EXTRACTION OF CASHEW NUT SHELL LIQUID FROM CASHEW NUT SHELL

BY

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Degree Of Bachelor Of Engineering
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DECLARATION

I hereby declare that project is solely the result of my work under the supervision of Dr.M.O. Edoga. All literature cited have been duly acknowledged in the reference.

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12-12-2005
DATE

CERTIFICATION

This project titled EXTRACTION OF CASHEW NUT SHELL LIQUID FROM CASHEW NUT SHELL by Aitzaz Ashraf of chemical Engineering Department, F.U.T., Minna ,meets the requirements for industrial development, and hence hold the merit for the award of the degree of Bachelor of Engineering in chemical Engineering of Federal University of Technology, and approved for its contributions for industrial development and utilization of local materials .

2005/1212

DR.M.O.EDOGA
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DATE

EXTERNAL EXAMINER

DATE

DEDICATION

This project is dedicated to my late Father Mohammed Ashraf Chaudhry.

ACKNOWLEDGEMENT

My greatest thanks go to ALLAH for his protection and guidance throughout my academic pursuit.

I express my profound gratitude to Dr. M.O. Edoga who supervised this project and for making his time available to meticulously scrutinize this project work despite his tight academic schedule.

I wish to thank the H.O.D and all the members of staff of Department of Chemical Engineering.

I also thank all my colleagues and friends who either consciously or unconsciously supported or encouraged me.

ABSTRACT

This research work was aimed at studying extraction of Cashew nut shell liquid (CNSL) in order to determine some of the parameters that may be required for the design of a full scale plant for the extraction of CNSL from cashew nuts. Specifically, the present work attempted separating the active principle (CNSL) in monohydric phenols and dihydric phenols, respectively.

The CNSL was extracted from cashew nut shell by both direct leaching (direct contact) and indirect leaching (soxhlet apparatus). The yields obtained from these two methods were compared. In this work, the only solvent used was hexane .The experiment was also conducted by varying the temperature and the yields obtained were compared. The infra red analysis of the samples was carried out.

Result shows that there is a higher yield by using the direct leaching approach than the use of soxhlet apparatus.The rate of extraction was found to increase with increase in temperature.

The separation of the polyphenol from the CNSL resulted into two distinct layers with the polyphenol taking a larger portion of the separating funnel; and being denser ,it was drawn out from bottom of the funnel .The shake-up experiment broke the polyphenol into momohydric phenols and dihydric phenols respectively.

The infrared spectra show the presence of (Phenyl-group) in the cashew nut shell liquid (CNSL).

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CHAPTER ONE

1.0 INTRODUCTION

1.1 Background

Cashew nut shell liquid (CNSL) obtained from the cashew tree botanically known as anacardium occidentale contains cardanol, which has a long aliphatic side chain .Cashew nut shell liquid (CNSL) is a versatile by-product of the cashew industry.

The nut has a shell of about 1/8 inch thickness inside which is a soft honey comb structure containing a dark reddish brown viscous liquid. It is called cashew nut shell liquid, which is the pericap fluid of the cashew nut. It is often considered as the better and cheaper source for unsaturated phenols. CNSL has innumerable applications in polymer based industries such as friction linings, paints, varnishes, laminating resins, rubber compounding resins, cashew cements, polyurethane based polymers, surfactants, epoxy resins, foundry chemicals and intermediates for chemical industry. It offers much scope and varied opportunities for the development of other tailored polymers. CNSL resins have been used extensively in the manufacture of friction-resistant components in applications such as brake and clutch linings. These resins are used in forming friction ingredients in form of time dust obtained from the completely cured resins and also as binders for friction ingredients. CNSL-aldehyde condensation products and CNSL-based phenolic resins are used in applications such as surface coatings, adhesives, varnishes and paints. Various polyamines synthesised from CNSL or cardanol are used as curing agents for epoxy resins. CNSL and its derivatives have been used as antioxidants, plasticisers and processing aids for rubber compounds and modifiers for plastic materials. Resins based on the reaction products of cardanol, phenol and formaldehyde are used to improve the resistance of rubber articles to cracking and ozone degradation. CNSL-based cardanol and cardol are all used to provide oxidative resistance to sulfur-cured natural rubber products. Cardanol-CNSL or sulfonated-CNSL is added to rubber gum stock or nitrile rubber to improve the processability, mechanical properties and resistance to crack and cutting properties of the vulcanites.

A number of products based on CNSL are used as antioxidants, stabilisers and demulsifiers for petroleum products. Metal xanthates of partially hydrogenated, sulfurised cardanol is used to lower the pour point of lubricating oils as well as acting as antioxidant and anticorrosive properties. Soluble metal derivatives of CNSL are used to improve the resistance to oxidation and sludge formation of lubricating oils. Oxidised CNSL and its derivatives are used as demulsifying agents for water in oil type petroleum emulsions.

1.2 Aims and Objectives

This study aims at extraction of CNSL in order to determine some of the parameters that may be required for the design of a full scale plant for the extraction of CNSL from cashew nuts. Specifically, the present work will attempt to separate the active principle extract (CNSL) in momohydric phenols and dihydric phenols respectively.

1.3 Approach

There are three different methods generally used in extracting cashew nut shell liquid from cashew nuts. They are mechanical, roasting and solvent extraction .Due to time and equipment limitation , only solvent extraction will be used in this study. Two types of methods of solvent extraction can be used.These include soxhlet extractor and the direct contact method . In this work, the only solvent is used is hexane. It is anticipated that from the combined methods (direct and indirect leaching) of the extraction that 90% cashew nut shell liquid would be recovered.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Cashew Tree

The cashew tree is native of Brazil. It was discovered by the Portuguese and introduced in their African and Asian colonies. At present, India is by far the largest producer of cashew nuts. The earliest reports about cashew in Brazil, come from French, Portuguese and Dutch observers. The French naturalist Thevet (1558) described the tree and provided the first drawing of the cashew showing the local people harvesting fruit and squeezing juice from the "apples" into a large jar. Gandavo (1576) was the first Portuguese writer to describe the cashew and state that its kernels were better tasting than almonds. Many others described both botanical and agricultural information about cashew and its local use. The Tupi natives of Brazil called the cashew "acaju". The name became "caju" in Portuguese, "cashew" in English, "cajuil" in Spanish and "acajou" or "cajou" in French. The latter term is used because acajou refers to mahogany.

Ayurveda, the Indian system of medicine originated 3000 years ago, has accepted this rich nut as a potent remedy as well as a nutritive food. The ayurvedic way of looking at health and healing is very different from the Western medical concepts : it is based on some philosophical aspects (how the body is related to its natural environment, to the cosmos, to time, what is its psychic role, what are its unconscious levels...). Ayurveda compares the body to a field in perfect harmony with nature, which can produce good or bad things. No bad thing such as illness can come from an external cause only: it is due to irregular living patterns and improper eating habits which destroy the natural balance of the body. When correcting this wrong way of living, the organism can renew its metabolic activities, particularly with natural diet : herbs, nuts, berries, a minimum amount of meat... The Ayurveda school of medicine is actually a compendium of herbal knowledge.

As for nuts in general, they have been popular with the people of the Indus valley: almonds were good for eye problems, walnuts were brain tonics, and cashew, introduced in Ayurveda texts in the 16th century, was used as a stimulant, a rejuvenator, and an appetizer medicine. Cashew nuts are also popular in African pharmacopoeia.

2.2 Cashew Nut Shell Liquid (CNSL)

The production of cashew nut shell liquid stems from the fact that in past years, the most desirable part of the cashew was the kernel of the nut .In order to get to this , one has the liquid of the nut perciarp (CNSL) to remove first . This liquid is both caustic and poisonous and any attempt to crack the nut without some form of traditional practice to extract the liquid by roasting will be difficult to handle. However, when the processing of cashew nut attained an industrial scale, it was discovered that cashew nut shell liquid , apart from being the by-product of kernel production also has a variety of uses .

The production of cashew nut shell liquid is a recent development which started at the beginning of this century, and gradually developed into an important foreign exchange earner with the annual production for export in various countries soaring considerably.Cashew nut shell liquid is a pale brown viscous oil made up of anacardic acid, cardanol and cardol. The development of the use of cashew nut shell liquid (CNSL) in commerce can be traced from

Cardolite Corporation and its predecessors 3M Company and Irvington Varnish and Insulator Company.

Harvel Corporation imported the first commercial quantity of CNSL in 1926 in the United States. CNSL was known for centuries prior to this, but no significant commercial uses had been developed. Dr. Harvey of Harvel corporation recognized that the unique chemical structure (a meta substituted alkenyl phenol) should have commercial use.

Harvel entered into a joint venture with Irvington Varnish and Insulator Company for the purpose of developing commercial uses of CNSL. Irvington and Harvel in the 1930's developed the first major (and still the largest) use of CNSL which is cashew friction particle in the brake lining industry. Other uses for coatings were developed using its phenolic nature and its properties as a drying oil, but none was long lasting. However, in 1946 the business was large enough to justify construction of a dedicated factory to the production of derivatives of CNSL in Newark, New Jersey.

In 1953, commercial production of cardanol began in Newark. Now, whole new families of products were possible. Cardolite© NC-510, n pentadecylphenol, found use as an intermediate in color film. In 1954, 3M developed the monoglycidal ether of cardanol . By the late 50's the NC-513, especially, found extensive use in epoxy chemistry, and its use continues to this day. Liquid cashew resins were introduced as binders in automotive strip linings. In the 1970's, 3M developed amine functional curing agents based on CNSL and gave them the generic name "phenalkamines." By this time phenalkamines had shown great promise in the heavy duty coatings market because of all season cure, good adhesion to poorly prepared surfaces, and good corrosion resistance. Cardolite (new name aquired from 3M Management luyout) and its few key customers pioneered the growth of phenalkamines in the late 80's and early 90's.

Cardolite Corporation continues to focus on advancing the use of CNSL in epoxy coatings and friction materials. Cardolite is, by far, the world's largest cardanol producer. Newer phenalkamines have been developed which preserve the properties of the first generation products but which have lower viscosities and lighter colors. Other new raw materials from CNSL are produced that will aid epoxy and friction formulators meet the demands of the next century.

2.3 Cost Effective Modern Material

In the search for cost effective modern materials, CNSL and its products have a significant role to play Being renewable, it offers much advantage over synthetics. Its versatility stems from its innumerable applications in many areas. Recent research has shown that the constituents of CNSL possess special structural features for transformation into speciality chemicals and high value polymers. This involves a value addition of many orders of magnitude and the chemical transformation provides 100% chemically pure products. Thus, CNSL offers vast scope and opportunities for the production of speciality chemicals, high value products and polymers. Production The total annual production of CNSL in India is around 15,000 tonnes whereas the potential availability is around 45,000 tonnes. The world production is around 125000 tonnes.

2.4 Composition of Cashew Nut Liquid

Natural (i.e. cold, solvent extracted) CNSL contains approximately 70% anacardic acid (Fig2.1), 18% cardol, and 5% cardanol, with the remainder being made up of other phenols and less polar substances. As can be seen in Figure 1, anacardic acid, cardanol and cardol consist of mixtures of components having various degrees of unsaturation in the alkyl side-chain.

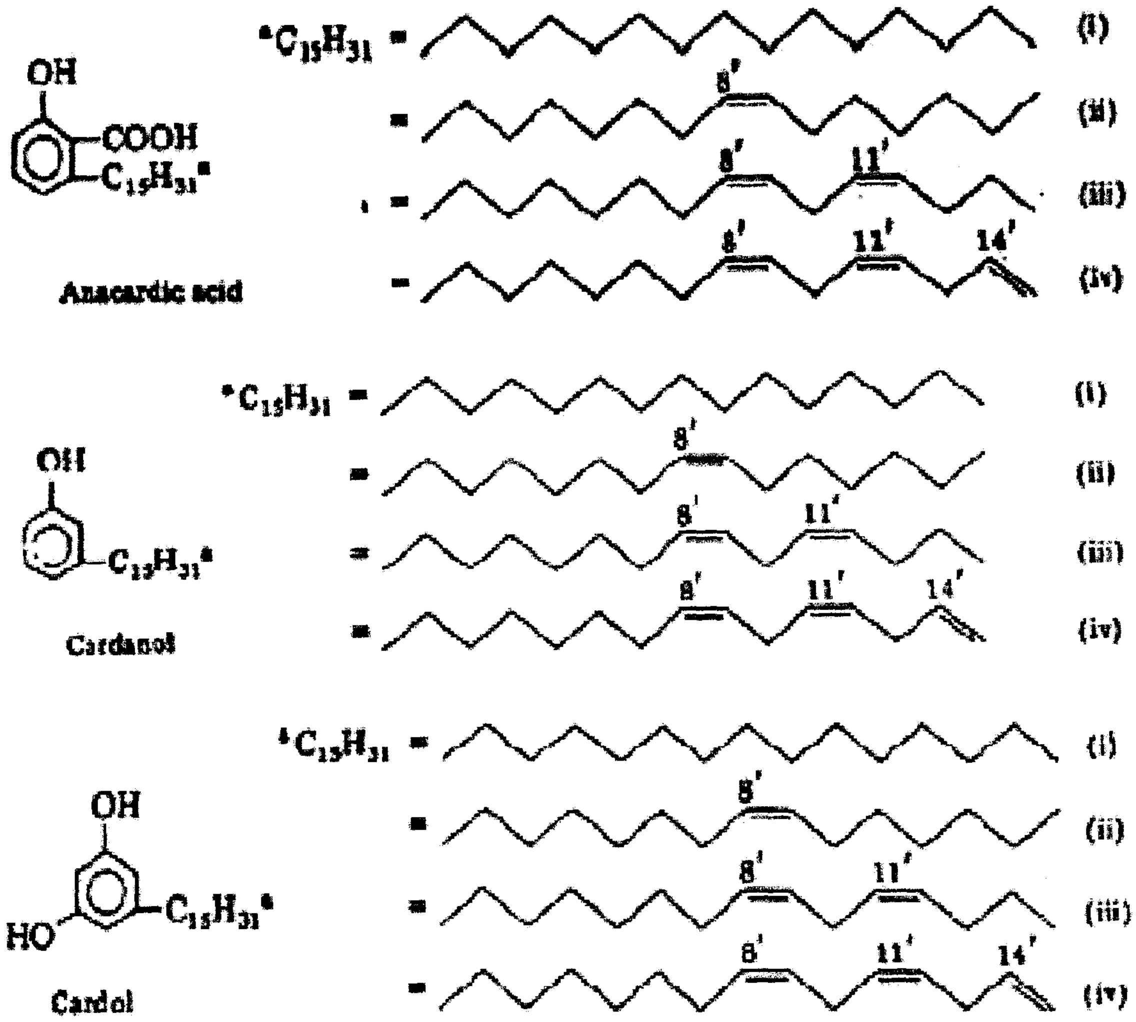


Figure 2.1: Structure of Anacardic Acid, Cardanol and Cardol

In technical (i.e. heat extracted) CNSL, the heating process leads to decarboxylation of the anacardic acid to form cardanol. Typically, the composition of technical CNSL is approximately 52% cardanol,10% cardol, 30% polymeric material, with the remainder being made up of other substances.

The technical CNSL is often further processed by distillation at reduced pressure to remove the polymeric material. The composition of distilled technical CNSL is approximately 78% cardanol, 8% cardol, 2% polymeric material and the remainder other substances.

Table 2.1 summarises the composition of typical batches of technical and distilled technical grades of CNSL.

Table 2.1: Composition of typical batches of Technical and Distilled CNSL

	Cardanol	Cardanol Monoen	Cardanol Diene	Cardanol Triene	Cardol diene	Cardol triene	Polymer	dentifed
T-CNSL	0.06	17.10	10.78	24.42	2.36	7.50	30.6	5.83
D-CNSL	-	25.9	16.2	35.8	2.04	5.90	2.5	9.70
AT-CNSL	0.09	24.7	15.6	35.3	3.41	10.8	-	8.42

T-CNSL = Technical grade, D-CNSL = Distilled grade, AT-CNSL = Technical grade component percentages adjusted for removal of polymer.

2.5 Products From CNSL

CNSL is a mixture of four components: all are substituted phenols - anacardic acid, cardanol, cardol and 2-methyl cardol. The first two are monohydric phenols whereas the other two are dihydric phenols. In the nut, CNSL occurs mainly as anacardic acid(~90%) and cardol around slightly lower than 10%. During the hot-oil bath process for extraction of CNSL, anacardic acid gets decarboxylated to cardanol. So in the technical grade CNSL, the main components will be cardanol and cardol and of course, some polymerised CNSL. CNSL can be extracted by the expeller method but the oil has to be heated after extraction to convert anacardic acid to cardanol. The expelled and heated CNSL will have less amount of polymerised CNSL. However, if there is a requirement for pure monomers, the best source will be solvent extracted CNSL. Each component again is a mixture of four structurally related monomers, the difference being only in the degree of unsaturation. Thus, cardanol is a mixture of a four components: saturated (~5%), monoene (~49%), diene(16.8%) and triene (29.3%). (This makes the chemistry of addition polymerisation essentially complex). Thus, CNSL contains a total of 16 components, which makes it a complicated system.

2.5.1 Advantage of CNSL based polymers

- Improved Flexibility and reduced brittleness.
- Solubility in Organic Solvents.
- Improved Processability.
- Low Fade Characteristics for Friction.
- Resistance to 'Cold Wear'.
- Good Electrical Resistance.
- Better Water Repellence.
- Improved alkali and acid resistance.
- Compatibility with other polymers
- Antimicrobial Property.
- Termite and Insect Resistance.
- Structural Features for Transformation into High Performance Polymers.

2.5.2 Polymerization characteristics of CNSL

CNSL can be polymerised by a variety of methods:

- Addition Polymerisation through the side chain double bonds using cationic initiators such as sulphuric acid, diethylsulphate etc..
- Condensation Polymerisation through the phenolic ring with aldehydic compounds.
- Polymerisation after Chemical Modification to introduce speciality properties.
- Oxidative Polymerisation.
- Various Combinations of the above.

2.5.3 Industrial significance of CNSL

- Low Cost Phenol
- Versatility in Polymerisation and Chemical Modification
- Possibilities for Development of High Performance Polymers
- Property advantage over phenolics in certain applications such as impact resistance; flexibility, faster heat dissipation etc. Reactivity CNSL undergoes all the conventional reactions of phenols.

2.6 Cardanol

Cardanol is a naturally occuring Phenol manufactured from Cashew Nut Shell Liquid. It is a monohydroxyl Phenol having a long hydrocarbon Chain (C15H27) in the meta position. The products obtained from Cardanol have many advantages over these manufactured from other substituted Phenols. It is therefore widely used in the manufacture of surface coatings, Insulaating

Varnishes, Oil and Alcohol soluble resins, Laminating resins, Rubber compounding, Azo dyes, etc. The components of commercial Cardanol differes in the degree of unsaturation of the side chain but for the practical purposes it can be represented by the following formula.

C6H4(OH)-(CH2)7-CH=CH-CH2-CH=CH(CH2)2 -CH3

The average unsaturation of about two double bonds in the side chain of the Cardanol molecules make cross linking easy and give a satisfactory gradual drying and baking properties to paints prepared from it. Because of its peculiar structure Cardanol Varnishes have high electric Insulation, greater resistance to water chemicals and good flexibility. The long hydrocarbon side chain imparts to Cardanol aldehyde condensate greater solubility in drying Oils such as linseed DCO or tung Oil and aliphatic hydrocarbons.

Cardanol can be substituted by 30% in phenolic resins used for phenol bonded resins for plywood and lamination industries. Water solubale PCF resin(phenol, cardanol pharmaldehyde resin): We develop PCF resin by substituting 30% cardanol in phenolic resins. The resins will give additional property of antifungus and antithermic when used in plywood industries. In lamination industries it gives additional advantage in non-edge cracking and also cost saving. Distillation of CNSL under reduced pressure gives cardanol. The residue will be rich in cardol and is generally known as residol, which is conveniently used in the preparation of friction dust for brake linings, and also in rubber compounding formulations.

2.6.1 Friction lining materials

CNSL and cardanol based resins have found extensive uses in automotive brake lining applications as binders/friction dust. Although CNSL-Formaldehyde (CF) resins alone wouldn't meet the required mechanical properties, it improves impact properties and reduces fade considerably by dissipating heat faster than phenol-formaldehyde (PF) resins. Moreover, it imparts better water repellence, which is required in wet condition? CF resins give rise to a softer material, which is more efficient in 'cold wear'. Above all, the cost of CF resins is always lower by factor of 3 or more than that of PF resins. Addition of friction dust gives a silent braking action, which is highly desirable in modern times. About 12,000 -15,000 tonnes of brake linings are produced for use in motor cars every year in the country With the increasing use of automobiles, there is large scope for increase in demand for brake linings.

In brake lining materials, two types of CNSL products are used:

- CNSL resin as matrix resin as a partial substitute for phenolic resin to reduce cost
- CNSL based friction material (friction dust) to modify the friction and wear properties of brake linings

Property Advantages of CNSL Resins in Brake Linings over Phenolics

- Faster Heat Dissipation
- Lower Fade
- Better 'Cold Wear'
- Improved moisture resistance

2.6.2 Modified CNSL resin

Modified CNSL Resin can substitute PF totally and meet all the specifications of brake linings. Additionally, it improves impact properties, reduces fade. Cashew modified phenolics and CNSL-furfural reaction products, although expensive, give superior properties to that of straight cashew binder.

2.6.3 CNSL based friction dust

Friction dust is added to brake linings to modify the frictional and wear properties of brake linings. It also provides similar properties as that of CNSL matrix resin. Formulations for improved skid resistance and low brake noise have been reported. The friction dust is generally prepared by cross-linking CF resin with hexamine and paraformaldehyde and powdering the product to the required specifications. Modified friction dust for applications in 'hot wear' conditions can be prepared from Modified CNSL Resin or from borated CNSL resin. Borated friction dust is known to be especially used in the production on air brake pads. Additionally, they wouldn't catch fire during transportation as is reported to have happened in the case of CF based friction dust.

2.6.4 Surface coatings

CNSL based surface coatings possess excellent gloss and surface finish with optimum levels of toughness and elasticity It is widely known that CNSL resin is added to synthetics by paint/varnish manufacturers to control properties and to reduce cost. Its anti-termite and anti- microbial properties are well known from very ancient times as its use in protecting bottom of the boat hulls speaks out. Because of its dark colour, its outlets are restricted to anticorrosion primers, black enamels, marine paints etc. Recently, the Regional Research

Laboratory, Thiruvananthapuram, has developed a transparent resin from CNSL that can be used as a base for paints of all colours.

CNSL resins give excellent lacquers with superfine surface finish and gloss. The dried film of this lacquer is superior to ordinary oil paints in resistance against vegetable and mineral oils, grease, moisture and chemicals. CNSL resins based varnishes possess good insulating properties apart from its high water repellence and low dielectric properties. Although CNSL and its resins are highly susceptible to fire and burn easily, they can be successfully made fire resistant by incorporating flame retardant elements chemically or flame retardant fillers physically. Chlorinated CNSL pigmented with sodium silicate, red mud titanium dioxide, mica powder or similar materials is known to be prepared and used by industries as flame retardant varnish. This flame retardant has to be blended with the surface coating for optimum performance and a self-extinguishable coating will be obtained.

CNSL resins alone or in combination with other resins show excellent water and weather proofing and can be used for protection of roofs. An anti-corrosive primer developed from CNSL shows excellent properties for application as protective coating for ships' bottoms. The coating withstand alkalinity normally encountered with cathodically protected steel hulls. Rust inhibiting zinc rich primers can be prepared from CNSL. Coatings giving tough elastic films are reported from CNSL-glycerine reaction products. A coating based on residol is used to protect the interior of ferro-concrete domes used for the collection of gobar gas.

2.6.5 Foundry core oil and other chemical

Application of CNSL as foundry core oil shows its versatility CNSL resins are known to impart good scratch hardness to sand cores after baking them. It also provides resistance to moisture and weathering, good green strength and surface finish to moulded articles. It particularly replaces linseed oil in this respect. Modified CNSL Resin when used as core binder was found to improve collapsibility of the core and enhances bench life and anti-damp behaviour in comparison to conventional core binders.

2.6.6 Laminating resin

To reduce brittleness and to improve flexibility of the laminates, CNSL or cardanol derivatives are extensively used in the laminating industry Resins of this type are produced by the co-condensation of phenol, CNSL and formaldehyde. The resins also exhibit improved age hardening and better bonding to the substrate. The lamination industry uses 900-1000 tonnes of CNSL for production of cardanol and for the manufacture of laminating resins. A typical formulation for resin production for particle board contain CNSL resin 90, xylene-10, 40% formaldehyde -35-85, sodium hydroxide-2, and water-10.

2.6.7 Rubber compounding resins

The rapid growth of rubber industry has accelerated demand for new ingredients which are used in the compounding of rubber for vulcanisation. Incorporation of CNSL products in rubber improves tensile strength and abrasion resistance, reduces fatigue, enhances self adhesion and rubber to cord adhesion and contributes to antioxidant and antiozonant activity. The fast curing cashew modified phenol-formaldehyde resins enhance the resistance of the product to ageing, chemical attack and the action of solvents and acids. The residol mentioned earlier is said to have properties much superior to that of pine tar, which thus gets replaced in rubber formulations.

2.6.8 Cashew cements

Polymer based cements are now widely used because they give good adhesion and are unaffected by moisture, acids and alkalis. The phosphorus modified CNSL resin is most suitable for this purpose. This material adheres well to porous bricks, steel and concrete and could be set by gentle heat or by addition of curing agents. Thus, Anorin-38 bonds bricks much more efficiently and is resistant to acids and alkalis so that it could be used to cement floors which are subject to chemical attack. One of the most useful applications will be to seal leaks in the concrete roofs. This material can be admixed with a curing agent and made in the form of a putty which can be introduced to the cavities of the leaks by mild heating when it sets to a solid to fill the cavities.

2.6.9 Epoxy resins

Epoxy resins offer properties much superior to those of polyester and phenolics. A subsidiary of 3M company USA is known to market a high impact adhesive by name 'Cardolite5' made from cardanol. It is reported that the epoxy polymer is made by acid catalysed electrophilic reaction of phenol with cardanol to get a'biphenol which is then epoxidised. The presence of the side chain assures improved flexibility and impact resistance over that of the conventional epoxies available in the market.

2.6.10 Wood composites and CNSL based adhesives

Speciality wood products have been made and marketed, by in-situ polymerisation of certain monomers after suitably incorporating them in wood. As cardanol as such fails to get polymerised by the conventional free radical or high energy irradiation methods, it requires special methods). CNSL based adhesives, however, are reported to exhibit admirable properties to meet the growing demand for quality and durability in bonding plywood. Various cashew-aldehyde resins when impregnated in low grade woods such as rubber wood show remarkable upgrading of quality These resins are equally applicable to the preparation of particle boards, bamboo boards, coconut leaf based boards etc. where both quality and cost effectiveness could be simultaneously achieved. Anorin-38 with a bonding capacity 500 times more than that of CF resins and with capabilities to reduce the flammability of the material stands a good chance for plywood and particle boards, particularly as there is a price advantage two to three times lower than the conventional phenolics adhesives.

2.6.11 Surfactants

CNSL can be advantageously used in the manufacture of anionic and non-ionic surface active agents. Like long chain fatty acids, cardanol possesses a typical lipid structure with a hydrocarbon hydrophobic group and a hydrophilic phenolic end group. This structure could be modified suitably to incorporate improved ion exchange capabilities such as introduction of a sulphonic acid group on the phenolic ring. The ion exchange resins are said to be good emulsifiers for oil-in-water and water-in-oil systems.

2.6.12 Industrial chemicals and intermediates for chemical industry

CNSL forms the basic raw material for a vast number of industrially important chemicals and chemical intermediates. Patents and reports cite a number of applications such as antioxidants, bacericides, fungicides, disinfectants, insecticides, dispersing and emulsifying agents, dye stuffs etc. Hydrogenation of cardanol gives 3-pentadecylphenol which stands a good chance for industrial utilization. Reports suggest its utiliation as a replacement for nonyl phenol and as a starting material for the preparation of 6-tertiarybutyl-3-pentadecylphenol and 3-pentadecyl-phenyl-glycedyl-ether. It's copolymerised product with phenol and formalehyde has been processed into speciality coatings by the Japanese. Suitable chemical modiication can convert the material into plastiizers that can replace the costly petrochemial based plasticizers.

2.7 Processing Methods

CNSL removal - before the shell is removed from the nut the CNSL is extracted. The traditional method of removing CNSL in East Africa involves roasting the nut in drums or baths. The roasting process not only removes the corrosive CNSL but also makes the shell brittle, thereby aiding the cracking process. This method results in the loss of most or all of the CNSL. To extract and retain CNSL the nuts are roasted in baths at a temperature of 180-185°C. Vents in the equipment dispel the unpleasant fumes. This method recovers 85-90% of the liquid.

The traditional method of extracting CNSL in India involves roasting the nuts in a shallow pan over open charcoal fires. Constant agitation is required to prevent the nuts from becoming scorched. This method is extremely unpleasant as the shells burst releasing CNSL and fumes with resulting losses.

An improved method involves roasting the nuts in a perforated pan with troughs placed underneath to catch the liquid. At a larger scale whole nuts are placed in rotating perforated cylinders inclined at an angle above a heat source. As the nuts fall downwards the shell liquid flows through the holes and is collected in troughs. The nuts are then water sprayed and set aside for cooling.

2.7.1 Roasting Methods

The application of heat to the nut releases the nut shell liquid and makes the shell brittle which facilitates the extraction of the kernel when breaking the shell open. Three methods of roasting exist: open pan, drum roasting and the 'hot oil' method. The latter is more suitable to medium scale operations with associated higher equipment costs and viability of CNSL collection.

2.7.2 Open pan

An open, mild steel, circular dished pan of around 2 feet in diameter is supported on a basic earth fireplace. When heated, 2-3lbs (1kg) of nuts are placed on the pan at one time and stirred constantly. The CNSL starts to exude and then ignites. This produces a long flame and black smoke. After approximately two minutes, the pan is dowsed and the charred, swollen and brittle nuts are thrown out of the pan. The moisture evaporates quickly leaving the nuts ready for shelling.

2.7.3 Drum roasting

The idea of continually feeding the nuts into a rotating drum over a fire developed from the pan method. A slight horizontal slope in the mounting ensures the movement of the nuts through the drum. The drum is pierced so that the flames touch the nuts and the smoke is controlled by a hood and chimney arrangement. The nuts are dowsed using a continuous spray. This process was further modified by using the heat from the burning CNSL being harnessed to roast the nuts some more. The roaster consists of a contained helical screw which moves the burning nuts at a controlled rate. The design was a distinct improvement, with little fuel being consumed and there being greater control on the roasting time.

2.7.4 Hot oil method

The principle employed in this method is that oil bearing i.e. substances the shells, when immersed in the same oil at high temperature, will lose their oil, thus increasing the volume of the oil in the tank. For this method, conditioning becomes important. The equipment consists of a tank of CNSL heated to a temperature of 185 - 190□C by a furnace underneath and a wire basket used to hold the nuts for immersion into the tank. The depth of the basket must be sufficient so that the rim remains well above the oil during the roasting. Immersion time can range from 1½ to 4 minutes. About 50% of the liquid is extracted from the nuts. Draining trays are needed at the end of the tank for the roasted nuts to dry and the residue oil can be returned to the tank. Caution must be taken not to heat the tank to over 200□C because at this point polymerization of the CNSL takes place. The temperature can be maintained by continuous firing. The tank should be emptied and cleaned after each day's roasting. The life of a tank made of an eighth inch thick mild steel plate should exceed one and a half years and can be constructed locally with welding facilities.

2.8 Solvent Extraction Methods

In the laboratory there are two types of solvent extraction available . These extraction methods are direct and indirect methods. In the direct method we use the batch extractor. For the indirect method soxhlet extractor is used . For the purpose of this experiment both direct and indirect method are used. The extraction of a soluble constituents from a solid by the use of liquid solvent is generally referred to as leaching. The mechanism of leaching may involve simple physical solution or dissolution made possible by chemical reaction. Leaching is primarily concerned with the recovery of oils from a nut or seed structure which has been prepared to Facilitate it's penetration by solvent. That is the size reduction followed by diffusion of the oil-solvent mixture to the Surface of the solid. The rate of transport of solvent into the mass to be leached, or of soluble fraction into the solvent, or of extract solution out of the insoluble material, or some combination of these three rates may be significant. A membraneous resistance may be involved and a chemical reaction rate may also affect he rate of leaching.

2.8.1 Advantages of solvent extraction

(i) Solvent extraction is free from undesired impurities due to injured cell of the nut seed, hence it gives higher quality of oil extracted.

(ii) It reduces residual oils in the extracted nut to less than 1% which is much less than the maximum oil content to which oil nut can be reduced by mechanical expression .

(iii) More oil is extracted especially when dealing with low oil content seed or nuts.

(iv) It constitutes the most efficient method for the recovery of oil from any oil bearing materials.

2.8.2 Disadvantages of solvent extraction

(i) Solvent with (low flash point) flammable present danger of fire and explosion .

(ii) Solvent extraction equipment are relatively expensive.

(iii) There are may be difficulty in the selection of solvent to be used . Unsuitable solvent will create problem during distillation or recovery of the oil from the oil\solvent mixture.

2.8.3 Factors influencing selectivity of the solvent

In many leaching operation, the suitability of the right solvent influences the type of solvent to be used .These characteristics are as follows:-

(1) Recoverability: After extraction it is expedient to recover the solvent for reuse. And this is done with the aid of distillation. Therefore it is required that the solvent should not form azeotrope with the extracted solute and the mixture should show high relative volability for low temperature recovery.

(2) Chemical Reactivity : The solvent choosen should be chemically stable and inert not only with solute but with the component of the system and common materials of construction.

(3) Relative Volatility: The ability of a liquid to change to vapour or gaseous form is termed volatility . It is required that the relative volubility of the solute to the solvent be favourable.

(4) Viscosity And Freezing Point: Viscosity is defined as the resistance to flow . It decreases with evaporation and vapour pressure . The greater the vapour pressure the lower the viscosity. The solvent chosen should be able to circulate freely to enable it reach pore and surface area, for extraction to take place. Therefore the solvent chosen should have a sufficient low viscosity and low freezing point to circulate freely.

(5) Purity: The solvent should be free from any impurities. So as to not interfere with extraction.

(6) Inflammability: For safety reasons it is very necessary that the solvent should have a high flash point above 33.2°C.Generally all liquids with flash point below 32.2°C are flammable.

(7) Availability: The solvent should be easily available.

(8) Toxicity: Generally for extraction of edible oil non toxic solvent are used, hexane etc. But since the uses of CNSL are non edible. There is no problem in using Benzene because benzene is considered toxic and moreover cancerous.

(9) Less Costly: The solvent should not be expensive

(10) Colour: Colour of solvent should be distinct from that of the solute

2.9 Leaching

The extraction of a soluble constituent from a solid by the use of liquid solvent is generally referred to as leaching. The mechanism of leaching may involve simple physical solution or dissolution made possible by chemical reaction.

The rate of transport of solvent into the mass to be leached or of a soluble fraction into the solvent, or extract solution out of this insoluble material or of the solute from the solution in contact with particle to the bulk of solution or some combination of these rates may be involved and a chemical reaction rate may also affect the rate of leaching.

Leaching is primarily concerned with the recovery of oils from a nut or seed structure which has been to facilitate it's penetration by solvent and diffussion of the oils solvent mixture to the surface of ht solid.

The structure of cashew nut shell (CNSL) is quite complicated. The oil is considered as being maintained in small tough cell walls. The C.N.S flakes are porous and in the cause of extraction the solvent migrate to the pores in order to extract oil.

The design of large scale solvent extraction apparatus must be determined by the rate at which equilibrium is attained between a lean miscella outside the shell particles, and oil solvent mixture within the particles. The design is also affected by the particle size distribution that would allow optimum extraction of oil.

Based on the porous nature of the flakes a mechanism has been postulated for explaining the leaching of oils. This is called the molecular diffussion theory.

2.9.1 Unit operation involved in CNSL extraction

Extraction of oil from cashew nut shell is basically a physical change but chemical conversion may be required in refining and further processing of oils. The unit operation involved in the extraction of CNSI are:-

1. Material handling
2. Drying (sun)
3. Size reduction
4. Extraction
5. Stripping
6. Filtration and flow of fluids

2.9.2 Physical characteristics of a solid

Aknowledge of physical characteristic of the carrier solid is important to determine weather a prior treatment is necessary to make the solute more accessible to the solvent.

These prior treatment may involve the following unit operations:

1. Crushing
2. Grinding
3. Cutting into pieces or reforming into special shapes such as flakes.

Solute particle may exist in the solid in a variety of ways. It may exist on the surface of the solid, may be surrounded by a nature hard material. May be chemically combined or may exist inside cells or as in the case of many vegetable and animal bodies Solute adhering to the solid body is readily remissible by the solvent.

When solute consists in Peres surrounded by a matrix of hard material the solvent has to diffuse to the interior of the solid to capture solute and then diffuse out before a separation can result in such case. Sub-division of the solid by grinding or cutting decreases the surface exposed to the solvent that is the solvent has to trail a shorter distance to remove the oil. However size reduction into fine particles has it's problems. Insure instead, the amount of solute to be removed is small in relation to the amount of material to be treated so that grinding becomes uneconomical too-fine division may result in packing of solids during extraction. Preventing free flow of solvent through the solid bed.

In such a case extraction is mush more difficult especially when finely divided solids are treated in an unagitated state.

Dispersion of the particles in liquid solvent by agitation permits thorough contacting of the solid with the solvent. Agitation, while giving good extraction may cause suspension of fine particles in out flowing solute, which may subsequently require a difficult filtration of clarification at a step.

In the case of materials with cellular structure, if the ell walls remain intact, the leaching action involves osmotic passage of the solute through cell walls. It is however impractical and undesirable to grind materials to rupture cell walls, since this may result in addition to the desired solute, creating a purification problem.

Therefore instead of restoring to excessive subdivision many solid of porous structure are cut into wedge shaped slices called "cossetles". Crushed and reshaped into flakes, as in the case of vegetable seeds to obtain increased surface, which permits free flow of solvent through the solid and allows a more selective extraction.

2.9.3 Factors affecting extraction

Several parameters affects the extraction process

1. Time:-In the extraction of most vegetable seeds and nuts, the rate of extraction initially increases rapidly for the first hour's but later decreases.
2. Solvent:- solvent affects the rate of extraction since some of its properties that affects the rate of extraction like viscosity, boiling point, density, surface tension and vapour pressure differ from solvent to solvent.

The size, the greater the interfacial area between the solid and the solvent hence the higher the rate of transfer of material. Smaller size also means that the solvent has to travel smaller distance within the solid thereby increasing the rate of extraction.

3. Flake Thickness:- Like particle size, thickness is also indirectly proportional to the rate of extraction. This is because as flake thickness decreases the cell walls. Thickness also decreases, resulting in higher diffusion.
4. Temperature:- This has the effect of decreasing the viscosity of oil and the solvent thereby increasing the diffusion coefficient. However care most be insured so as to prevent explosion of solvent when dealing with flammable solvent.
5. Moisture Content:- This is expressed as the amount of water contained in the shell. As the moisture content of shell decreases, the rate of extraction increases. Decreases in moisture content lead to decrease in the water in the tissue which encircles the oil from which it is not easy to extract the oil.

CHAPTER THREE

3.0 METHODOLOGY

3.1 Materials and Equipment

The materials and equipment used in the present study are presented in Tables 3.1 and 3.2, respectively

Table:3.1 list of materials used for the Experiment.

Materials	Source	Research Code	Comment
Cashew Nut shell	Cashew nut, Minna	NA	Dried and crushed
Water	Tap connection Chem. Lab. FUT. Minna	NA	Liquid 30°C
N – Hexane	Aldrich-Chem. Gillingham	n-Hexane 99%	Volatile Liquid
CNSL	Cashew Nut shell	NA	Amber – coloured viscous oil
Alanine 336	Aldrich-Chem .Gillingham	NA	White Crystal solid
H_2SO_4	Avondale Lab; England	UN 1164	Acidic and conc(98%W/V)
NaOH	Avomdal Lab; England	UN1145	Alkaline (33%W/V)

Table 3.2 List of New Equipment used for the Experiment.

Equipment	Sources	Research Code	Comment
Soxhlet Apparatus	Pyrex. England	BDB24	Glass apparatus
Weighing balance	Ohaus, U.K	B300	Digital display
Measuring cylinder	Clifton, England	CE94	Glass apparatus
Beakers	Clifton, England	CE20	Glass apparatus
Round bottom Flask	Clifton, England	CE56	Glass apparatus
Thimble	Lambsman, India	NA	Filter apparatus
Reflux condenser	Pyrex, Engand	BDB24	Glass apparatus
Spatula	Chem. Eng. Lab FUT, Minna	Na	Stainless steel
Cork and wool	Chem. Eng. Lab FUT, Minna	NA	Stoppers
Thermometer	Zeal, England	MM76	Mercury in glass Immersion
Separating funnel	Clifton, England	CE119	Glass apparatus
Mortar and piston	Chem. Eng. Lab FUT, Minna	NA	Crushing equipment
Heating Mantle	Electrothermal, Britain	B300	Stainless case
pH meter	Kent Ind. Ltd. England	KE869	Digital display
Viscometer	Pyrex, England	PE 101869	U-tube glass apparatus
Oven	Gallenkamp, England	CE94	Vacuum drier

3.2 Experimental Procedures

3.2.1 Gathering and screening of the cashew nuts

For a successful leaching operation there are some basic physical operations that must be carried out to improve both the quantity and quality of the extract that will be obtained. Some of such processes are washing. Drying, shelling and size reduction

WASHNING: The nuts after discarding the apples are washed thorough with detergent to remove any dins and contaminant that might he attached.

DRYING: This is best done by spreading the washed nuts in the open sun for as many days as possible so as to reduce the" moisture content as much as possible. The furnace is not used (though faster) because the high temperature of furnace may affect some physical characteristics of the liquid .

SHELL1NG: This is the breaking of the nuts to separate the nut from the shell. The shell is recovered for use while the nut is discarded. This is best done by means plier while wearing glooves.

SIZE REDUCTION: This is done by means of a crusher to reduce the size of the shell and increase the surface area. Size reduction is very important because the particle of a soluble material is surrounded by a matrix of insoluble matter, so also this will allow the solvent to penetrate into the particle to allow the extract to diffuse out.

3.2.2 Extraction using direct leaching

In this process, 10g of ground cashew nutshell was measured into a round bottom flask, which contain the solvent, hexane. This mixture was placed on an electric heater and the thermostat was adjusted to maintain a constant heating rate at 65°C. The mixture was allowed to boil for an experimental time of 2 hours.

The vapour from the boiling solvent was made to condense and return to the mixture by means of a reflux condenser, which was mounted, on the flask through which water was constantly flowing. After the extraction has been completed the healer was switched oft and the solvent decanted. The raffinate was weighed and recorded while other operations like filtration; solvent recovery and weighing were further carried out on the extract phase to obtain the extracted liquid.

These procedures were repeated for a temperature of 75°C and 85°C one after the other while still maintaining hexane as the solvent. The set up is shown in Figure3.1.

The results obtained were tabulated as shown in Table4.1, Table4.2 and Table4.3.

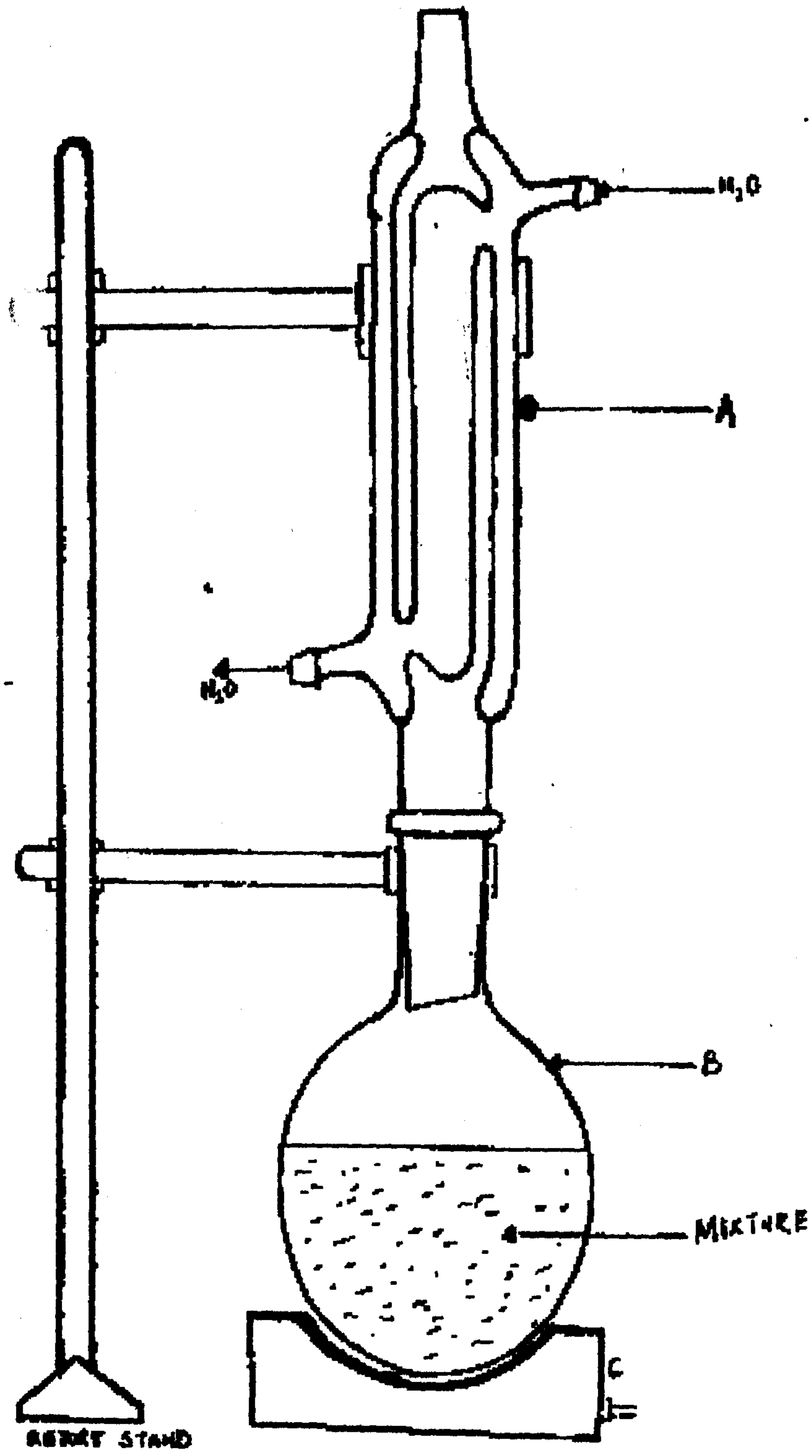


FIG3.1: Direct leaching Experimental Set-up

A	REFLUX CONDENSER
B	ROUND BOTTOM FLASK
C	ELECTRIC HEATER

3.2.3 Extraction using indirect leaching (soxhlet apparatus)

In this process, 10g of the sample was placed inside a thimble (A) and inserted into the inner tube of the apparatus. This apparatus was then filled to a round bottom flask (C), which contained the solvent (hexane). A reflux condenser (D) was also mounted and fitted on the apparatus. The solvent was boiled at a temperature of 65°C for 2 hours. The vapour passes up through the tubes (E), condensed by the condenser and the condensed solvent falls into the thimble and lowly fills the body of the Soxhlet. When the solvent reached the top of the tube (F), it siphons over into the flask (C) and thus removed the portion of the sample that has been extracted in the thimble. The process repeats itself automatically for the 2 hours demonstration time and the apparatus was dismantled.

These procedures were repealed four time using fresh samples of the same quantity and the same solvent (hexane) but with a variation of temperature at 65°C,75°C and 85° C for each samples. The results obtained were tabulated as in Table 4.4, Table4.5 and Table4.6.

The set up of the Soxhlet extraction apparatus is shown in fig 3.2.

Note that, the same solvent, sample quantity and equal time was used throughout the extraction.

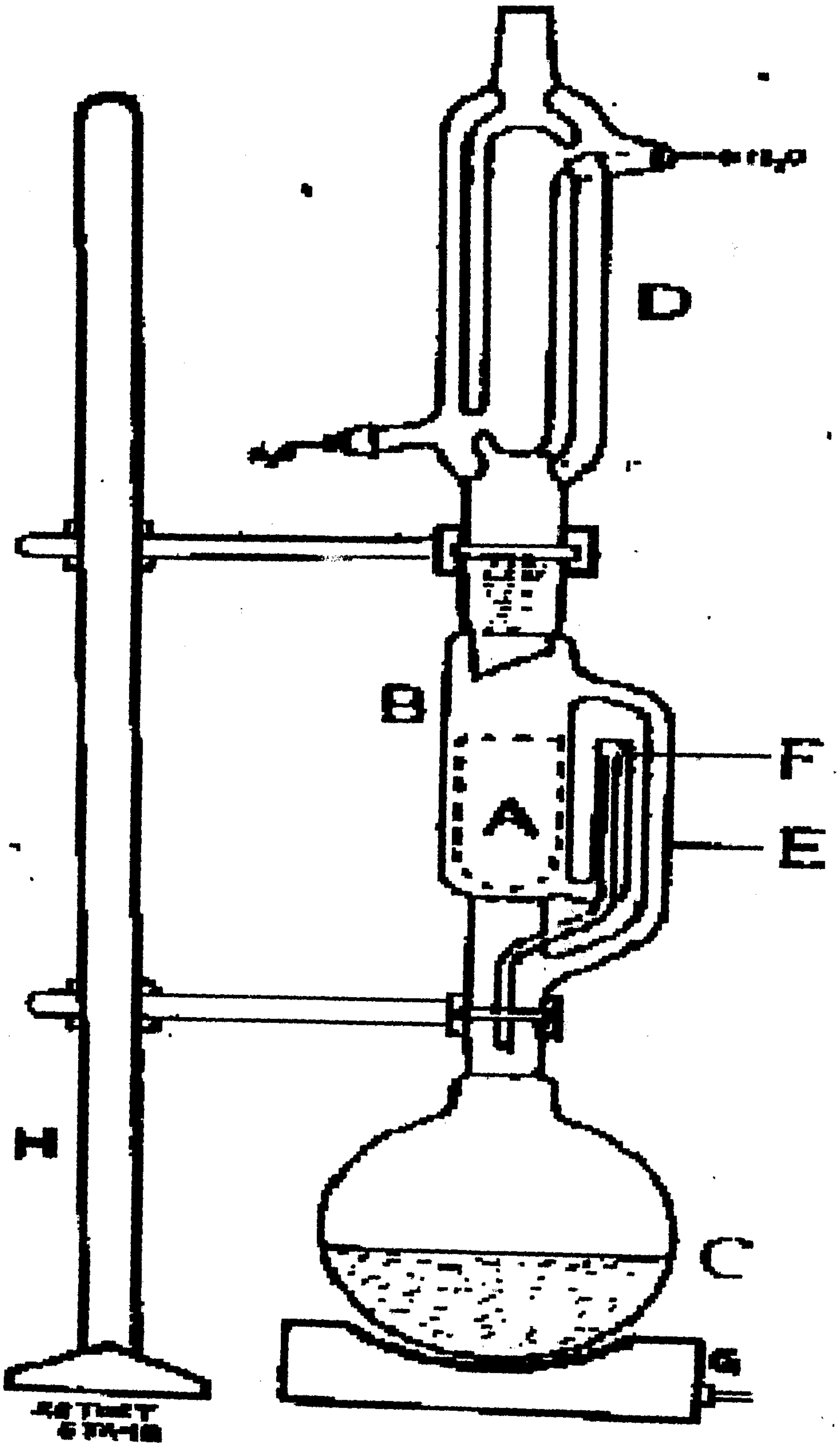


Figure 3.2 Indirect Leaching (Soxhlet) Experimental Set-up

A THIMBLE

B SOXHLET UNIT

C ROUND BOTTOM FLASK

D REFLUX CONDENSER

E VAPOUR DUCT

F SIPHON TUBE

G ELECTRIC HEATER

3.3 Treatment of the Liquid after Extraction

After the extraction has been duly completed, some physical operations were carried out to improve the quality of the extract. Some of these operations are separation of the extract from the raffinate, filtration, evaporation and solvent recovery.

i. Separation: This is the process of separating the raffinate phase from the extract phase. It was then carried out on the direct leaching process where the sample was heated directly in the solvent and is allowed to interact. Here decanting the extract phase from the ratlinate phase carried out the separation. The extract was decanted into a beaker.

ii. Filtration: The extract phase decanted, which still contained some panicles, was then filtered via a filter paper to obtain a product which is purely a mixture of the liquid and the solvent.

iii. Solvent recovery; this is the process of separating the liquid extracted from the solvent used. In this process, the mixture was heated above the boiling point of the solvent, which then vaporized and condensed into another container via a reflux condenser where it was recovered form use.

iv Evaporation: This is the process of removing moisture from the extracted liquid or reducing the moisture content to a bearest minimum. Here the liquid was boiled directly while it was opened to atmosphere.

v. Weighing: The extracted liquid and the raffinate were weighed on an electronic weighing balance to determine their weight while the recovered solvent volume was measured using a measuring cylinder.

3.4 Separation of Monohydric Phenols and Dihydric Phenols in CNSL

3.4.1 Diagram

The experiment set-up as shown below:

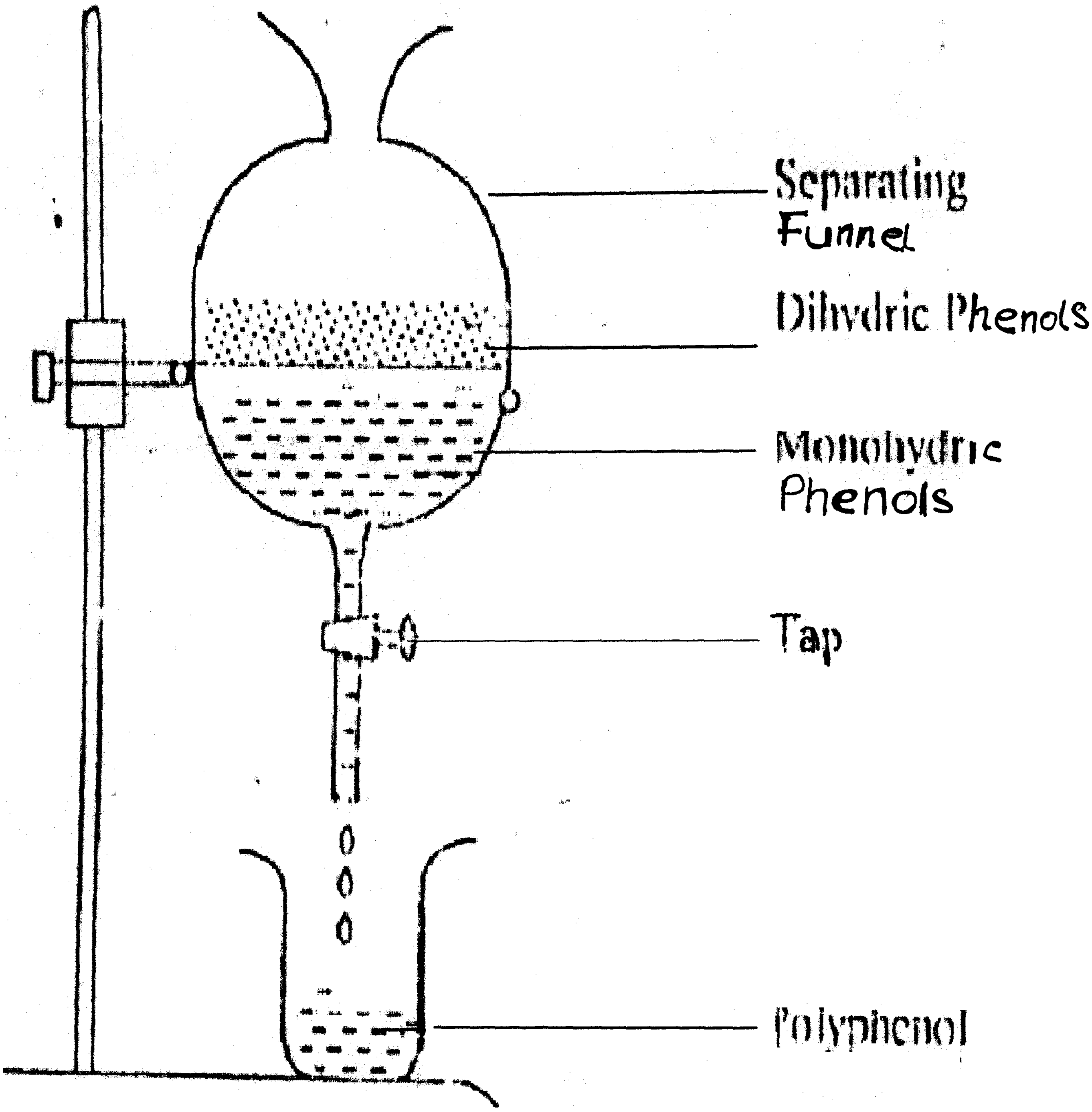


Fig.3.3: Experimental Set-up for Shaking Apparatus

3.4.2 Procedure

The experimental set-up was as shown in the diagram above. The extraction and stripping experiment were performed using shake-put methods. Phenol and resorcinol were chosen as' representatives of monohybrid and dihydric (polyhydric phenols) and their starting concentration in aqueous phase were about 5g/l and 2g/l respectively. Equilibrations were carried out in 500ml separating funnels at ambient temperature $(25^{o}C)$ after 5 minutes of vigorous shaking with Alkaline 336 in n-hexane diluents at 1:1 volumetric phase ratio. Effect of pH on equilibrium was studied by adjusting the pH of the aqueous phase by either drop-wise addition of concentrated sulphuric acid or Sodium hydroxide.

3.5 Determination of Physicochemical Properties

3.5.1 pH measurment

The pH of the polyphenol recovered was measured by means of pH meter electrode. About 30ml of the sample was put into a beaker and the electrode dipped into it. The value was read directly from the instrument and recorded.

3.5.2 Measurement of viscosity

The measurement of viscosity of the polyphenol was carried out using viscometer bath with an inserted u-tube capillary. A known quantity of the sample was poured into the u- tube capillary and corked. The u-tube was suspended into the viscometer bath containing water maintained at a temperature of 30'C. The cork w as removed and the sample begins to flow through the capillary. The time taken for the liquid to flow through the capillary of fixed length was recorded and used for the calculation of the viscosity of the liquid, this was recorded.

3.5.3 Determination of density and specific gravity

A clean dry 1ml density bottle was accurately weighed and recorded. The flask was then filled to the mark with the polyphenol and reweighed. The difference between these weights gives the density of the liquid and this was recorded. The polyphenol was then removed and bottle cleaned and allows drying. (This was accomplished easily by rinsing with ethyl alcohol and allowed to stand briefly). The cleaned dried bottle was again accurately weighed.

3.5.4 Determination of refraction index

The instrument used for the determination of refractive index of the polyphenol (monohydric phenols and dihydric phenols) is refractometer. The surfaces of the prisms were cleaned even with ethanol and allowed to dry before use because the index of refraction is sensitive to even a small amount of contaminant.

The polyphenol sample was placed on the lower prism and ensured that it covered the entire width of the prism plate, a dropper was used for this purpose. I lie upper prism was brought into contact with the lower prism so that the polyphenol forms an unbroken layer between the two prisms. The controls were manipulated to bring the light and dark fields into focus with the cross hairs. The readings were then made. This warned out at a temperature of bout 25°C.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Results

The result obtained by varying the methods of extraction and temperature are given in Tables 4.1 - 4.6.

Table 4.1: Results of Direct Leaching of CNSL at 65°C

Mass of CNS before extraction(g)	Mass of CNS after extraction(g)	CNSL extracted (g)
10	6.9	3.1
10	7.3	2.7
10	7.1	2.9
10	7.2	2.8
10	7.3	2.7

Table 4.2: Results of Direct Leaching of CNSL at 75°C

Mass of CNS before extraction(g)	Mass of CNS after extraction(g)	CNSL extracted (g)
10	6.4	3.6
10	6.7	3.3
10	6.5	3.5
10	6.4	3.6
10	6.6	3.4

Table 4.3:Results of Direct Leaching of CNSL at 85°C

Mass of CNS before extraction(g)	Mass of CNS after extraction(g)	CNSL extracted (g)
10	6.2	3.8
10	6.1	3.9
10	6.1	3.9
10	6.0	4.0
10	6.3	3.7

Table 4.4: Results of Indirect Leaching of CNSL at 65°C

Mass of CNS before extraction(g)	Mass of CNS after extraction(g)	CNSL extracted (g)
10	8.2	1.8
10	8.4	1.6
10	7.9	2.1
10	8.2	1.8
10	8.3	1.7

Table 4.5: Results of Indirect Leaching of CNSL at 75°C

Mass of CNS before extraction(g)	Mass of CNS after extraction(g)	CNSL extracted (g)
10	7.2	2.8
10	7.3	2.7
10	7.1	2.9
10	7.0	3.0
10	7.2	2.8

Table 4.6: Results of Indirect Leaching of CNSL at 85°C

Mass of CNS before extraction(g)	Mass of CNS after extraction(g)	CNSL extracted (g)
10	6.9	3.1
10	6.5	3.5
10	6.5	3.5
10	6.6	3.4
10	6.7	3.3

4.1.2 Physicochemical properties

The results of the various physicochemical analysis conducted on the polyphenol (monohydric phenols and dihydric phenols) CNSL can simply be presented in form of table as below:

Table 4.7: Physicochemical Properties of samples

Parmeters	Monohydric Phenols	Standard value Monohydric Phenols	Dihydric Phenols	Standard value Dihydric Phenols
pH	4.72	5	2.57	3.0
Refractive index	6.2	6.65	1.9	1.65
Specific gravity	1.18	1.25	0.95	1.1
Viscosity	58.8Cp	-	301.2 Cp	-
Percentage(%)	79.36 %	75 %	20.64 %	25%

4.2 Infrared Spectra

Almost any compound having covalent bonds, whether organic, will be found to absorb various frequencies of electromagnetic radiation in the infrared region of the spectrum. A second and more important use of the infrared spectrum is that it gives structural information about a molecule.

The absorptions of each type of bound (N-H, C-H, O-H, C-X, C-O, C-O, C-C,C C,=C, C=N etc) are regularly found only in certain small portions of the vibrational infrared region.(8)

Figure(A) give the infrared spectra of sample O,(CNSL). It is observed that there is an absorption at region 3,600-3650cm at peak the absorption is sharp and the analysis shows that there is presence of phenol in conc. Solution with O-H functional group at this the intensity of the absorption is medium (M).

At peaks 2850- 3000cm-1 the intensity of the absorption is strong s island sharp which is an indication of C-H stretching vibration. Thus –CH2- group is confirmed present.

The absorption at region 1600-1680cm1 is sharp and an intensity of medium-weak (m-w) it show the presence of C=C group. While at peak 1450 cm-1 the absorption indicates the presence of Alkanes C+B group with bending vibration and he intensity is medium.

Though at sharp peak it shows the presence of C=N Nitriles group. At peaks 1300-1000cm-1 the absorption shows the presence of C-O group, Alcohols, Ethers, Esters, carboxylic acid the intensity is sharp.

4.3 Discussion of Results

The various methods used as well as temperature variation were compared with respect to the yield obtained in each case. The results obtained from the methods adopted in carrying out the extraction shows that the yield obtained from the direct leacking is higher than that using soxhlet apparatus.

This is because, in the direct leaching technique, the solvent is allowed to interact directly with the sample in the vessel while heating, therefore is an inimate contact between the two In case of soxhlet apparatus,the solvent will have to vapourise, condense and fall back into the thimble where the extraction take place.

Also in direct leaching, both the solvent and the samples are contained in the flask placed on heating elememt, therefore they are both heated. From common knowledge, extraction is always influenced by temperature and in most case increase with increase in temperature, this therefore accounts for the higher yield. But in case of Soxhlet equipment, there is no direct heating of the sample or the sample is not heated at all. The solvent that fall into the thimble containing the sample is not in its vapoure state but rather conbdensed and cooled, hence a lower yield when compared with direct leaching.

Since the experiment was conducted under a specific time frame, there was no time lag in case of direct leaching as against indirect method where the solvent will have to vapourise condensed, fall back into the thimble and soak the sample before extraction can begin.

Temperature is important factor influencing leaching and other extraction operations. Increase in temperature, increase the rate of extraction. This is due to the fact that in most cases, solubility increase with increase with increase temperature hence giving a higherrate of extraction.

Similary, the diffusion coefficient is expected to increase with increasing temperature. It can therefore be seen from the results that the amount of CNSL extracted increased appreciably with every rise in temperature.

The separation of the polyphenol from the CNSL result to two distinct layers with the polyphenol taking a larger portion of the separating funnel ; and being denser, it was drawn out from bottom of the funnel.The shake-up experiment broke the polyphenol into monohydric phenols and dihydric phenols. It was found that the cashew nut shell liquid contained 79.36% monohydric phenols and 20.64%dihydric phenols .

The results of various physicochemical properties tests conducted, such as pH, viscosity, specific gravity and refractive index. Form the table4.7 ,the pH values of monohydric phenols and dihydric phenols are 4.47 and 2.57 respectively from the pH table ,these values shows that the two layers are acidic.The knowledge of the pH is necessary because most condensation reaction in adhesive formulation are carried out at specific pH level. This will help to know the type of buffer solution or stabilizer that will be required when it is to be used.

The Refractive index of monohydric phenols and dihydric phenols are 6.2 and 1.9 respectively . This shows that the two layers have a close Refractive, with their standard values which are 6.65 and 1.64.

The specific gravity of monohydric phenols and dihydric phenols are 1.18 and 0.95 respectively .This shows that the two layers have a close , specific gravity with their standard values which are 1.25 and 1.1.

The analysis of red spectra (fig. A) shows that the following function group were present in the structure of the samples. In the case of sample O1, CH, C = C, CH3, C = N, C – O Alcohols, ethers ester, and carboxylic acid and reasonably quantity of phenol was confirmed present.

CHAPTER FIVE

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The results obtained justified the basis of research work. Through the comparison, we were able to deduce a better method that should be used for the extraction. The results obtained show that the yield from the direct leaching was more than that obtained from the use of soxhlet extraction equipment.

Extraction is known to proceed favourably with increasing temperature, the result of the present experiment is supportive of the fact. It was seen that the yield increased progressively with temperature increase. Despite this general assertion, there is a limit to which the temperature can be increased. The extraction therefore should be done at an optimum temperature.

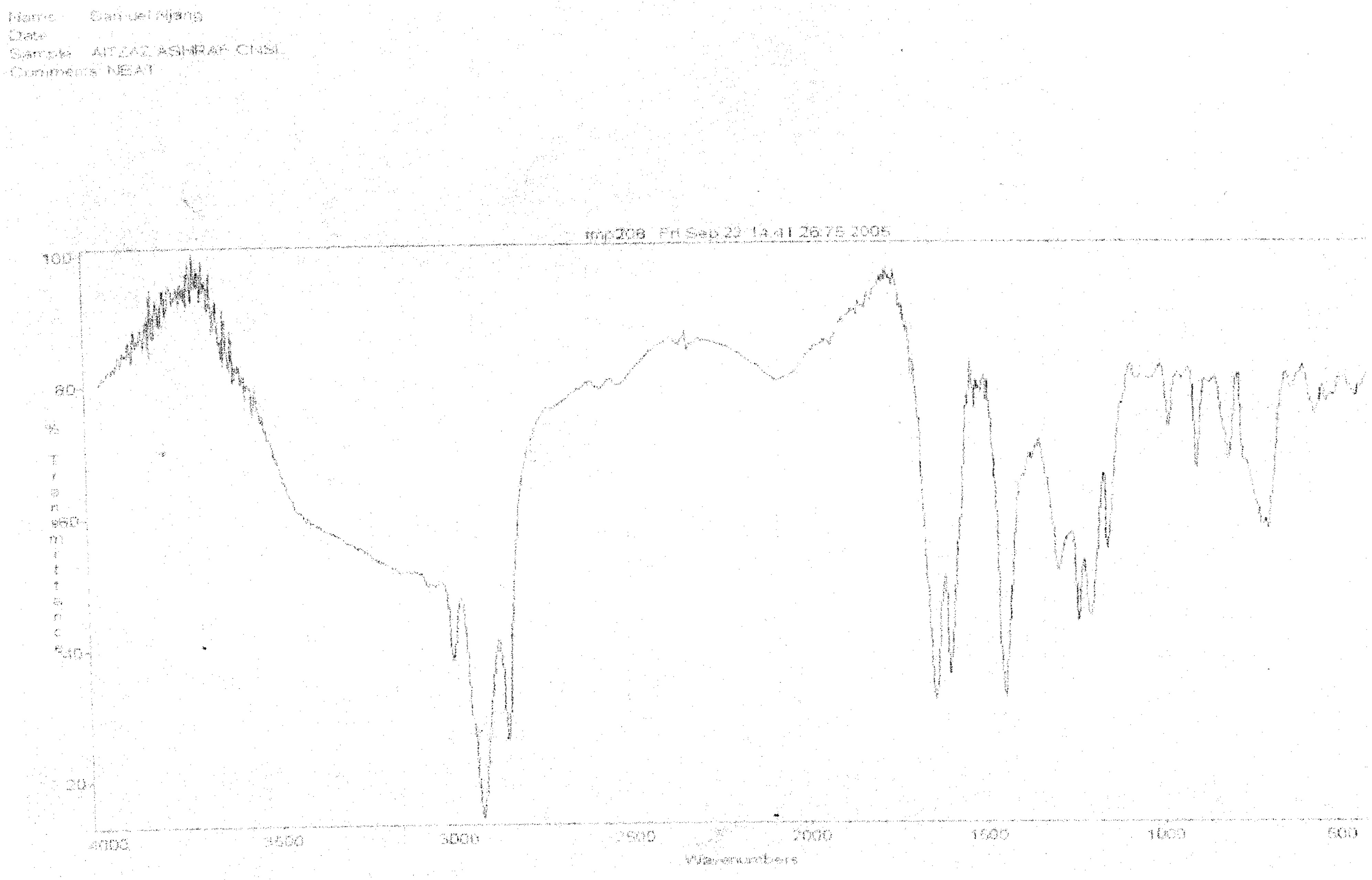
. The use of the amine facilitates the separation of monohydric and dehydric phenols within the process of extraction and offers an effective means of removing polyhydric phenols from the raffinate more than is possible with the commonly used commercial solvents. The principle of extraction by amime, which exploits the physico-chemical characteristics of polyphenol, has been explained. The results of various instrumental analysis carried out show that polyphenol is a useful polymeric (organic) substance that can serve as an alternative raw material for process industry.

5.2 Recommendations

Although the objective of the research work was achieved, but other areas which may have not been touch should be investigated such as:

- Variation of particle size with extraction time i.e constant time
- Other organic solvent other than hexane should be used.

Thus, the University should try to make available, such necessary analysis equipment like viscometer, pH meter, IR and UV machines. They are means of generating revenue to school and it will help students carry out their work at easily.



Name Samuel Njang
Date
Sample AITZAZ ASHRAF CNSL
Comments NEAT

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APPENDIX

APPENDIX A

Total mass of sample = 157.53g

Mass of first layer (monohydric phenols) = 125.02g

Mass of second layer (dihydric phenols) = 32.51g

$$\text{Percentage of monohydric phenols} = \frac{\text{Mass of first layer}}{\text{Total mass of sample}} \times 100\%$$

$$\text{Percentage of monohydric phenols} = \frac{125.02}{157.53} \times 100\%$$

Percentage of monohydric phenols = 79.36%

$$\text{Percentage of dihydric phenols} = \frac{\text{Mass of second layer}}{\text{Total mass of sample}} \times 100\%$$

$$\text{Percentage of dihydric phenols} = \frac{32.51}{157.53} \times 100\%$$

Percentage of dihydric phenols = 20.64%