EXTRACTION OF POLYPHENOLS FROM CASHEW NUT SHELL AND RED ONION SKIN

BY

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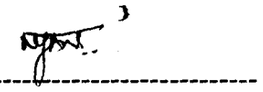
DECEMBER, 2005.

DECLARATION

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



14/12/05

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Date.

CERTIFICATION

This project titled EXTRACTION OF POLYPHENOLS FROM CASHEW NUT SHELL AND RED ONION SKIN by Njang Samuel Odia meets the requirements for the award of the degree of Bachelor of Engineering (Chemical Engineering) of Federal University of Technology, Minna and is approved for its contributions to scientific knowledge and literary presentation.

2005-12

Dr. M. O. Edoga — 2005-12-12

Supervisor — Date

Dr. F. Aberuagba — Date

Head of Department.

External Supervisor — Date

DEDICATION

This project work is dedicated to my lord Jesus Christ and my dearly beloved parents for the gift of education they gave to me.

ACKNOWLEDGEMENT

To God be the glory for being my guide and counsellor throughout the duration that this research work lasted and throughout my life.

Depth of gratitude to my noble and erudite supervisor **Dr. M. O. Edoga** for his professional assistance, counselling and motivation that ensured this work a huge success. He went through the manuscript patiently, making all necessary corrections. He accepted, tolerated, encouraged and instructed me to strive for the best things in life. May our heavenly father reward him.

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Special regards to my ever-caring guardian **Hadjia Ndakpayi** and her beloved family and also to **Bologi Ahmed** for his friendly assistance.

Great regards to you all for your contributions and concerns towards the success of this noble work.

ABSTRACT

The aim of this research work was to carry out a study of the extraction of polyphenols from cashew nut shell and red onion skin powder so as to determine some of the parameters necessary for the design and operation of a full – scale plant. Some physicochemical properties of the extracts were also determined.

Results of the infrared analyses (IR) revealed the presence of phenyl group in the range of 3500 to 3800cm^{-1}. The pH values of the polyphenols are in the range of 6.75 to 6.83. Refractive indices are in the range of 1.4360 to 1.4632. Specific gravities are in the range of 0.9137 to 0.9248. The viscosities are in the range of 1.8762 to 1.8870cs. The yield of the polyphenols increased as temperature increased.

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NOMENCLATURE

1	CNS	- Cashew Nut Shell.
2	ROSP	- Red Onion Skin Powder.
3	DIPE	- Di-isopropyl Ether.
4	LDL	- Low Density Lipoprotein
5	CNSL	- Cashew Nut Shell Liquid
6	IPCS	- International Programme on Chemical Safety
7	N / A	- Not Applicable

CHAPTER ONE

1.0 INTRODUCTION

In some developed and developing countries, it has become empirical that the amount of natural fuels such as petroleum and coal are beginning to dwindle and also very expensive due to high demand for this non-renewable fuels.

Polyphenols are the polymerization products of phenols. Phenols are aromatic alkanols gotten or found in aqueous streams of coal coking as part of coal tar and in petroleum residues. Due to the high polar and complex nature of phenol, the extraction of phenol presents one of the most difficult industrial separation problems.

Coffee and chocolate products contain a range of polyphenolic anti-oxidants known as flavanoids (flavanoids are naturally occurring phenolic compounds in plants), which provide health benefits such as prevention of heart diseases (Harborne, 1980 and Hour et al., 1999). Polyphenols are the most abundant group of compounds in fresh tealeaves and are found in green and black tea beverages (Hour et al., 1999). Polyphenols are found as anti-oxidants in extracts of onions where they are helpful in diabetic retinopathy (Girretson et al., 1995).

Polyphenols are a large family of natural compounds found at high concentrations in onions, apple, red wine, beverages and so on. They are also found in shells of cashew nut. Polyphenols can be extracted from red onion skin and cashew nut shell via direct and indirect leaching techniques using suitable solvents.

In Nigeria, red onion skin and cashew nut shell are seen as wastes and as such they are discarded. Polyphenols contained in these materials are very useful as a sort of barrier or protection against ultra violet rays from the sun, of which in its raw form are toxic and poisonous to man.

For instance, in a few developed countries such as U.S, local raw materials are been looked into for the provision of these polymerized phenols as a possible substitute of these non-renewable natural fuels that are dwindling in their available amounts and are quite expensive.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Polyphenol

Polyphenols are a group of vegetable chemical substances, characterized by the presence of more than one phenolic group. Their phenolic reactions produce gelatins, alkaloids and other proteins (Chung et al., 1998).

The polyphenol have been shown to be strong anti-oxidants with potential health benefits. Green tea and extra virgin olive oil are a high content source of polyphenols (Frankel et al., 1993).

Polyphenol is an aqueous stream, a result of the polymerization products of phenols, thus they have similar trends such as colourless liquid, sweet odour with melting point of 43°C and boiling point of 183°C. But the increased number of hydroxyl group makes these substances more water-soluble and also contributes to an increased tendency to become oxidized.

It inflicts blisters on the skin. It reacts with formaldehyde to give dry oil, with furfural to give a molding plastic and with other chemicals to give rubber-like masses, which could be, used as electric insulators (Dupont & Gokel, 1989).

The polyphenol is one of the best known disinfectants and the germ-killing power of other chemicals is usually based on a comparison with it. It is a valuable chemical raw material for the production of plastics, dyes, pharmaceuticals and other products (Samson, 1995).

2.1.1 Effects of polyphenol

Polyphenol exhibit a wide range of biological effects as a consequence of their anti-oxidant properties. They inhibit LDL in vitro (Frankel et al, 1993). Moreover, LDL is isolated from volunteers supplement with red wine or red wine polyphenol show reduced susceptibility to oxidation (Fuhrman et al, 1995; Nigdikar et al, 1998). Thus, polyphenols probably prevent LDL oxidation in vivo with significant consequences in atherosclerosis and also prevent DNA from oxidative damage with important

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consequences in the age-related development of some cancers (Halliwell, 1999). In addition, flavanoids have anti-thrombotic and anti-inflammatory effects (Gerritsen et al, 1995; Muldoon and kritchevsky, 1996). The antimicrobial property of polyphenolic compounds has been well documented (Chung et al, 1998).

Several types of polyphenols (phenolic acids, hydrolysable, tannins and flavanoids) show anti-carcinogenic and anti-mutagenic effects. Polyphenol might interfere in several of the steps that leads to the development of malignant tumors, inactivating carcinogens, inhibiting the expressions of mutant genes and the activity if enzymes involved through activation of procarcinogens and activating enzymatic systems involved in the detoxication of xenobiotics (Bravo, 1998). However, some polyphenols have been reported to be mutagenic in microbial assays and co-carcinogenes or promoters in inducing skin carcinogens in the presence of other carcinogens. This latter possibility warrants further research (Chung et al., 1998).

Several studies have shown that in addition to their anti-oxidant protective effect on DNA and gene expression, polyphenols, particularly flavanoids, inhibit the initiation, promotion and progression of tumors, possibly by a different mechanism (Chung et al, 1998).

Wine contains many polyphenolic compounds that apparently exhibit anti-cancer properties, including gallic acid, caffeic acid, ferulic acid, catechin, quertecin and revertrol among others. Gallic acid is anti-mutagenic with the Ames' test (Hour et al, 1999) and hepato protective for carbon tetra chloride toxicity (Kanai and Okano, 1998). In an experiment with transgenic mice that spontaneously develop skin tumors, the addition of red wine solid extract to their diet led to marked delay in tumor development (Clifford et al, 1996). Some investigators have proposed that red wine has the highest cardioprotective effects because of its high concentration of polyphenols, which inhibit the oxidation of human low-density lipoprotein (LDL) in vitro. White wine does not have a high concentration of polyphenols (DeRijke YB et al, 1996; Fuhrman B et al, 1995; Nigdikar SV et al, 1998).

2.1.2 Extracts of polyphenol

On the 18th of August 2003, a Spanish company, specialized in developing nutraceuticals from vegetable fats, has patented a process to obtain a concentrated polyphenol product from the wastewater of olives (www.nutraingredients.com/recent polyphenol extractives).

The extract, hytolive, can be added to numerous foods to boost their anti-oxidant activity, claims Malaga-based Genosa.

The active agent in hytolive is hydroxytyrosol, a potent anti-oxidant recognized by the European Olive Oil Medical Library. The phenolic compound is obtained with more than 99.5% purity in the new process, according to the company, and has higher anti-oxidant activity than ascorbic acid, one of the antioxidants more commonly used in food industry (www.nutraingredients.com/recent polyphenol extractives).

Hytolive can be applied to wide range of foodstuffs, including diary products, oils and spreads, chocolates, drinks and cereals.

Genosa, which also develops nutraceuticals, cosmetics and pharmaceuticals, recently launched a vegetable-based functional oil that helps control cholesterol and triglyceride levels. The oil is said to reduce build-up of atherosclerotic plagues at higher rates than virgin olive oil. It also showed neuroprotection attributed to its polyphenol content.

There is considerable scientific evidence to suggest that polyphenols play a role in protecting against cardiovascular diseases and risk factors for cancer. They also appear to show ageing and promote respiratory health (www.nutraingredients.com/recent polyphenol extractives).

2.1.3 Classes of phenolic compounds

Table 2.1 below gives the major classes of phenolic compounds in plants;(Harborne, 1980)

2.2 Phenol

Phenols are a class of aromatic organic compounds derived from benzene by the addition of one or more hydroxyl groups. Examples include the cresol, xylenols, resorcinol and itself known as benzophenol (hydroxybenzene). It has a molecular formula of C_6H_6O or C_6H_5OH. In the U.S (1979), it was the 35th highest volume of chemical produced (Walter L. Badge et al, 1995).

2.2.1 Derivation of phenols

Most of the phenols made in the U.S are by the oxidation of cumene giving acetone as by-product. The first step in the reaction produces hydropereoxide, which on reaction with dilute sulphuric acid decomposes to the primary products including phenyl dimethyl carbinol and acetophenone. Several other benzene-based processes have been used in the past. It can also be derived from benzoic acid (Saburi, 1998).

The polyphenol is a polymerization product of one or more of the following phenolic compounds:

Monohydric : Phenol
Cresol
Xylenol and so on.

Dihydric : Catechol
Resorcinol
Quinol

Trihydric : Pyrogallol
Hydroquinol
Phloroglucinol

The monhydrics are those with on hydroxyl group. The dihydrics and trihydrics are those with two and three hydroxyl groups respectively (Malcolm P. S., 1999).

Number of carbon atoms	Basic Structure	Class	Examples
6	C_6	Simple phenols	Catechol, hydroquinone
		Benzoquinones	2,6-Dimethyl benzoquinone
7	C_6-C_1	Phenolic acids	Gallic, salicyclic
8	C_6-C_2	Acetophenones	3-Acetyl-6-methoxybendahyde
		Tyrosine derivatives	Tyrosol
		Phenylacetic acids	p-hydroxyphenylacetate
9	C_6-C_3	Hydroxycinnamic acids	Caffeic, ferulic
		Phenylpropenes	Myristicin, engenol
		Coumarins	Umbelliferon, aesculetin
		Isocoumarins	Bergunon
		Chromones	Eugenin
10	C_6-C_4	Naphthoquinones	Juglone, plumbagin
13	C_6-C_1-C_6	Xanthones	Magniferin
14	C_6-C_2-C_6	Stilbenes	Resveratrol
		Anthraquinones	Ennodin
15	C_6-C_3-C_6	Flavanoids	Quercetin, cyanidin
		Isoflavanoids	Genistein
18	$(C_6$-$C_3)_2$	Lignans	Pinoresinol
		Neolignans	Eusiderin
30	$(C_6$-C_3-$C_6)_2$	Biflavanoids	Amataflavone
n	$(C_6$-$C_3)_n$	Lignins	
	$(C_6)_n$	Catechol melanins	
	$(C_6$-C_3-$C_6)_n$	Flavolans (Condensed Tannins)	

2.2.2 Properties of phenol

- White and sometimes colourless crystalline mass, which turns pink or red under the influence of light.
- It possesses hydrophilic properties.
- It liquefies distinctive odour, sharp burning taste.
- It forms weak solution with a sweetish taste.
- It has a specific gravity of 1.07 and a flash point of 78°C with a melting point of about 40.6°C.
- It is soluble in alcohol, ether, chloroform, glycerol and other common organic solvents.

2.2.3 Uses of phenol

Phenolic resins, epoxy resins, nylon, selective solvent for lubrication oil refining, adipic acids, salicylic acids, phenolphthalein, pentachlorophenol, acetophenetidine, puric acids, germicidal paints, pharmaceuticals, laboratory reagents, dyes and indicators, general disinfectants, rubber and so on.

2.2.4 Hazards

- Inflicts blisters on skin absorption.
- Strong irritant to tissue.
- Toxic by ingestion and inhalation.
- Has a tolerance of 5ppm in air.

Table 2.2: Vapour Pressure of Phenol up to 1atm

Pressure (mmHg)	1	5	10	20	40	60	100	200	400	760
Temperature (60°C)	40.1	62.5	73.8	86.0	100.1	108.4	121.4	139	160	181.9

2.3 Cashew *(Anarcadium Occindentale)*

The main commercial product of the cashew tree is the nut. In the main producing areas of East Africa and India, 95% or more of the crop is not eaten, as the taste is not popular.

However, in some parts of South America and West Africa, local inhabitants regard the apple as well as the nut as delicacies. In Brazil, the apple is used to manufacture jams, and soft and alcoholic drinks. Goa in India, it is used to distill a cashew liquor called "feni".

The cashew fruit is unusual in comparison with the other tree nuts since the nut is outside the fruit. The cashew apple is an edible false fruit, attached to the externally born nut by a stem. In its raw state, the shell of the nut is leathery, not brittle. It contains the thick vesicant oil, CNSL, within a sponge-like interior. A thin tasta skin surrounds the kernel and keeps it separated from the inside of the shell. The primary products of cashew nuts are the kernels, which have value confectionary nuts. Cashew Nut Shell Liquid (CNSL) is an important industrial raw material for resin manufacture and shells can be burnt to provide heat for the decorticating operation (Raghavendra, 1970).

2.3.1 Cashew nut shell liquid (CNSL)

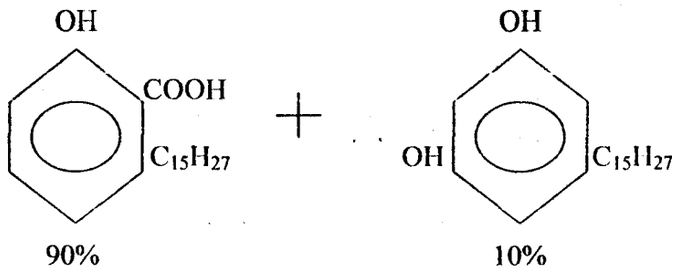
This is a by-product of cashew industry. The cashew nut has a shell of about 1/8 inch thickness inside with a soft honeycomb structure containing a dark brown viscous liquid. It is called CNSL, which is pericarp fluid of the cashew nut.

2.3.2 Characteristics of CNSL

IS 840 governs the characteristics of CNSL. The specification of CNSL is shown in table 2.3 below (Raghavendra, 1970).

2.3.3 CNSL Structure

The chemical structure of CNSL is shown below;



90% Anarcadic Acid

10% Cardol

Table 2.3: Characteristics of CNSL

Specifications	Values
Specific gravity 30/30°C	0.95 to 0.97
Viscosity at 30°C, in centipoises	300 (max)
Moisture, % by weight, max	1.0
Loss in weight on heating, max	2.0
Ash, % by weight, max	1.0
Iodine value,	
(a) Wij's method	250
(b) RK method	290
Polymerization time in min, max	4

2.4 Onion *(Allium cepa)*

This could be in different forms depending on how they are formed, grown or produced thus, leading to their different names such as common onion or bulb onion for bulb forming types, spring onions, green or bunching onions for onions which produced edible leaves.

Onion is a biennial herb normally grown as an annual. The bulb is formed of the basis of thickened food-storage leaves. The hollow foliage leaves are produced from a flattened, basal, conical stem. Group of 5-10 flowers are produced in umbels and up to 100cm tall is the growth of the whole inflorescence (Rice et al, 1992).

2.4.1 Polyphenolic compounds in onions

Compounds found in onions may have the ability to prevent certain types of cancer.

In laboratory tests, quercetin, a polyphenol found in onions, was shown to inhibit pancreatic cancer growth in cell and animal studies. Quercetin is also found in the skins of apples. Resveratrol is another polyphenol that is believed to have cancer-fighting

properties. The two of them are also being credited with the ability to help prevent LDL cholesterol from oxidizing and sticking to artery walls (Frankel et al., 1993)

2.5 Extraction

Extraction of oil involves meal-leaching operation, solvent recovery from meal and oil separation from miscella oil solvent (Saburi, 1998).

2.5.1 Extraction process

There are three distinct process usually employed in leaching operations,

- Dissolving the soluble constituent
- Separating the solution, so formed, from the insoluble solid residue.
- Washing the solid residue in order to free it of unwanted soluble matter.

The process of leaching is been mainly carried out in batch, but many continuous plants have also been looked into, developed and modified. The equipment type depends on the nature of the solid; whether coarse or fine and cellular or granular. The normal distinction between coarse and fine particles is that the former have sufficiently large settling velocities for them to be readily separable from the liquid whereas the latter can be maintained in suspension with the aid of small amount of agitation. Generally, the solvent is allowed to percolate through beds of coarse materials, whereas fine solids offer too high a resistance.

As already pointed out, the extraction rate will in general be a function of the relative velocity between the liquid and the solid. In most plants the liquid flows through the stationary solid bed of particles while in others both the liquid and the solid move in a counter current fashion (Saburi, 1998).

But for this very research work, we will be working on CNS and ROS. These two materials are solid in nature and the method of extraction is the leaching process that is to be employed.

2.5.2 Leaching process

Leaching is a separation technique that is often employed to remove a solute from a solid mixture with the aid of a solvent. It is originally referred to the removal of a

soluble component via a fixed bed, but of recent, it is applied to solid – liquid extraction generally.

The separation operation involving washing of a solute adhering to the surface of a solid is also considered solid – liquid extraction. Because of its application in several industries. Leaching is known by such other name as decoction (use of solvent at its boiling point), lixiviation, percolation, infusion, elutriation, decantation and settling. The presence of a solid component distinguishes all these techniques from liquid – liquid extraction (Schweitzer, 1979).

Basics of Leaching

Leaching generally refers to the removal of substance from a solid through a liquid extraction media. The desired component diffuses into the solvent from its natural solid form. Examples of leaching include the removal of sugar from sugar beets with hot water and the removal of nickel salts or gold from their natural solid beds with sulfuric acid solutions.

There are many different types of equipment used for leaching. Most of these pieces of equipment fall into one of two categories:

Categories of leaching

1. **Percolation:** The solvent is contacted with the solid in a continuous or batch method. This method is popular for in-place ore leaching or large scale "heap" leaching popular for extreme amounts of solid.
2. **Dispersed solids:** The solids are usually crushed into small pieces before being contacted with solvents. This is a popular leaching method when an especially high recovery rate can economically justify the typically higher operating cost.

Whether the leaching is taking place via percolation or by dispersed solids, there are three important factors that aid in leaching: temperature, contact time/area and solvent selection. Temperature is adjusted to optimize solubility and mass transfer.

Liquid-to-solid contact is essential for the extraction to take place and maximize contact area per unit volume reduces equipment size.

Solvent selection plays an important role in solubility as well as the separation eps that follow leaching. Nearly all leaching equipment employs some type of agitation aid in mass transfer and to ensure proper mixing. The most popular leaching quipment can be seen in Perry's Chemical Engineers Handbook www.cheresources.com/basics of leaching).

.6 The Extractor Equipment

Extractor selection and design for oil production are based on efficiency, cost, peration and capacity so also time. Recent designs are mere modifications so as to ccount for improvement on these factors. There exist no single extractor that can be said o be the most efficient or best constituting all these three factors alone, but all are ompromise of one or a few of these factors. Extractors are primarily distinguished as either batch or continuous process extractor.

2.6.1 Batch process extractors

They are suitable for less than 50tons of feed per day operation. The upper section is filled with the change of seed flake, which is sprayed with fresh solvent from a distributor. The solvent percolates through a bed of solid and drains into a lower compartment where together with any water extracted is continuously boiled off by means of steam coil. Other forms of extraction are as follows: Basket type, vertical u-tube and vertical cavity extractors (Saburi, 1998).

2.6.2 Continuous process extractors

A typical laboratory apparatus that operates o the principle of the continuous process extractors is the "soxhlet extractor"

The Soxhlet Extractor

A soxhlet extractor is a type of laboratory glassware. It was originally designed for the extraction of liquid from a solid test material, but can be used whenever it is difficult to extract any compound from a solid (Dingler, 1879). The soxhlet extractor named after its designer is used to keep a continuous supply of fresh solvent to a sample being extracted.

Typically, by test material is placed inside a "Thimble" made from filter paper, hich is headed into the main chamber of the soxhlet extractor. The extractor is attached a flask containing a solvent (commonly ether or petroleum ether) and a condenser. The olvent is heated, causing it to evaporate. The hot solvent vapor travels up the extractor hrough the vapor tube and up into the condenser, where it cools and converted into iquid. This liquid, which is completely free from dissolved components, drips down into he main chamber on top of the test material (Sample). This continues until a level of olvent in the chamber reaches the highest level of the return tube. When the solvent eaches this level, it starts a siphon, which empties the main chamber through the return ube back into the flask.

This cycle may be allowed to repeat many times. After many cycles (usually a few hours), the flask will contain the extract products dissolved in the solvent with the solid material left in the thimble. The thimble generally made out of glass or pressed cellulose. Filter paper will work just fine as will a coffee filter or even a piece of paper towel in a pinch. (Dingler, 1879).

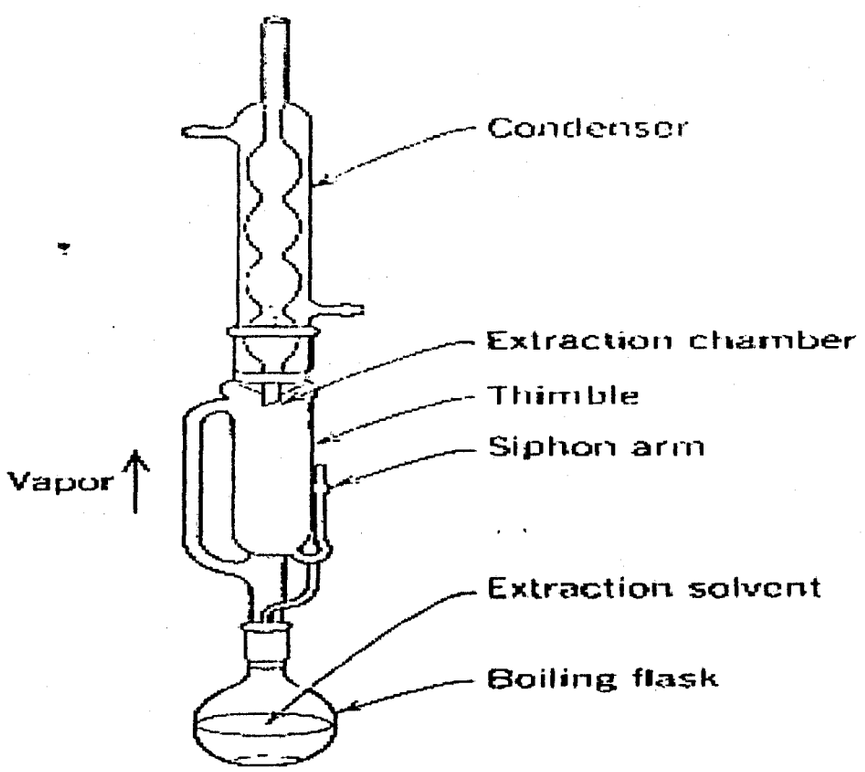


Figure 2.1: A Schematic Representation of a Soxhlet Extractor.

2.7 Solvents

2.7.1 Types/classification and properties

The physical properties of some organic solvents made them suitable for oil extraction. These include selective solubility, volatility, boiling point and other thermal characteristics. Solvent that has been used in this research work as extractive agent is classified as follows.

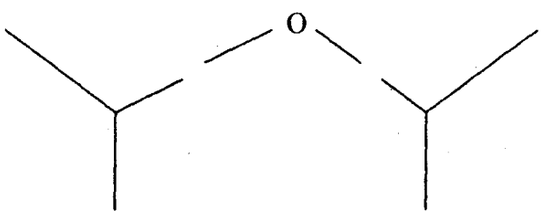
The Solvent (Diisopropyl Ether)

The solvent used for extraction of polyphenols from cashew nut shell and red onion skin is a newly formed solvent produced in 1995 and reviewed in March 1996. It is known as diisopropyl ether also known as isopropyl ether, 2,2[1] – oxybispropane, 2-isopropoxypropane or diisopropyl oxide (IPCS & CEC, 2004).

It's molecular formula shown below;

$$C_6H_{14}O \,/\, (CH_3)_2\,CHOCH(CH_3)_2$$

It has a molecular mass of 102.18 gmol. It's structure is shown below;



It is a colourless liquid with characteristic odour and a boiling point of 69°C. See appendix for more information on the solvent (Diisopropyl ether).

2.7.2 Organic solvent selectivity

Properties of a solvent such as its boiling point, density and viscosity affect its suitability, acceptability and selectivity to affect more complete and economical extraction of the solute so desired. The extent and rate of solution of the desired solute and other impurities in the solid by a solvent influence considerably the size of an extractor overall operating costs, type and cost of separation as well as solvent and solute recovery equipment and the quality of products. The solvent should be selective, relatively cheap, non-toxic and readily available. (Schweitzer, 1979)

Other properties to be considered are the safety properties of solvents which determine hazard associated with the use of solvents so as to set conditions for safe use. Some of these properties include: Flash points, Flammable limits, minimum ignition temperature (M I T), carcinogenicity, threshold limit value (T L V) and among others.

Thus, it is important to note or acknowledge the fact that the ability of an organic substance to dissolve other substance to form a homogenous solution makes its use as extractive solvent reliable and feasible. (Schweitzer, 1979)

2.8 Physicochemical Properties of Liquids

When the physical properties of substances depends on their chemical composition and properties, we say those physical properties are referred to as the physicochemical properties of that substance.

These properties aid in determining and defining the features and possible functions in which the liquid can display and how they can be developed, modified or reacted with other substances for more useful purposes. Some of these properties are: the molecular weight, specific gravity (or density), viscosity, refractive index and so on.

2.8.1 Molecular weight

A materials' molecular weight (M W) is the summation of all the atomic weight of the atoms constituted in it. In a polymeric material, M W is an important variable relating directly to a polymer physical property.

In determining the M W of polymers, however, more complex than that of non-polymeric materials whose M W can be determined via the application of the principle basics of mass spectrometry. So also freezing point depression and boiling point elevation (i.e. cryoscopy and ebulliometry respectively).

This is because, one; in any polymerization process, it is virtually impossible for all growing polymer chains to terminate at the same size, hence one must necessarily deal with average M W.

Two; the freezing point depression, boiling point elevation and titration techniques are effective only with relatively low M W polymer less than 40,000. the

conventional techniques of mass spectrometry have not been used extensively in the polymer field beyond the characterization of polymer degradation product because of the requirements for volatilization samples.

In general, the higher the M W the tougher the polymer and too high a M.W can lead to difficulties in procession. Common techniques used for determining M W of polymer are: osometry, measurement of solution viscosities, light scattering and ultra-centrifugation. (Perry, 1997)

2.8.2 Viscosity

This is the resistance a substance offers to flow when subjected to shear stress. It is a measure of the internal friction that arises when there are velocity gradients within the system. For fluid, its meaning is conceptually and operationally defined.

In the viscosity of liquids, momentum transfer between shearing layers also underlies the viscous behavior of simple liquids, but since the mean free path has little meaning for liquids, no simple relation exist for them. In contrast to the behavior of gases, temperature decrease the viscosity of simple liquids and its effect it's larger.

The temperature dependence of the viscosity of simple liquid bear no simple relationship to gas kinetic theory but instead generally follow an exponential law of the form:

$$\eta = Ae^{(B/RT)}$$

where A and B are parameters characteristic of the liquid and are reasonably constant over finite range of temperature.

R is a gas constant

T is absolute temperature

The measurement of dilute solution viscosity in polymer, provide the simplest and most widely used technique for determining M W in a routine manner. It is not an absolute method; each polymer system must first be calibrated with absolute M W determinations run on fractionated polymer samples.

Viscosity is measured at concentration of about 0.5g/100ml of solvent by determining the flow time of certain volume of solution via a capillary of fixed length. Viscosities are run at constant temperature usually 30.0 +/- 0.01° C. (Perry, 1997)

2.8.3 Refractive index

This is defined as the ratio of the speed of light through a vacuum to the speed through a given sample. The velocity of light through the medium will therefore, be related to the structure of the molecules and in particular to what functional groups present.

In general, organic materials have refractive indices that falls within the range of 1.3 – 1.6. At the lower ends are alcohols (related structurally to water with refractive index of 1.33) and ketones, and at the higher end are found such compounds as chloroform, benzene, nitro-benzene and aniline.

Refractive indices are usually measured by refractometer and two of these are in common use; these include: Fisher refractometer and Abbe- Spencer refractometer. Both these refractometers are useful for observing refractive index value between1.3 and 1.7. (Perry, 1997)

2.8.4 Specific gravity (S G)

The S G of a material is defined as the ratio of its density to the density of some standard materials such as water at a specific temperature usually at 40°C.

S G is a convenient concept because it is usually easier to measure than density and its value is the same in all system of units. S G is a constant at varying temperature. (Perry, 1997)

2.8.5 Infrared Spectra

In the infrared region of the spectrum, electromagnetic radiation of various frequencies are absorbed by almost any organic or inorganic compound possessing covalent bonds. The spectrum gives structural information about a molecule.

Only certain small portions of the vibrational infra red region reveal the absorption of different type of bond (N-H, C-H, O-H, C-X, C=N, C=O, C-C, C=C and so on) (Donald L Pavia et al, 1976).

CHAPTER THREE

3.0 EXPERIMENTAL METHOD

3.1 Experimental Apparatus and Materials

The materials and apparatus used for the extraction of polyphenols from cashew nut shell and red onion skin powder are as follows:

Table 3.1: List of Materials Used for the Experiment

Materials	Sources	Research Code	Comment
Cashew nut shell	Cashew nut, Minna	CNS	Crushed
Red Onion Skin	Onion, Minna	ROS	Blended
Diisopropyl Ether	BDH Chemicals	DIPE	Volatile liquid
Water	Tap connection, FUT Minna.	N / A	Liquid

Table 3.2: List of Apparatus Used for the Experiment

Materials	Sources	Research Code	Comment
Blending Machine		N / A	Electrical
Reflux Condenser	Pyrex, England	BDB24	Glass apparatus
Soxhlet Apparatus	Pyrex, England	BDB24	Glass apparatus
Oven	Gallenkamp, England	CE94	Vaccum drier
Viscometer	Pyrex, England	PE101869	U-tube glass apparatus
pH meter	Kent Ind. Ltd, England	KE896	Digital display

2 Experimental Procedure

2.1 Gathering and pretreatment of cashew nut shell and red onion skin

The cashew nuts were purchased at Minna central market and was screened to remove foreign bodies.

The nuts were allowed to dry for several days under atmospheric temperature of the sun to enhance easy removal of the shell. A knife was employed to remove the seed after which the shell was properly crushed to small sizes to create a better surface area for contact with the solvent and consequently for easy removal of oil.

The above pretreatment steps were also taken to prepare the red onion skin .

3.2.2Extraction of polyphenols from ROSP and CNS by direct and indirect leaching

The extraction was carried out in three stages on the basis of temperature variation. It was carried out at 65°C, 70°C, and 75°C.

At each of these extraction temperatures, both direct and indirect leaching techniques were employed for each sample, with extraction time, mass of sample and volume of solvent kept constant for each case.

Direct leaching procedure

Figure 3.1 below is the experimental set-up for the direct leaching procedure. About 150ml of the solvent (DIPE) was measured using the measuring cylinder and then poured into the round bottom flask. The crushed CNS (5.0g) was also introduced into the flask. Thus, establishing a direct contact between the solvent and the sample of CNS.

The reflux condenser was mounted on the flask with water connection in and out for cooling. The mixture in the flask was then heated to the designated temperatures for some time . At each particular temperature the vapour of the solvent leaves the mixture in the flask upwards trying to escape but it was condensed by the reflux condenser back to the flask as liquid, thus creating an avenue for a more severe contact between the sample and the solvent.

The process was continued for a period of 3 hours after which the heat was removed and the apparatus allowed to cool. The remaining sample residue and the

solvent mixture (now containing the extract) still in the flask, were then decanted through a filter paper into a beaker to remove the residue left in the mixture after extraction time has elapsed. The residue filtered out was then dried in an oven for some time and weighed to determine the mass/amount of oil that has been extracted from it.

Indirect leaching procedure

The experimental set-up for indirect leaching procedure is shown in Figure 3.2. About 150ml of the solvent (DIPE) was measured using the measuring cylinder and then pored into the round bottom flask. The crushed CNS sample (5.0g) was placed inside the thimble and inserted into the main chamber of the soxhlet apparatus. The soxhlet apparatus was then mounted unto the flask. The reflux condenser was then mounted on the apparatus with water connection in and out for cooling.

The solvent was heated at the designated temperatures for some time; the vapour of the solvent moves up the vapour tube but was condensed on its way by the condensing effect of the reflux condenser. The condensed solvent drops into the thimble creating an avenue for contact with the sample. After a period of time, a mixture of the solvent and the extract fills up the main chamber of the soxhlet apparatus, which was then siphoned back to the flask leaching out the oil (polyphenol) from the sample.

The system was then cooled and the thimble removed and dried in an oven. The dried thimble was then weighed to determine the difference in weight after extraction has been completed, consequently, determining the amount of oil that has been extracted from the sample. The whole process was repeated using fresh samples until a sufficient amount of oil was gotten.

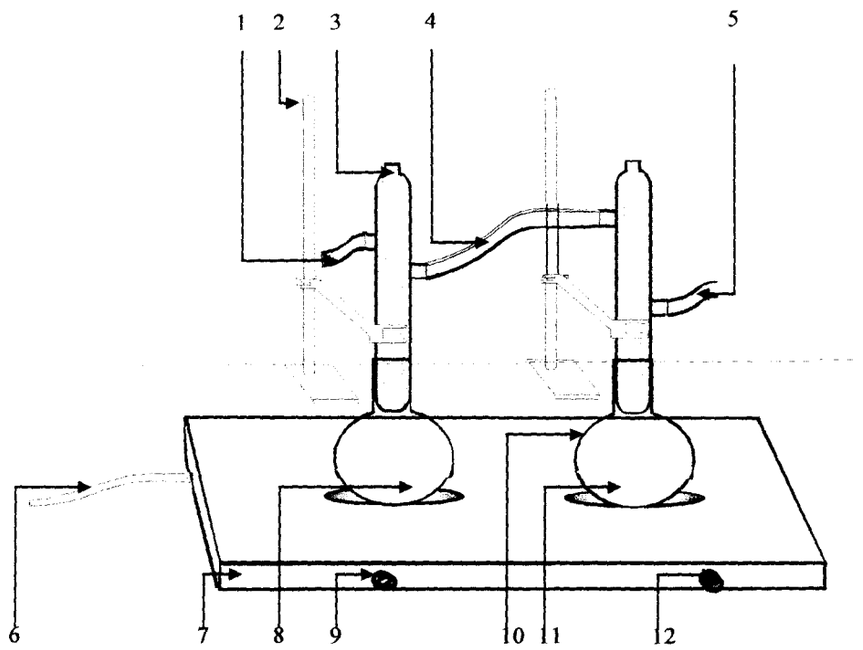


Figure 3.1: Experimental Set – up for Direct Leaching Method.

1	Cooling water in
2	Retort stand
3	Condenser apparatus
4	Pipe connection
5	Cooling water out
6	Link to power supply
7	Heating mantle
8	Mixture of solvent and sample A
9	On/Off switch
10	Conical flask
11	Mixture of solvent and sample B
12	Temperature regulator.

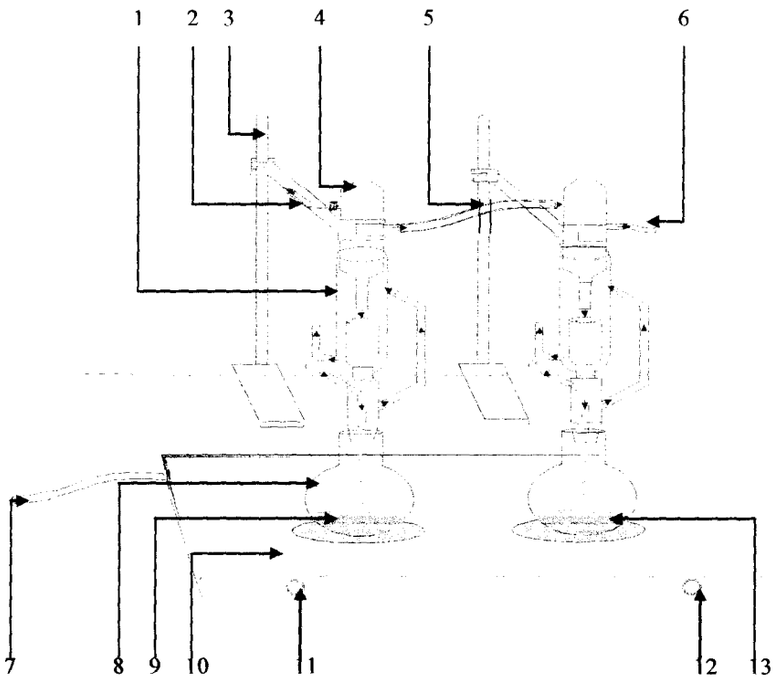


Figure 3.2: Experimental Set-up for Indirect Leaching Method.

1	The soxhlet apparatus
2	Cooling water in
3	Retort stand
4	Condenser apparatus
5	Pipe connection
6	Cooling water out
7	Link to power supply
8	Conical flask
9	Solvent mixture of sample A extract
10	Heating mantle
11	On/Off switch
12	Temperature regulator
13	Solvent mixture of sample B extract.

3.2.3 Solvent recovery

Now the solvent mixture contains both the oil (polyphenol) and the solvent (DIPE). The mixture was heated to the boiling point of the solvent (70°C) so that it just evaporates and condenses out of solution leaving only the extract in the flask for further analysis.

3.3 Characterization of Polyphenol Extract

3.3.1 pH measurement

The pH of the polyphenol extract was measured using the pH meter. 30ml of the sample was collected into a beaker of 50ml and the pH value was determined by inserting the electrode into it. The value was now read from the instrument and was recorded.

3.3.2 Specific gravity measurement

The empty SG bottle was weighed and its weight recorded. The same SG bottle was filled with the sample and weighed and its weight was recorded. The difference in weight between the SG bottle filled with phenol and the empty SG bottle gave the density.

3.3.3 Viscosity measurement

The standard method of viscosity determination was used using the viscometer bath capillary inserted into the viscometer bath. A reasonable quantity of the sample was poured into the U-tube viscometer with capillary and then corked. The U-tube was suspended into the viscometer bath containing water and the temperature of the bath 33°C.the cork was removed and the time taken for the content to run up starting from the top mark to the middle mark was noted using a stopwatch. From this result the viscosity of the sample was calculated.

3.3.4 Refractive index measurement

Certain quantity of pre-heated phenol was introduced into the prism chamber and allowed to stabilize for some time.

The refractometer reading was taken by turning the compensation room of the refractometer until there was a sharp division between the dark portions of the sample. The accurate refractometer was taken as the whole number and the decimal units shown on the micrometer screw gauge.

3.3.5 Infrared analysis

The infrared analysis was carried on the phenol extract from CNS and ROS using the MATISON USA 1998 model, GENESIS II FTIR spectrometer.

CHAPTER FOUR

4.0 RESULTS

Tables 4.1 to 4.12 show the results obtained during the extraction of polyphenols from ROSP and CNSL.

Table 4.1: Direct Leaching at 65°C (ROSP)

Sample	Mass of sample before extraction, m_1 (g)	Mass of sample after extraction, m_2 (g)	Average mass of sample extraction, (g)	Percentage yield (%)
A	5.00	4.54		
B	5.00	4.56		
C	5.00	4.53	4.54	9.20
D	5.00	4.54		
E	5.00	4.54		

Table 4.2: Direct Leaching at 65°C (CNSL)

Sample	Mass of sample before extraction, m_1 (g)	Mass of sample after extraction, m_2 (g)	Average mass of sample extraction, (g)	Percentage yield (%)
A	5.00	3.46		
B	5.00	3.48		
C	5.00	3.46	3.47	30.60
D	5.00	3.47		
E	5.00	3.48		

Table 4.3: Indirect Leaching at 65°C (ROSP)

Sample	Mass of thimble, m_T (g)	Mass of thimble + sample before extraction, m_1 (g)	Mass of thimble + sample after extraction, m_2 (g)	Percentage yield, (%)
A	4.07	9.07	8.81	5.20
B	4.11	9.11	8.86	5.00
C	4.09	9.09	8.84	5.00
D	4.11	9.11	8.85	5.20
E	4.10	9.10	8.86	4.80

Table 4.4: Indirect Leaching at 65°C (CNSL)

Sample	Mass of thimble, m_T (g)	Mass of thimble + sample before extraction, m_1 (g)	Mass of thimble + sample after extraction, m_2 (g)	Percentage yield, (%)
A	4.05	9.05	8.07	19.60
B	4.15	9.15	8.17	19.60
C	4.13	9.13	8.16	19.40
D	4.12	9.12	8.15	19.40
E	4.11	9.11	8.16	19.00

Table 4.5: Direct Leaching at 70°C (ROSP)

Sample	Mass of sample before extraction, m_1 (g)	Mass of sample after extraction, m_2 (g)	Average mass of sample extraction, (g)	Percentage yield, (%)
A	5.00	4.51		
B	5.00	4.52		
C	5.00	4.50	4.51	9.80
D	5.00	4.52		
E	5.00	4.51		

Table 4.6: Direct Leaching at 70°C (CNSL)

Sample	Mass of sample before extraction, m_1 (g)	Mass of sample after extraction, m_2 (g)	Average mass of sample extraction, (g)	Percentage yield, (%)
A	5.00	3.43		
B	5.00	3.45		
C	5.00	3.44	3.44	31.20
D	5.00	3.43		
E	5.00	3.46		

Table 4.7: Indirect Leaching at 70°C (ROSP)

Sample	Mass of thimble, m_T (g)	Mass of thimble + sample before extraction, m_1 (g)	Mass of thimble + sample after extraction, m_2 (g)	Percentage yield, (%)
A	4.13	9.13	8.81	6.40
B	4.10	9.10	8.78	6.40
C	4.12	9.12	8.80	6.40
D	4.10	9.10	8.76	6.80
E	4.11	9.11	8.80	6.20

Table 4.8: Indirect Leaching at 70°C (CNSL)

Sample	Mass of thimble, m_T (g)	Mass of thimble + sample before extraction, m_1 (g)	Mass of thimble + sample after extraction, m_2 (g)	Percentage yield, (%)
A	4.10	9.10	8.11	19.80
B	4.09	9.09	8.10	19.80
C	4.08	9.08	8.09	19.80
D	4.11	9.11	8.11	20.00
E	4.10	9.10	8.09	20.20

Table 4.9: Direct Leaching at 75°C (ROSP)

Sample	Mass of sample before extraction, m_1 (g)	Mass of sample after extraction, m_2 (g)	Average mass of sample extraction, (g)	Percentage yield, (%)
A	5.00	4.48		
B	5.00	4.49		
C	5.00	4.48	4.47	10.60
D	5.00	4.47		
E	5.00	4.45		

Table 4.10: Direct Leaching at 75°C (CNSL)

Sample	Mass of sample before extraction, m_1 (g)	Mass of sample after extraction, m_2 (g)	Average mass of sample extraction, (g)	Percentage yield, (%)
A	5.00	3.43		
B	5.00	3.42		
C	5.00	3.41	3.41	31.80
D	5.00	3.40		
E	5.00	3.40		

Table 4.11: Indirect Leaching at 75°C (ROSP)

Sample	Mass of thimble, m_T (g)	Mass of thimble + sample before extraction, m_1 (g)	Mass of thimble + sample after extraction, m_2 (g)	Percentage yield, (%)
A	4.14	9.14	8.79	7.00
B	4.13	9.13	8.79	6.80
C	4.12	9.12	8.78	6.80
D	4.14	9.14	8.78	7.20
E	4.11	9.11	8.77	6.80

Table 4.12: Indirect Leaching at 75°C (CNSL)

Sample	Mass of thimble, m_T (g)	Mass of thimble + sample before extraction, m_1 (g)	Mass of thimble + sample after extraction, m_2 (g)	Percentage yield, (%)
A	4.12	9.12	8.12	20.00
B	4.11	9.11	8.14	19.40
C	4.10	9.10	8.09	20.20
D	4.10	9.10	8.07	20.60
E	4.12	9.12	8.11	20.20

4.1 Physicochemical Properties of Polyphenol Extract

Table 4.13 below shows the values of some of the properties of the polyphenol produced from CNSL and ROSP both by direct and indirect leaching.

Table 4.13: Properties of polyphenol produced

Property	CNS-Direct Leaching	CNS-Indirect Leaching	ROSP-Direct Leaching	ROSP-Indirect Leaching
pH (32°C)	6.83	6.82	6.76	6.75
Specific gravity (32°C)	0.9137	0.9135	0.9248	0.9244
Refractive index (32°)	1.4360	1.4340	1.4632	1.4633
Viscosity (32°C)	1.8870	1.8863	1.8764	1.8762
Operating temperature (°C)	65, 70 & 75	65, 70 & 75	65, 70 & 75	65, 70 & 75
Operating pressure (atm)	1	1	1	1
Percentage yield (%) at;				
65°C	30.60	19.40	9.20	5.04
70°C	31.20	19.92	9.80	6.44
75°C	31.80	20.08	10.60	6.92

4.2 Infrared Spectra

Figure 4.21 and 4.22 give the infrared spectra of the polyphenol extracted from sample of CNSL produced by direct and indirect leaching, respectively. It is observed that there is absorption at region 3600 – 3800cm^{-1} for both figures with Fig. 4.22 being more intense than Fig. 4.21. At this peak, the absorption is sharp and the analysis shows that there is presence of phenol in concentrated solution with O-H functional group.

At peaks 2850-3000cm^{-1}, the intensity of the absorption is strong and sharp for both Figures 4.21 and 4.22, which is an indication of C-H stretching vibrations. Thus, -CH_2- group is confirmed present.

At peaks 1600 – 1800cm^{-1}, there is a medium-weak (m-w) absorption intensity which shows the presence of C=C group for both Figures 4.21 and 4.22. While there is an indication of the presence of alkanes C+ at peak 1450cm^{-1}. Though sharper peaks indicates the presence of C=N nitriles group as seen in Fig. 4.22.

At peaks 1300-1000cm^{-1}, the absorption shows the presence of C-O group, alcohols, esters, ethers, carboxylic acid and the intensities are sharp.

Fig. 4.23 and Fig. 4.24 give the infrared spectra of the sample of ROSP produced directly and indirectly respectively. It is observed that the absorptions at region 3500 – 3800cm^{-1}, the peak of the absorption shows the presence of phenol in concentrated solutions with O-H group, at this intensity the absorption is medium .

At peaks 3400cm^{-1}, the absorption shows the presence of alcohols and phenols having O-H functional group and H bonded (hydrogen bond), the intensity of absorption is more in Fig. 4.24. While at peaks 3000-2850cm^{-1} show a strong intensity of absorption more in Fig 4.24, a C-H group presence with the stretching vibration. Thus –CH_2- group is confirmed.

The peak of 2400cm^{-1} shows the presence of C=N group and nitriles with sharp intensity. While peaks 1725-1700cm^{-1}, the absorption has a bit of sharpness with strong intensity indicating the presence of C=O and also COOH groups. And also at peaks 1600-1400cm^{-1} several bit of sharpness with C=C group and aromatics with the intensity at m-w. The peaks at these regions have more intensity in Fig. 4.24.

At peaks 1300cm^{-1}, the absorption is strong but sharper in Fig. 4.24 to 4.23 and this shows the presence of C-O group, alcohol, esters, ethers, and carboxylic acids.

At peak 800cm^{-1}, a very sharp intensity of absorption is observed in Fig. 4.23, this shows the presence of C-X, chloride with strong intensity.

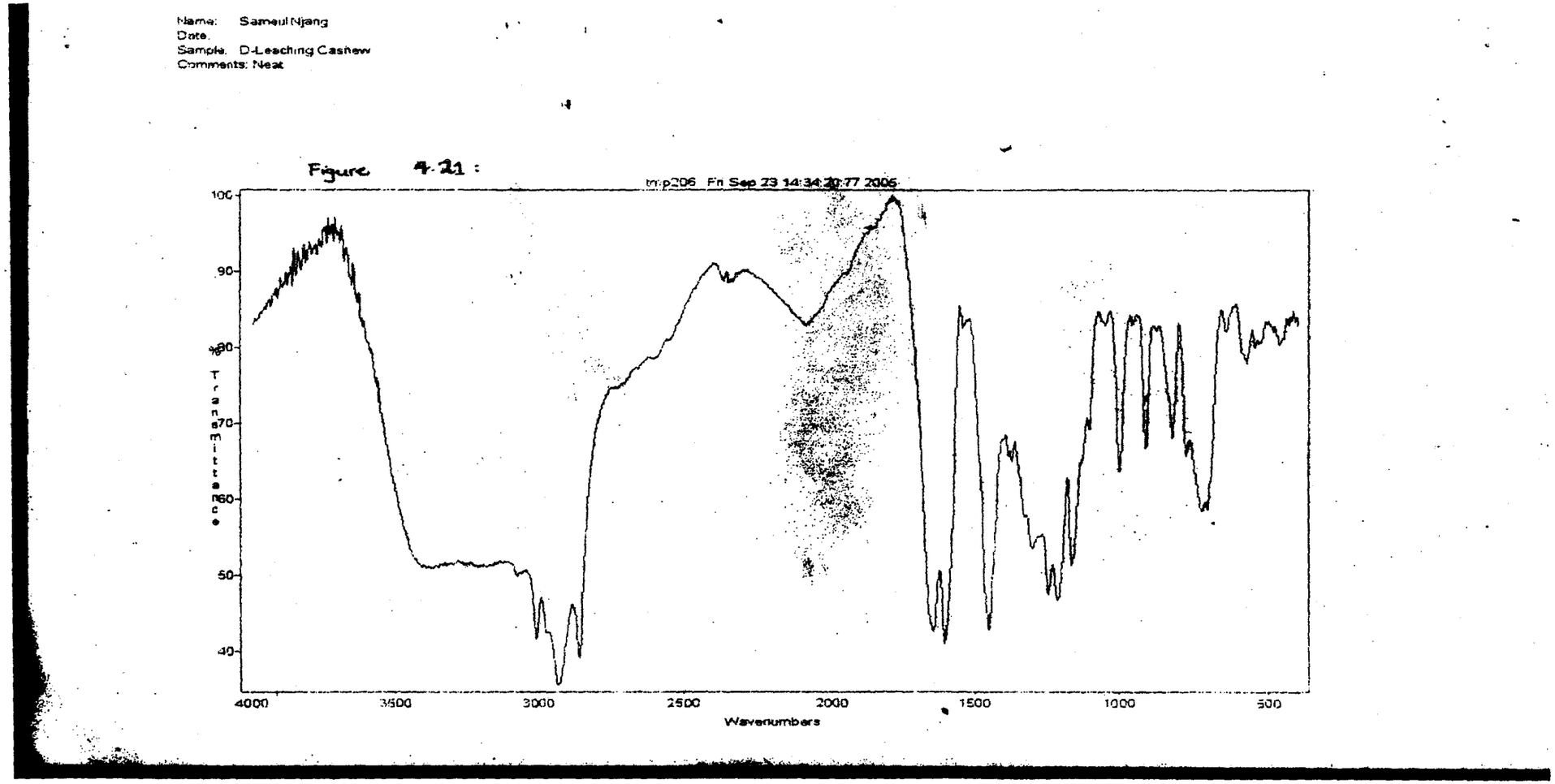
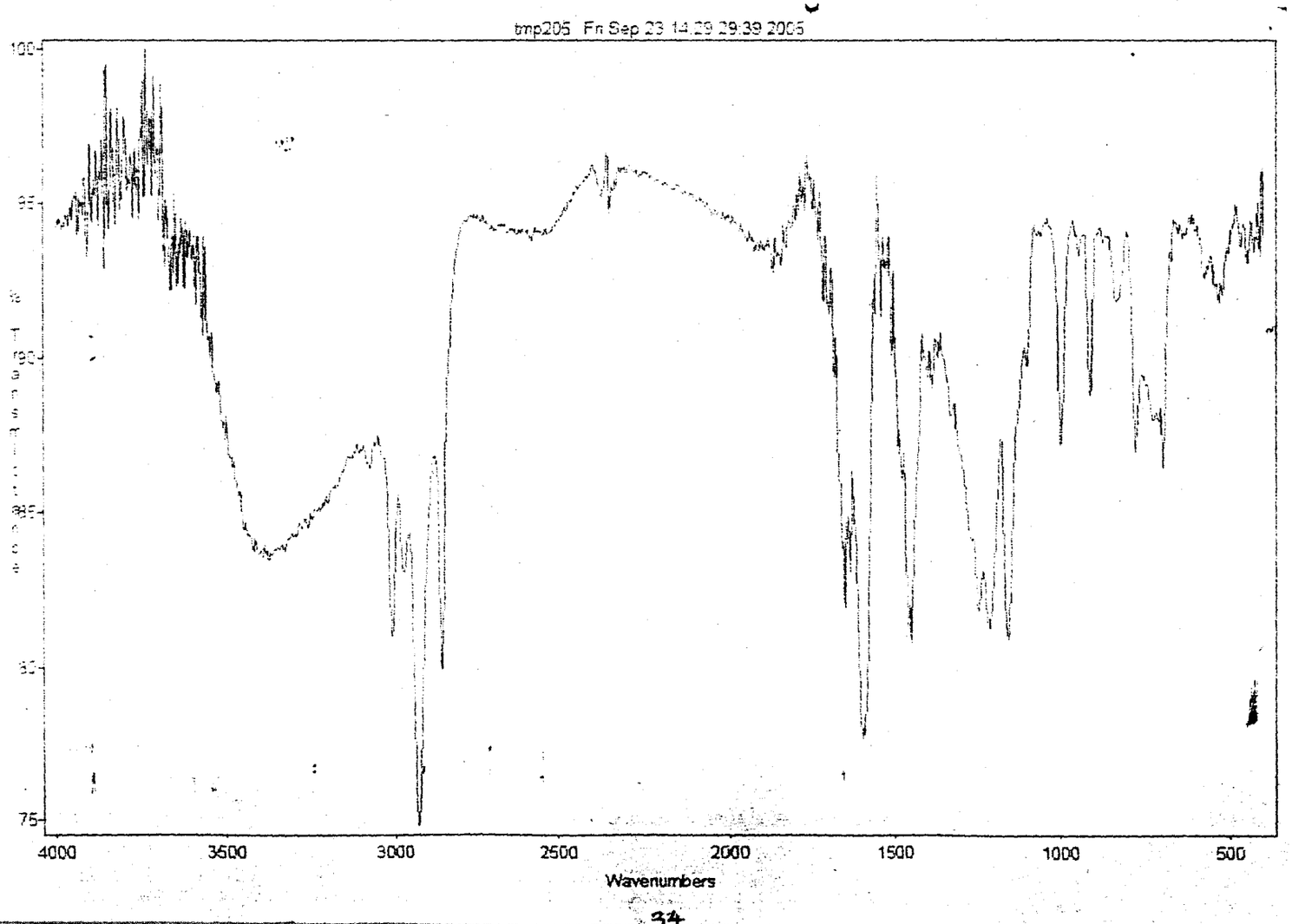


Figure 4.21: IR Analysis for Direct leaching of CNS.

Name: samuel Njang
Date:
Sample: Indirect L Cashew shell
Comments: Neat

Figure 4.22 :



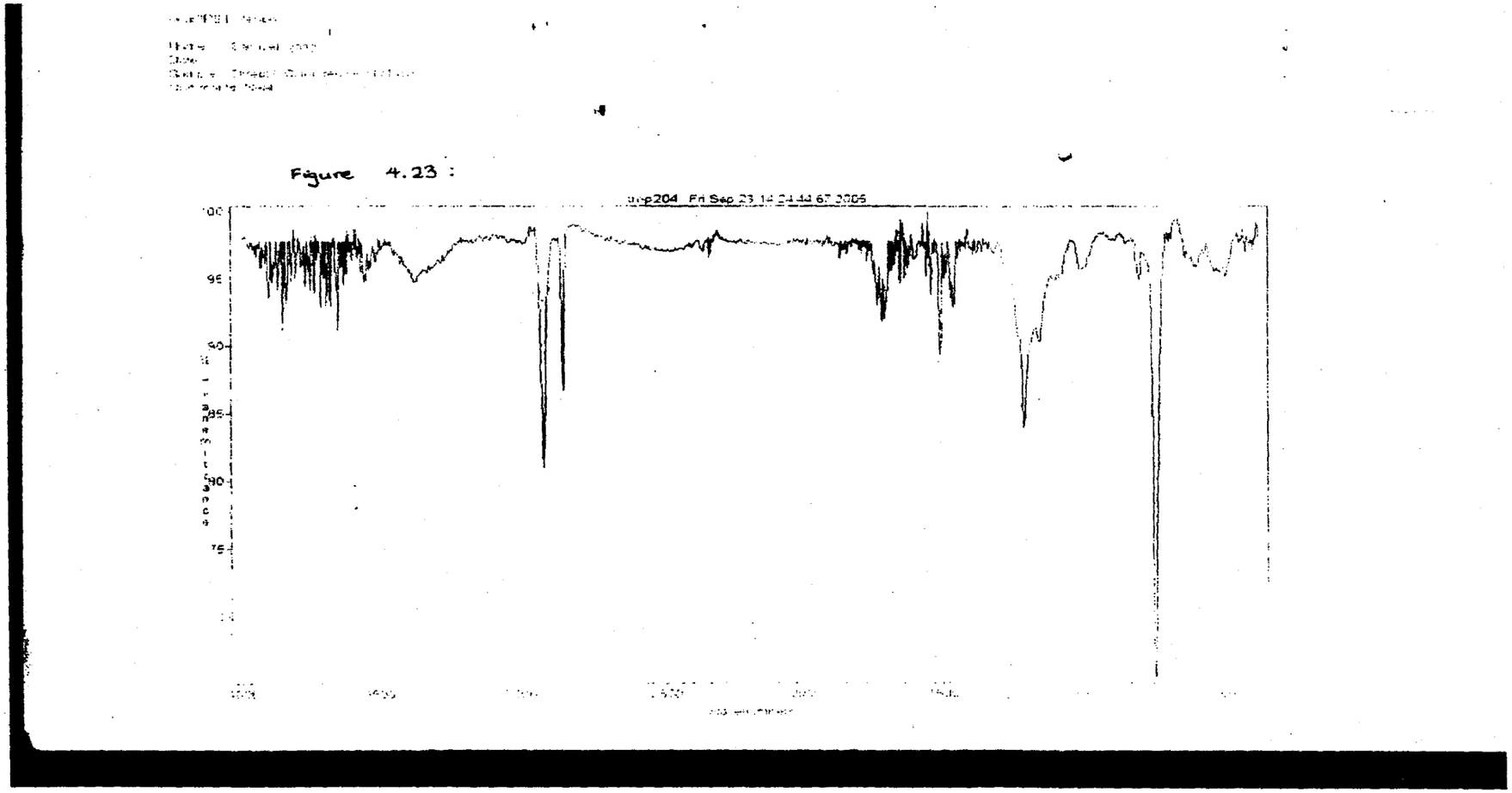
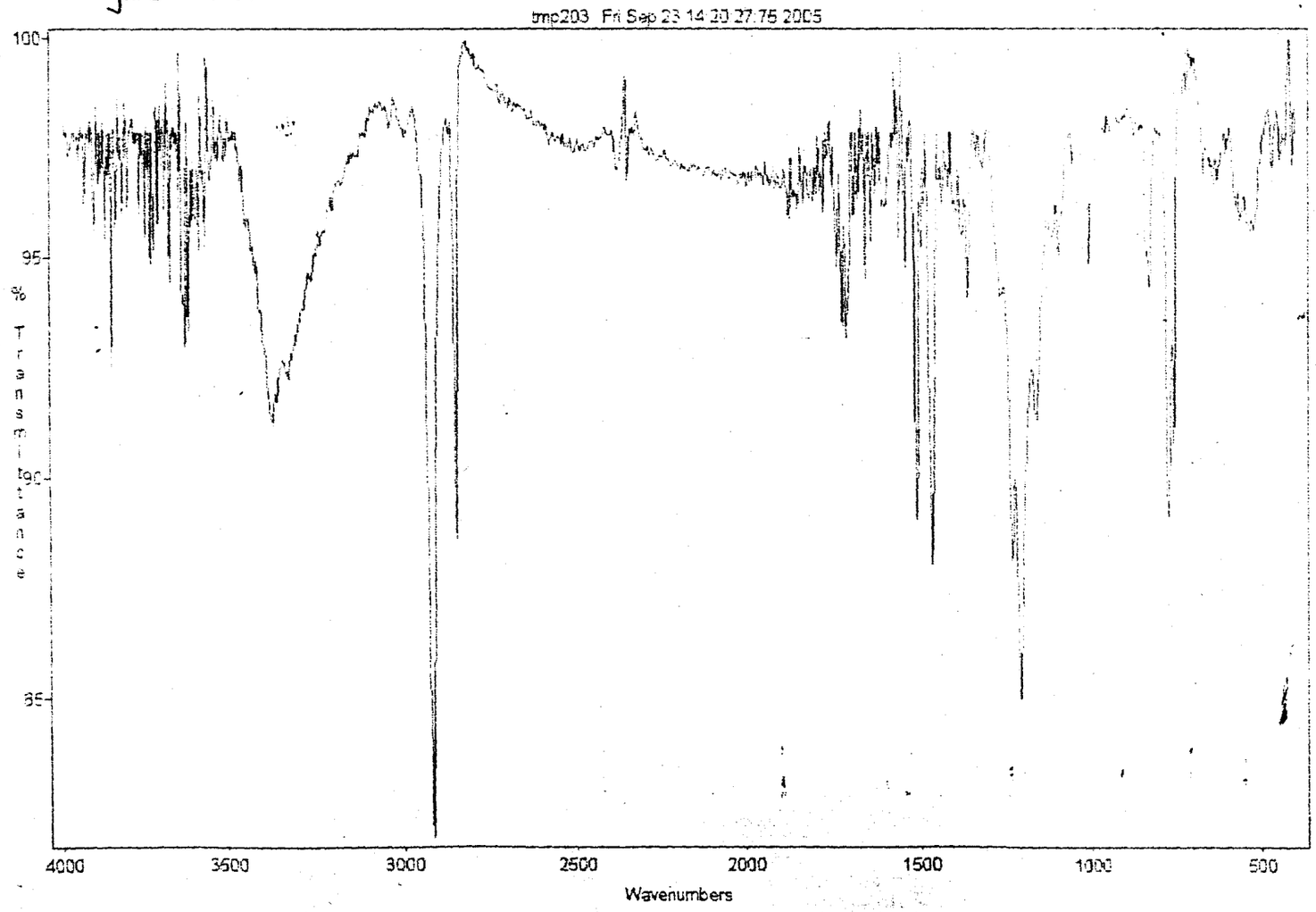


Fig 4.23: IR Analysis for Direct Leaching of ROS.

Name: Samuel Njang
Date:
Sample: Indirect L onion red skin
Comments: Neat

Figure 4.24:



CHAPTER FIVE

5.0 DISCUSSION OF RESULTS

Tables 4.1 to 4.12 show the results obtained from the extraction of polyphenols from CNS and ROSP using both direct and indirect leaching techniques at temperatures of 65, 70 and 75 degree Celsius.

5.1 Physicochemical Properties of Polyphenol Extract

Table 4.20 shows the values of the various properties of the samples extracted. From this table, the pH values show that both samples are acidic with that of ROS being more acidic than that of CNS.

The specific gravity values of both samples were found to be less than the literature values but approximately they could be said to be in harmony with the literature value.

The refractive index of both samples was found to be relatively close to the literature value with only a difference of about 0.03.

The percentage yield also shows that as the temperature of extraction (operating temperature) increased, the yield also increased. This is in harmony with the theory, which brings out the fact that the higher the temperature of extraction the higher the extract (yield) produced.

5.2 Infrared Spectra

The analysis of the Fourier Transform Infrared spectra (FTIR) shows that the following functional groups were found present in the structure of the samples. Both samples contained reasonable amounts of phenol, alcohol, ethers, esters, and COOH groups. Sample of CNS extract contained groups such as CH, C=N, C=O, C=C, CH_3, while the ROS extract contained C-H, C-X, chlorides, C=N, C=C, C=O, CH_2.

CHAPTER SIX

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

The solvent used diisopropyl ether (DIPE) was able to effectively extract polyphenol from CNS and ROS.

The values of some physicochemical properties of the samples produced show that they are relatively close to the literature values.

The analysis of the infrared spectra shows that the two samples contain a reasonable amount of phenol (phenyl group). Both samples can be treated properly to give phenolic resins, selective solvent for refining oils, germicidal paints, cosmetics and pharmaceuticals, etc.

6.2 Recommendations

Efforts with respect to seeking more advanced information on the properties of the samples is still necessary to enable one establish a standard specification, since IR means structural properties.

To improve on the validity of this research work, further analysis should be carried out to determine some important properties such as thermal properties and more definite structural properties, so as to produce more reasonable and concrete results.

Unavailability of most of these analytical equipment is one of the major constraints that delayed the progress and details of the work, and as such the university should try to procure more relevant equipment such as FTIR, NMR, DSC, DMA, etc and other analytical equipment of high technological quality. They would assist a great deal for students to carry out their research work effectively and qualitatively, and also generate reasonable amount of capital for the department and the university at large.

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24 International Programme on Chemical Safety and the Commission of European communities, 2004.

APPENDIX

Appendix A1

A1: Calculation of percentage yield (%)

From Table 4-01;

Average Mass of sample after extraction;

$= (4.54 + 4.56 + 4.53 + 4.54 + 4.54) / 5$

$= 4.542$

$\%\ Yield = ((m_1 - m_2) / m_1) * 100$ ...I (Horwitz, W., 1970)

$= ((5 - 4.54) / 5) * 100$

$= 9.20\%$

From Table 4-02:

Average;

$= 3.47$

$\%\ Yield = ((5 - 3.47) / 5) * 100$

$= 30.60\%$

From Table 4-03:

% Yield is given by;

$|(m_1 - m_2) / (m_1 - m_T)| * 100$ ...II (Horwitz W., 1970)

Sample of ROS – A, $m_1 = 9.07$

$m_2 = 8.81$

$m_T = 4.07$

thus A % yield = 5.20%

Sample of ROS – B, $m_1 = 9.11$

$m_2 = 8.86$

$m_T = 4.11$

thus B % yield = 5.00%

and so on for sample of ROS - C, D, and E.

From Table 4-04

% Yield with reference to equation II

Sample of CNS – A; $m_1 = 9.05$

$m_2 = 8.07$

$m_T = 4.05$

Thus A % yield = 19.60%

Sample of CNS – B; $m_1 = 9.15$

$m_2 = 8.17$

$m_T = 4.15$

Thus B % yield = 19.60%

And so on for sample of CNS – C, D, and E.

Appendix B

Table B1: Important Data on Diisopropyl Ether

DIISOPROPYL ETHER	
IMPORTANT DATA	
PHYSICAL STATE; APPEARANCE: COLOURLESS LIQUID, WITH CHARACTERISTIC ODOUR. **PHYSICAL DANGERS:** The vapour is heavier than air and may travel along the ground; distant ignition possible. As a result of flow, agitation, etc., electrostatic charges can be generated. **CHEMICAL DANGERS:** The substance can readily form explosive peroxides if unstabilized and explode on shaking. **OCCUPATIONAL EXPOSURE LIMITS:** TLV: 250 ppm as TWA; 310 ppm as STEL; (ACGIH 2004). MAK: 200 ppm, 850 mg/m^3; Peak limitation category: I(2); Pregnancy risk group: D; (DFG 2004).	**ROUTES OF EXPOSURE:** The substance can be absorbed into the body by inhalation of its vapour. **INHALATION RISK:** A harmful contamination of the air can be reached rather quickly on evaporation of this substance at 20°C. **EFFECTS OF SHORT-TERM EXPOSURE:** The substance is irritating to the eyes, the skin and the respiratory tract. The substance may cause effects on the central nervous system. Exposure above the OEL could cause lowering of consciousness. **EFFECTS OF LONG-TERM OR REPEATED EXPOSURE:** Repeated or prolonged contact with skin may cause dermatitis.
PHYSICAL PROPERTIES	
Boiling point: 69°C Melting point: -60°C Relative density (water = 1): 0.7 Solubility in water: poor Vapour pressure, kPa at 20°C: 15.9 Relative vapour density (air = 1): 3.5	Relative density of the vapour/air-mixture at 20°C (air = 1): 1.5 Flash point: -28°C Auto-ignition temperature: 443°C Explosive limits, vol% in air: 1.4-7.9
ENVIRONMENTAL DATA	
NOTES	
Usually contains p-benzylaminophenol as stabilizer. An added stabilizer or inhibitor can influence the toxicological properties of this substance, consult an expert. Check for peroxides prior to distillation; eliminate if found. Card has been partly updated in October 2004. See sections Occupational Exposure Limits, EU classification, Emergency Response.	
ADDITIONAL INFORMATION	
LEGAL NOTICE Neither the CEC nor the IPCS nor any person acting on behalf of the CEC or the IPCS is responsible for the use which might be made of this information	

Table B2: Hazards of Diisopropyl Ether

TYPES OF HAZARD / EXPOSURE	ACUTE HAZARDS / SYMPTOMS	PREVENTION	FIRST AID / FIRE FIGHTING
FIRE	Highly flammable.	NO open flames, NO sparks, and NO smoking.	AFFF, powder, alcohol-resistant foam, water spray, carbon dioxide.
EXPLOSION	Vapour/air mixtures are explosive.	Closed system, ventilation, explosion-proof electrical equipment and lighting. Prevent build-up of electrostatic charges (e.g., by grounding).	In case of fire: keep drums. etc.. cool by spraying with water.
EXPOSURE			
Inhalation	Cough. Drowsiness. Sore throat.	Ventilation, local exhaust, or breathing protection.	Fresh air, rest. Refer for medical attention.
Skin	Dry skin. Redness.	Protective gloves.	Remove contaminated clothes. Rinse skin with plenty of water or shower.
Eyes	Redness.	Safety spectacles.	First rinse with plenty of water for several minutes (remove contact lenses if easily possible), then take to a doctor.
Ingestion	(Further see Inhalation).	Do not eat, drink, or smoke during work.	Rinse mouth. Rest. Refer for medical attention.

Spillage Disposal:

Evacuate danger area! Consult an expert! Ventilation. Collect leaking and spilled liquid in sealable metal containers as far as possible. Absorb remaining liquid in sand or inert absorbent and remove to safe place. Do not wash away into sewer. Personal protection: self-contained breathing apparatus.

Safe Storage:

Fireproof. Cool. Keep in the dark. Keep in a well-ventilated room. Store only if stabilized.