DETERMINATION OF SOME CHEMICAL PROPERTIES OF CASTOR SEED OIL (*Ricinus communis*)

AYANNIYI,TUNDE GBENGA

2000/9488EA

SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY

DEPARTMENT OF AGRICULTURAL ENGINEERING

FEDERAL UNIVERSITY OF TECHNOLOGY
MINNA

NOVEMBER, 2006

DETERMINATION OF SOME CHEMICAL PROPERTIES OF CASTOR SEED OIL (*Ricinus communis*)

AYANNIYI TUNDE GBENGA

2000/9488EA

A FINAL YEAR PROJECT SUBMITTED TO THE DEPARTMENT OFAGRICULTURAL ENGINEERING IN FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF BACHELOR OF ENGINEERING (B. ENG.) DEGREE IN AGRICULTURAL ENGINEERING

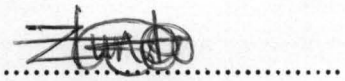
DEPARTMENT OF AGRICULTURAL ENGINEERING

FEDERAL UNIVERSITY OF TECHNOLOGY
MINNA

NOVEMBER, 2006

DECLARATION

I hereby declare that this project is a record of a research work that was undertaken and written by me. It has not been presented before for any degree, diploma or certificate at any University or Institution. Information derived from personal communication, published and unpublished works of others were duly referenced in the text.



Ayanniyi, Tunde

10/11/2006

Date

CERTIFICATION

This Project entitled "Determination of Some Chemical Properties of Castor Seed Oil" by Ayanniyi, Tunde meets the regulations governing the award of Bachelor of Engineering (B.ENG) of the Federal University of Technology, Minna, and it is approved for its contribution to scientific knowledge and literary presentation.

Engr. Dr. D. Adgidzi
Supervisor

12.11.2006
Date

External Examiner

02/11/2006.
Date

Engr. Dr. Mrs. Z. D. Osunde
Head, Department of Agricultural Engineering

13/11/2006
Date

DEDICATION

This project is dedicated to my dear and lovely parents Mr. and Mrs. Adebayo Ayanniyi and also to my wonderful siblings.

ACKNOWLEDGEMENT

First and foremost, my gratitude goes to the Almighty God, the one who was, who is, and who is to come. He is the pillar that holds my entire life.

I cannot but appreciate the effort and assistance of my dear supervisor Dr.D.Adgidzi who through his kindness, support and guidance has contributed immensely in making this work a great success. The Lord bless him in all his life endeavours. I also appreciate Engr. Dr. O. Chukwu, Dr. (Mrs.) Z.D Osunde, Mr. Peter Adeoye and all other lecturers and staffs of the department for their various impartations right from my inception into this school till my final year on this campus.

My sincere appreciation and gratitude will I also express to my dear and loving parent Mr. and Mrs. Ayanniyi for their love and care beginning from my first day on earth through my primary and secondary education up to date. What could I have been without you? The Almighty will bless and satisfy you with long life and prosperity. Amen!

I also say thanks to my younger brother Yemi for his assistance in typing this work and his complementary effort in making this work a beautiful one. To the rest of my siblings, I also say thanks for their prayers and support.

Finally, I appreciate my dear friends Tunde, Martins, Joseph, Charles, Adams, Samuel, Femi and also a special one to Joseph Odesanya who has been so caring to me during my stay on campus. Also to my dear friend back at home, Isaac Aina I say thanks for his support.

ABSTRACT

The castor oil plant (*Ricinus communis*) is a plant species of the Euphorbiaceae and the sole member of the genus *Ricinus* and of the subtribe Ricininae. The oil of the castor seed which is one of its major constituents was extracted with the aid of the solvent extractor. Some of the chemical properties which are of utmost relevance to industrial application were selected for study. Standard experiment were undertaken in studying the chemical properties of the seed and the values obtained for each parameters are as follows; acid value 3.84; hydroxyl value 165.01; Saponification value 146.42; pH 6.14; iodine value 87.8; refractive index 1.469; peroxide value 2.47; viscosity 8.93. The result thus obtained from these tests fully describes the chemical properties of the seed. Modification of the oil is achieved by a variety of chemical processes including oxidation, hydrogenation and thermal treatments to produce products for specific applications.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER ONE

1.0 INTRODUCTION

In handling, storage and design of processing machineries for agricultural materials, knowledge of the basic properties of these materials are required. The chemical properties, mechanical properties among others are important designing of the machine and equipment for various agricultural operations example in design of processing machineries and storage, the chemical properties of the materials content such as acid content of seed coat etc must be known so as to allow for the use of the right materials or component during construction and also to enhance proper storage after processing.

There are various oilseeds of which the castor seed is one. Oilseeds have been utmost importance to man because of the great benefits derived from it. Castor seed produces seed oil in which nearly 90% of the acyl residues are ricinoleic acid. Such a degree of homogeneity and the unique chemical properties of the seed content makes it a valuable industrial raw material. Castor is a warm season plant and is indigenous to eastern Africa. Now the bulk of the crop is utilized in industry. It is water resistant and is used for fabrics and other protective coverings. Although castor seed was not grow on a commercial scale until late 1930, the unique chemical properties its content has made it desirable and it has recently been introduced the New World and is now found growing in a naturalized state all over tropical Americans, also in many tropical and subtropical countries the world at large. The major producers of Castor seed are shown below.

Table 1.1 Major Producers of Castor Seed

COUNTRY	1969-1971	1978	1979	1980
Brazil	363	317	327	281
India	125	217	236	233
China	87	98	225	120
U.S.S.R	67	43	62	58
Thailand	40	43	37	26
World	844	852	907	845

Source : FAO, Production Yearbook,1981

1.1 Aim and objective

The aim and objective of this project is to determine some of the chemical properties of castor seed.

1.2 Statement of problem

The derivatives of castor seed though many, still remain unknown and unfamiliar to so many people in Nigeria. The seed itself is not widely known and this results in low productivity nation-wide. To this effect, scientific data as regarding its engineering properties are rarely available.

1.3 Justification

The study of some of the chemical properties of oil seed have been an attempt to provide objective measurement resulting in more meaningful data in the design of processing machineries, food processing and storage. These data will generally aid food industries to select appropriate method of processing this seed which will result in maximizing the product.

1.4 Scope of study

Some chemical properties of relevant industrial application have been selected for study within the scope of this project. These include the acid value, iodine value, saponification value, hydroxyl value, peroxide value, pH, refractive index, viscosity.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Origin of castor seed and ecology

Castor seed originated in Africa and grows wild in East and North Africa, the Yemen and the Middle East. It was cultivated in ancient Egypt as long as 400BC. It was taken at an early date to India and beyond, and was recorded in China in the Tang period, AD 618-90. It was introduced shortly after Columbus. The castor plant is now naturalized in many tropical and subtropical countries.

Castor requires a warm climate and is killed by frost. At least 140-180 day growing season is required before the first killing frost. It can be grown over a wide altitude range in the tropics and with both low and medium rainfall. Heavy rainfall and water logging should be avoided. At sustained temperatures above $100^{O}F$ seed may fail to set. The best soils for cultivation are rich well-drained sandy or clayey loams.

2.2 Castor seed

2.2.1 Botany and structure

Castor (Ricinus Communis) belongs to the Euphorbiaceae or spurge family, containing a vast number of plants native to the tropics. In its widespread naturalized state, castor is usually a fairly tall, many-branched perennial, but when cultivated commercially, it is short lived, erect, little branched, and treated as an annual. The castor plant varies greatly in its growth habit, color of foliage, stems, seeds size, color, and oil content, so that varieties often bear little resemblance to

each other colour differences in leaves, stems and inflorescent aid in the reflection of horticultural and ornamental plants (Weiss,1983)

Castor plants can be basically divided into two types, tall and short, commonly known as giant and dwarf castors. The period from emergence to maturity varies with variety and is greatly influenced by the environment (14 -160 days). Giant types have a large, well-developed taproot system reacting several meters in length with profuse laterals and secondary roots. In dwarf types, the tap root is less apparent and their root system is well developed and often deeply penetrating to take maximum advantage of soil moisture, making the plant fairly drought resistant. The stem is round, glabrous or glaucous, and covered with a waxy bloom, giving red or green stems a bluish appearance leaves are very large and usually dark glossy green palmate, with 5 to 11 lobes and prominent veins on the under surface. The leaves and stems of ornamental varieties vary form light green to dark red, depending on the level of anthocyanins present. Leaves are alternate, except, except for two opposite leaves at the node immediately above the cotyledon, and are borne on long, stout petioles. Young leaves can be mildly toxic to animals and some insects. Flowers are produced over an extended period, the giant and perennial types may flower year-round under suitable climate. The fruit is a globular capsule, spiny to some degree, becoming hard and brittle when ripe, and occasionally shattering at maturity.

The seed colour may vary from white, brown, and buff to black or red, usually everal classes occurring as an attractive mottling on the testa.

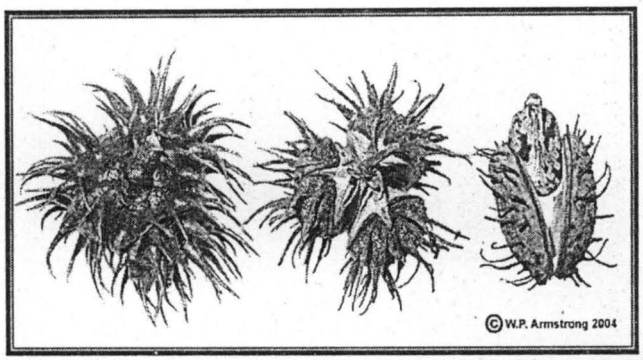


Fig.2.1 Castor bean fruit (**Ricinus communis**): The spiny, globose seed capsule (left) dries and splits into 3 sections called carpels (center).

Fig 2.2 Flower cluster (inflorescence) of castor bean (**Ricinus communis**).

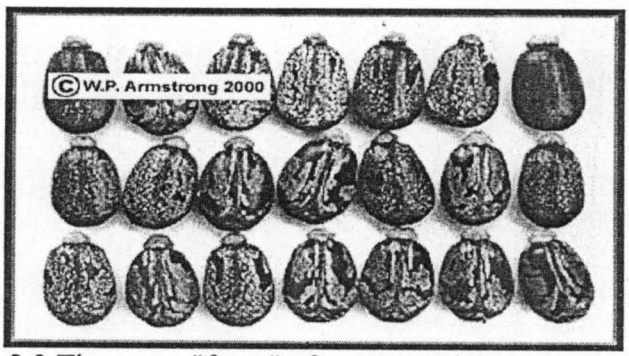


Fig 2.3 The many "faces" of castor seeds.

Root: Well-developed tap-root with prominent laterals, which produces a surface and prominent leaf scars. A single stem is first produced which terminates in an inflorescent at 6th-10th node in dwarf early cvs, at 8th-16th node in later maturing cvs, and at 40th or more nodes in tall and wild plants. As the panicle develops, 2-3 sympodial branches grows out, one from each node immediately below it, these end in inflorescences and one or more sympodial branches grow out from the nodes immediately below them and the process is continued. These development along each axis is sequential and a plant will have inflorescence at various stages of development. Degree of branching varies considerably.

Leaves: Spirally arranged with a phllotaxis of 2/5, peltate, stipules 1-3cm long, united, sheating bud, deciduous, petiole pale green or reddish, round, 8-5ocm long with 2 nectiferous glands at junction to lamina, 2 glands on either side at base and 1 or more glands on upper surface towards base; lamina or bicular, 10-75cm in diameter, palmately 5-11 particle for about half length, segment ovate or lanceolate, acuminate, serrate, dark green or reddish above, paler green beneath.

Flowers: Borne in terminal, many flowered panicles to 10-40cm long, unisexual, with male flowers at the base and female flowers on the top 30-50 percent of inflorescence, some commercial hybrids entirely female. Male flowers in 3-16 flowered cymes, pedicles 0.5-1.5cm long, sepals 3-5, ovate, spreading, green, 5-7mm long, petals absent, stamens numerous, 5-10mm long, with much branched filaments, each branch ending in a small, spherical, pale yellow anther. Female flowers in 1-7 flowered cymes, pedicles 4-5mm long, sepals 3-5 green, 3-5mm long, connate and bursting irregularly, quickly falling, petals absent, ovary

superior, 3-celled, 1 ovule per cell, ovary wall covered with fleshy soft green spines each terminating in a transparent bristle which breaks off as fruit develops, style very short, stigmas 3, deeply bifid, fleshly, red papillate, long persistent.

Fruits: Globose capsule with elongated pedicel, 3-lobed, 1.5-2.5cm in diameter, usually spinney, unripe flowers green, but red in some cvs, turning brown on ripening, woody remains of pistil at apex. Dehisce by woody pericarp splitting along dorsal suture, in wild plants and some cvs splitting is violent ejecting seeds, most modern cvs are indehiscent and seed is held in capsule for several weeks.

Seeds: Ovrid, compressed dorsally, tick-like, shinning, pale grey pale buff to almost black with darker mottling, yellowish white caruncle at base, very variable in size, 0.5-1.5cm long, 450-5,000 seeds per lobes. Testa brittle, about 20 percent weight of seed, endosperm copious, embryo small, cotyledons thin and papery (Alyadural *et al.,* 1963)

2.2.2 Germination and pollination

Sand seed will retain their viability for 2-3 years. The brittle testa is easily damaged and crushed seeds may caves clogging in mechanical planters. It is advisable to treat the seeds to prevent subsequent damping off. More even germination may be obtained by pairing boiling water on the seeds and leaving them to soak for 24hours. Some Castor seeds show dorm only, but this may be broken by removing the carbuncle and cutting a small hole in the testa. Germination is epigeal and emergence takes 7-10 days, sometimes longer. Castor plants are protogynous: Most of the female flowers have set seed and the fruit are developing before the male flowers open on the same inflorescence. The

anthers bursts explosively on drying or when touched scattering copious pollen. After shedding pollen male flowers soon abscises. It is usually stated that castor is mainly wind pollinated. At the same time as the flowers are opening the glands on the young leaves on the sympodial branches below the inflorescence exude copious nectar, so it seems probable that insects play some part in pollination.

2.2.3 Cultivation and management

There are many varieties of wild and cultivated castor, varying in plant height, colour of stem and seeds, bean size e.t.c. Preferred variation do not shatter when ripe and mature evenly. The tropical tall types are planted at spacing ranging from 90cm by 60cm to 150cm by 120cm. seeding rate is 10-20kg per hectare. Tall cultivars are often topped to produce more branches. Complete fertilizers should be applied as the crop has high nutrient requirements. The bunches of fruits are cut manually when ripe. Yield of up to 900kg of seeds per hectare were obtained. Seed damage should be avoided as this causes the oil to go rancid (David et al, 1992). Often the plants are lopped when 1m high to encourage branching. In the USA with mechanized production, seed rate of 13-15kg/ha are used, planting 3-7cm deep, at 22-28cm spacing in rows 1m apart. Castor exhausts the soil of nutrients, and although in Africa and Asia there is little or no fertilizer application, in the USA 44-135kg/ha nitrogen is usually applied in split dressings. In pure stand seed yields of up to 1000kg/ha are found in India, with an average of 500kg/ha, in the USA under intensive cultivation with irrigation, yields of 1000kg/ha can be obtained. The meal after oil extraction contains poisonous

substances and should not be fed to livestock. It is a useful fertilizer (Alyadural *et al.*, 1963)

2.2.4 Harvesting, handling and maturity

Harvesting and hulling are the most difficult and time-consuming operations in castor growing, although suitable machines and varieties for large scale and mechanical operations are presently available. In India, harvesting operations are generally spread over a period of 5-10 weeks or more, as all the capsules or fruits on the fruiting branches or spikes do not mature at the same time. The firs-formed fruits open and begin to dry in about 120 to 150 days from planting, depending open soil and variety. The normal practice is to harvest the crop when a few fruits show signs of drying. The immature kernels are light in weight and low in oil content compared to fully matured ones. Delaying the harvest results in heavy loss due to dehiscence of mature capsules and consequent shedding of the sees, if harvesting is done after all the fruits have dried. Mechanical harvesting consists of removing seed capsules from standing plants, using a better mechanism that strikes plant directly under the lowest raceme. The capsule fall outs a conveyer and thence move to the huller. The hulls are removed in a drum which rolls the capsules between two rubber surfaces moving at different speeds. Uneven ripening and varying thickness of the capsule wall produce a large proportion of unhulled or broken seeds. Fields should preferably be sown with one variety to reduce such loses. The harvesting period may be extended in separate blocks within the field, where several varieties are sown. Capsules must be completely

dry when harvesting commences, and hulling rollers should be accurately adjusted.

It is important to maintain correct working speed by adjusting the machinery frequently. The number or amount of seed remaining in the surface of mechanically harvested fields is related to both plant height and speed of the harvesting machine. There was more seed at the field edges, indicating that as the harvester slowed at row ends, seed losses increases. Several run around the margins prior to harvesting the main block reduced such losses (Smith, 1972) Most harvesting are designed to operate when the relative humidity is below 45%, as moist capsules may remain attached to racemes and do not hull easily. Defoliants like paraquat or diquat at varying rates have been used successfully 10 to 15 days before harvest, where frost or the on set of the dry season does not desiccate plant (Scarpe, 1975). Harvesting of seed crops requires great care and special skill in operating combines. Hand or manual harvesting may be preferred in smaller plots. Hulling can be mechanical, provided they are run at slow speeds, feeding dry capsules regularly. Hulled seeds should be sorted immediately to remove the damaged ones. After harvest, castor plants should be destroyed, preferably by burning, to reduce the subsequent insect and disease infestation. Clusters reaped slightly green or with wet capsules must be dried before hulling. Sun drying or artificial heating may be employed for this purpose prolonged exposure to sun or heat may affect the oil content of seed. Uniform density is required to be maintained while loading a drier with capsules of high moisture content to enhance efficiency of drying. Drying of unshelled castor with moisture

content varying from 14.4 to 34.4% did not produce significant chemical change, although the acid value was altered to some extent (Schoenleber et al, 1966)

In India the harvested produce is usually stored in gunny bags or in bamboo baskets. Experiments have indicated that whatever the method of storage-whether in bags, baskets, or in the open-there is no significant change in the oil content of the seed up to a period of three years. In most parts of India, the spikes or capsules after harvest are collected into heaps over which cow dung water is sprinkled to soften the husk and facilitate dehusking. The heaps thus treated are covered with straw or some such material and weighed to exclude air. In Northern India, the common practice is to bury the capsules in pits and to cover them with dung and earth (Gideon, 1965). This is supposed to ripen the immature fruits, besides softening the capsules to facilitate dehusking. After 4 to 10day, the capsules are spread out in the sun to dry before threshing. Both these practices are undesirable as they reduce the oil content of the beans 2 to 5% and increase the free fatty acid content of the resulting oil.

Investigations carried out in Madras have shown that of the different methods tried, the most efficient and economical method for harvesting and processing castor seed begins when a few capsules are shelled and the beans are extracted (Gideon, 1965).

2.2.5 Storage

Castor seeds are large and occupy considerable space in the store house in relation to their weight. Unlike bagged groundnuts, castor seed cannot be stored in the open except for short periods as both heat and sunlight reduce oil content and

quality. The castor seed must be bagged carefully and handled as little as possible. It is advisable to use wooden scoops or shovels and rubber conveyor belts. Most countries have regulations in force regarding transport of castor seed, relating to size and colour restrictions on carriage as food or feedstuffs. An increase in the fatty acid content of castor oil is normally noticed in about 3 months from the commencement of storage of seeds. This is generally tower than the increase which occurs on storage of crude oil and higher than the increase which takes place on the storage of refined oil

2.2.6 Post-harvest losses of castor seed (pests and diseases)

Castor seed is attached by a variety of pests and diseases, causing a severe decrease in yield and quality. Agioles (Euxon) cut worm, crickets (aryllotalpa sp., Branchytrupes sp., and Grylles sp.); flee beetles (Aphihona whitfieldi Bry (sudan). Hermaeophage ruficellis luc (Israel). Stem borders [Ostrinia nubilasis Hb, Xyleules capensis wlk. (Africa), sphenopetra Arabica, H. armigera, spadoptera liturn and s. littoralis, Euproctis sp. (Africa, Asia)]; and a number of other pests like jassids, leafhoppers and whiteflies attack the castor plants, including inflorescences and seed (Weiss, 1983)

Few pests attack stored castor seed if the testa is unbroken, but damaged seed and press cake can become infested. The most common storage insects pests include the tropical warehouse beetle (Ephestia Cautella), cigarette beetle (Lasioderma Serriocorne), and red flour beetle (Tribolium Castaneum). All these are cosmopolitan types, occurring all over the world.

An important disease attacking the castor flower and fruit is capsule mold caused by Alternaria ricini, Botritis ricini, and sclerotinia ricini. Seeds and seedlings are also attacked by various root rots. (Fusarium sp., Pithium sp., Rhizoctonia sp., and sclerotium sp) and a seedling blight (Phytophthora sp.,). Bacteria leaf spot (Alternaria ricini, Cercospora ricinells, and Pseudomonas sp.) and leaf rust (Melampsora ricini and Pseudomonas sp.) and other important diseases of the castor plant (Weiss, 1983)

2.3 Chemical composition

Castor seed contains between 40% and 60% oil that is rich in triglycerides mainly ricinolein. It consists of two protein ricin and RCA (ricin communis agglutinin). The RCA protein is made up of with four subunits having a molecular weight 120,000 Daltons and ricin 62,000 to 65, 000 Daltons. The proximate composition of castor seed ranges as follows: oil, 45 to 51.8% moisture, 3.1 to 8% protein, 12 to 16% carbohydrate, 3.1 to 7% fiber, 23.1 to 27.2% and ash 2 to 2.2%. (Godin *et al.*, 1971)

2.4 Biological effects of castor seed

Castor seed is an active poison, as the toxic protein, ricin, and the alkaloid, ricinine, are present in it. The seeds of castor beans are very poisonous to people, animals and insects. Just one milligram of ricin (one of the main toxic proteins in the plant) can kill an adult. It acts by inhibiting protein synthesis. The seed is only toxic as the outer shell is broken or chewed open. Due to the above, the seeds should be kept away from children. RCA is not toxic but agglutinates red blood cells of mammals while ricin is toxic but does not agglutinate red blood cells.

2.5 Castor oil: a derivative of castor seed

2.5.1 Chemical composition of castor oil

Castor oil triglyceride is unique in that its major fatty acid is the unsaturated, hydroxylated 12-hydroxy 2-octadecanoic acid, familiar known as ricinoleic acid. The fatty acid composition of typical castor oil is palmitic acid, 2%, stearic acid, 1%, oleic acid, linoleic acid, 3%, and ricinoleic acid, 87%. The fatty acid composition of castor oil is shown in the table below.

Table 2.1 Fatty acid composition of castor oil

Fatty acid	Percent of total
Ricinoleic	89.5
Linoleic	4.2
Oleic	3.0
Stearic	1.0
Palmitic	1.0
Dihydroxylstearic	0.7
Eicosanoic	0.3
Linolenic	0.3

Source : Weiss,1983

2.5.2 Extraction and refining of castor oil

The seed is separated from the spiny husk mechanically (using dehusking machines) or by sun drying in the open until the casing splits. The dehusked beans are cleaned by screening, dehulled from the hard shells by an impact or disk mill decorticator. The hulls and kernels are the separated by aspiration.

Castor oil is extracted by a variety of processes or combination of processes such as hydraulic presses continuous screw presses, and solvent extraction. The most satisfactory approach is via hot pressing using an hydraulic press, followed by solvent extraction to remove the bulk of oil remaining in the press cake (Marter, 1965). Both pressing and solvent extraction may be carried out on a batch or continuous basis, the latter is likely to be a more economical method. Crude oil is a pale straw colour, but turns colourless after refining and bleaching. It has distinct odor, but it can be easily deodorized in the refining process. The unpleasant flavour, slightly acrid with a nauseating after taste remains in all qualities of oil and is usually masked in castor used or medicinal purposes.

Hot pressing via hydraulic presses extracts between 75 and 85% of the oil contained in the castor seed, while the remaining press case has about 12% oil content. Subsequent solvent extraction yields the bulk of the remaining oil together with castor meal with 1 to 2%.

Cold press oil is often sufficiently high quality for immediate end uses, but oil from both hot hydraulic pressing and solvent extraction requires refining. The degree of refining varies partly as function of intended end use, but deacidification, bleaching, and cleaning may be employed.

Alkali refining of castor oil is rarely necessary, since it does not contain a high amount of free acidity in the seed or even after extraction and storage. Solvent-extracted oils and oils obtained from damaged or improperly stored seeds may have higher acidity and colour and may need to be refined (Kulkarni *et al.,* 1977)

2.5.3 The hydraulic screw process

First stage of extraction is pre - pressing using a high pressure continuous screw press - expeller. The expeller usually consists of a barrel containing a stainless steel helical screw. The pitch of the screw flights gradually decreases towards the discharge end, to increase the pressure on the pulp as it is carried through the barrel. (Fellows, 1996). Extracted oil is filtered, and collected in a settling tank. Material removed from the oil, called foot, is fed back into the stream of fresh material. Material discharged from the press, called cake, contains 8 to 10 percent oil. It is crushed into coarse meal, and subjected to solvent extraction with hexane or heptane. Continuous processing is used, based on the principle of counter current flow of solvent and oil bearing material. The oil is removed effectively, as the material comes into contact with increasingly purer solvent. After extraction, solvent is removed by distillation, and their resulting oil is processed in similar manner as oil from the pressing step. (Weiss, 1971)

2.5.4 Solvent extraction of castor oil

The Soxhlet apparatus is used for the solvent extraction of castor oil from the seeds. About 300ml of normal Hexane is poured into round bottom flask and 10g of the seed is placed in the thimble and inserted in the centre of the extractor. The

Soxhlet should be heated at 60°c. When the solvent starts boiling, the vapour rises through the vertical tube into the condenser at the top. The liquid condensate drips into the filter paper thimble in the centre, which contains the solid sample to be extracted. The extract seeps through the pores of the thimble and fills the siphon tube, where it flows back down into the round bottom flask. This should be allowed to continue for 30 minutes and can then be removed from the tube, dried in the oven, cooled in the desiccators and weighed again to determine the amount of oil extracted.

2.5.5 Castor Oil Recovery and Purification

After extraction, the solvent is removed by distillation, and their resulting oil is processed in similar manner as oil from the pressing step. (Weiss, 1971). The oil is further processed so as to obtain it in a purer form.

The steps to refining the crude oil include:

- Settling and Degumming of the oil - Done to remove the aqueous phase from the lipids, and to remove phospholipids from the oil.
- Bleaching - Bleaching results in the removal of coloring materials and oxidation products. (Oil Processing & Purification Information from ANARAC)
- Neutralization - The neutralization step is necessary to remove free fatty acids from the oil. This can be done in one of two ways: (a) Alkali (Chemical) or (b) Steam Stripping (Physical) means.(Vegetable Oils & Fats Processing Info from IISC, India)

- Alkali/Chemical Method: Caustic soda (alkali) is mixed in the proper amounts and the aqueous solution is removed, leaving the neutral oil behind.
- Steam Stripping: This is done under vacuum, to remove moisture, free fatty acids, odor bodies, and other impurities from the oil. As it is performed under vacuum conditions, the oil can be kept at a low temperature, preserving its chemical structure by not subjecting it to temperatures in which undesirable dehydration reactions can occur. (Distillation & Stripping Systems from Sutcliffe & Speakman)

- Deodorization of the oil - Deodorization results in the removal of odour from the oil

2.6 Storage of castor oil

Crude castor oil is generally not stored for a long period. The colour and acidity of crude castor oil stored at high atmospheric temperature do not increase appreciable even after one month storage. Refined castor oil can be stored up to six months to one year with little change in colour or acidity. Both crude and refined castor oil can be stored for one or two years without accumulation of peroxide in significant amounts or increasing the oxidative rancidity (Kulkarni,L.G et al,1977)

2.7 Utilization of castor oil

Owing to its peculiar structure, castor oil is particularly versatile and most of its uses derive from the presence of the hydroxyl group and the close proximity of the hydroxyl group to the double bond in the ricinoleic acid molecule. The castor

oil from cold pressing or refined castors oil from hot pressing can be used directly in a number of end uses: medicines, cosmetic, lubricants, coatings, and disinfectants. In most major castor oil consuming countries, a wide range of modified products of castor oil are manufactured (Marter,1981).The following describes the modification processes applied to castor oil with a diversity of end uses.

- Oxidation, which provides a range of intermediates of varying viscosity, can be employed to produce plasticizers, adhesives, bases for lubricants and hydraulics.
- Sulfonation yields a range intermediates used in textile and lather goods as welting and drying agents.
- Alkoxylation yields propoxylates used in lubricants and hydraulics and ethoxylated derivatives with a range of uses from deformers to cosmetics, detergents, and cutting oils.
- Hydrogenation produces hydroxylstearic acid, which can be used in alkoxylation and may be incorporated as metallic soaps in a wide range of greases.
- Thermal decomposition yields methyl 10-undecylenate or undecylenic acid and heptaldehyde. Methyl 10-undercylenate ultimately yields nylon 11 which has a wide range of applications. The modified undecylenic acid is used in insecticides and fungicides. The heptaldehyde and its derivatives are used in synthetic flavorings or perfumes and lubricants, respectively.

- Thermal alkali treatment, depending upon the conditions or reactions, yields, 10-hydroxyldecanoic acid or sebacic acid. The former is used in the textile industry as a softening agent and for non shrink purposes. The sebacic acid derivatives include nylon 6-10, plasticizers for vinyl, and lubricants for jet engines. (Marter,1981)

Thus, modification processes provide a wide range of intermediate and end uses and in several instances provide a single modification process yielding a variety of industrial products.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Chemical composition of castor seed

The material used in carrying out this project work is the castor seed. It contains between 40% and 60% oil that is rich in triglycerides, mainly ricinolein. It contains ricin and the alkaloid ricinine. Ricin is one of the small groups of phtotoxins which also include arbin, cirnin, crotin and robin. It also contains allergens and one off the allergens has been identified as chlorogenic acid. The fatty acid composition of typical castor oil is palmitic acid, 2%, stearic acid, 1%, oleic acid, 77%, linoleic acid, 3%and ricinoleic acid, 87%. The proximate composition of castor seeds ranges as follows: oil, 45 to 51.8%, moisture, 3.1 to 5.8%, protein, 10 to 16%, carbohydrate, 3.1 to 7%, fiber 23.1 to 27.2% and ash, 2 to 2.2%. (Godin et al, 1971)

3.2 Determination of saponification value

2g of the sample was weighed into a conical flask; 25ml of 0.1N ethanoic potassium hydroxide was then added. The content which was constantly stirred was allowed to boil gently for 60min. A reflux condenser was placed on the flask containing the mixture. Few drops of phenolphthalein indicator was added to the warm solution and them titrated with 0.5m Hcl to the end point until the pink colour of the indicator just disappeared. The same procedures were used for other samples and blank.

3.3 Determination of acid value

The specific quantity of the sample was weighed and 50ml of an ethanol ether mixture was added. The resulting solution was used as the test solution. The solution was cooled and few drops of phenolphthalein TS was added and titrated with 0.1mol/l ethanoic potassium hydroxide to the solvent until its pink colour of the solution persists for 30seconds.

3.4 Determination of hydroxyl value

1g of the sample was weighed accurately and transferred into a round-bottom flask and 5ml of acetic anhydride pyridine was added. A small funnel was placed on the neck of the flask and heated for 1hour while immersing the flask to a depth of about 1cm from the bottom into an oil bath at 95-100 degree celsius. The solution was cooled and 1ml off water was added. It was shaken well and heated for 10minutes. After cooling, the funnel and the neck of the flask was rinsed with 5ml of ethanol and titrated with 0.5mol/l ethanoic potassium hydroxide with 1ml of phenolphthalein TS added as indicator.

3.5 Determination of iodine value

0.4g of the sample was weighed into a conical flask and 20ml of carbon tetra chloride was added to dissolve the oil. 25ml of Dam's reagent was added to the flask using a safety pipette in fume chamber. A stopper was inserted and the content of the flask was vigorously swirled. The flask was placed in the dark for two hours 30 minutes. At the end of this period, 20ml of 10% aqueous potassium iodide and 125mlof water was added using a measuring cylinder and titrated with 0.1M of sodium thiosulphate solutions until the yellow colour almost disappeared.

thiosulphate solutions until the yellow colour almost disappeared. Few drops of 1% starch indicator was added and the titration continued by adding thiosulhate drop wise until blue coloration disappears after vigorously shaken.

3.6 Determination of pH value

2g of the sample was poured into a clean dry 25ml breaker and 13ml of hot distilled water was added to the sample in the beaker and stirred slowly. It was then cooled in a cold water bath to 25^0C. The pH electrode was standardized with buffer solution and the electrode immersed into the sample and the pH value was read and recorded.

3.7 Determination of viscosity

A clean, dried viscometer with a flow time above 200 seconds for the fluid to be tested was elected. The sample was filtered through a sintered glass (fine mesh screen) to eliminate dust and other solid material in the liquid sample. The viscosity meter was charged with the sample by inventing the tube's thinner arm into the liquid sample and suction force was drawn up to the upper timing mark of the viscometer, after which the instrument was turned to its normal vertical position. The viscometer was placed into a holder and inserted to a constant temperature bath set at 29^0C and allowed approximately 10minutes for the sample to come to the bath at 29^0C. The suction force was then applied to the thinner arm to draw the sample slightly above the upper timing mark. The afflux time by timing the flow of the sample as it flow freely from the upper timing mark to the lower timing mark was recorded.

3.8 Determination of peroxide value

5g of the sample was weighed into a 250ml Erlenmeyer flask. 30ml acetic acid, chloroform solution (3:2) was added under a fume hood and swirled to dissolve the oil. 0.5ml saturated KI solution was added and swirled for 1minute. 1ml of indicator was also added and titrated with the solution until the blue colour disappeared and the peroxide value was taken.

3.9 Determination of Refractive index

The refractive index was determined by setting up the refractometer. A drop of the oil sample was applied on the glass and the bulb was shown directly in front of the prism. The control knobs were adjusted and readings were obtained and recorded.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Saponification value

The saponification value is the number of mg of potassium hydroxide (KOH) to saponify the esters in 1 g of the sample and neutralize the free acids in 1 g of a sample.

$$\text{Saponification value} = \frac{(a-b) \times 28.05}{\text{Weight (g) of the sample}}$$

where a = volume (ml) of 0.5 mol/l hydrochloric acid consumed in the blank test,
b = volume (ml) of 0.5 mol/l hydrochloric acid consumed in the test.

The experiment goes thus:

Titration (a) = 11.2

Titration (b) Blank = 23.2

Weight in g of sample = 2.2989

$$\text{Saponification Value} = \frac{(23.2-11.2) \times 28.05}{2.2989}$$

$$= 146$$

The highest accuracy in the titration is most important. The blank can be omitted if aqueous alkali is used (Hartman and Antunes, 1971).

4.2 Acid value

The acid value is the number of mg of potassium hydroxide (KOH) required to neutralize 1 g of a sample.

$$\text{Acid Value} = \frac{\text{Titration (ml)} \times 5.16}{\text{Weight of sample used}}$$

Titration (ml) = 1.4ml

Weight (wt) of sample used = 2.0611

(1ml 0.1M sodium hydroxide = 0.0282g oleic acid) = 0.0282g

Percentage = 100

$$\text{To Calculate FFA} = \frac{1.4\text{ml} \times 0.0282 \times 100}{2.0611\text{wt}}$$

$$= 1.92$$

To Calculate Acid Value = FFA (1.92) x 2 = 3.84

4.3 Hydroxyl Value

The hydroxyl value is the number of mg of potassium hydroxide (KOH) required to neutralize acetic acid combined to hydroxyl groups, when 1 g of a sample is acetylated under the following conditions.

$$\text{Hydroxyl value} = \frac{(a-b) \times 28}{\text{Weight of sample}} + \text{acid value}$$

$$\text{Hydroxyl value} = \frac{(23-11.2) \times 28}{2.05} + 3.84$$

$$= 165.01$$

4.4 Iodine value

The iodine number (or "iodine adsorption number" or "iodine value") in chemistry is the mass of iodine in grams that is consumed by 100 grams of a chemical substance. From the experiment, the iodine value was found to be 87.8

4.5 pH value

pH is a measure of the activity of hydrogen ions (H^+) in a solution and, therefore, its acidity or alkalinity. For dilute solutions, however, it is convenient to substitute the activity of the hydrogen ions with the molarity (mol/L) of the hydrogen ions (however, this is not necessarily accurate at higher concentration. Though a pH value has no unit, it is not an arbitrary scale; the number arises from a definition based on the activity of hydrogen ions in the solution. The precise formula for calculating pH is:

$$pH = -\log_{10} [H^+]$$

From the experiment, the pH of castor oil was found to be 6.14

4.6 Refractive index

This is the angle through which a beam of light is bent when passing through a thin film of melted fat or oil. For determining the refractive index using the Abbe refractometer and sodium vapour lamp the following temperatures are employed, 20°C for oil, 40°C for solid fats which are fully molten at that temperature, 60°C for hydrogenated fats and 80°C for waxes. Equations for correction readings for temperature are given by the International Unit of Pure and Applied Chemistry IUPAC (1979). From the experiment, the refractive index at 25.3°C = 1.4690

4.7 Peroxide value

The PV is defined as the meq. peroxide/kg_{sample}. The test is a volumetric one where I_2, formed from potassium iodide in the presence of peroxides, is titrated with thiosulfate. This means $meq._{peroxide} = meq._{thiosulfate}$ at the equivalence point. Since meq. = NV when volume is in units of ml, we have

$PV = meq._{thio}/kg_{sample}$

$= N_{thio}V_{thio}/kg_{sample}$

$= (0.01\ meq./ml)(V_S - V_B)1000/g_{sample}$

$= 10(V_S - V_B)/g_{sample}$

V_S = volume of thiosulfate required to titrate the sample

V_B = volume of thiosulfate required to titrate the blank

Peroxide Value = $\frac{(V - V_o)\ T \times 10^3 mEq/Kg}{M}$

Titration minus (-) Blank = 2.0

Molarity of thiosulphate solution = $0.002 \times 10^3 \times mEq/Kg$

Weight of sample = 1.6173

Calculation Goes Thus:

$$\frac{(\text{Titration} - \text{Blank}) \times \text{molarity of thiosulphate solution}}{\text{Weight}}$$

$$\frac{2.0 \times 0.002 \times 10_3 mEq/Kg}{1.6173} = 2.47mEq/Kg$$

4.8 Viscosity

The viscometer was used in determining the viscosity and the value was found out to be 8.93.

Table 4.1 Results of Chemical Analysis of Castor Oil

PARAMETERS	RESULTS
pH	6.14
Refractive Index at 25.3°C	1.4690
% FFA As Oleic Acid	1.92
Acid Value	3.84
Iodine Value	87.8
Hydroxyl Value	165.01
Peroxide Value	2.47
Saponification Value	146.42

Table 4.2 International Specifications of Castor Oil

Characteristic	**British Standard**	**U.S. No. 1**
Acid value	4	3
Saponification value	177-187	179-185
Iodine value	82-90	82-88
Hydroxyl value	156	161-169
Refractive index, 20°c	1.477-1.481	1.473-1.477
Specific gravity	0.958-0.969	0.961-0.963

Source: Weiss, E. A., 1983

4.9 Discussion of results

From the result obtained, the Saponification number was found to be 146.42. This gives information concerning the character of the fatty acids present in the castor oil and in particular concerning the solubility of the soap derived from it in water. The higher the Saponification number, the more soluble the soap that can be made from it. From International specifications, the Saponification value is said to be between the values of 177 and 187. The value derived from the experiment is quite low and this might be due to the inefficiency of the equipment used during the experiment. The iodine number from the experiment carried out is found out to be 87.8. This simply tells us about the degree of unsaturation of the oil. When compared to International specifications, it is okay. The value of the iodine number obtained from the experiment indicates high drying qualities and this is very much needed in the paint manufacturing industry. Most of the results obtained such as the hydroxyl value, acid value e.t.c are in agreement with International specifications. The pH from the experiment was found out to be 6.14. This parameter determines the shelf life of the oil as regarding its storage. The lower the value, the shorter the shelf life of the oil as this makes the oil goes rancid over a short period of time. The peroxide value was found out to be 2.47mEq/kg. Fresh oils usually have peroxide values well below 10mEq/kg. A rancid taste often begins to be noticeable when the peroxide value is between 20 and 40 mEq/kg. In interpretation such figures, however it is necessary to take into account the particular oil or fat involved.

From the experiment, the value of the viscosity was determined to be 8.93 which when compared to International specifications is quite within the range. Viscosity as a property of castor seed oil must be known because its packaging as well as the storage depends largely on it.

The refractive index was also obtained to be 1.4690 at 25.3^0C. This depicts the angle through which a beam of light is bent when passing through the oil and is very applicable at different temperatures in making waxes. International specifications shows that at 20^0C which is a lower temperature compared to that of the experiment, the refractive index is averagely 1.48

The acid value was also gotten to be 3.84 from the experiment and this falls within the range of International specification.

CHAPTER FIVE

5.0 CONCUSION AND RECOMMENDATION

5.1 Conclusion

To an extent, this work has been made to determine some of the chemical properties of castor seed. A proper knowledge of the various parameters such as the acid value, hydroxyl value, pH, iodine value, viscosity, refractive index, peroxide value studied during the course of this work will further enhance scientific research and development as regarding its processing and usage. It will also help in the design of machines used for castor processing as well as plants.

5.2 Recommendations

Some of the limitations encountered in the course of this study include non-familiarity as well as scarcity of the seeds in some regions of the country. I recommend that the seed should be widely grown in large quantities in all parts of the country. Also effort should be made to create a public awareness of the seed itself as well as its derivatives and usage. Owing to its poisonous ability, research institutes should take it upon themselves to design and fabricate machines which will help in the grinding and processing of castor seed and other seeds with related chemical properties.

REFERENCES

Canvin, B. T (1879, 1963). The Formation of Oil in the Seed of Ricinus Communis L., Can. J. Biochem., Page 41.

David Gibbon and Adam Pain., Crops of the Drier Regions of the Tropics

FAO, Production Yearbook, (1981). Vol. 34, Food and Agriculture Organization, Rome,

Gideon, C. A, (1965) Castor Cultivation in India, Indian Central Oilseeds Committee. Hyderabad, India.

Godin, V. J. and Spensely, P. C, (1971). Oils and Oilseeds, Crop and Product Digest. No. 1, Tropical Products Institute, London, Page 8.

Kulkarni, L. G. and Ramanamurthy, G. V, (1977). in Castor, Rev. ed., Jaiswal, P. L., Wadhwani, A. M., and Singh. R., Eds., Indian Council of Agricultural Research, New Delhi

Marter, A. D, (1981). Castor: Markets, Utilization and Prospects, Tropical Products Institute (G152), London..

Popova, E. V, (1963). Castor Oil Plant, Lenin Academy of Agriculture, Scientific and Industrial Application. Botany Men. Culture, Lenin, U.S.S.R.,

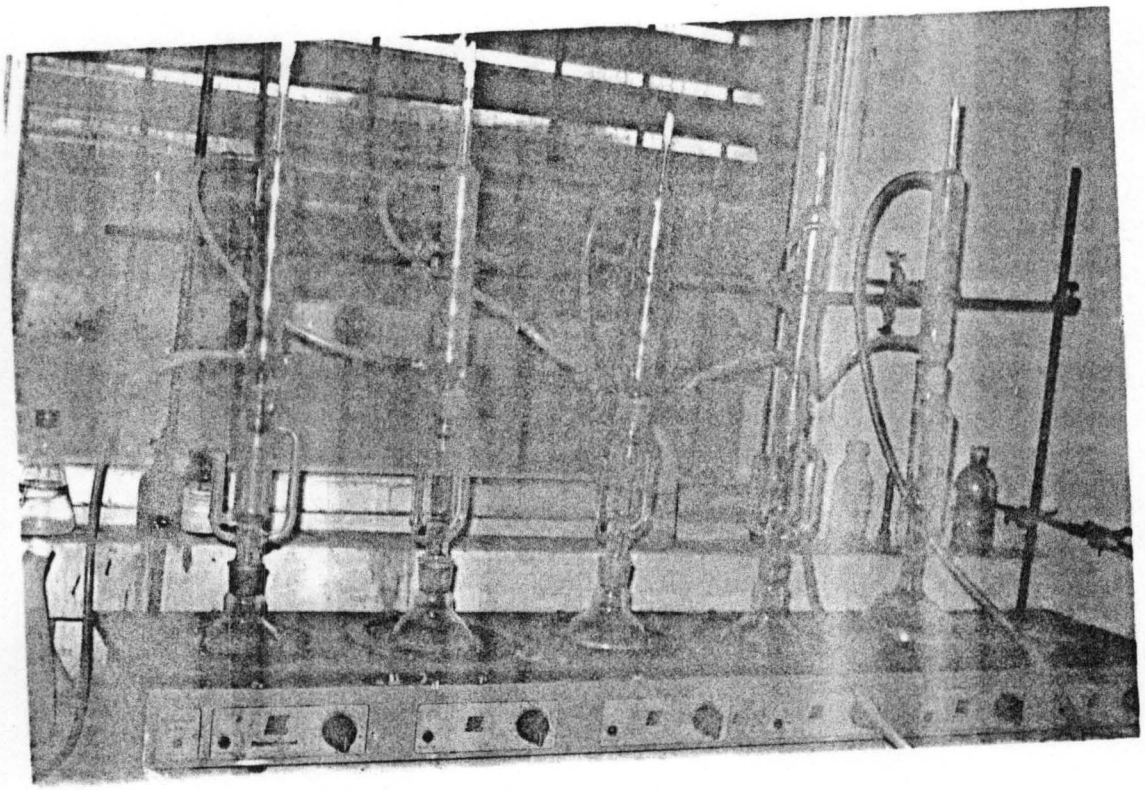
Smith, D. T, (1972). Survival and Emergence of Volunteer Castor Seed in soil, Argon. J., 64(6), 799,

Salunkhe D. K and Desai B. B Post Harvest Biotechnology of Oilseeds

Weiss, E. A, (1971). Castor, in Sesame and Safflower. Leonard Hill, London,

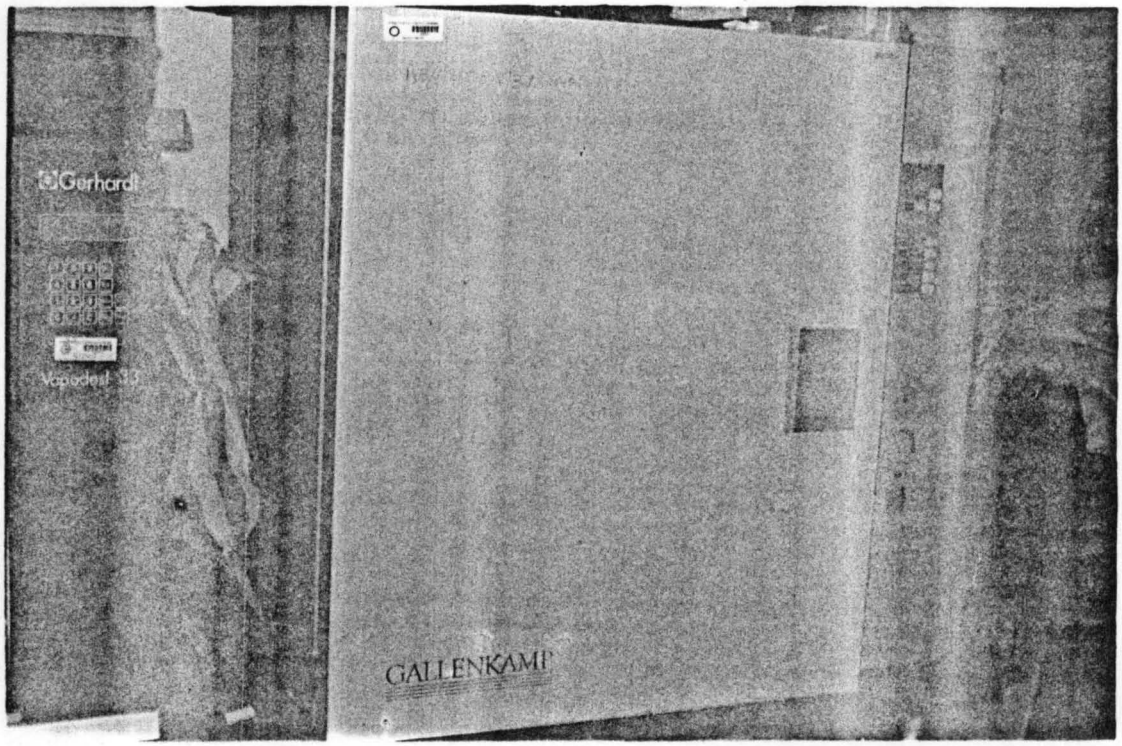
Weiss, E. A. (1983), Oilseed Crops, Tropical Agriculture Series, Longman, London.

APPENDIX A



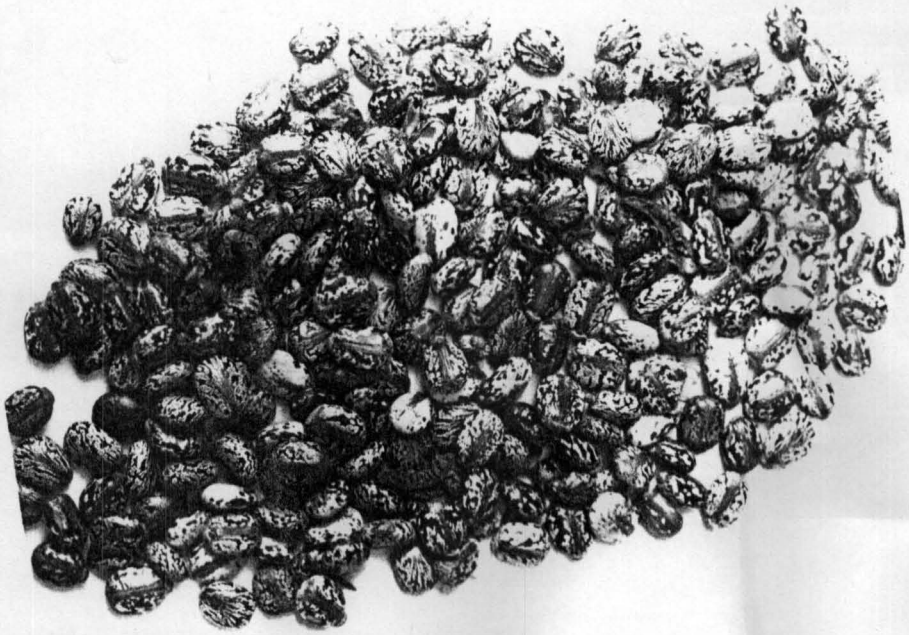
The Soxhlet Apparatus used in extracting the oil from the castor seed

APPENDIX B



The Oven used in drying the seeds before grinding and extraction of the oil from the castor seed

APPENDIX C



A Picture Showing Unshelled Castor Seeds