seed is swallowed without chewing, and there is no damage to the seed coat, it will most likely pass harmlessly through the digestive track. However, if it is chewed or broken and then swallowed, the toxin will be absorbed by the intestines. Ricin is contained in the seed pulp (cake) following the separation of the oil from the bean. No ricin remains in the oil and it is inactivated during extraction if done under heated condition.

Interestingly though, during this study it was observed that doves (bird) like and eat castor seeds.

Report on ricin content of a castor bean vary, but is probably in the range of 1-5 %.

However, with population awareness on the dangers of mis-handling the castor seeds, such dangers can be avoided.

2.8.1 Ricin and its activity

Many plants contain poisonous substances that have attracted the attention of both the physicians who have frequently found the sparing use of plant toxins to be therapeutically beneficial as well as criminals who have tended to use the same for harmful activities. Ricin fits readily into this classification. Purified ricin is a white powder that is soluble in water and stable over a wide range of pH values. Several scientists working in Norway showed that the toxicity of ricin is due to its catalytic action on eukaryotic ribosomes

[Lord *et al*, 1994]. Ribosomes are cell organelles where protein synthesis occurs. The ricin enzymatic activity renders the ribosome unable to synthesis proteins, this consequently prevent cell replication leading to cell death. The molecular nature of ricin allows it to bind to the mammalian cell surface and enter through endocytic uptake and deliver the catalytically active polypeptide into the cell cytosol. Due to its activity, ricin and its associated compounds are being investigated as possible anti-cancer chemotherapies [Bradberry *et al*, 2003]. Consequently, ricin was used in the world war as a chemical weapon and today it is rated as a very potent warfare.

2.9 Physicochemical properties of oils

2.9.1 Introduction

The extraction of oils from seeds is by one or a combination of mechanical pressing and solvent extraction. In mechanical pressing, the seeds are crushed and then adjusted to low moisture content by warming. Thereafter, the crushed seeds are loaded into hydraulic presses and they are pressed by mechanical means to extract the oil. However, mechanical pressing will only remove about 45 % of the oil present [Ogunniyi, 2006]. The remaining oil in the cake can only be removed by solvent extraction. In the solvent extraction method, the crushed seeds are extracted with a solvent in a Soxhlet extractor or commercial extractor. Solvents used for extraction include heptane, hexane, petroleum ether, diethyl ether among others. The solvent chosen is influenced by the polarity of the oil, ease of recovery, safety and its price.

2.9.2 Purification of castor oil

The oil obtained by mechanical expelling or solvent extraction is termed ‘crude oil’. It contains a number of impurities. Some of the impurities are small seed fragments and meal fines which are easily removed by filtration. Other impurities include free fatty acids, hydrocarbons, ketones, tocopherols, glycolipids, phytosterols, phospholipids, proteins, pigments and resins. All these form colloidal suspensions in the oil. Most of these impurities have unfavorable effects on the flavor, odor, appearance and shelf life of the oil.

This necessitates the removal of these impurities in a process called degumming. There are various ways of degumming including use of water and acids [Zufarov *et al*, 2008]. Bleaching is a procedure in the processing of oils. It involves the removal of compounds that brings about color to a substance. Various methods are employed. Use of activated clay and charcoal have been cited and used [Akpan *et al*, 2006]. Castor oil has high viscosity and because of this, it cannot pass through most fine filter papers. In the laboratory, it is filtered when hot with the help of cotton wool.

2.9.3 Kinematic viscosity

Viscosity is a measure of the resistance of a fluid to flow under some force. It is used for establishing identity or detecting adulteration in fatty oils because most commonly used oils, except castor oil have viscosities that do not differ much from each other [Kamau, 1993]. In drying oil industries, the heat-bodying, blowing and other methods for modifying oils cause large changes in viscosity. Viscosity is hereby used as a test for process control. In design of pumps, piping and other process equipments, viscosity of the oils is a very important factor to consider.

Kinematic viscosity is a measure of the time for a fixed volume of liquid to flow by gravity through a capillary. In the petroleum industry, kinematic viscosity is usually expressed in centistokes, cSt, where 1 St = 100 cSt. Kinematic viscosity can be determined using the U-tube viscometer. This device is also known as glass capillary viscometers or Ostwald viscometers, named after Wilhelm Ostwald. Another version is the Ubbelohde viscometer, which consists of a U-shaped glass tube held vertically in a controlled temperature bath. In one arm of the U is a vertical section of precise narrow bore (the capillary). Above this is a bulb; with it is another bulb lower down on the other arm. In use, liquid is drawn into the upper bulb by suction, and then allowed to flow down through the capillary into the lower bulb. Two marks (one above and one below the upper bulb) indicate a known volume. The time taken for the level of the liquid to pass between these marks is proportional to the kinematic viscosity [IP Standards for Petroleum and its

Products, 1973]. Most commercial viscometers are provided with a conversion factor, or can be calibrated by a fluid of known properties. The time required for the test liquid to flow through a capillary of a known diameter of a certain factor between two marked points is measured. By multiplying the time taken by the factor of the viscometer, the kinematic viscosity is obtained.

2.9.4 Specific gravity

The density of fats and oils at temperatures which they will be stored, handled and processed need to be known for design of tanks, piping and processing equipment for these materials. Beside the obvious utility in chemical engineering, data on density or specific gravities of oils have been used in connection with analysis and identification of these oils [Eckey, 1954].

2.9.5 Iodine value

The iodine value (I.V) is a reflection of the degree of unsaturation in an oil sample. Many methods for determining I.V have been devised [Kamau, 1993]. The results are commonly expressed as percent iodine absorbed, i.e., grams of iodine per 100 grams of sample, whether or not the halogen used is actually iodine. Three methods can be used; these are Wijs, Hanus and Hubl. The methods of Wijs and Hanus are recommended for industrial and commercial analysis [IUPAC, 1979]. The Hubl method is still official in some countries (except for commercial transactions). The statement of results must mention the method used, as different methods give different figures for the same oil [IUPAC, 1979].

2.9.6 Acid value

Fatty acids in oils and fats occur in either as free fatty acids or as esters, where they are bound to the triglycerol molecule. Acid value therefore, is an indication of the amount of free fatty acids present in the sample. Results of this determination are often expressed as free fatty acids, as percent oleic, percent palmitic or percent lauric acid in the sample, depending on the kind of oil or fat being handled. This relationship is shown in Table 2.4. Expressing results as acid value is preferable, since it involves no assumptions concerning composition of the acid present. Free fatty acids as oleic acid is numerically approximately half the acid value [Akpan *et al*, 2006].

Table 2.5: Various oils and fats and how they are expressed.

Nature of fat or oil	**Expressed as**	**Molecular weight**
Coconut, palm kernel and similar oils	Lauric acid	200
Palm oil	Palmitic acid	256
Castor oil	Ricinoleic acid	298
All other oils	Oleic acid	282

[IUPAC, 1979]

There are two methods used in determination of free fatty acid or acid values. These are the indicator method and the potentiometric method which is mostly used for highly colored oils and fats.

2.9.7 Saponification value

Saponification value represents the number of milligrams of potassium hydroxide required to saponify 1 gram of fat or oil under the conditions specified. It is a measure of the average molecular weight of all the fatty acids present. As most of the mass of a fat or oil is in the three fatty acids, it allow for comparison of the average fatty acid chain length. The long chain fatty acids found in fats have a low saponification value because they have fewer numbers of carboxylic functional groups per unit mass of the fat as compared to short chain fatty acids. If more moles of the base are required to saponify a small amount of fat or oil, then there are more moles of the fat or oil with fatty acids of short chain length.

2.9.8 Unsaponifiable matter

The unsaponifiable matter of oils comprises the substances which are soluble in oils but which, after saponification, are insoluble in water and soluble in the solvent used for the determination. It includes the naturally occurring unsaponifiable substances (e.g. sterols, alcohols, hydrocarbons) as well as the foreign organic unsaponifiable substances [BS 242:1969]. These substances influence the quality and shelf life of oils.

2.9.9 Refractive index

Refractive power of fats and waxes varies somewhat widely and is chiefly governed by the proportion and degree of unsaturated matter present. In fat industries, it provides a most rapid means of following the progress of hydrogenation of fat or oil [Kamau, 1993]. Refractive index of a medium is the ratio of the speed of light at a definite wavelength in a vacuum to its speed in the medium. The refractive index of a given substance varies with the wavelength of light and with the temperature. Generally, the speed of light in air is taken in the place of the speed in a vacuum [IUPAC, 1979]. Unless specified, the wavelength chosen is that of D-lines of sodium i.e. 589.6 nm.

2.9.10 Flash point

Flash point is a measure of the oil flammability. It is used primarily to determine the safety precautions necessary for transport and storage. Vegetable oils generally have flash points much higher than that of mineral oils and thus provide an advantage in terms of safety. Accordingly, flash point can be used to determine the purity of vegetable oils [World Agroforestry Research Report, 2009].

2.9.11 Lovibond color

Color is an important indication of product composition, purity and degree of deterioration [Yew *et al*, 2006]. In the case of vegetable oils e.g. castor oil, each type of oil has its own color specification. Conventional methods of color measurements are by visual comparative techniques.

2.9.12 Set to touch time of drying oils

The set to touch time is a physical test done by the use of the finger. This is specified by ASTM Method D 1640, Drying, Curing, or Film Formation of organic coatings at room temperature. The directions include expressions such as ‘touch lightly’, ‘feel sticky’ and ‘firm pressure’ are used. However, these terminologies may mean different things to different people [Sward, 1972].Thus, another relatively simple method of evaluating the dryness of a film estimation of the adherence or non adherence of sand or paper to the film under various loadings. Glass beads, typewriter paper can also be used [Sward, 1972]. Both methods can be used to determine the set to touch time of the dehydrated castor oil.

METHODOLOGY

3.1 Sampling area.

Five Kenyan counties were selected as areas where wild castor oil plants seems to blossom and can be cultivated extensively. The semi-arid regions in these counties were selected as best sites for sample collection. These areas are shown in Table 3.1. The plant was characterized at the East African Herbarium as *Ricinus communis* L. A copy of the identification certificate is attached at the Appendix.

Table 3.1: Counties where samples were collected

	COUNTY	AREA SELECTED
1.	Nakuru	Naivasha
2.	Laikipia	Rumuruti
3.	Murang'a	Maragwa
4.	Makueni	Kibwezi
5.	Kiambu	Ndeiya (Limuru)

3.1.1 Naivasha sub county in Nakuru County.

The area is located in the Rift valley part of Kenya and about 100 KMs east of Nairobi city. It is bounded by latitudes 0^0 30 ' S to 1^0 00' S and by longitudes 36^0 00' E and 36^0 30' E. The climate is semi arid with less than 200 mm of rainfall per year [Thompson and Dodson, 1963]. The area is sparsely populated mainly due to less productivity. Mara ngishu Location is in Naivasha where the samples were collected. Figure 3.1 shows the location of Mara ngishu.

3.1.2 Rumuruti sub county of Laikipia County

Laikipia County lies between latitudes 0^0 17 S, 0^0 45 N and longitudes 36^0 15'E, 37 ^{0}20' E. It occupies an area of approximately 9,666 KM^2. Rumuruti is one of the sub county of Laikipia County located between longitude 36^0 12 '17" to 36^0 45 '16" E and latitude 0^0 28'51" N and 0^0 7'28" S with altitudes ranging from 1780 to 1835 meters above sea level. The soils in this area are classified as red brown sandy clay loam lovi soils which are fertile and suitable for forest and crop production (Ojwang' *et al*, 2010).The average annual rainfall varies from 400 mm to 750 mm with most part experiencing semi arid conditions. Rumuruti receives less than 500 mm of rainfall annually (Mwita, 2013). The Figure 3.2 shows the location of Rumuruti.

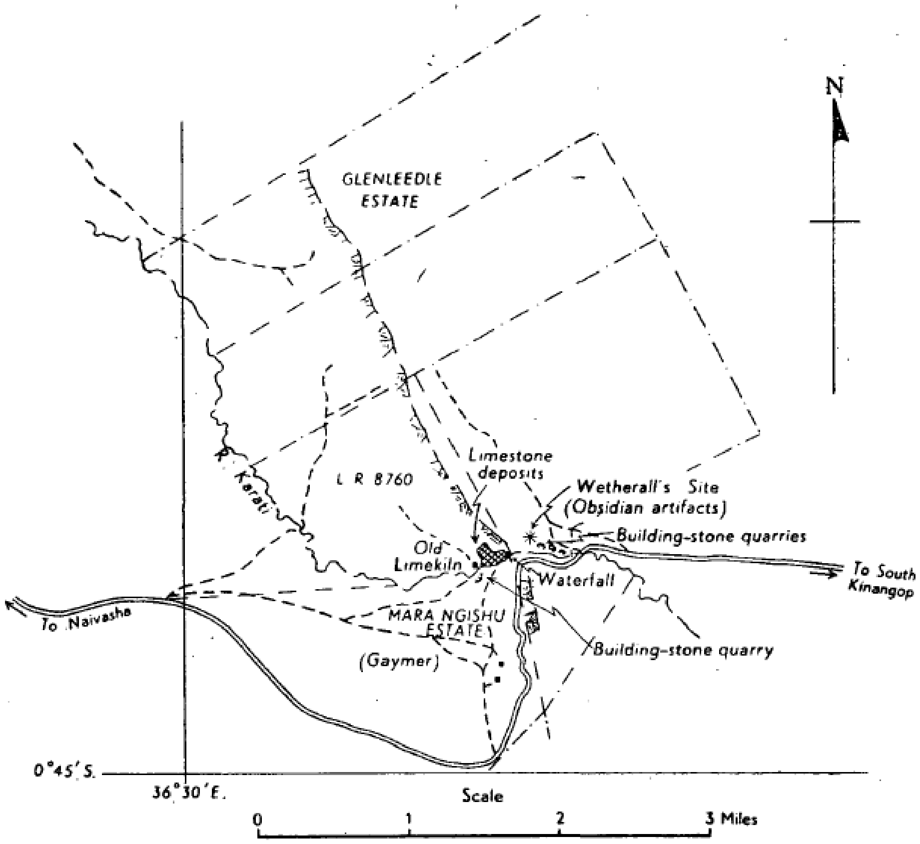


Figure 3.1: Map of Mara Ngishu in Naivasha where the samples were collected [Thompson, 1962].

3.1.3 Maragua sub county of Murang'a County

Murang'a county is located on co-ordinates 0^0 43' 0" South and 37^0 9' 0" East. Maragua area experiences dry conditions especially the area around Makuyu [Murang'a East District Landslide Report, 2009]. Figure 3.3 is a map of Murang'a County showing the location of Maragua.

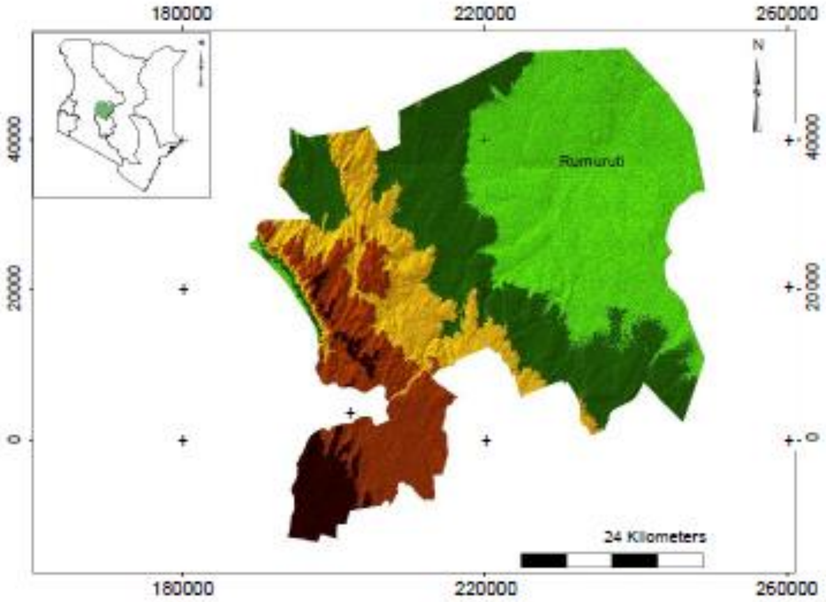


Figure 3.2: The location of Rumuruti [Mwati, 2013]

Figure 3.3: The location of Maragua in Murang’a County [Murang’a East District Landslide Report, 2009]

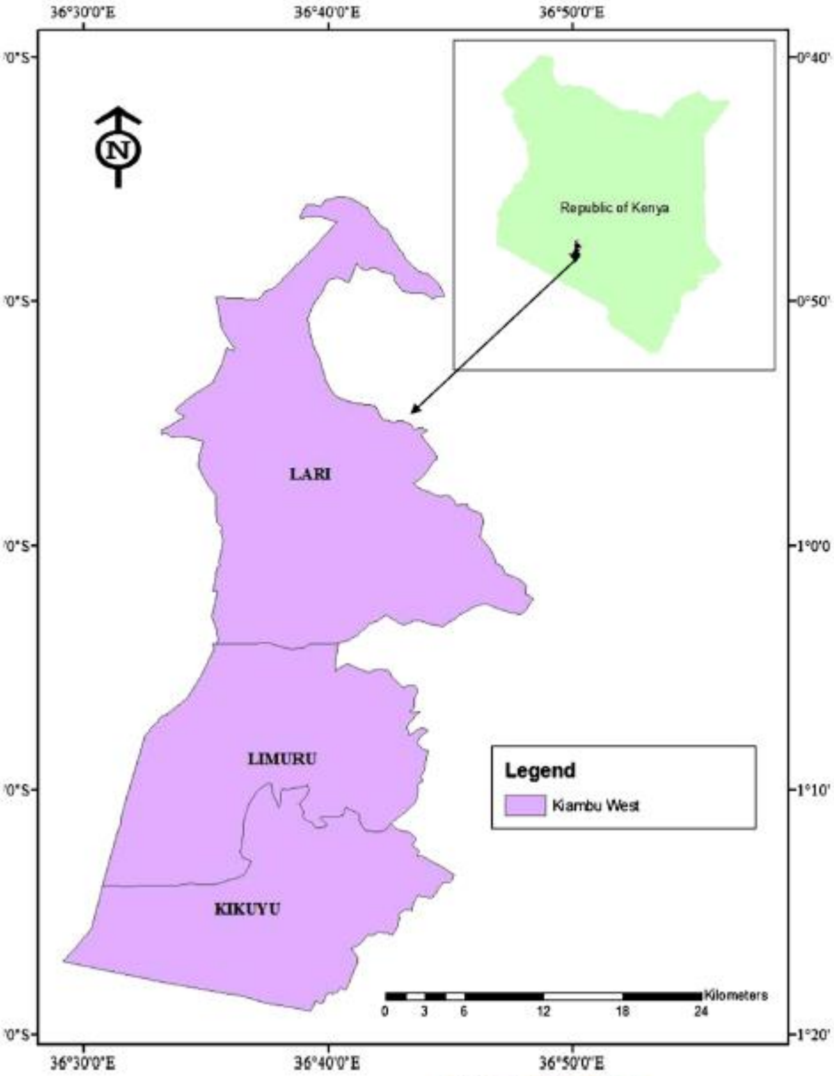
3.1.4 Kibwezi sub county of Makueni County

Kibwezi is located at the southern tip of Makueni County in Kenya. It lies between the latitudes 2^0 6' S and 3^0 S and longitudes 37^0 36' E and 38^0 30' E respectively. The altitude varies from 600 to 1,100 meters above sea level. Climate is typical semi arid with low and unreliable rainfall [Kibwezi-Kiboko observatory baseline survey, 2011]. Figure 3.4 shows the location of Kibwezi.

Figure 3.4: The location of Kibwezi sub-county [Integrated Smart Survey Report, 2011]

3.1.5 Limuru Division of Kiambu County

Kiambu county covers an area of 1, 207.4 KM^{2}. It lies between latitude 0^{0} 75 and 1^{0} 20’ south of the Equator and longitudes 36^{0} 54’ and 36^{0} 85’ East. Ndeiya is one of the Locations in Limuru Division of Kiambu west Sub County as shown in Figure 3.5. Ndeiya and Karai Divisions have high incidences of malnutrition and food insecurity due to the arid conditions of the area [Wabwoba and Wakhungu, 2013]. The area receives less than 500 mm of rainfall annually. Figures 3.6 to 3.10 shows different typical seeds collected from the selected areas while Figure 3.11 shows one of the stages in sample preparation.



source : Researcher, 2011

Figure 3.5: Location of Limuru [Wabwoba and Wakhungu, 2013]

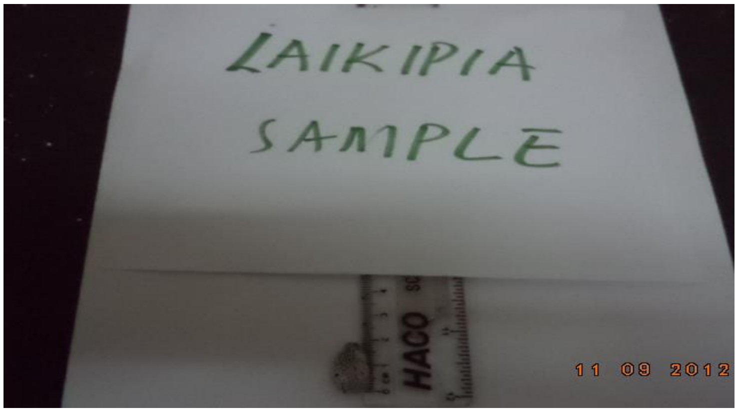


Figure 3.6: A sample of castor seeds collected from Laikipia.

Figure 3.7: A sample of castor seeds collected from Naivasha.

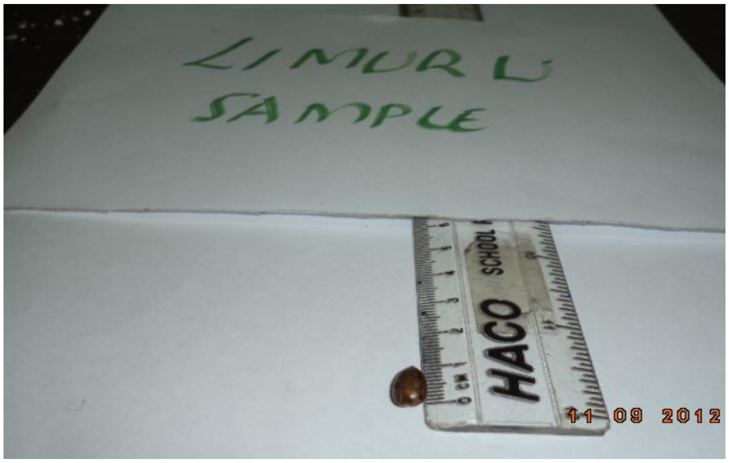


Figure 3.8: A sample of castor seeds collected from Limuru.

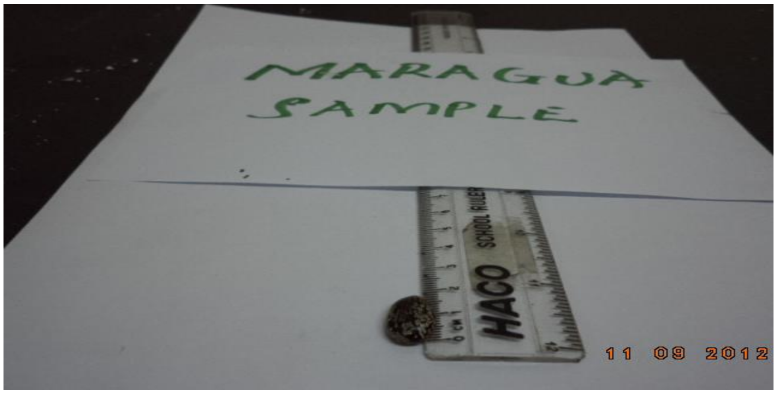


Figure 3.9: A sample of castor seeds collected from Maragua.

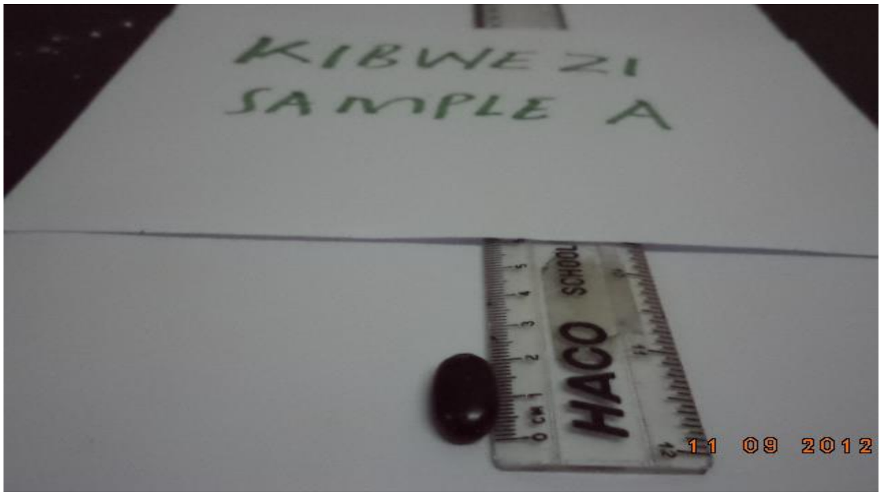


Figure 3.10: A sample of castor seeds collected from Kibwezi.

Figure 3.11: Spreading the castor seeds to air dry in the laboratory.

3.2 Raw material preparation

The ripe castor fruit were hand plucked off from their attachment. The fruits were sun dried in the open until the casing split and shed the seeds. The seeds were further dried in the oven at 60^{o}C for seven hours to reduce their moisture content. The seeds were ground using a pestle and mortar to increase the surface area and weaken the cell walls to release castor fat for extraction.

3.3 Determination of moisture and volatile matter content of the seeds

Approximately 50.0 g of the ground seeds sample were weighed and dried in the oven at 105°C for 8 hours and the weight taken after every 2 hours. After each 2 hours, the sample was removed from the oven and put in a dessicator to cool for 30 minutes. It was then weighed. The percentage moisture and volatile matter content of the seeds was calculated using the following formula:

$$\text{Moisture, \% m/m} = \frac{\text{Weight of seeds before drying (g)} - \text{Weight of seeds after drying (g)}}{\text{Weight of seeds before drying (g)}} \times 100 \ldots\ldots\ldots\ldots \textbf{(\textit{Eqn 1})}$$

3.4 Extraction

3.4.1 Solvent extraction and determination of oil content

The Soxhlet method was used. The weight of flask was noted as M_1. About 10 g of the ground castor seeds was accurately weighed (M_2) and put in the thimble. The thimble was placed in the extraction apparatus. 100 ml of diethyl ether were poured into the flask. The flask was fitted into the extraction apparatus and heated using water bath on a heating mantle as shown in Figure 3.12.

Figure 3.12: Extraction of castor oil using the soxhlet extractor in a water bath.

The heating mantle was set at a rate to ensure that the solvent in the flask boiled gently.

After extracting for 4 hours, the flask was allowed to cool; the thimble and the extractor were dismantled. The thimble was allowed to dry in the open. The thimble content (cake) was poured out and inspected for any remaining oil in the sample. If any, the cake was returned to the thimble and extraction continued. The solvent in the flask was removed by distillation on a boiling water bath. The flask was further heated in the oven at 105 ^{O}C for about 20 minutes and allowed to cool in the dessicator and then weighed. The flask was heated again in the oven for the same period then cooled and weighed. This was repeated until the difference between two weighing was less than 0.01 g. The final weight was noted as M_3. The oil content expressed as a percentage by mass of the grounded seeds was calculated using the equation below:-

$$\text{Oil content, \% m/m} = \frac{(M_3 - M_1)}{M_2} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \textbf{(\textit{Eqn 2})}$$

3.4.2 Cold press extraction

The castor seeds were first heated in an oven at 105 ^{O}C for 1 hour. This cooking helped to coagulate proteins and to make extraction efficient [Belaid *et al,* 2000]. The different samples were pressed with a hydraulic press located at the University of Nairobi. The solid impurities in the oil obtained were allowed to settle at the bottom of the beaker before it was decanted then purified by de-gumming and bleaching.

3.4.2.1 De-gumming of the cold pressed oil

The extracted oil was degummed by adding boiling water to the oil in a separating funnel. The mixture was shaken vigorously for two minutes and allowed to stand and separate. The aqueous layer was run off and the procedure repeated three more times to ensure complete removal of the gum [Zufarov, 2008]. The oil was then dried in the oven at 105 ^{0}C.

3.4.2.2 Bleaching and filtration of the castor oil

A weighed amount of the oil was mixed with 2 % m/m activated charcoal and heated at 105 ^{0}C for 20 minutes, the oil was then filtered using cotton wool in a hot oven set at 105 ^{0}C. The weight of final purified castor oil obtained was noted and percentage oil loss calculated.

3.5 Characterization of the crude oil

3.5.1 Determination of kinematic viscosity

The viscosity was measured by a calibrated U-tube viscometer as shown in Figure 3.13. It was fixed to a viscometer holder and inserted in an oil bath maintained at 40 ^{0}C [IP Standard Methods, 1973]. The viscometer was left inserted for two minutes to attain the temperature of the oil bath. The oil sample was warmed to 40 ^{0}C before putting it in the viscometer. A stop watch was used to determine the time taken by the sample to pass between two viscometer marks. Four readings were done for each sample. For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity i. e,

$$V = T X \S \text{ ....} \textbf{(Eqn 3)}$$

Where V is the kinematic viscosity, T is the time in seconds and § is the viscometer factor.

Figure 3.13: Set up for determination of kinematic viscosity

3.5.2 Determination of specific gravity

25ml pycnometers were used [IP Standard Methods, 1973]. They were thoroughly washed and rinsed with distilled water. The pycnometers were dried in an oven then cooled in a dessicator. Distilled water was added to the pycnometer and then the stopper added. It was then put in a dessicator connected to a vacuum pump where all the air was sucked out. The water was then refilled. The pycnometer was then put in a water bath at 30 ^{0}C for 30 minutes after which it was thoroughly wiped and the weight taken. The water was then poured off and the same procedures repeated for the sample castor oil. The specific gravity (S.G) was calculated as follows;

$$S.G = (W_1-W_0)/(W_2-W_O)\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\textbf{(\textit{Eqn 4})}$$

Where: -

W_1 Weight of pyconometer with castor oil in grams.

W_0 Weight of empty pyconometer

W_2 Weight of pyconometer with distilled water in grams

3.5.3 Determination of iodine value

This is the measure of the number of grams of iodine absorbed per 100 g of oil and denotes the degree of unsaturation. There are three main methods of iodine value determination. These include the Wij's and the Hanus method [IUPAC, 1979]. The method specified by IUPAC was used.

Reagents:

3.5.3.1 Preparation of Wij's reagent

7.8 grams of iodine trichloride was weighed and put into a flask of 1500 ml capacity. A mixture of 700 ml acetic acid and 300 ml carbon tetrachloride was added to the iodine trichloride and swirled.

The halogen content in the resulting mixture was determined as follows:-

5 ml of this solution was taken in a conical flask, 5 ml of a solution of potassium iodide (100g/l) and 30 ml of water was added. This solution was titrated with 0.1 N sodium thiosulphate in the presence of three drops of starch indicator. This volume was noted as 10.2 ml.

To the iodine trichloride solution, 10 g of powdered iodine was added and dissolved by shaking. Halogen content was determined as previously done. The new titer volume was noted to be 15.4 ml i.e. one and a half times the original titer volume. The solution was allowed to stand for 24 hours and then decanted into a brown bottle.

3.5.3.2 Preparation of 0.1N sodium thiosulphate

Standard 0.1 N sodium thiosulphate was prepared by dissolving the content of an ampoules prepared by Sigma-Aldrich ^{TM} into a 1000 ml volumetric flask and filling to the mark with distilled water.

3.5.3.3 Preparation of starch solution

1.0 g of soluble starch solution was dissolved in 100 ml distilled water, boiled then cooled.

Procedures for determination of the iodine value;

0.2 g of the sample was weighed into a conical flask and 20 ml of carbon tetrachloride was added to dissolve the oil. Then 25 ml of Wij's reagent was added to the flask using a safety pipette in a fume chamber. The mixture was stopped and vigorously swirled. The flask was placed in the dark for 1 hour. After this, 20ml of 10% aqueous potassium iodide and 125ml of water was added. The content was titrated with standard 0.1 N sodiumthiosulphate solutions until the yellow color almost disappeared. A few drops of 1% starch solution indicator was added and titration continued by adding sodiumthiosulphate solution drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test and other samples.

The iodine value (I.V) was calculated using the following expression:

$$I.V = 12.69\,C\,(V_1 - V_2)/M \qquad \textbf{(\textit{Eqn 5})}$$

Where;

I.V- Iodine value

C-concentration of sodium thiosulphate used in Normality.

V_1-Volume of sodium thiosulphate used for blank in ml.

V_2-Volume of sodium thiosulphate used for determination in ml.

M- Mass of sample in grams.

3.5.4 Determination of acid value

The acid value is defined as the number of milligrams of potassium hydroxide required to neutralize 1 g of the sample [BS 242: 1969].

2g to 10 g of the sample was weighed to the nearest 0.01g. 50 ml of ethanol toluene (1:1) mixture was added to the sample and shaken until a solution formed. The solution was titrated with 0.1N ethanolic potassium hydroxide solution in the presence of phenolphthalein as indicator. The 0.1 N ethanolic potassium hydroxide was prepared from an ampoules prepared by Sigma-Aldrich ^{TM}.

The acid value was calculated as follows:

Acid Value= 56.1 TN/M ……………………………………………………………(***Eqn 6)***

Where; T- Volume of ethanolic potassium hydroxide solution in ml

N- Normality of the ethanolic potassium hydroxide solution.

M- Mass of the sample in g.

3.5.6 Determination of saponification value

Saponification value is the number of milligrams of potassium hydroxide required to neutralize the fatty acids resulting from the complete hydrolysis of 1 gram of the oil sample.

It is inversely proportional to the mean molecular weight of the fatty acids present in the oil. The indicator method specified by IUPAC was used [IUPAC, 1979]. 2 grams of the sample was weighed into a conical flask; 25 ml of 0.5N ethanolic potassium hydroxide was added. The content was constantly stirred using boiling chips while being boiled and refluxed gently on a hot plate for 60 minutes. Few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5N HCl to the end point i.e. until the pink color of the indicator just disappears.

The same procedure was used for all the other samples and the blank. The expression for saponification value (S.V) was calculated as follows;

S.V =56.1 N (V_0-V_1)/M **……………………………………………………………………(*Eqn 7)***

Where;

N- Actual concentration in Normality of the HCl used.

V_0-The volume of the solution used for blank test in ml.

V_1- The volume of the solution used for the sample in ml.

M- Mass of the sample taken in grams.

3.5.7 Determination of unsaponifiable matter

The determination was done according to IUPAC [IUPAC, 1979], whose procedures are as follows:

5 g of the sample (M_2) was weighed to the nearest 0.01 g. This was put into a 250 ml bottom flask. 50 ml of the 2N ethanolic potassium hydroxide was added. Boiling chips were added to help in stirring. A reflux condenser was connected and the mixture refluxed for one hour. The condenser was detached from flask and the contents transferred to a separating funnel. The flask was rinsed several times with a total of 100 ml of water and the water added into the separating funnel. The flask was also rinsed with a total of 100 ml of diethyl ether and the contents added to the separating funnel. The contents were allowed to cool to between 20^0C and 25^0C, stopped and shaken vigorously for one minute releasing the pressure intermittently. The mixture was allowed to stand until the two layers clearly separated. The soap solution was run off into the flask used for the saponification. The diethyl ether solution was poured through the top of the funnel into a second separating funnel containing 40 ml of water.

Extraction of the soap solution was done two more times in the same manner, each time with 100 ml of diethyl ether, using the first separating funnel, and combining the three extracts in the second separating funnel. The funnel containing the combined extracts and 40 ml water was rotated gently and after settling, the water layer was run off. The diethyl ether solution was washed as follows:

i. Twice with 50 ml of water.
ii. Once with 50 ml of 0.5N potassium hydroxide solution.
iii. Once with 50 ml of water.

The last two washings were repeated and then washed with water until neutral to phenolphthalein.

The diethyl ether solution was transferred quantitatively through the top of the funnel into a weighed round bottom flask, rinsing the funnel with small amounts of diethyl ether. The diethyl ether was evaporated by heating in a water bath, 6 ml of acetone added and the solvent evaporated completely by mean of gentle stream of air while the flask was being held obliquely and almost submerged in a boiling water-bath and rotated. The residue was dried by placing the flask horizontally in a drying oven at 80^0C. The flask and the contents were allowed to cool in a dessicator and weighed (M_1). The drying was repeated at an interval of 15 minutes until the loss between two weights was less than 0.0015 g.

The residue was dissolved in 20 ml of the 95% v/v neutralized ethanol and titrated with 0.1 N ethanolic potassium hydroxide solutions in the presence of phenolphthalein as indicator.

Unsaponifiable matter (U.M), % m/m was calculated as follows:

$$U.M = [M_1 - 0.28VT]/M_2 \times 100 \quad \ldots\ldots\ldots\ldots \textbf{\textit{(Eqn 8)}}$$

Where; M_1- mass of residue in g. V-volume in ml of KOH used

M_2- mass of the sample in g T-Normality of KOH used

3.5.8 Determination of refractive index

Refractive index is the physical attribute of triglyceride measured by the angle through which a beam of light is bent when passing through a thin film of oil. The determination was performed with the use of an Abbe refractometer at 25^0C as shown in Figure 3.10 [Mensah and Ochran, 2005].

Figure 3.14: Determining the refractive index using an Abbe refractometer.

3.5.9 Determination of flash point

Flash point is the temperature at which a heated substance sparks when an open flame is passed near it. The Cleverland open cup method according to ASTM D92 66 was done [IP Standards, 1973]. The test cup was filled to a specific level with the sample. The temperature of the sample was increased rapidly at first and then at a slow constant rate using a hot plate as the flash point was approached. A fixed thermometers' bulb was immersed in the oil but not touching the base of the cup. At specified intervals a small test flame was passed across the cup. The lowest temperature at which application of the test flame causes the vapors above the surface of the liquid almost to ignite was taken as the flash point.

3.5.10 Determination of color by the Lovibond tintometer

The Lovibond tintometer was used to determine the color of the samples. The color of the samples was determined by comparison with a series of red, yellow and blue standard color glasses [IP Standards, 1973]. Light reflected from a white background passed through the cell containing the sample, and thence to the viewing-tube, where it illuminates one half of the field of view. The other half of the field of view was illuminated by light from the background which passed through the color standards. A blue 'daylight' filter was incorporated in the viewing-tube. The color standards were mounted in racks of up to nine slides each, arranged so that the colors could be moved into the field of view as required. The value of the standard or combination of standards which gives the best match was recorded as the color of the sample.

3.6 Neutralization of crude castor oil

100g sample of the oil was heated to 55^0C. To the oil, an equivalent amount of 2N NaOH solution was added to neutralize the free fatty acids. The mixture was stirred as it was being heated at 55^0C. Completion of neutralization was determined by testing the mixture with phenolphthalein indicator [Akpan *et al*, 2006]. When the indicator color of the sample mixture turned to pink, neutralization was deemed to have been completed. Hot sodium chloride solution was added to the mixture to ensure adequate salting or graining out of soapstock. Then the mixture was poured into a separating funnel. After three hours, the lower layer of separated mixture was drained out. The upper layer was washed with hot water. Washing was done until the color of phenolphthalein indicator did not change to pink. After complete washing, the oil was dried at 100^0C in an oven until the weight of dried oil remained unchanged. The neutralized oil was weighed to calculate percentage oil loss as follows:

$$\text{Percentage oil loss} = \frac{\text{weight of beaker and crude oil sample} - \text{weight of beaker and neutralized oil}}{\text{Weight of beaker and crude oil sample}} \text{X100}$$

……………………………….(Eqn 9)

3.7 Dehydration of castor oil

Various catalysts have been recommended for the dehydration of castor oil. One of the common catalysts is sodium hydrogen sulphate ($NaHSO_4$). However, preliminary work done showed that the dehydrated oil became very dark and formed an after tack film on cooling. This however was reduced by adopting the following proposed procedures [Sivasamban *et al,* 1954].

50.34 g of solvent extracted and 50.02 g of cold pressed oil were put in a 100 ml round bottomed flask. The set up was put as shown in Figure 3.15 [Hlaing and Mya, 2008]. The oil was heated using a heating mantle up to a temperature of 220 ^{0}C. The vacuum was disconnected and very fast, 1.0281 g of $NaHSO_4$ (representing about 2 % m/m of the oil) was added to the oil and vacuum restored. The temperature was maintained for 30 minutes. After this, the vacuum was again disconnected and 1.0340 g of CaO (representing 2 % m/m of the oil) was added. Heating was stopped, vacuum was reconnected and the oil was left to cool to room temperatures. This was repeated for the cold pressed oil. The iodine values of the dehydrated oils were determined as follows. As specified in the IUPAC test method for oils with iodine values between 100 and 150, an amount close to 0.13 g was weighed [IUPAC, 1979]. The procedures specified in section 3.5.3 were done to obtain the iodine number while procedures in section 3.5.1 were done to obtain the kinematic viscosity of the dehydrated oil. Set to touch time and drying time of the dehydrated oil was also determined.

Fig 3.15: Laboratory set up for dehydration of castor oil.

3.4 Determination of set to touch time

Both cold press and solvent dehydrated oils were applied on three sets of metal plates. Solvent and cold pressed crude oils were also applied. The plates were exposed in air under the shade. At intervals of two hours, the surface of the plate was touched firmly with the finger. When no oil adhered to the finger after touching, this was taken as the set to touch time [Koleske, 1972].

3.9 Determination of drying time

A drying time tester was used to determine the drying time. The oil was applied using a brush on a metal plate to form a thin film. After every 24 hours, the drying time tester machine (Figure 3.16) was used to test whether the oil film coat had dried. A one kilogram weight was placed on the upper pan. This helps to increase grip between the lower axle covered with a cotton cloth. By switching on from a knob, the axle rotates for 10 seconds and in the process scratching the film on the metal plate due to friction. The plate was inspected for any abrasion. When no oil was scrubbed, the oil was deemed to have dried [Koleske, 1972]. A collection of neutralized crude and dehydrated oil are shown in Figure 3.17

Figure 3.16: Drying time testing machine.

Figure 3.17: Neutralized, crude solvent and cold pressed oil and dehydrated castor oil.

RESULTS AND DISCUSSIONS

4.1 Percentage moisture and volatile matter content

The moisture and volatile matter content of castor seeds is the loss in mass which the product to be analyzed undergoes on heating at 105 ^{O}C. It was reported as a percentage of the ground castor seeds analyzed. This was done by putting a weighed amount of ground castor seeds in a crucible and heating in an oven. The content was removed at a 2 hour intervals, cooled in a dessicator then weighed. The results are shown in Table 4.1.

Table 4.1: Percentage moisture and volatile matter content in castor oil

Name of sample	Moisture and volatile matter content, % m/m	Standard deviation.
Laikipia	6.0	0.2
Naivasha	5.9	0.1
Limuru	5.3	0.3
Maragua	4.8	0.2
Kibwezi	4.9	0.1

The seed moisture and volatile matter content are very vital parameters which influence the seed quality and storage life of the seed. Seed moisture quality also influences seed infestation by pests and pathogens. The results obtained showed the average moisture and volatile matter content ranged from 4.8 to 6.0 %. The average moisture content for all the seeds was 5.38 % m/m. The recommended moisture content for castor seeds is 5 % [Ace Commodity Exchange, 2013]. Earlier work had reported moisture content of 4.15 % [Akpan *et al*, 2006] and 5.8 % [Bagali *et al*, 2010].

4.2 Percentage oil content

Castor oil was taken as the whole of the substances extracted by the solvent from the grounded seeds. It was expressed as a percentage by mass of the seeds. The results obtained are shown in Table 4.2.

Table 4.2: Percentage oil content of the castor seeds samples.

Name of sample	Oil content, % m/m	Standard deviation.
Laikipia	44.53	0.85
Naivasha	51.44	1.05
Limuru	43.07	1.61
Maragua	46.77	1.15
Kibwezi	42.11	0.49

From the results, it can be concluded that the castor oil collected from Naivasha gave the highest yield of 51.44 % while those from Kibwezi had the lowest yield of 42.11 %. The average oil content of all the seeds was 45.58 %. A contract specification quotes a specification of 47 % oil content [Ace Commodity Exchange, 2013]. The oil content of the seeds influences the economic value of the seeds.

Earlier research reported castor oil yields of 53.2 % for castor seeds from Nigeria using normal hexane as the extracting solvent[Akpan *et al*, 2006] and 57 % for castor seeds from Ghana extracted using di-ethyl ether [Mensah and Ochran, 2005]. Castor seeds have been reported to contain on average, oil content of 40 to 60 % by weight [Ogunniyi, 2006]. The oil content in the seeds depends on the seed variety, climatic conditions and whether it grows wild or under cultivation [Mensah and Ochran, 2005].

4.3 Percentage oil loss after purification

100.0014 g of crude degummed cold pressed oil was put in a beaker. 2.2346 grams of activated charcoal (representing approximately 2 % of the oil taken) was added to the oil. The oil was heated in an oven for 30 minutes. After which the oil was filtered using cotton wool fitted in a filter funnel. Three filtrations were done before obtaining the final clear product. The weight of the final product was noted to be 63.0843 g. Percentage loss was calculated to be:

$$(100.0014-63.0843)/100.0014 \times 100 = 36.865, \% \text{ m/m}$$

The amount of oil lost during filtering using cotton wool is massive. This loss is occasioned by the fact that the cotton wool absorbs a lot of the castor oil being filtered.

4.4 Specific gravity

The specific gravity of a substance is defined as the ratio of the mass of a given volume of the substance at a certain temperature to the mass of an equal volume of pure water at the same temperature. The results are shown in Table 4.3.

Table 4.3: Specific gravity of various castor oil samples

Name of sample	**Method of extraction**	**Specific gravity at 30 ^{O}C**
Laikipia	Solvent extraction	0.9741
	Cold press extraction	0.9773
Naivasha	Solvent extraction	0.9689
	Cold press extraction	0.9768
Limuru	Solvent extraction	0.9752
	Cold press extraction	0.9787
Maragua	Solvent extraction	0.9750
	Cold press extraction	0.9783
Kibwezi	Solvent extraction	0.9551
	Cold press extraction	0.9755

From the results, it can be noted that other than the castor oil obtained from Naivasha, the solvent extracted oils had slightly lower specific gravity than the cold press oil. The Kenya standard for castor oil for cosmetic industry KS 03-799:1989 specifies a specific gravity value of 0.954-0.960. An analysis certificate of Naivasha cold pressed oil is attached at the Appendix. A S.G of 0.9587 for solvent extracted has been reported [Akpan *et al*, 2006] and 0.9633 [Hlaing and Mya, 2008]. Others reported a value of 0.962 for cold pressed oil [Mensah and Ochran, 2005]. The results obtained indicate that only the Kibwezi solvent extracted sample meet the requirement of the standard.

4.5 Refractive indexes

The oils in a beaker were incubated in a water bath set at 40 ^{0}C. A small amount of the oil was picked with a 1 ml pipette and applied on the refractometer. Setting was done and the value was read directly from the scale of the Abbe refractometer. The results are shown in Table 4.4. Verification of the results was done by taking the refractive index of distilled water and comparing with the literature value. This value was noted as 1.333

Table 4.4: Refractive indexes of the various castor oil samples at 40 ^{0}C.

Name of Sample	Method of extraction	Refractive index at 40 ^{0}C.
LAIKIPIA	Solvent extracted	**1.472**
	Cold pressed	**1.472**
NAIVASHA	Solvent extracted	**1.470**
	Cold pressed	**1.472**
LIMURU	Solvent extracted	**1.472**
	Cold pressed	**1.472**
MARAGUA	Solvent extracted	**1.471**
	Cold pressed	**1.472**
KIBWEZI	Solvent extracted	**1.472**
	Cold pressed	**1.472**

The results showed that both the cold and solvent extracted oils had similar refractive indexes. The KS 03-799: 1989 Specifies the ideal castor oil for use in cosmetic industry should have a refractive index of between 1.4700-1.4750 at 40^{0}C. This shows that all the samples met the requirement of this specification and therefore can be used in industries in Kenya.

Refractive indexes of 1.477 for cold pressed oil at 25 ^{O}C was reported [Mensah and Ochran, 2005] and 1.469 at 28 ^{0}C [Akpan *et al*, 2006].

4.6 Flash point

Flash point is the temperature at which, when a substance is heated gives off flammable vapors. The results are shown in Table 4.5.

Table 4.5: Flash point of various samples of castor oil

Name of sample	**Method of extraction**	**Flash point in ^{O}C**	**Standard deviation**
Laikipia	Solvent extraction	241	1
	Cold press extraction	276	2
Naivasha	Solvent extraction	265	1
	Cold press extraction	271	2
Limuru	Solvent extraction	263	1
	Cold press extraction	269	1
Maragua	Solvent extraction	262	2
	Cold press extraction	274	1
Kibwezi	Solvent extraction	271	2
	Cold press extraction	272	2

The results indicate that the solvent extracted oils had slightly lower flash points than their corresponding cold pressed oils. Flash point is usually a safety precaution parameter i.e. it is indicative of how fast a certain substance can catch fire.

The KS 03-799: 1989 Specifies a flash point minimum of 127 ^{0}C. This means that all the samples whether extracted using the solvent or by cold pressing were within the specification. The results also show that the castor oil can be heated to generally high temperatures e.g. during dehydration without one worrying about the risks of it catching fire. A flash point of $305^{0}C$ has been reported [Vashist and Ahmad, 2011] and 271 ^{0}C [World Agroforestry Research Report, 2009]. Castor oil from Nigeria extracted using hexane had a flash point of $225 \pm 2^{0}C$ [Abitogun *et al*, 2009]

4.7 Kinematic viscosity

Kinematic viscosity is a measure of the time for a fixed volume of liquid to flow by gravity through a capillary. The results are shown in Table 4.6.

Table 4.6: Kinematic viscosity of castor oil samples at 40 ^{0}C

Name of sample	Method of extraction	Kinematic viscosity in cSt at 40 ^{O}C	Standard deviation
Laikipia	Solvent extraction	168.62	0.46
	Cold press extraction	199.13	0.40
Naivasha	Solvent extraction	169.89	0.44
	Cold press extraction	188.93	0.62
Limuru	Solvent extraction	178.23	0.45
	Cold press extraction	196.09	0.46
Maragua	Solvent extraction	166.94	0.31
	Cold press extraction	179.70	0.30
Kibwezi	Solvent extraction	180.14	0.26
	Cold press extraction	205.82	0.53

The results indicate that the kinematic viscosity ranged from 168.62 cSt to 205.82 cSt. Cold pressed castor oil had higher viscosity than their corresponding solvent extracted oil. The small impurities in the cold pressed oils may have been responsible for the high viscosity of these oils. Viscosity of biofuel of which castor oil is proposed, is an important aspect due to its effect on volume flow and injection spray characteristics. The recommended kinematic viscosity of diesel is 38 cSt. This means that for castor oil to be used as a biofuel, trans-esterifications, blending with other fossil fuels as well as heating are necessary to lower the viscosity. A kinematic viscosity of 182.25 cSt for cold pressed castor oil obtained in Kenya was reported [World Agroforestry Research Report, 2009]. Castor oil from India extracted by use of solvent was reported to have a kinematic viscosity value of 156.25cSt [Vashist and Ahmad, 2011].

4.8 Acid value

The acid number is the measure of free fatty acids in the oil, which is usually dependent on the method of extraction. It is the number of milligrams of potassium hydroxide required to neutralize the free fatty acids in 1 g of the fat. Since the expected acid value was between 1-10, amounts as close to 10 g was weighed into a conical flask. 150 ml of neutralized solvent mixture of 1: 1, 95% ethanol: diethyl ether mixture was added. The mixture was stirred and titrated against 0.1 N ethanolic KOH that had been prepared from an ampoules prepared by Sigma Aldrich ™. Two drops of phenolphthalein indicator was used and the end point noted when a permanent pink color formed that persisted for at least 10 seconds when the mixture was shaken vigorously. The results are shown in Table 4.7.

Table 4.7: Acid values of castor seed oil samples

Name of sample	Method of extraction	Acid value	Standard deviation
Laikipia	Solvent extraction	13.34	0.19
	Cold press extraction	5.35	0.12
Naivasha	Solvent extraction	5.35	0.15
	Cold press extraction	1.17	0.05
Limuru	Solvent extraction	18.67	0.10
	Cold press extraction	5.77	0.10
Maragua	Solvent extraction	7.46	0.18
	Cold press extraction	2.35	0.10
Kibwezi	Solvent extraction	19.38	0.37
	Cold press extraction	10.24	0.09

From the results obtained, it was evident that castor seeds from different location yielded oils with different acid values. It was also clear that in all the samples, the solvent extracted oil had a higher acid value than their corresponding cold press extracted oil. The KS 03-799: 1989 specifies an acid value of 2.0 maximum. It is thus evident that most of the oil samples except for the sample from Naivasha which is extracted by cold press did not meet that requirement. However, addition of small quantities of alkali can be done to lower these acid values [World Agroforestry Research Report, 2009]. Castor oil, like all other vegetable oils, has different physical and chemical properties that vary with the method of extraction. Cold pressed castor oil has low acid value than its solvent extracted counterpart [Ogunniyi, 2006]. An acid value of 2.7 for cold pressed oil has been reported [Mensah and Ochran, 2005] and 1.148 [Akpan *et al*, 2006]. Other researches reported an acid value of 9.7 [Sivasamban *et al*, 1954], 19 [Hlaing and Mya, 2008] and 14.8 ± 0.14 for hexane extracted castor oil [Abitogun *et al*, 2009]. Heating the oil has also been shown to favor hydrolysis of the oil to form free fatty acids [World Agroforestry Research Report, 2009]. During extraction of both the solvent and the cold pressed oils, heating was done e.g. to evaporate the solvent and during filtration of the cold pressed oil. These factors may have contributed to the high and varied acid values obtained. Neutralization of the seed oil is one of the procedures in oil processing.

4.9 Saponification value

0.5 N ethanolic KOH and 0.5 N HCl were prepared from ampoules prepared by Sigma Aldrich ^{TM}. 25 mls of 0.5 N KOH was titrated against 0.5 N HCl using phenolphthalein indicator so as to obtain the blank. Weights of oils samples as close as possible to 2.0 g were weighed into a round bottomed flask. Using a pipette, 25 mls of 0.5 N ethanolic KOH was added and some boiling marbles. The mixture was refluxed for 1 hour after which two drops of phenolphthalein was added and the mixture titrated against 0.5 N HCl until the pink color disappeared completely. The results of the determination are shown in Table 4.8

Table 4.8: Saponification values of castor seed oil samples

Name of sample	Method of extraction	Saponification value in mgKOH/g	Standard deviation
Laikipia	Solvent extraction	181.91	0.86
	Cold press extraction	184.99	0.59
Naivasha	Solvent extraction	181.18	0.52
	Cold press extraction	183.93	0.26
Limuru	Solvent extraction	180.70	0.74
	Cold press extraction	184.99	0.50
Maragua	Solvent extraction	178.90	1.51
	Cold press extraction	182.31	1.15

Kibwezi	Solvent extraction	180.32	1.75
	Cold press extraction	174.85	3.30

From the results, it was observed that other than the Kibwezi sample, the saponification value for cold press extracted oil had a higher saponification value than the solvent extracted. The KS 03-799: 1989 Specifies a saponification value of between 177 and 185 mgKOH/g. Saponification values of 181 mgKOH/g [Bagali *et al*, 2010] and 177 mgKOH/g [Mensah and Ochran, 2005] have been reported. Other values reported are 185.83 mgKOH/g [Akpan *et al*, 2006] and 178 ± 0.42 mgKOH/g [Abitogun, 2009]. Cold pressed oils have higher saponification values than their solvent extracted counterparts [Ogunniyi, 2006]. During cold press extraction, other impurities may also be extracted together with the oils. These substances may react with the alkali increasing the saponification values of these oils.

4.10 Iodine value

Iodine values are indicative of the level of unsaturation in the oil. The results obtained are shown in Table 4.9.

Table 4.9: Iodine values of castor seed oil samples

Name of sample	Method of extraction	Iodine value	Standard deviation
Laikipia	Solvent extraction	85.92	0.63
	Cold press extraction	83.17	0.47
Naivasha	Solvent extraction	84.80	0.42
	Cold press extraction	83.62	0.45
Limuru	Solvent extraction	86.72	0.38
	Cold press extraction	82.61	1.00
Maragua	Solvent extraction	87.20	0.87
	Cold press extraction	83.84	0.67
Kibwezi	Solvent extraction	86.43	0.34
	Cold press extraction	84.43	0.23

The results obtained showed the iodine values for all the cold press extracted oils being lower than the corresponding solvent extracted oils. The KS 03-799: 1989 specifies an iodine value of between 82 and 90. The results confirmed that all the samples met the requirement of this standard.

Reported iodine values are as follows, 87 [Bagali *et al*, 2010], 85.2 [World Agroforestry Research Report, 2009] for castor extracted from Kenya. Others are 82 [Mensah and Ochran, 2005] and 84.4 [Deligiannis *et al*, 2009]. The impurities in the cold extracted oils increase the weight taken but do not contribute to increasing the unsaturation, this could explain the differences observed in the iodine values. Iodine value only indicates the level of unsaturation but not the position of the double bonds.

4.11 Unsaponifiable matter

The unsaponifiable matter is the whole of the products present in the substance analyzed which, after saponification thereof with an alkaline hydroxide and extraction by a specified solvent, remain non-volatile under the define conditions of test. Weighed amounts of the castor oil were carefully taken through the procedures described in section 4.5.7. The results obtained are shown in Table 4.10.

Table 4.10: Percentage unsaponifiable matter in the castor seed oil samples

Name of sample	Method of extraction	Unsaponifiable matter, % m/m	Standard deviation
Laikipia	Solvent extraction	0.75	0.06
	Cold press extraction	0.09	0.01
Naivasha	Solvent extraction	0.72	0.04
	Cold press extraction	0.15	0.02
Limuru	Solvent extraction	0.74	0.05
	Cold press extraction	0.14	0.02
Maragua	Solvent extraction	0.92	0.07
	Cold press extraction	0.15	0.02
Kibwezi	Solvent extraction	0.78	0.12
	Cold press extraction	0.19	0.01

The results obtained showed that solvent extracted oils had high amounts of unsaponifiable matter than their cold pressed extracted counterparts. This can be explained by the fact that after extraction, the cold pressed oils underwent filtration, degumming and bleaching. During this processes, a lot of the unsaponifiable matter was removed. However, all the values obtained for both solvent and cold pressed oils were within those specified by the KS standard.

4.12 Color in a 1” cell Lovibond scale

These measurements were done at the Kenya Bureau of Standards using the Lovibond Tintometer and reported as lovibond values. This technique involved matching the color of light transmitted through the oil put in a 1inch cuvette with the color of light transmitted from same light source through a set of colored glass slides. The results are shown in Table 4.11. A copy of test result for Naivasha solvent extracted oil is also attached at the Appendix.

Table 4.11: Lovibond color numbers of the various castor oil samples

Name of Sample		**Photometric color number (Lovibond).**
LAIKIPIA	Solvent extracted	**0.6**
	Cold pressed	**0.2**
NAIVASHA	Solvent extracted	**0.5**
	Cold pressed	**0.2**
LIMURU	Solvent extracted	**0.6**
	Cold pressed	**0.2**
MARAGUA	Solvent extracted	**0.5**
	Cold pressed	**0.2**
KIBWEZI	Solvent extracted	**0.5**
	Cold pressed	**0.2**

The KS standard specifies maximum color number of 4.0 in a 1 inch cell on the lovibond scale. This implies that all the samples met this requirement.

4.13 Percentage oil loss after neutralization of the castor oil

From the weights taken and the procedures in section 3.6, the percentage loss after neutralization was calculated. The results are shown in Table 4.12.

Table 4.12: Percentage oil loss after neutralization

Type of oil	**Oil loss after neutralization, % m/m**	**Standard deviation**
Solvent extracted	8.02	0.76
Cold press extracted	7.57	1.67

Free fatty acids in the oil react with the sodium hydroxide added to form the sodium stearate soap. This reduces the amount of the oil that goes to the next stage of dehydration. 8.448 percentage losses have been reported [Hlaing and Mya, 2008]. Information about percentage loss of the oil after neutralization is important especially when calculating the costs of production of the dehydrated castor oil.

4.14 Iodine values of the dehydrated castor oil

The procedures as specified in section 4.5.3 were followed. The results are shown in Table 4.13.

Table 4.13: Iodine values of dehydrated castor oil.

Type of oil	**Iodine value before dehydration**	**Iodine values after dehydration**	**Standard deviation**	**ASTM D961-86 requirement**
Solvent extracted	86.24	140.71	0.92	125-145
Cold press extracted	81.79	137.44	1.67	125-145

A drying oil is a vegetable oil which dries by oxidation. The ability to dry and the rate of drying is influenced by the number and position of the double bonds in the molecule of these oils. Castor oil has only one double bond and therefore it can never dry by oxidation. It is classified as non-drying. However, by dehydrating castor oil, an extra double bond is added which makes it a drying oil thus suitable to be used in the coatings industries. This increase in unsaturation is evident in the increase in the iodine value of the oils from an average of 86.24 to 140.71 for solvent extracted oils and 81.79 to 137.44 for cold pressed oils. These values fall within the limits specified by the ASTM D 961-86 for dehydrated castor oil. Dehydrated castor oil with iodine values of 140.01 and 139.05 have been reported [Hlaing and Mya, 2008] while another research reported iodine values of 127.5, 127.6 and 133.8 [Sivasamban *et al*, 1954].

4.15 Kinematic viscosity of dehydrated castor oil

Dehydrated castor oil was subjected to the procedures specified in section 3.5.1. The results are shown in Table 4.14.

Table 4.14: Kinematic viscosity of the dehydrated castor oil

Type of oil	**Average kinematic viscosity in cSt**	**Standard deviation**	**ASTM D961-86 requirement**
Dehydrated solvent extracted	156.96	1.10	150-180
Dehydrated cold pressed oil	161.52	0.58	150-180

The iodine value and viscosity are indicators of the extent of the dehydration process. The results obtained shown in Table 4.13 and Table 4.14 indicated that the parameters tested on dehydrated oil were within those specified by the Standard. After dehydration of castor oil, ricinoleic acid looses the OH group. This group is responsible for the high kinematic viscosity obtained in crude oil due to hydrogen bonding. The reduction in kinematic viscosity is therefore another indication of dehydration. A kinematic viscosity of 160.0 and 168.6 cSt was obtained after using $NaHSO_4$ catalyst and a vacuum pressure of 600-640 mmHg to dehydrate the castor oil [Hlaing and Mya, 2008].

4.16 Set to touch time and drying time of the dehydrated castor oil

Both cold press and solvent dehydrated oils were applied on three sets of metal plates. Solvent and cold pressed crude oils were also applied. The plates were exposed in air under the shade. At intervals of two hours, the surface of the plate was touched firmly with the finger. When no oil adhered to the finger after touching, this was taken as the set to touch time. This time was noted as 36 hours for both the solvent and cold pressed dehydrated oils. No drying was noted on the plates where crude oils had been applied.

After each day, the plates where tested using the drying time tester machine. By applying friction on the plate using the rough cloth on the machine, the time taken when no abrasion was left by this scrubbing was noted. This time was noted as 3 days. No drying of the crude oil was noted even on the third day.

The drying time and the iodine values obtained are within those recommended for a good drying oil [Obiero, 2013]. The oils only limitation was its dark color. Because of its color, it could only be used in the manufacture of dark colored paints.

4.17 Summary

The results obtained for each of the castor oil sample was compared with the KS Standard as shown in Table 4.15.

Table 4.15: Comparison of the results of castor oil analysis with KS standard

Test parameter	**KS 03-799:1989 Requirement**	**LAIKIPIA**		**NAIVASHA**		**LIMURU**		**MARAGUA**		**KIBWEZI**	
		Solvent extracted	Cold pressed	Solvent extracted	Cold pressed	Solvent extracted	Cold pressed	Solvent extracted	Cold pressed	Solvent extracted	Cold pressed
Refractive index @ 40 ^{0}C	1.4700-1.4750	1.472	1.472	1.470	1.472	1.472	1.472	1.471	1.472	1.472	1.472
Specific gravity	0.954-0.960	0.9741	0.9773	0.9689	0.9768	0.9752	0.9787	0.9750	0.9783	0.9551	0.9755
Saponification value	177-185	181.91	184.99	181.18	183.93	180.70	184.99	178.90	182.31	180.32	176.60
Iodine value (Wijs)	82-90	85.92	83.17	84.80	83.60	86.72	82.61	87.20	83.84	86.43	84.43
Acid value, max	2.0	13.34	5.35	5.35	1.17	18.67	5.77	7.46	2.35	19.38	10.24
Unsaponifiable matter, % m/m max	0.8	0.75	0.09	0.72	0.15	0.74	0.15	0.92	0.15	0.78	0.19
Flash point, min ^{0}C	127	241	276	265	271	263	269	262	274	271	272
Color in a 1' cell on the Lovibond scale, max	4.0	0.6	0.2	0.5	0.2	0.6	0.2	0.5	0.2	0.5	0.2
Kinematic viscosity @ 40 cSt	-	168.62	199.13	169.89	188.93	178.23	196.09	166.94	179.70	180.14	205.82

CONCLUSIONS AND RECOMMEDATIONS

5.1 Conclusions

A number of findings were made during the course of this research. The percentage oil content of the seeds was noted to depend on the variety of the seed and the locality it was growing in the wild. The physicochemical properties of the castor oil extracted had very slight variations between them. Thus we can conclude that the quality of the oil is not affected by the geographical location where the castor plants were growing. There were variations between solvent and cold pressed extracted oils. This implies that the quality of the oil can be affected by the method of extraction. However, for our case all the oil extracted using both cold and solvent extraction met most of the requirements of the KS 03-799:1989, specification for castor oil for cosmetic industry. Value addition of the castor oil by dehydrating it so as to be used in the coating industry was also shown to be a viable undertaking.

5.2 Recommendations.

Kenya's Vision 2030 envisages that Kenya will attain medium income status by the year 2030. For a country to achieve this, it must strive to boost its exports, reduce imports and develop its processing and manufacturing industries. Castor oils industry presents one of the opportunities of turning around the economy of this country. To this end, we recommend the following:

1. The government of Kenya should consider commissioning a feasibility study into castor oil cultivation, extraction, value addition and marketing. Such a study was done in South Africa in the year 2000 [Belaid *et al*, 2000] and 2011 [International Trade Administration of South Africa, 2011]

2. During commercialization of castor seeds and oil, the price of the seed should be based on the seed type and the geographical location of the source of the seeds since they affect the output of oil content.
3. The Kenya Bureau of Standards should revise its KS 03-799: 1989 standard, clause 2.1 and clause 3 part (v) with regard to specific gravity parameter as well as the requirement that all castor oil used in cosmetics should be extracted using cold press. This is because the study has shown very slight difference exists between cold extracted and solvent extracted oils. In any case, both oils meet the requirements of the Standard.
4. Some physicochemical properties were not analyzed, these included, hydroxyl value, acetyl value and analysis of the fatty acids in the castor oil. It is thus recommend that further study be done for these parameters.
5. More study need to be done to understand how doves (birds) *Zenaida macroura,* are able to feed on castor seeds without being intoxicated by ricin and its associated toxins.
6. Plant bleeders and geneticists should strive to develop castor plants that do not produce ricin and other toxics. Meanwhile, a study into the use of ricin as a chemotherapy for cancer would be most welcomed.
7. Public awareness into the economic potentials as well as the risks associated with ingesting castor beans should be initiated.
8. A detailed study to determine factor that favors formation of either the conjugated or the non conjugated isomer of the dehydrated castor oil should be investigated. This will help in producing stereo specific dehydrated oil.

Finally, with majority of the youths in Kenya being jobless, and the Government of Kenya lauching initiatives such as The Uwezo Fund and the Youth Enterprise Development Fund, it would be very ideal if the youths could form castor oil growers’ co-operatives so as to access the funds and get agricultural extension services.

During this research, it was observed that though cold pressing was a cheap method of extraction, it had its own limitations. First, not all the oil in the seed can be extracted by this method thus the need for solvent extraction. Due to the high viscosity of the oil, purification by filtering using fine filters is almost impossible. This necessitates the use of cotton wool while heating the oil in an oven. The cotton wool absorbs a lot of the oil and thus reduces the final yield. Heating the oil also increases the acid value of the oil. A better purification method could help solve this limitation. Solvent extraction was observed to be a better method of extraction especially when using diethyl ether. However, diethyl ether is an expensive solvent and highly flammable. A substitute to this could be petroleum ether.

During dehydration we had difficulties in obtaining a set up where one could add the catalyst and CaO to the hot oil without disturbing the vacuum. The dehydrated castor oil that was obtained was darker in color than the recommended one. The oil would be ideal as a primer for making dark colored paints like the so called Lead oxide [Obiero, 2013]. However, with a more powerful vacuum pump and a better set up, clearer oils can be obtained.

Starch, Iodine, acetone, Ethanol, potassium hydroxide, sodium hydroxide, potassium iodide and phenolphthalein were manufactured by May & Baker Company. Sodium bisulphate and Iodine trichloride was from BDH Chemicals Ltd. Diethyl ether was from Panreac while activated charcoal was from Howse & McGeorge Ltd. Calcium Oxide was from Hopkin & Williams Ltd while Sodium Chloride was from Scientific Ltd. Ampoules of Sodiumthiosulphate, Sodium hydroxide and Hydrochloric acid were from Sigma Aldrich.

The viscometers used were from Fungilab size no. 4 and 6. The refractomer was from Hilger & Watts Ltd.

Data obtained was analyzed using MS Excel.

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APPENDICES

4-1

Moisture content in the castor seed samples

Name of sample		Weight of sample (g)	Final weight loss of sample (g)	Percentage moisture content, % m/m	Average moisture content, % m/m	Standard deviation
Laikipia	A	50.0	3.1	6.2	6.0	0.2
	B	50.0	3.1	6.2		
	C	50.0	3.0	6.0		
	D	50.1	2.9	5.8		
Naivasha	A	50.0	2.9	5.8	5.9	0.1
	B	50.0	3.0	6.0		
	C	50.0	3.0	6.0		
	D	50.1	2.9	5.8		
Limuru	A	50.0	2.5	5.0	5.3	0.3
	B	50.1	2.7	5.4		
	C	50.0	2.7	5.4		
	D	50.0	2.8	5.6		
Maragua	A	50.1	2.3	4.6	4.8	0.2
	B	50.0	2.5	5.0		
	C	50.0	2.3	4.6		
	D	50.1	2.5	5.0		
Kibwezi	A	50.0	2.4	4.8	4.9	0.1
	B	50.0	2.4	4.8		
	C	50.0	2.5	5.0		
	D	50.0	2.4	4.8		

4-2

Percentage oil content in the castor seeds

Name of sample		Weight of sample seeds taken (g) W0	Weight of beaker and oil (g) W1	Weight of empty dry beaker (g) W2	Weight of empty dry beaker (g) W2	Oil content, % m/m =(W1-W2)/WO x 100	Average % oil content, m/m	Standard deviation
Laikipia	A	10.06	72.68	68.20	4.48	44.53	44.53	0.85
	B	10.08	87.27	82.58	4.69	46.53		
	C	10.08	72.32	67.78	4.54	45.04		
	D	10.05	72.34	67.80	4.54	45.17		
Naivasha	A	10.05	86.61	81.44	5.17	51.44	51.44	1.05
	B	10.03	72.42	67.41	5.01	49.95		
	C	10.01	77.52	72.42	5.10	50.95		
	D	10.10	84.24	78.94	5.30	52.48		
Limuru	A	10.03	85.90	81.58	4.32	43.07	43.07	1.61
	B	10.00	72.24	67.55	4.69	46.90		
	C	10.03	86.09	81.65	4.44	44.27		
	D	10.12	80.12	75.62	4.50	44.47		
Maragua	A	10.07	72.33	67.62	4.71	46.77	46.77	1.15
	B	10.07	86.23	81.67	4.56	45.28		
	C	10.03	86.43	81.63	4.80	47.86		
	D	10.06	73.45	68.85	4.60	45.73		
Kibwezi	A	10.07	85.90	81.66	4.24	42.11	42.11	0.49
	B	10.05	71.81	67.51	4.30	42.79		
	C	10.04	86.02	81.70	4.32	43.03		
	D	10.08	72.00	67.76	4.24	42.06		

4-4

Specific gravity of castor oil

Name of sample		Weight of density bottle and distilled water at 30 ^{O}C (g) W1	Weight of density bottle and Castor oil at 30 ^{O}C (g) W2	Specific gravity W2/W1
Laikipia	Solvent extracted	46.0592	44.8651	0.9741
	Cold press extracted	46.0828	45.0376	0.9773
Naivasha	Solvent extracted	46.0592	44.6284	0.9689

	Cold press extracted	46.0828	45.0116	0.9768
Limuru	Solvent extracted	46.0592	44.9185	0.9752
	Cold press extracted	46.0828	45.1034	0.9787
Maragua	Solvent extracted	46.0592	44.9099	0.9750
	Cold press extracted	46.0828	45.0808	0.9783
Kibwezi	Solvent extracted	46.0592	43.9933	0.9551
	Cold press extracted	46.0828	44.9528	0.9755

4-6

Flash points of castor oil

Name of sample		Flash Point ^{o}C				Average Temperature ^{o}C	Standard deviation
		A	B	C	D		
Laikipia	Solvent extracted	240	241	240	243	241	1
	Cold pressed	278	275	276	276	276	2
Naivasha	Solvent extracted	265	265	264	267	265	1
	Cold pressed	269	271	272	270	271	2
Limuru	Solvent extracted	263	264	262	263	263	1
	Cold pressed	270	268	270	269	269	1
Maragua	Solvent extracted	262	263	260	261	262	2
	Cold pressed	273	273	274	274	274	1
Kibwezi	Solvent extracted	270	268	272	273	271	2
	Cold pressed	273	270	270	273	272	2

4-7

Kinematic viscosity of castor oil

Name of sample	Type of oil		Time taken in seconds	Viscometer factor	Viscosity in cSt	Average kinematic viscosity in cSt	Standard deviation
Laikipia	Solvent extracted	A	1754	0.095942	168.28	168.62	0.46
		B	1760	0.095942	168.86		
		C	1763	0.095942	169.15		
		D	1753	0.095942	168.19		
	Cold press extracted	A	2076	0.095942	199.18	199.13	0.40

		B	2070	0.095942	198.60		
		C	2080	0.095942	199.56		
		D	2076	0.095942	199.18		
Laikipia	Solvent extracted	A	1627	0.10461	170.20	169.89	0.44
		B	1619	0.10461	169.36		
		C	1622	0.10461	169.68		
		D	1628	0.10461	170.31		
	Cold press extracted	A	1802	0.10461	188.51	188.93	0.62
		B	1810	0.10461	189.34		
		C	1812	0.10461	189.55		
		D	1800	0.10461	188.30		
Laikipia	Solvent extracted	A	1700	0.10461	177.84	178.23	0.45
		B	1703	0.10461	178.15		
		C	1710	0.10461	178.88		
		D	1702	0.10461	178.05		
	Cold press extracted	A	1880	0.10461	196.67	196.09	0.46
		B	1870	0.10461	195.62		
		C	1872	0.10461	195.83		
		D	1876	0.10461	196.25		
Laikipia	Solvent extracted	A	1736	0.095942	166.56	166.94	0.31
		B	1740	0.095942	166.94		
		C	1744	0.095942	167.32		
		D	1740	0.095942	166.94		
	Cold press extracted	A	1875	0.095942	179.89	179.70	0.30
		B	1872	0.095942	179.60		
		C	1876	0.095942	179.99		
		D	1869	0.095942	179.32		
Laikipia	Solvent extracted	A	1722	0.10461	180.14	180.14	0.26
		B	1722	0.10461	180.14		
		C	1719	0.10461	179.82		
		D	1725	0.10461	180.45		
	Cold press extracted	A	1974	0.10461	206.50	205.82	0.53
		B	1964	0.10461	205.45		
		C	1963	0.10461	205.35		
		D	1969	0.10461	205.98		

4-8

Acid values of castor oil

Name of sample	Type of oil		Weight of sample (g) M	Titre (T) value 0.1 N KOH (ml)	Acid value =56.1 TN/M	Average acid values	Standard deviation
Laikipia	Solvent extracted	A	10.0057	23.9	13.40	13.34	0.19
		B	10.6193	25.1	13.26		
		C	10.0001	23.4	13.13		
		D	10.0089	24.2	13.56		
	Cold press extracted	A	9.9799	9.7	5.45	5.35	0.12
		B	10.2413	9.7	5.31		
		C	10.0082	9.7	5.44		
		D	10.0224	9.3	5.21		
Naivasha	Solvent extracted	A	10.3843	9.4	5.08	5.25	0.15
		B	10.9008	10.1	5.20		
		C	10.1112	9.5	5.27		
		D	10.0099	9.7	5.44		
	Cold press extracted	A	10.0519	2.0	1.12	1.17	0.05
		B	10.0267	2.1	1.17		
		C	10.0112	2.1	1.18		
		D	10.0601	2.2	1.23		
Limuru	Solvent extracted	A	10.0227	33.4	18.69	18.67	0.10
		B	10.0082	33.5	18.78		
		C	10.0755	33.3	18.54		
		D	10.0771	33.5	18.65		
	Cold press extracted	A	9.6652	9.9	5.75	5.77	0.10
		B	10.0489	10.4	5.81		
		C	10.0242	10.5	5.88		
		D	10.0295	10.1	5.65		
Maragua	Solvent extracted	A	10.0957	13.4	7.45	7.46	0.18
		B	10.0084	13.4	7.51		
		C	10.0034	12.9	7.23		
		D	10.6123	14.5	7.67		
	Cold press extracted	A	10.0238	4.3	2.41	2.35	0.10
		B	10.0765	4.0	2.23		
		C	10.0211	4.1	2.30		
		D	10.0489	4.4	2.46		

Kibwezi	Solvent extracted	A	10.5268	35.9	19.13	19.38	0.37
		B	10.1440	36.0	19.91		
		C	10.6192	36.2	19.12		
		D	10.5423	36.4	19.37		
	Cold press extracted	A	10.0150	18.4	10.31	10.24	0.09
		B	10.0728	18.2	10.14		
		C	10.0450	18.5	10.33		
		D	10.0626	18.3	10.20		

4-9

Saponification values of castor oil

Name of sample	Type of oil		Weight of sample (g) M	Titre (T) value 0.5 N KOH (ml)	Saponification value =56.1 TN/M	Average saponification values	Standard deviation
Laikipia	Solvent extracted	A	1.9941	12.9	181.46	181.91	0.86
		B	1.9986	13.0	182.45		
		C	1.9949	13.0	182.79		
		D	1.9999	12.9	180.93		
	Cold press extracted	A	2.0014	13.2	185.00	184.99	0.59
		B	2.0079	13.3	185.80		
		C	2.0078	13.2	184.41		
		D	2.0043	13.2	184.73		
Naivasha	Solvent extracted	A	1.9961	12.9	181.28	181.18	0.52
		B	2.0358	13.2	181.87		
		C	2.0019	12.9	180.75		
		D	2.0012	12.9	180.81		
	Cold press extracted	A	1.9956	13.1	184.13	183.93	0.26
		B	1.9953	13.1	184.16		
		C	1.9991	13.1	183.81		
		D	2.0012	13.1	183.62		
Limuru	Solvent extracted	A	2.0065	13.0	181.73	180.70	0.74
		B	1.9945	12.8	180.02		
		C	2.0056	12.9	180.42		
		D	2.0034	12.9	180.62		
	Cold press extracted	A	1.9998	13.2	185.15	184.99	0.50

		B	2.0086	13.2	184.34		
		C	1.9956	13.2	185.54		
		D	2.0019	13.2	184.95		
Maragua	Solvent extracted	A	1.9999	12.6	176.72	178.90	1.51
		B	2.0028	12.8	179.27		
		C	2.0078	12.9	180.22		
		D	2.0013	12.8	179.40		
	Cold press extracted	A	1.9993	12.9	180.99	182.31	1.15
		B	2.0013	13.1	183.61		
		C	2.0098	13.1	182.83		
		D	2.0055	13.0	181.82		
Kibwezi	Solvent extracted	A	1.9997	13.0	182.35	180.32	1.75
		B	2.0067	12.9	180.32		
		C	2.0043	12.9	180.53		
		D	2.0004	12.7	178.08		
	Cold press extracted	A	2.0043	12.5	174.94	174.85	3.30
		B	2.0076	12.4	173.25		
		C	2.0012	12.8	179.41		
		D	2.0900	12.8	171.79		

NB: To obtain the blank, three titrations were done and the results were 25.1, 25.1, and 25.1. The mean was thus taken as 25.1 mls. The titer value was obtained by subtracting the volume used in titrating the refluxed mixture from the blank i.e. 25.1 ml. This is the volume of KOH that was used to saponify the oil.

4-10

Iodine values of castor oil

Name of sample	Type of oil		Weight of sample (g) M	Titre (T) value 0.1 N Sodiumthiosulphate (ml) V_2	Titre (T) value 0.1 N Sodiumthiosulphate (ml) $V_3=V_1-V_2$	Iodine value= 12.69 NV_3/M	Average iodine value	Standard deviation
Laikipia	Solvent extracted	A	0.2084	41.2	14.2	86.47	85.92	0.63
		B	0.2015	41.9	13.5	85.02		
		C	0.2078	41.3	14.1	86.11		
		D	0.2034	41.6	13.8	86.10		
	Cold press	A	0.2091	41.6	13.8	83.75	83.17	0.47

	extracted	B	0.1982	42.4	13.0	83.23		
		C	0.1997	42.4	13.0	82.61		
		D	0.2047	42.0	13.4	83.07		
Naivasha	Solvent extracted	A	0.2047	41.8	13.6	84.31	84.80	0.42
		B	0.2043	41.7	13.7	85.10		
		C	0.2011	41.9	13.5	85.19		
		D	0.2055	41.7	13.7	84.60		
	Cold press extracted	A	0.2066	41.7	13.7	84.15	83.62	0.45
		B	0.2108	41.6	13.8	83.07		
		C	0.2045	41.9	13.5	83.77		
		D	0.2067	41.8	13.6	83.49		
Limuru	Solvent extracted	A	0.2115	41.0	14.4	86.40	86.72	0.38
		B	0.2023	41.5	13.9	87.19		
		C	0.2089	41.1	14.3	86.87		
		D	0.2056	41.4	14.0	86.41		
	Cold press extracted	A	0.2078	42.0	13.4	81.83	82.61	1.00
		B	0.2009	42.1	13.3	84.01		
		C	0.2090	41.9	13.5	81.97		
		D	0.2043	42.1	13.3	82.61		
Maragua	Solvent extracted	A	0.2017	41.5	13.9	87.45	87.20	0.87
		B	0.2006	41.8	13.6	86.03		
		C	0.2045	41.2	14.2	88.12		
		D	0.2067	41.2	14.2	87.18		
	Cold press extracted	A	0.2058	41.9	13.5	83.24	83.84	0.67
		B	0.2077	41.6	13.8	84.31		
		C	0.2011	42.2	13.2	83.30		
		D	0.2087	41.5	13.9	84.52		
Kibwezi	Solvent extracted	A	0.2069	41.3	14.1	86.48	86.43	0.34
		B	0.2001	41.7	13.7	86.88		
		C	0.2078	41.3	14.1	86.11		
		D	0.2089	41.2	14.2	86.26		
	Cold press extracted	A	0.2020	42.0	13.4	84.18	84.43	0.23
		B	0.2043	41.8	13.6	84.48		
		C	0.2067	41.6	13.8	84.72		
		D	0.2076	41.6	13.8	84.36		

NB: The titer values of 0.1 N sodium thiosulphate used for the blank were 55.4 ml, 55.5 ml, 55.4 and 55.3 ml. The average titer value (V_1) calculated was 55.4 ml. The titer value of the Wij's reagent used (V_3) was obtained by subtracting the titration value from the titer value of the blank i.e. $V_1 - V_2$.

4-11

Percentage unsaponifiable matter of castor oil

Name of sample	Type of oil		Weight of sample (g) M	Weight of residue (g)	% m/m unsaponifiable matter	Average Unsaponifiable matter	Standard deviation
Laikipia	Solvent extracted	A	5.0045	0.0362	0.72	0.75	0.06
		B	5.0062	0.0401	0.80		
		C	5.0042	0.0393	0.79		
		D	5.0023	0.0340	0.68		
	Cold press extracted	A	5.0086	0.0036	0.07	0.09	0.01
		B	5.0026	0.0050	0.10		
		C	5.0095	0.0041	0.08		
		D	5.0078	0.0050	0.10		
Naivasha	Solvent extracted	A	5.0005	0.0354	0.71	0.72	0.04
		B	5.0018	0.0391	0.78		
		C	5.0003	0.0362	0.72		
		D	5.0012	0.0340	0.68		
	Cold press extracted	A	5.0026	0.0071	0.14	0.15	0.02
		B	5.0086	0.0081	0.16		
		C	5.0024	0.0065	0.13		
		D	5.0089	0.0091	0.18		
Limuru	Solvent extracted	A	5.0043	0.0345	0.69	0.74	0.05
		B	5.0016	0.0366	0.73		
		C	5.0078	0.0375	0.75		
		D	5.0053	0.0404	0.81		
	Cold press extracted	A	5.0024	0.0074	0.15	0.14	0.02
		B	5.0071	0.0068	0.14		
		C	5.0012	0.0054	0.11		
		D	5.0003	0.0081	0.16		
Maragua	Solvent extracted	A	5.0032	0.0501	1.00	0.92	0.07
		B	5.0022	0.0422	0.84		

		C	5.0012	0.0455	0.91		
		D	5.0043	0.0468	0.94		
	Cold press extracted	A	5.0033	0.0078	0.16	0.15	0.02
		B	5.0026	0.0072	0.14		
		C	5.0040	0.0065	0.13		
		D	5.0034	0.0091	0.18		
Kibwezi	Solvent extracted	A	5.0063	0.0463	0.92	0.78	0.12
		B	5.0045	0.0412	0.82		
		C	5.0067	0.0362	0.72		
		D	5.0013	0.0320	0.64		
	Cold press extracted	A	5.0027	0.0095	0.19	0.19	0.01
		B	5.0013	0.0085	0.17		
		C	5.0019	0.0093	0.19		
		D	5.0078	0.0103	0.21		

NB: The final products formed were slightly alkaline (confirmed by adding phenolphthalein indicator) and thus no titration was done with the 0.1 N ethanolic KOH

4-13

Percentage oil loss after neutralization

Type of Oil		Weight of empty beaker (g) M1	Weight of beaker plus sample taken (g) M2	Weight of beaker plus neutralized oil obtained (g) M3	Weight of oil taken (g) M4=M2-M1	Weight of neutralized oil (g) M5=M3-M1	Percentage weight loss m/m=(M4-M5)/M4 X 100	Average percentage weight loss m/m	Standard deviation
Solvent extracted	A	136.7	237.9	228.7	101.2	92.0	9.09	8.02	0.76
	B	154.3	254.5	247.1	100.2	92.8	7.39		
	C	112.5	212.5	204.5	100.0	92.0	8.00		
	D	118.7	218.6	211.0	99.9	92.3	7.61		
Cold press extracted	A	220.2	320.2	313.4	100.0	93.2	6.80	7.57	1.67
	B	224.6	324.5	318.6	99.9	94.0	5.91		
	C	214.6	314.6	306.8	100.0	92.2	7.80		
	D	218.3	318.4	308.6	100.1	90.3	9.79		

4-14

Iodine values of dehydrated oils

Type of oil	Iodine value before dehydration	Weight of sample (g) M		Titre (T) value of 0.1 N Sodiumthiosulphate (ml)	Iodine value = 12.69 x TN/M	Average Iodine values	Standard deviation
Solvent extracted	86.24	A	0.1364	15.1	140.48	140.71	0.92
		B	0.1359	15.2	141.93		
		C	0.1344	14.9	140.69		
		D	0.1326	14.6	139.72		
Cold press extracted	81.79	A	0.1302	14.3	139.38	137.44	1.67
		B	0.1313	14.3	138.21		
		C	0.1320	14.2	136.51		
		D	0.1319	14.1	135.66		

NB: The titers obtained for the blank were 55.2 ml, 55.3 ml, 55.2 and 55.1 ml. The average volume of the blank was calculated as 55.2 ml. The titer volume was obtained by subtracting the volume of 0.1 N sodiumthiosulphate titrated with the sample from the volume used in the blank.

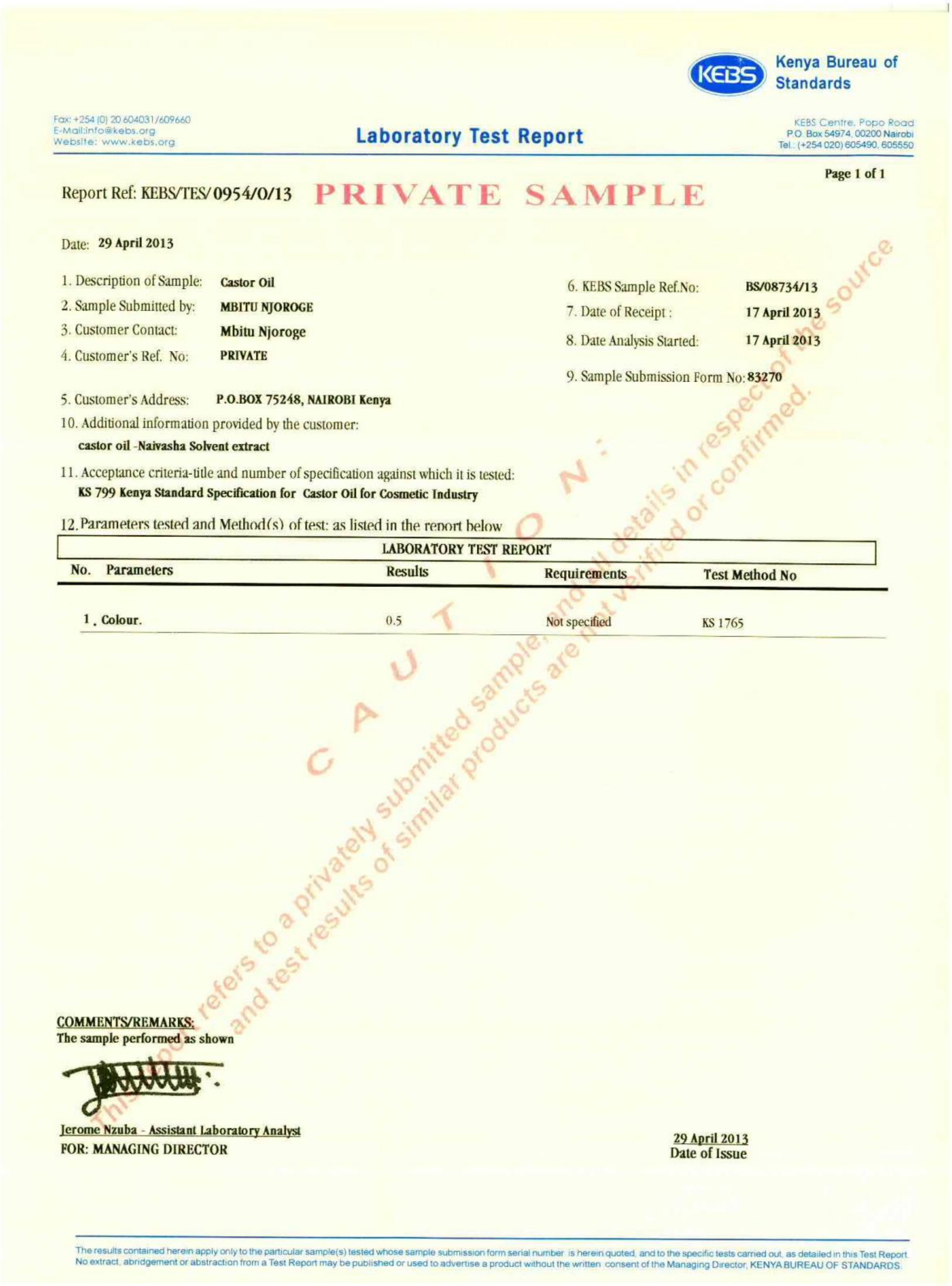
4-15

Kinematic viscosity of dehydrated castor oil

Type of oil		Time taken in seconds	Viscometer factor	Viscosity in cSt	Average kinematic viscosity in cSt	Standard deviation
Dehydrated solvent extracted	A	1637	0.095942	157.06	156.96	1.10
	B	1620	0.095942	155.43		
	C	1640	0.095942	157.34		
	D	1647	0.095942	158.02		
Dehydrated cold press extracted	A	1690	0.095942	162.14	161.52	0.58
	B	1687	0.095942	161.85		
	C	1677	0.095942	160.89		
	D	1680	0.095942	161.18		

A-1

TEST RESULTS FOR COLOR FOR NAIVASHA SOLVENT EXTRACTED CASTOR OIL



Kenya Bureau of Standards

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E-Mail:info@kebs.org
Website: www.kebs.org

Laboratory Test Report

KEBS Centre, Popo Road
P.O. Box 54974, 00200 Nairobi
Tel.: (+254 020) 605490, 605550

Page 1 of 1

Report Ref: KEBS/TES/**0954/0/13** **PRIVATE SAMPLE**

Date: **29 April 2013**

1. Description of Sample: **Castor Oil**
2. Sample Submitted by: **MBITU NJOROGE**
3. Customer Contact: **Mbitu Njoroge**
4. Customer's Ref. No: **PRIVATE**

6. KEBS Sample Ref.No: **BS/08734/13**
7. Date of Receipt : **17 April 2013**
8. Date Analysis Started: **17 April 2013**

9. Sample Submission Form No: **83270**

5. Customer's Address: **P.O.BOX 75248, NAIROBI Kenya**

10. Additional information provided by the customer:
castor oil -Naivasha Solvent extract

11. Acceptance criteria-title and number of specification against which it is tested:
KS 799 Kenya Standard Specification for Castor Oil for Cosmetic Industry

12. Parameters tested and Method(s) of test: as listed in the report below

LABORATORY TEST REPORT

No.	Parameters	Results	Requirements	Test Method No
1 .	Colour.	0.5	Not specified	KS 1765

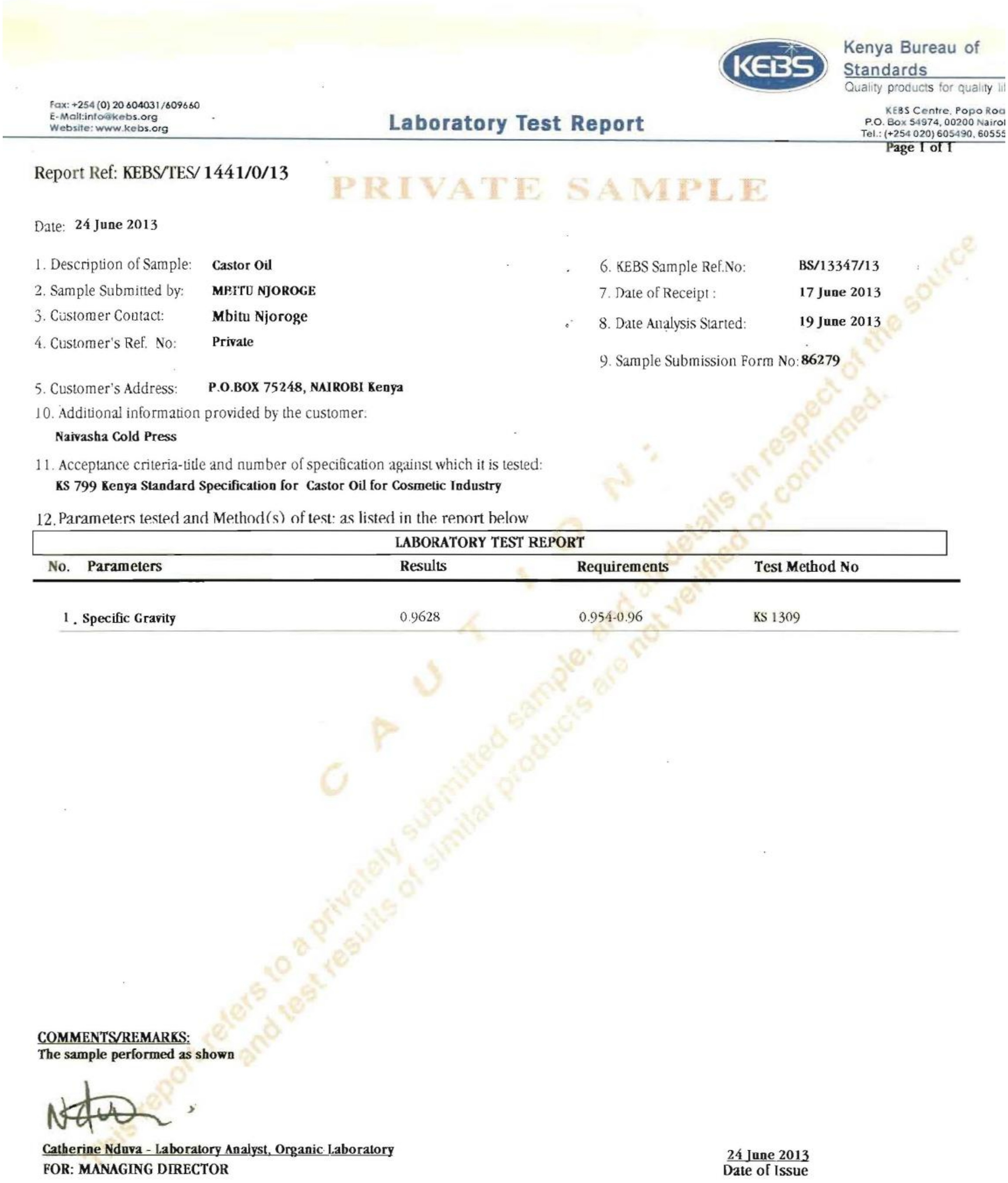
COMMENTS/REMARKS:
The sample performed as shown

Jerome Nzuba - Assistant Laboratory Analyst
FOR: MANAGING DIRECTOR

29 April 2013
Date of Issue

A-2

TEST RESULTS OF SPECIFIC GRAVITY FOR NAIVASHA COLD EXTRACTED CASTOR OIL



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Laboratory Test Report

Page 1 of 1

Report Ref: KEBS/TES/ **1441/0/13**

PRIVATE SAMPLE

Date: **24 June 2013**

1. Description of Sample: **Castor Oil**
2. Sample Submitted by: **MBITU NJOROGE**
3. Customer Contact: **Mbitu Njoroge**
4. Customer's Ref. No: **Private**
5. Customer's Address: **P.O.BOX 75248, NAIROBI Kenya**
6. KEBS Sample Ref.No: **BS/13347/13**
7. Date of Receipt : **17 June 2013**
8. Date Analysis Started: **19 June 2013**
9. Sample Submission Form No: **86279**
10. Additional information provided by the customer:
    Naivasha Cold Press
11. Acceptance criteria-title and number of specification against which it is tested:
    KS 799 Kenya Standard Specification for Castor Oil for Cosmetic Industry
12. Parameters tested and Method(s) of test: as listed in the report below

LABORATORY TEST REPORT

No.	Parameters	Results	Requirements	Test Method No
1 .	Specific Gravity	0.9628	0.954-0.96	KS 1309

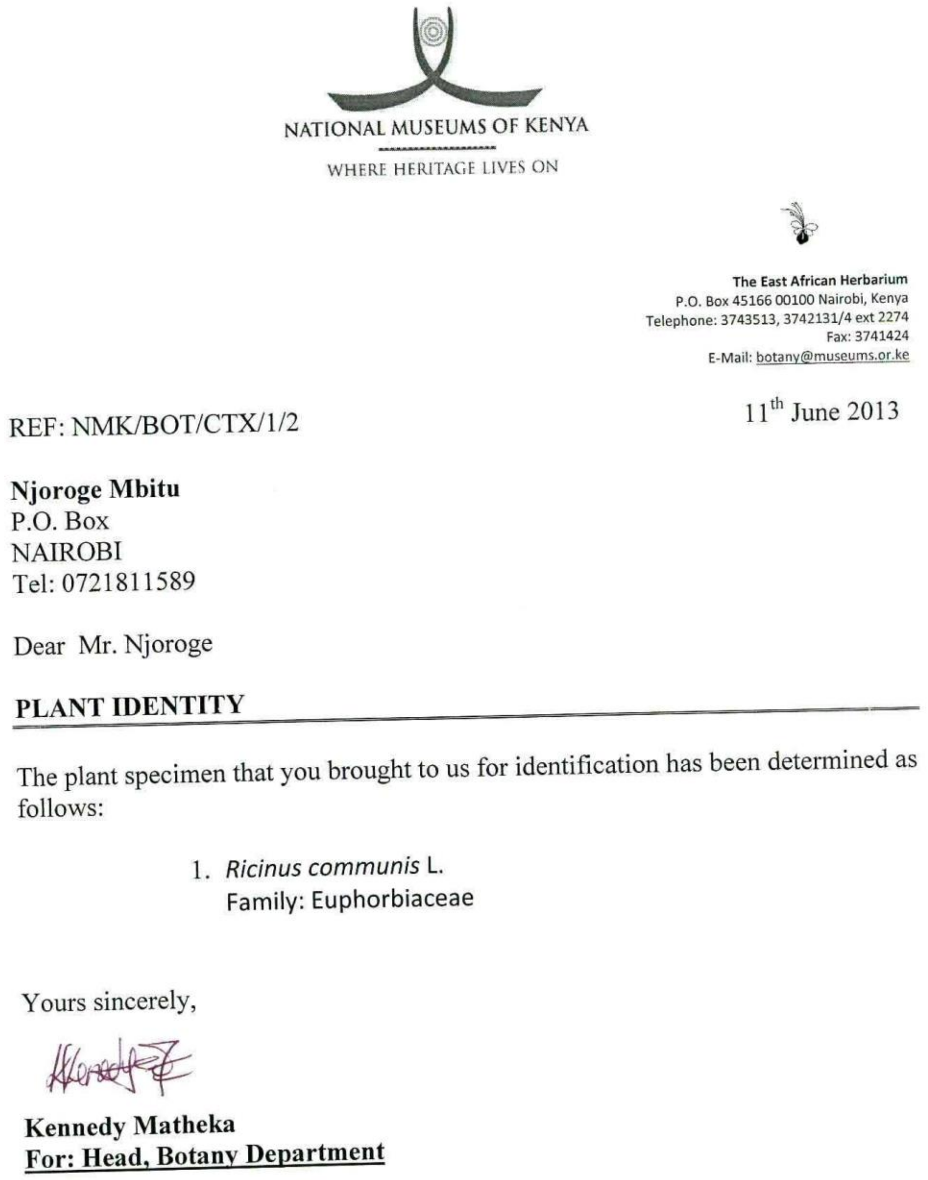
COMMENTS/REMARKS:
The sample performed as shown

Catherine Nduva - Laboratory Analyst, Organic Laboratory
FOR: MANAGING DIRECTOR

24 June 2013
Date of Issue

A-3

A COPY OF THE PLANT IDENTIFICATION CERTIFICATE



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REF: NMK/BOT/CTX/1/2

11th June 2013

Njoroge Mbitu
P.O. Box
NAIROBI
Tel: 0721811589

Dear Mr. Njoroge

PLANT IDENTITY

The plant specimen that you brought to us for identification has been determined as follows:

1. *Ricinus communis* L.
   Family: Euphorbiaceae

Yours sincerely,

Kennedy Matheka
For: Head, Botany Department