PHYSICOCHEMICAL, SPECTROSCOPIC AND RHEOLOGICAL STUDIES OF *IRVINGIA GABONENSIS (O'RORKE)* BAILL AND *CELTIS ZENKERI (ESA)* ENGL GUM EXUDATES

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ZARIA, NIGERIA

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PHYSICOCHEMICAL, SPECTROSCOPIC AND RHEOLOGICAL STUDIES OF *IRVINGIA GABONENSIS (O'RORKE)* BAILL *AND CELTIS ZENKERI (ESA)* ENGL GUM EXUDATES

BY

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Declaration

I hereby declare that the work in the thesis titled "Physicochemical, Spectroscopic and Rheological Studies of *Irvingia gabonensis* and *Celtis zenkeri* Gum Exudates" was performed by me in the Department of Chemistry A.B.U, Zaria under the supervision of Prof. E.B Agbaji and Prof. N.O Eddy. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this work has been presented for another diploma or degree in any Institution.

Mamman Ibrahim SANI _____ _____

Signature Date

Certification

This project report titled "Physicochemical, Spectroscopic and Rheological Studies of *Irvingia gabonensis* and *Celtis zenkeri* gum exudates" meets the regulations governing the award of the degree of Master of Science of Ahmadu Bello University, Zaria, and is approved for its contribution to knowledge and literary presentation.

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Dedication

This work is dedicated to Almighty Allah (S.W.T) who gave me the life, opportunity and guidance throughout the programme.

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All praises and gratitude are due to Almighty Allah, the most knowledgeable, the wisest, the creator and sustainer of the world. May the peace and blessings of Allah be upon His Messenger and Prophet Muhammad (S.A.W), the members of his household and all those who follow him on the path of righteousness.

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Abbreviations

AS GUM = *Acacia senegal* Gum Exudates

CZ GUM = *Celtis zenkeri* Gum Exudates

FTIR = Fourier Transform Infrared Spectroscopy

GCMS = Gas Chromatography Mass Spectroscopy

IG GUM = *Irvingia gabonensis* Gum Exudates

SEM = Scanning Electron Microscope

Abstract

Irvingia gabonensis (IG) Baill and *Celtis zenkeri* (CZ) plant are used for various purposes such as timber and medicine. Physicochemical, spectroscopic and rheological studies of IG and CZ were carried out on the exudates. The analyses were carried out using standard procedures and the results were tested and compared with that of *Acacia Senegal* gum exudates. The results of the physiochemical analysis of *Irvingia gabonensis* and *Celtis zenkeri* gum exudates revealed that the gums are highly soluble in water with high amount of total dissolved solids. The exudates were odourless, tasteless and with pH values of 5.42 and 4.61 for *Irvingia gabonensis* and *Celtis zenkeri* gum exudates respectively. The viscosity values of the gum exudates were found to decrease with increase in temperature while increase in concentrations of the exudates was found to increase the viscosity of the gums. Addition of electrolytes such as KCl, KBr and $AlCl_3$ was found to increase the viscosity of the gums. The concentration of metals presence in the study gums are manganese (213-146mg/kg), iron (375-311mg/kg), zinc (41-10mg/kg), lead (63-15mg/kg), magnesium (2285-2274mg/kg), cadmium (1.6-1.0mg/kg), calcium (2315-652mg/kg), copper (16.6-16.4mg/kg) and nickel (79-48mg/kg) for IG and CZ gums respectively. Surface morphology analyses indicate the shapes and dimension of the particles in addition to the amorphous nature of the gum particles. The measurement of fibre and pore shows that the pore areas for IG gum and CZ gums were 0.71μm^2 and 0.02μm^2 respectively while the fibre length ranged from 1.03 to 21.34µm and 53.35 to 1.52µm respectively. The Fourier Transform Infrared Spectroscopy (FTIR) analysis revealed the presence of C-C bending, CO_2 bending, C-O stretch, C-H wag, C-H in plane bending, C=C stretch, C-D and C=O asymmetric stretch for both 1G and CZ gums. The data obtained from the different analyses give an insight into the possibility of using the plant gums as food additive, pharmacological and industrial applications.

CHAPTER ONE

1.0 INTRODUCTION

A gum in general, is any water-soluble or water-swellable polysaccharide that is extractable from marine and land plants, or from microorganisms that possess the ability to contribute viscosity or gelling ability to their dispersions (Abu Baker *et al.*, 2007). The most fundamental property of a gum therefore is its water solubility and high viscosity in aqueous dispersions. Among the advantages of natural gums over their synthetic counterparts are their biocompatibility, low cost, low toxicity (eco-friendliness) and relative widespread availability (Odeku, 2005; Emeje *et al.*, 2009; Nep and Conway, 2010; Ogaji and Okafor, 2011).

Plant gums are organic substances obtained as an exudation from fruits, trunk or branches of the trees spontaneously or after mechanical injury of the plant by incision of the bark or after the removal of the branch or after invasion by bacteria or fungi (Ahmed *et al*., 2009). They are capable of displaying colloidal properties in an appropriate solvent or swelling agent. According to Ahmad *et al*. (1994), most plant gums are polyelectrolytes, which is a class of polymers that bears a large number of ionizable groups on the main chain. Gums have numerous applications in several industries. For example, *Albizia zygia* and some *Albizia lebbeck* gums have been found to be useful as natural emulsifiers for food and pharmaceuticals (Mhinzi, 2002). According to DePaula *et al*. (2001) *Albizia lebbeck* gum exudate is also used as a substitute for Arabic gum in the metallurgical industries. Guar and some other gums have a number of applications in the mining and mineral processing industry (Ma and Pawlik, 2007). In the froth flotation of base metal and platinum group metal ores, guar gum is used as a depressant of naturally hydrophobic waste minerals such as talc. The role of the polysaccharide is to adsorb on the talc surface, render it hydrophilic and prevent its flotation (Ma and Pawlik 2007).

The principal use of plant gums is in foodstuffs because of their ability to impart desired qualities to foods by influencing their viscosity, body and texture; most frequently in confectionery food, flavouring and soft drinks. They also have pharmaceutical and industrial applications as demulcents, adhesives in pill manufacture, lithography, paints, inks, corrosion inhibitors and as emulsifying agents. The use of natural gums exudates and extracts of plants, have been given a strong consideration due to its high value for industrialization and to the international market, example being *Gum Arabic* which in current production potential, is around 30,000 to 40,000 tonnes per annum, Virtually all gum arabic in the Sahelian zone is exported, either immediately or after a period of storage or stockpiling. Sudan dominates the world exports, accounting for 70% to 80%, the balance being accounted for by the Sahelian countries of West Africa (Nigeria, Mali, Niger, Burkina Faso, Chad, Tanzania and Kenya). One billion pounds are consumed in the United States each year where the growth in demand exceeds 8% per year (Yusuf *et al.*, 2006).

Another example is the cashew gum, which by natural exudation or by means of incisions, produces a gum or resin of a yellowish colour, soluble in water, and which presents a great potential for industrialization, appears on the trunk and branches of the cashew tree. It is similar to gum arabic and may be used as a substitute for liquid glue for paper. In the pharmaceutical and cosmetic industry it is used as an agglutinant for capsules and pills while in the food industry as a stabilizer of juices, beer and ice cream, as well as for clarification of juices, and can also be utilized in the making of cashew wine. Besides proving to be strong wood glue when mixed with water, it presents a fungicidal and insecticidal action and because of this it is much used in book binding. Research already exists on its utilization in the making of inks and varnishes (Emeje *et al*., 2009)

These gums find wider application because of their physical, rheological and chemical properties such as properties include solubility, water sorption, swelling capacity, pH, effect of temperature, and viscosity among others (De Paula *et al*., 2001). *Ficus glumosa* is an example of lesser known plant producing gum the plant commonly called African rock fig tree in English and 'Kawuri' in Hausa belongs to the family, Moraceae. *Ficus glumosa,* a tall plant of about 24 meters in height and about 3 meter in diameter, occurs on rocky outcrops, where it splits rocks; along dry water course or in open boundry; frequently in valleys, where it reaches its greatest size (Keay, 1989) The species also occurs in fringe forest in savannah areas, especially in swampy ground, and in swamp forest in coastal areas (Arbonnier, 2004). The bark also contains abundant sticky white latex which is used in Northern Nigeria like bird-lime to trap crickets (Ameh, *et al*., 2012). The exudate is also chewed (chewing-gum) and used for fastening arrowheads to their shaft.

1.1 Justification of the Study

There is increasing demand for gums globally because of their vast applications leading to increase in prices of the existing ones in the local and international markets. For the price to be stable and less expensive there is need to look for alternative suitable natural gums which will have similar characteristics to the existing ones. In view of the numerous applications that have been discovered for plant gums and the dependence of the applications on physicochemical, rheological, surface morphology and other properties of the gums, there is the need to assess these properties for gums that have not been adequately studied and thereby providing a means of references for future use.

1.2 Aim of the Study

The aim of the study is to determine the physical and chemical properties of the plant gums from *Irvingia gabonensis and Celtis zenkeri*, and compare the properties of the plant gums with Gum Arabic *(Acacia senegal)* which is one of the most commercially used plant gum.

1.3 Objectives of the Study

The above aim will be achieved through the following objectives:

i. Tapping and purification of the gum exudates from the parent plant species;
ii. Investigating the functional groups present using fourier transform infrared spectroscopy (FTIR);
iii. Analyzing the chromopores present within the chemical components using ultra violet spectroscopy (UV);
iv. Identifying the metals present using atomic absorption spectrometry;
v. Determining the rate of flow of the gums by the use of a cannon ubbelohde cappilary viscometer;
vi. Determining the effects of temperature, concentration and addition of electrolytes on the viscosity of the gums;
vii. Determining the fibre and pores measurement using scanning electron microscope;
viii. Analyzing the surface morphology by the use of scanning electron microscope;
ix. Determining the elements present using scanning electron microscope.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Gums

The term gum is generally applied to a wide variety of colloidal substances that are similar in appearance and have peculiar characteristics (Columbia Encyclopedia, 2006). Ghani (1988) described gums as a group of non- crystalline polysaccharides which usually contain sugars such as mannose and galactose and their uronic acid derivatives. The term gum is defined as a molecular structure with high molecular masses, a substance usually with colloidal properties, that in an appropriate solvent produce gels or suspensions of high viscosity or solutions of low matter content that can absorb water at ten times their weights (Umoren *et al.,* 2006). Plant gum are obtained as an exudates from fruit, trunk or branches of tree, the exudates become hard nodules or ribbons on dehydration to form a protective sheath against microorganism. They form clear glassy masses which are usually coloured from dark brown to pale yellow. The gums are high molecular weight polymeric compounds composed mainly of C, H, O and N. They are capable of possessing colloidal properties in an appropriate solvent or swelling agent at low dry weight. They occur naturally as salts (especially of calcium and magnesium). Gums are either hydrophobic or hydrophilic. Hydrophobic gums are insoluble in water and these include resins and rubber, whereas hydrophilic gums are soluble in water and can be subdivided into natural, semi-synthetic and synthetic gums (Sarah, 1998).

Gums serve as a food reserve or to check excessive transpiration, Tannin protects against frost, animals and fungi while Resins acts as protection against plant injury. These substances occur in larger quantities in some plants as compared to others (Sarah, 1998).

2.1.1 Origin of Gums

There is no agreement as to the origin of gum exudates. Some thought that they are a product of normal plant metabolism and some suggest that they arise from pathological conditions (Smith and Montgomery, 1959). Others observe that it is likely to be as a result of non-parasitic diseases and bacteria activities (Ghani, 1988 and Sanni, 1992).

2.1.2 Classification of Gums

Gums, whether they are occurring naturally or synthetically, are divided into two groups. Group I contains acid gums of which the acid components are L- glucuronic acid, D-glucuronic acid, D-galacturonic acid, sulphate and phosphate groups. Group II comprises the neutral gums of which the neutral components are hexoses, 6-deoxyhexoses, pentoses, sugar alcohols and ethers of all these classes (Davidson, 1980). Synthetic gums on the other hand contain basic groups and have not been classified along with these two groups and therefore, provision has been made for the synthetic gums to be classed into group III, of which the components are amino acids, polypeptides or proteins and O- and N- alkyl derivatives of all these classes of compounds.

In the classification of plant gums, substances such as resins which are not true gums are often included (Davidson, 1980). True gums exuded by plants are soluble in water or if not soluble, absorb large amount of water to form gluey solutions. Resins on the other hand, are completely insoluble in water but soluble in ethanol, while true gums are precipitated in the presence of ethanol. The chemical nature of true gums is complex. Generally, they contain carbon, hydrogen, oxygen, calcium, magnesium and potassium in the form of metallic salts which occur in various organic compounds. Ha and Thomas (1988) were able to distinguish the components of various gums, They reported that significant quantities of xylose and fructose

were the only sugars found in tragacanth gum while ghatti gum has low quantities of these sugars. Cherry gum has low levels of xylose and rhamnose, and a large amount of arabinose. On the other hand, Karaya, guar and locust bean gums contain no arabinose while a large quantity of mannose occurs in the locust bean and guar gums. Trangacanth, ghatti and arabic gums all contain arabinose and rhamnose (Ha and Thomas, 1988).

The gums can be distinguished from one another by the levels of sugars they contain. Higher levels of these sugars are seen in tragacanth followed by ghatti while gum arabic has the least content as compared to the other two gums. Karaya and trangacanth were found to contain galacturonic acid while gum arabic, cherry and ghatti contain glucoronic acid. Khaya gum has been reported to contain both D- glucoronic and D-galacturonic acids. Analysis of the gum showed that approximately 50% was in the free acid form and the remainder was largely the calcium salt (Aslam *et al.,* 2006)

2.1.3 Types of Plant Gums

Plant gums are polyanionic polysaccharides obtained from trees of genera *Sterculia, Albizia, Acacia, Astragalus* and *Anogeissus.*

Gum Karaya is defined as the dried exudate obtained from *Sterculia Urens Roxd.* And other related species of *Sterculia*, and variously known as *Sterculia* gum, Indian gum, kadayo gum, katilo gum, kullo gum, kutero and mucara gum. (JECFA-FAO-1988). Gum *Karaya* consists mainly of high molecular weight acetylated polysaccharides which on hydrolysis yields galactose, rhamnose and arabinose together with a small amount of glucuronic acid (Anderson *et.al*, 1982). Gum Karaya contains approximately 0.17% w/w nitrogen presumably in amino acid form (Anderson *et.al*, 1982). Gum Karaya possesses high viscosity at extremely low gum concentration and commercial gum has specific rotation of +58° (Anderson *et. al*, 1982) and it

has a low solubility in water so it tends to swell rather than dissolve in water and a coarse particles of gum Karaya can absorb water and swell to 60-100 times its original volume (Meer, 1980), a property useful in bulking agents.

Albizia gum derived from trees of the genus Albizia is formed as round elongated wax of variable size and colour ranging from yellow to dark brown (Mital and Adotey, 1973).

Chemical analysis reveals the presence of L-arabinose D- galactose D-glucuronic acid, Dmannose and 4-0-methyl glucuronic acid and L-rhamnose with slight acetylation and methylation. Metal ion analysis suggests that gum exudates derived from the genus *Albizia* are complex salts *of* calcium, potassium, magnesium and sodium, in decreasing proportions. Other metals like zinc, copper, iron, lead and aluminium are present in trace amounts (Anderson and Morrison. 1990). Albizia gum is soluble in water forming a colourless mucilage with a bland taste (Mitai and Adotey, 1973).

Gum arabic is the dried gummy exudation *of Acacia Senegal* or closely related species of Acacia family leguminosae (JECFA-FAO, 1990). It occurs as a mixture of calcium magnesium and potassium salts of arabic acid and is composed of six carbohydrate moieties namely galactose, arabinopyranose, arabinofuranose, rhamnose, glucuronic acid and 4-0 methyl glucuronic (Anderson and Karamalla, 1966). The gum is highly soluble in water and solutions of up to 60% w/v gum concentration can be prepared (Glicksman, 1983).Gum arabic solution is slightly acidic with maximum viscosity obtained at the neutral pH. It is principally used in the food and pharmaceutical industries as stabilizer, thickener, suspending and binding agent in the manufacture of confections, dairy products, beverages, cotton -seed oil emulsion and tablets (Glicksman, 1983).

Gum Tragacanth is the dried exudation obtained from stems and branches of *Astragalus gummifer labillardiere* and other Asiatic species of *Astragalus* (Family leguminosae (JECFAFAO, 1986). Gum tragacanth is a complex mixture of acid polysaccharides containing galacturonic acid, galactose, fructose, arabinose, xylose and small amounts of rhamnose and glucose. It swells in water to give thick gel like dispersion which shows typical pseudo plastic behaviour (Gordon, 1992). Gum tragacanth has been used extensively in food products (Gordon, 1992).

Gum Ghatti is a dried gummy, translucent exudate obtained from *Anogeissus latifiolia* (family combretaceae), a large tree found in India and Sri Lanka. The exudations are natural but the yield can be increased by making artificial incisions. Gum ghatti occurs naturally as a calcium and magnesium salt of complex polysaccharides acid. Acid hydrolysis has shown the gum to consist of L-arabinose, D-galactose, D-mannose, D-xylose and D-glucuronic acid in the ratio 10:6:2:1:2 (Jefferies *et.al.,* 1977). On dispersion in water, gum ghatti forms viscous solutions, intermediate between those of gum arabic and gum karaya. The dispersions have emulsifying and adhesive properties equivalent or superior to those described for gum arabic (Jefferies *et.al.,* 1977). Gum ghatti is used as food additive besides being used in medicines, textiles and adhesives (Topalian and Elsesser, 1966).

2.2 Gums Selected for the Study

2.2.1 *Irvingia gabonensis* (*Aubry lecomte ex O'Rorke)* Baill

Irvingia gabonensis Is a species of tree that grown in West Africa. It produces a fruit that is similar to mangos. People use the flesh and seeds of this fruit to make food and medicines. This fruit is very high in fiber, protein, and healthy fats (FAO 1993-2007). Recent studies on volunteers in Yaounde, Cameroon shows that extracts from this fruit can help to reduce fat,

lower cholesterol, and increase hormones that regulate glucose levels in the body (Ngondi *et al*., 2005). Extracts from African Mango seeds have many positive effects on human health, Studies shows that African Mango (*Irvingia gabonensis*) can increase the hormone, Adiponectin in the body (Ngondi *et al.,* 2009).

Irvingia gabonensis is indigenous to the humid forest zone from the northern tip of Angola, including Congo, DR Congo, Nigeria, Côte d'Ivoire and south-western Uganda. It is cultivated in south-western Nigeria, southern Cameroon, Côte d'Ivoire, Ghana, Togo and Benin (Ngondi *et al.,* 2005). *Irvingia gabonensis* is a species of African trees in the genus *Irvingia*; sometimes known by the common names wild mango, African mango, bush mango or dika. It bears edible mango-like fruits, and is especially valued for its fat- and protein-rich nuts.

2.2.2 *Celtis zenkeri* Engl (Esa)

Celtis zenkeri is one among the different species of Celtis; Celtis comprises of about 100 species and is widespread in all tropical, subtropical and temperate regions. For tropical Africa 11 species have been recorded, 2 of which are endemic to Madagascar. Celtis is taxonomically a difficult genus, showing much morphological variability. Traditionally, it has been treated as part of the family Ulmaceae, but later it was often considered to belong to a separate family Celtidaceae, whereas from most recent research it was proposed to take up the latter family into Cannabaceae (Bekele-Tesemma, 2007).

Celtis zenkeri grows fast, 1–2 m per year. First fruits may appear when trees are 4 years old. Flowering trees have been recorded from August to October in southern Africa, and in the month of May in Ghana. The flowers are usually pollinated by insects such as bees. Fruits ripen about 2 months after flowering. They are relished by birds such as bulbuls, mousebirds and barbet, they are an important component of the diet of colobus monkeys, and are also eaten by

baboons (Bekele-Tesemma, 2007). All these animals may contribute to seed dispersal. *Celtis zenkeri* occurs in a very wide range of habitats, from savanna to dry evergreen forest, riverine forest, montane rainforest and coastal forest, from sea-level up to 2400 m altitude. In Somalia it is typical of evergreen Juniperus and Buxus forest at altitudes of 1650–2000 m. Although it prefers relatively fertile and deep moist soil, it can also be found on sandy dunes and river banks, as well as on rocky soils, but under these conditions it usually only develops into a shrub. It is moderately drought resistant and can withstand light frost. In South Africa, trees have been recorded to be severely affected by water logging conditions, leading to death of trees.

2.2.3 Reference Gum **(***Acacia senegal***)**

Gum arabic is an edible, dried, gummy exudate from the stems and branches of *Acacia senegal* and *Acacia seyal* that is rich in non-viscous soluble fiber (Williams and Phillips, 2000). It is defined by the FAO/WHO Joint Expert Committee for Food Additives (JECFA) as 'a dried exudation obtained from the stems of *Acacia senegal* which will be closely related species of Acacia (family Fabaceae)' (FAO, 1999). In 1982 JECFA classified Gum Arabic as 'ADI not specified' (FAO, 1982). However, as a result of subsequent research, the specifications for GA have been severally revised (FAO, 1986, 1990, 1999; WHO, 1990). GA (Gum arabic) has wide industrial uses as a stabilizer, thickening agent and emulsifier, mainly in the food industry (e.g. in soft drinks syrup, gummy candies and marshmallows), but also in the textile, pottery, lithography, cosmetics and pharmaceutical industries (Verbeken et al., 2003).

2.3 Properties and Application of Plant Gum

The most fundamental property of a gum which makes it unique amongst polysaccharides generally is its solubility and viscosity. (Meer, 1980).The majority of gums dissolve in water at different concentrations (e. g gum Arabic can form solutions of up to 60% forming viscous

solutions). These properties of gums are exploited in many applications. The major application of gum is in food industry where emulsifying and stabilizing properties are utilized. It is also used in the pharmaceutical and medical field, in addition to other industries (cosmetic, adhesives, paints and inks). According to Glicksman and Schachat (1959) the major use (55%) of gum (gum *Arabic)* in the USA is in the food industry, primarily in confectionery. In Western Europe food uses of gum *arabic* accounted for 76% of the market (Gordon, 1992). Its main functions in confectionery are the prevention of sugar crystallization and as an emulsifier in fat-based sweets, e.g. toffees. The gum acts as an emulsifier by keeping the fat evenly distributed throughout the toffee there by preventing the fat from "leaching out' and forming an oxidizable film on the surface (Meer, 1980).

The gum is also used to incorporate flavours in confectionery such as pastilles and gum drops and in the preparation of lozenges. Gum arabic has also been used to stabilize frozen dairy products such as ice cream and sherbets due to its high water- absorbing properties. In this context the gum imparts a smooth texture to the frozen product by inhibiting the formation of ice crystals (Glicksman and Schachat, 1959).

In baking industry, gum *arabic* is used in glazes and topping, and in the encapsulation of spray-dried flavours into foods. In another application when used as flavour fixative, the gum forms a thin and impenetrable film around the flavor particle protecting it from oxidation, evaporation and absorption of moisture. In the soft drinks industry, gum is used as a flavour emulsifier for oil-in water emulsions and as a foam-stabilizing agent (Glicksman, 1969).

2.4 Physicochemical Properties of Plant Gums

The biological functions of natural biopolymers from plant sources depend on their chemical composition and molecular structure. In addition, the extraction and processing

conditions can significantly influence the chemical and molecular structure of plant biopolymer(Amid *et al*., 2012). In view of this Amid *et al*., (2012) conducted a study on *Durio zibethinus* seed gum to characterize its chemical and molecular structure. Size-exclusion chromatography coupled to multi angle laser light-scattering (SEC-MALS) was applied to analyze the molecular weight (Mw), number average molecular weight (Mn), and polydispersity index (Mw/Mn). The results obtained revealed that monosaccharides present in the carbohydrate of durian seed gum were galactose (48.6-59.9%), glucose (37.1-45.1%), arabinose (0.58-3.41%), and xylose (0.3-3.21%). The predominant fatty acid of the lipid fraction from the gum were palmitic acid (C16:0), palmitoleic acid (C16:1), stearic acid (C18:0), oleic acid (C18:1), linoleic acid (C18:2), and linolenic acid (C18:2). The most abundant amino acids were: leucine (30.9-37.3%), lysine (6.04-8.36%), aspartic acid (6.10-7.19%), glycine (6.07-7.42%), alanine (5.24-6.14%), glutamic acid (5.57-7.09%), valine (4.5-5.50%), proline (3.87-4.81%), serine (4.39-5.18%), threonine (3.44-6.50%), isoleucine (3.30-4.07%), and phenylalanine (3.11-9.04%).

Yusuf, (2011) determined and compared the physicochemical properties of gum exudates from three Acacia tree species (*A senegal*, *A. sieberiana* and *A. nilotica*) in Batagarawa, Katsina State. Data generated from the study confirmed that there are a number of physicochemical differences between the gum exudates. Physicochemical properties were also found to vary among the three gum samples within the following ranges: moisture (13.40-16.20%); water solubility at 300°C (38-45%); pH of 25% solution (4.50-5.00); relative density of 20% solution (300°C) (1.23-1.32); melting temperature (289-320^{0}C); relative viscosity of 1% solution (20.18-24.80); total ash (3.30-3.54%); nitrogen (0.38-0.42%); protein (2.51-2.77%) and total soluble fibre (77.99-80.41%). Analysis of all the samples showed no tannin content. Determined cationic composition of the gum samples showed calcium, magnesium, iron, sodium and

potassium as the predominant minerals. Copper, nickel, cobalt, manganese, chromium, zinc and lead were not detected in the study. Despite the physicochemical differences among the samples studied, values of physicochemical parameters obtained compared well with those reported in previous studies on *Acacia* gums in many parts of the world.

Sufficient work has been carried out and reported on physicochemical properties of gum samples (moisture, ash, nitrogen, protein, specific rotation, relative viscosity, refractive index, equivalent weight, pH, uranic acid, reducing sugar and tannin content). For example, Ahmed *et al*., (2009), studied *Anogeissus leiocarpus* gum samples collected as natural exudates from 3 different locations in Sudan, namely Abojebiha (season 1994-1995), Elfula and Rosaries (season 1996-1997). Results obtained showed significant differences within each location in most parameters studied except in the refractive index value which was found to be constant in all samples (1.334). The effect of location on the properties of gum samples was also studied and the analysis of variance showed insignificant differences ($p<0.05$) in all properties studied except in ash content and this was attributed to the differences in soil types at the various locations. Mean values obtained were, 9.2% moisture, 3.4% ash, 0.72% nitrogen, 4.74% protein%, -35.5° specific rotation, 1.68 relative viscosity, 4.2 pH, 1.334 refractive index, 14.3% uronic acid, 0.44% reducing sugar 1336.0% equivalent weight and 0.68% tannin content. UV absorption spectra of gum samples were determined and the maximum absorption points were found to range between the wave length values of 243 and 285 nm. Cationic composition of gum samples was also determined and the results showed that magnesium had the highest value in all samples followed by iron, sodium, potassium, calcium, zinc and trace amount of manganese, copper, nickel, cadmium and lead. Functionality (water holding capacity and emulsifying stability of *Anogeissus leiocarpus* gum were studied. The water holding capacity value was found to be

65.5% and emulsifying stability value was found to be 1.008. Insignificant differences were observed between *Anogeissus leiocarpus* gum and *Acacia senegal* gum for the 2 parameters studied.

In another comparative research, Gyedu-Akoto *et al.* (2008) studied the physicochemical properties of cashew gum (CG) collected from four cashew growing districts, Sampa, Wenchi, Bole and Jirapa in Ghana to help promote the utilization of cashew gum in the food industry. The gum was collected from trees of two different age groups, those that were 10 years and below and those above 10 years. Physicochemical properties of CG were compared to those of gum Arabic. Parameters studied included pH (3.8 - 4.2), total ash (0.5 - 1.2%), protein content (1.27 - 1.80%), total sugars (0.96 - 2.10 mg/g), total phenols (0.21 - 2.26%), moisture content (9.8 - 13.2%) and insoluble matter (1.9 - 4.8%). Gum from mature trees was generally found to have higher levels of protein, moisture, sugars and phenols than that from young trees, with the exception of pH which was lower in gum from mature trees. There were also variations in some of the physicochemical properties of the CG from the different locations. The predominant minerals in cashew tree gum were Ca, K, Na and Fe. This study showed that CG possesses good physicochemical properties and high levels of minerals.

In order to establish some of the physicochemical properties of *Khaya senegalensis* gum grown abundantly in Zaria, Nigeria as an excipient in pharmaceutical formulations, Mahmud *et al.*, (2008) measured the physicochemical properties such as pH, water sorption, swelling capacity and viscosities at different temperatures using standard methods. Khaya gum was found to be colourless to reddish brown translucent tears. 5 % w/v mucilage had a pH of 4.2 at 28 °C. The gum was slightly soluble in water and practically insoluble in ethanol, acetone and chloroform. They also found that the gum swells to about 10 times its original weight in water.

Water sorption studies revealed that it absorbs water readily and is easily dehydrated in the presence of desiccants. A 5 %w/v mucilage concentration gave a viscosity value which was unaffected at temperature ranges (28 – 40 °C). At concentrations of 2 and 5 %w/v, the gum exhibited pseudo plastic flow pattern while at 10 %w/v concentration the flow behaviour was thixotropic. The results indicate that the swelling ability of *Khaya senegalensis* gum may provide potentials for its use as a disintegrant in tablet formulation, as a hydro gel in modified release dosage forms and the rheological flow properties may also provide potentials for its use as suspending and emulsifying agents owing to its pseudo plastic and thixotropic flow patterns. In addition, Khaya gum should be stored in air tight containers with desiccators.

Little research is currently underway on gums from African plants, yet Africa imports a lot of gums for pharmaceutical and food industries (Gundidza *et al*., 2011a). Nigeria produces four grades of *Acacia* species in commercial quantities which are not used for local industrial applications but ex ported for foreign earnings (Ademoh and Abdullahi, 2009).The grades 1 and 2 that are preferred by the exporting countries are used in the pharmaceutical, confectionary, food, textile and beverage industries. Nigeria as country uses imported materials for binding their synthetic casting sands due to non-development of locally available materials like *Acacia* species exudates. In their research, Ademoh and Abdullahi (2009) investigated the physical and chemical properties of Nigerian *Acacia* species determine its viability for binding sand.

Alakali *et al*., (2009) investigated the rheological characteristics (consistency and flow behaviour indices) of food gum (C*issus populnea)* exudates obtained from the fresh leaves and stem as well as dried leaves and stem were determined at 20, 30, 40, 50 and 60°C using a rotational viscometer at shear rates of 0.1, 0.2, 0.5, 1.0 and 1.5 rpm for effective design and simulation of its momentum transfer process and system. The experimental design used for the

study of consistency and flow behavior indices of *C. populnea* at 20 - 60°C was Randomized Complete Block Design (RCBD). The study revealed that consistency index (K) of C. *populnea* exudates generally increased with increase in temperature. The K values for the fresh were generally higher than the dried materials. The K values of the fresh stem exudates, which is more viscous, were significantly ($p < 0.05$) different from the less viscous fresh leaves exudates, but did not show significant ($p \geq 0.05$) difference with the dried leaves which became concentrated due to drying. The flow behaviour index did not show any defined trend with changes in temperature, and was not significantly ($p \geq 0.05$) different. The exudates generally exhibited pseudo plastic behaviour at all the temperatures studied. A study of the apparent viscosity of the exudates between temperatures of 20 – 150°C shows that apparent viscosity increased with increase in temperature below the boiling point of 70°C. However, above the boiling point, the apparent viscosity decreased with increase in temperature. The viscosity-temperature data were fitted in Arrhenius-type equation and yielded activation energies between 0.997 to 2.431 kJ/mol°C, corresponding to temperature range between 20-70°C and 1.632 to 2.580kJ/mol with rate constant corresponding to temperature range between 70-150°C.

2.5 Measurement of Viscosity

Viscosity is a measure of the resistance of flow due to internal friction when one layer of fluid is caused to move in relationship to another layer (Acevedo *et al.*, 1990). The Poise represents absolute viscosity, the tangential force per unit area of either of two horizontal planes at unit distance apart, the space between being filled with the substance. A liquid with an absolute viscosity of one Poise requires a force of one dyne to maintain a velocity differential of one centimeter per second over a surface one centimeter square. When the ratio of shearing stress

to the rate of shear is constant, as is the case with water and thin motor oils, the fluid is called a Newtonian fluid. In the case of non-Newtonian fluids, the ratio varies with the shearing stress and viscosities of such fluids are called apparent viscosities.

2.5.1 Dilute Solution Viscosity

The viscosity of a dilute solution of a polymer, measured under prescribed conditions, is an indication of the molecular weight of the polymer and can be used to calculate the degree of polymerization. Dilute solution viscosity includes; relative viscosity, specific viscosity and reduced viscosity.

2.6 Studies on GC-MS, FTIR and SEM of Gums

Bamiro *et al.,* (2010) evaluated the physicochemical properties of *Terminalia randii* gum (Family Combretaceae) as binding agent in carvedilol tablet formulations in comparison with standard binders - polyvinylpyrrolidone (PVP) and corn starch by scanning electron microscopy (SEM), fourier transform infrared spectroscopy (FTIR), X-ray powder diffraction (XRPD), particle size analysis, pH and viscosity determinations. The mechanical properties of the tablets were assessed using the crushing strength and friability tests while drug release properties were assessed using the disintegration and dissolution times. The crushing strength of the tablets increased while the friability decreased with increase in the concentration of the binding agents. The ranking of the crushing strength of the tablet was formulations containing PVP > Terminalia gum > corn starch while the ranking for friability was the reverse.

Nep and Conway (2010) used spectroscopic techniques including x-ray photoelectron spectroscopy (XPS), Fourier-transformed infrared (FT-IR), solid state nuclear magnetic resonance (NMR), ^{1}H and ^{13}C NMR techniques to characterize grewia gum that was extracted from the inner stem of *Grewia mollis* plant. They characterized the gum by several techniques

such as gas chromatography (GC), gel permeation chromatography (GPC), scanning electron microscopy (SEM), differential scanning calorimetry (DSC) and thermogravimetric analysis of the extracted sample. The result obtained showed that *Grewia* gum is a typical amorphous polysaccharide gum containing glucose, rhamnose, galactose, arabinose and xylose as neutral sugars. The measured average molecular weight of the gum was 5925kDa expressed as pullulan equivalent. The gum was found to be thermally stable and had potentials for application as stabilizer or suspending agent in foods, cosmetics and in pharmaceuticals.

A multi-scale characterization of the fluidized-dried gum extracted from the fresh fruits of the plant *Abelmoschus esculentus* was done by Emeje *et al*. (2009). They described the physical, thermal, sorptional and functional properties of this natural gum. Elemental analysis, scanning electron microscopy (SEM), particle size analysis, X-ray powder diffraction (XPRD), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), Fourier transmittance infra-red (FT-IR), and nuclear magnetic resonance (NMR) spectroscopy were used to characterize the gum sample. *Abelmoschus esculentus*gum (AEG) had a glass transition temperature (T_g) of 70°C and no melting peak. It showed a 14.91% loss in weight at 195°C. X-ray diffractogram showed numerous broad halos for AEG. Elemental analysis showed that AEG contains 39.5, 7.3, 51.8 and 1.4% carbon, hydrogen, oxygen and nitrogen respectively. The results obtained in their study established the fundamental characteristics of AEG and suggested its potential application in the food, cosmetic and pharmaceutical sectors.

Indian forests are reported to be a major source of large number of non-wood forest products. One such product is an exudate tree gum, regionally called gum Kondagogu (*Cochlospermum gossypium* DC.), belonging to the family Bixaceae. This gum is collected by different tribes in the state of Andhra Pradesh and marketed by Girijan Cooperative

Society, Andhra Pradesh, India. Experimental work carried out by Vinod and Sashidhar (2010) on this gum has resulted in assigning a separate identity to this gum as compared to the well-established and commercially exploited gum Karaya. Gum kondagogu has unique physiochemical properties as compared to other tree gums. Proximate analysis of the gum revealed that it's highly acidic and with high water binding capacity. Elemental composition of gum kondagogu was determined by Energy dispersive X-ray fluorescence spectrometry (EDXRF). Surface morphological studies based on SEM analysis showed irregular shape with sharp edges in the native gum, while the deacetylated gum showed a fibrilar and porous structure. Atomic force microscope (AFM) analysis indicated that native gum was visualized as spherical lumps, suggesting an inter- or intra-molecular aggregation. Transmission electron microscope (TEM) image of native gum kondagogu showed that the polymer was an extending linear chain with branch points. FT-IR spectrum of native gum indicated the predominant presence of acetyl group (12%w/w). Analytical data on gum kondagogu indicated that the major neutral sugars were arabinose, mannose, α-D-glucose, β-D-glucose, rhamnose and galactose, whereas uronic acids (D-Glucuronic acid, β-D-galacturonic acid and α-D-galacturonic acid) were the major acidic sugars. Structural assignment was carried out using acid hydrolysis, Smith degradation and NMR studies [^{1}H, ^{13}C, 2-D NMR (TOCSY and NOESY)]. Smith degradation analysis indicated that the back bone structure of gum kondagogu was that of α-D-Gal*p*A-(1→4)-α-L-Rhap and can be grouped under rhamnogalacturonan type of gum. The experimental work provided by this work give an insight into the possibility of using this natural biopolymer in food, textile and pharmaceutical industry.

2.7 Studies on Rheology of Gums

The flow behaviour of six species of Iranian gum tragacanth dispersions had been investigated at different temperatures and ionic strengths, within a concentration range (0.05-1.5 %w/w) using a controlled shear rate rheometer. Simas-Tosin *et al*., (2009) showed via the steady shear measurements that the dispersions of the gum had shear-thinning natures. The power law model was used to describe the rheological properties of dispersions and Arrhenius model was used to evaluate the temperature effect. Composition analysis, surface tension measurement, particular size analysis, and colour measurement of all the species were also carried out. The results indicated that six species of gum tragacanth studied exhibited significantly different physicochemical properties. Therefore, various species of gum tragacanth can be used instead of different hydrocolloids in a wide range of applications (Simas-Tosin *et al.,* 2009).

Zakaria *et al.,* (1997) carried out an investigation on *Anacardium occidentale* using potentiometric titration and also determined the common metal contents in the gum. They discovered that the solubility and viscosity differences of the fractions may be attributable to the difference in the degree of acylation and the content of divalent cations present in the fractions. The fractions with higher degree of acylation form a gel but when partially deacylated disperse in water to form stable gel dispersion. The higher viscosity exhibited by the soluble fraction was attributable to the higher content of divalent cations.

In order to assess its application to industry, *Commiphora Africana* gum resin was isolated and subjected to rheological, moisture and ash content studies (Gundidza *et al*., 2011). For rheological studies, a rotational viscometer which had the ability to characterize both Newtonian and non-Newtonian systems was used. The gum resins from C. *africana* exhibited low shear stress even at high concentration of the gum resins. The change in shear stress

with temperatures produced almost a linear graph with a gradient of 0.06. In addition, the gum resin from this plant species was affected by the addition of salts and would have little application in formulations that contain salts. The moisture content obtained was 10.6 ±0.04.The low level of moisture in this gum resin appeared to be desirable since it will attract little bacterial or fungal growth in the formulation. The ash content was 3.64±0.01. The findings of this study has demonstrated that this gum has potential as a product for the cosmetic, pharmaceutical and food industries provided further studies are carried out to identify the phyto-constituents in the gum as well as toxicity studies.

In search for a cheap and effective natural excipient that can be used as an effective alternative for the formulation of pharmaceutical suspensions the properties of *Albizia zygia* gum (family Mimosoideae) were evaluated comparatively with those of *Tragacanth*, *acacia* and *gelatin* at concentration range of 0.5 – 4.0%w/v in sulphadimidine suspension (Femi-Oyewo *et al*., 2003). Characterization tests were carried out on purified *Albizia zygia* gum. Sedimentation volume, rheology and particle size analysis were employed as evaluation parameters. The values obtained there were used as basis for comparison of the suspending agents studied. The results obtained revealed that *Albizia zygia* gum is devoid of alkaloids, anthraquinones and carbohydrates which ensure its "inertness". *Albizia zygia* gum (2.5%w/v) produced a comparable suspending ability as 4%w/v Compound *Tragacanth*. Also, the suspending ability of all the materials was found to be in the order: *Albizia zygia*> Compound *Tragacanth* gum > Acacia gum > Gelatin. At all concentrations employed, *Albizia zygia* gum had the strongest suspending ability relative to the other materials. The results suggest that, due to the high viscosity of *Albizia zygia* gum, its mucilage can be a stabilizer of choice when high viscosity is desired. It can also serve as a good thickening agent in both

pharmaceutical and food industries (Femi-Oyewo *et al.,* 2003)

Ferula galbaniflua is one of the natural plants of Iran. Its exudates, Barijeh gum, can be used in food industry as Milani *et al*., (2007) explored the properties of a new gum extracted from tubers of *Ferula galbaniflua*. The composition of the gum was analyzed using HPLC. The effects of temperature (50, 70, 90 ^{0}C) and pH (4, 7, 10) on the extraction yield, viscosity and on the purity of extracted gum were investigated. It was found extraction yield in acidic pH was highest and rising temperature leal to more yield. By the other way, increasing temperature and reducing pH reduced the protein content in extracted gum, also increasing the temperature of the extraction medium leal to a decrease in viscosity of gums suspension. Also, alkaline extraction yield caused less viscosity of gum suspension.

Hesnandez-Tinoco *et al.,* (2004) studied the rheological and micro-structural characteristics of curds added with mesquite seed gum (MESG) and soy protein (SP). Two types of curds were prepared either with 100% raw commercial milk or with a 50% low-fat milk and the effect of the addition of 0.15% (w/w) and 0.45% (w/w) of MESG and/or 0.3% (w/w) and 0.6% (w/w) soy protein content was analyzed.

Dressler *et al.,* (2008) investigated the rheological properties of aqueous guar gum solutions at different concentrations and temperatures in a cone-and-plate rheometer in the linear and non-linear viscoelastic regimes. In the linear viscoelastic regime a low amplitude oscillatory shear experiments was performed to measure the complex modulus. They adopted the time-temperature and the time-concentration superposition principle to increase the window of experimentally accessible frequencies. The time-concentration superposition principle allowed for a frequency shift of approximately one decade towards the high frequency regime. Preliminary modeling efforts in the framework of the theory of linear viscoelasticity have been

undertaken to give a theoretical description of the measured data. In the non-linear viscoelastic regime they found for low shear rates a monotonous increase of the transient shear viscosity, whereas at higher shear rates a pronounced overshoot in the transient rheological properties was detected.

In another research conducted by Song *et al*. (2006) using a strain-controlled rheometer, the dynamic viscoelastic properties of aqueous xanthan gum solutions with different concentrations were measured over a wide range of amplitudes. The linear viscoelastic behaviour in small amplitude oscillatory shear flow fields was investigated over a broad range of angular frequencies. In their article, both the strain amplitude and concentration dependences of dynamic viscoelastic behaviour were reported as obtained from the experimental data from strain-sweep tests. In addition, the linear viscoelastic behaviour was explained in detail and the effect of angular frequency and concentration on this behaviour were discussed using well-known power-law type equations. Finally, a fractional derivative model originally developed by Barbosa *et al,* (2005) was employed to make a quantitative description of linear viscoelastic behaviour and the applicability of this model was examined with a brief comment on its limitations.

2.7.1 Effect of Electrolyte on Gums Solution

Available literature revealed that very few studies have ever been published on the adsorption of gums from high ionic strength brines (Ma and Pawlik, 2006b; Pawlik and Laskowski, 2006) and the solution chemistry of the polymer in concentrated electrolytes is still poorly understood. However, Mao and Chen (2006) studied the effect of lithium, sodium, potassium, and cesium chlorides on the properties of dilute guar gum solutions through viscosity measurements. The results they obtained showed that the intrinsic viscosity of guar gum was not significantly affected by the salts up to an electrolyte concentration of 4.1moldm^{-3}. In the same

ionic strength range, however, clear differences were observed between individual salts in their ability to influence the Huggins constant. At even higher electrolyte concentrations, only lithium and sodium chlorides markedly increased the intrinsic viscosity of the polymer, pointing towards the formation of a new type of structure in those solutions. The observed trends were attributed to the chaotropic (K^+, Cs^+) or kosmotropic (Li^+, Na^+) properties of the background salts. It was suggested that chaotropic electrolytes were capable of enhancing the dissolution of colloidal guar gum aggregates normally present in poly-saccharide solutions under ambient conditions. On the other hand, the presence of high concentrations of kosmotropic ions led to enhanced aggregation of guar gum due to competitive hydration for free water molecules between the polysaccharide chains and the strongly hydrated cations. Comparative tests with the use of urea showed that the solute affected the behavior of dilute guar gum solutions in a way similar to NaCl, so its dispersing/hydrogen bond-breaking capabilities towards the polymer were not very clear in the studied system. Ma and pawlik (2005 and 2006a) also found that the adsorption of polysaccharide on KCl and NaCl solutions proceeds divergently, and attributed the effects to the chaotropic and kosmotropic properties of $K^+$and Na^+cations, respectively.

Khouryieh *et al*., (2007) used an oscillating capillary rheometer to investigate the dynamic viscoelastic and intrinsic viscosity properties of deacetylated xanthan (0.025%), native xanthan (0.025%), guar gum (0.075%), and xanthan–guar mixtures in dilute solutions. Influence of ionic strength on xanthan conformation and interaction with guar gum was elaborated. As the salt concentration increased, significant ($p < 0.05$) decrease in viscosity and elasticity values were observed for both native xanthan–guar mixtures and deacetylated xanthan–guar mixtures. In water and 2mM NaCl solution, the relative viscosity and elasticity of both native xanthan–guar mixtures and deacetylated xanthan-guar mixtures were much higher than those calculated for

mixtures assuming no interaction, whereas no pronounced increase were found for polysaccharide mixtures in 40mM NaCl. The intrinsic viscosities of deacetylated xanthan–guar mixtures in water and 2mM NaCl were higher, whereas the intrinsic viscosities of native xanthan–guar mixtures were lower than those calculated from the weight averages of the two individually, assuming no interaction. These results demonstrated that intermolecular interaction has occurred between xanthan and guar mixtures in water and 2mM NaCl, but may not occur in 40mM NaCl, and mutual incompatibility may occur (Khouryieh *et al.*, 2007). The authors suggested that the degree of disordering of xanthan played a critical role in xanthan–guar interaction and may explain the differences in viscosity, elasticity and intrinsic viscosity measurements between 2 and 40 mM NaCl.

In aqueous solutions, the structure of xanthan gum has been found to under goes a thermally induced transition from an ordered (helical) to a disordered conformation. This conformational transition was also found to depend on ionic strength, nature of electrolyte, pH, and acetyl and pyruvate contents (Holzwarth, 1976; Morris *et al.,* 1977; Baradossi and Brant, 1982; Paoletti *et al*., 1983; Norton *et al*., 1984). At high temperature and low ionic strength, xanthan existed in solutions as a disordered structure, but reduction in temperature and/or addition of salts induces an ordered structure (Norton *et al*., 1984). In distilled water at 298 K, the backbone of xanthan is disordered (or partly ordered in the form of a randomly broken helix) but highly extended due to the electrostatic repulsions from the charged groups on the side chains (Rochefort and Middleman, 1987). When salt was added to the solution at 298 K, a disordered transition occur in which the backbone take on a helical conformation, and the charged trisaccharide side chains collapse down onto the backbone (due to charge screening effects) and stabilize the ordered conformation (Muller *et al*., 1986).

In an effort to investigate the effect of salts on the rheology of xanthan and *locust bean* gums, Higiro *et al*., (2007) also found that high temperatures favour the ‘‘disordered’’ transition, whereas high ionic strength favors the ‘‘ordered’’ transition. Their findings supported those reported elsewhere (Morris *et al.,* 1995). Lopes *et al.,* (1992) studied the interaction of xanthan and guar gum at low temperature in water and 20 mM NaCl by using viscosity methods. The authors also noticed a small synergistic effect between the two gums in 20 mM NaCl; the effect became more pronounced in water. They concluded that xanthan adopted a disordered conformation in water, whereas the conformation was in an ordered form in 20 mM NaCl. These findings were supported by Dalbe (1992). They used small deformation oscillation methods to study xanthan–glucomannan mixture and reported that addition of 85.55 mM NaCl or 67.07 mM KCl to gum mixtures led to a dramatic reduction in gel strength, which was not altered by further addition of electrolytes. Similar results were reported by Wang *et al*. (2002).

2.8 Spectrophotometry and Spectroscopy

2.8.1 Absorption Spectrophotometry

Is the measurement of the selective absorption by atoms, molecules, or ions of electromagnetic radiations having a definite and narrow wavelength range of a monochromatic light. Absorption spectrophotometry encompasses the following wave-length regions: Ultra-violet (185 nm to 380 nm), visible (380 nm to 780 nm), near infra-red (780 nm to 3,000 nm) and infrared (2,500 nm to 40,000).

2.8.2 Ultra-Violet (UV) Spectroscopy

Ultra-violet is not used primarily to show the presence of individual functional groups, but rather to show the relationship between functional groups chiefly conjugation either between carbon-carbon or carbon-oxygen double bond, between double bonds in aromatic ring. It can in

addition reveal the number and location of substituents attached to the carbons of the conjugated systems (Morrison and Boyd, 1978).

2.8.3 Atomic Absorption Spectroscopy

Atomic absorption spectroscopy is the measurement of the radiation absorbed by the unexcited atoms of the chemical substance that has been aspirated into a flame or in the absence of a flame directly into the path of radiation. Although nuclear magnetic resonance (NMR) has lately been used with great success in the differentiation among the fine structures of very similar polysaccharide molecules e.g. *Acacia Senegal* gum and *Acacia seyal* gum, resembling different "finger prints" (Defaye and Wong, 1986).

2.8.4 Scanning Electron Microscope

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals derived from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1cm to 5microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD). The design and function of the SEM is very similar to the EPMA and considerable overlap in capabilities exists between the two instruments (Goldstein, 2003).

Accelerated electrons in an SEM carry significant amounts of kinetic energy and this energy is dissipated as a variety of signals produced by electron-sample interactions when the incident electrons are decelerated in the solid sample. These signals include secondary electrons (that produce SEM images), backscattered electrons (BSE), diffracted backscattered electrons (EBSD that are used to determine crystal structures and orientations of minerals), photons (characteristic X-rays that are used for elemental analysis and continuum X-rays), visible light (cathodoluminescence–CL), and heat. Secondary electrons and backscattered electrons are commonly used for imaging samples: secondary electrons are most valuable for showing morphology and topography on samples and backscattered electrons are most valuable for illustrating contrasts in composition in multiphase samples (i.e. for rapid phase discrimination). X-ray generation is produced by inelastic collisions of the incident electrons with electrons in discrete orbitals (shells) of atoms in the sample. As the excited electrons return to lower energy states, they yield X-rays that are of a fixed wavelength (that is related to the difference in energy levels of electrons in different shells for a given element). Thus, characteristic X-rays are produced for each element in a mineral that is "excited" by the electron beam. SEM analysis is considered to be "non-destructive"; that is, x-rays generated by electron interactions do not lead to volume loss of the sample, so it is possible to analyze the same material repeatedly (Egerton, 2005).

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

Samples of crude *Irvingia gabonensis* gum were obtained as dried exudates from their parent trees grown at Zaria city, in Zaria Local Government, Kaduna State, Nigeria. The sample was collected early November 2013 during the day time. The plant material had earlier been identified and authenticated in the Herbarium section of Department of Biological Sciences Ahmadu Bello University, Zaria, Nigeria.

The crude *Celtis zenkeri* gum samples was obtained as dried exudate from the parent trees grown in Gagarawa village in Gagarawa Local Government Area, Jigawa State, Nigeria. The crude sample was collected late November 2013. The gums along with its parent plant were also identified at the Herbarium section of Department of Biological Sciences Ahmadu Bello University, Zaria, Nigeria.

3.2 Tapping of Gums

The gums were collected from the plant species by tapping (Smith and Montgomery, 1959) for *Irvingia gabonensis* (IG) gum and for *Celtis zenkeri* (CZ) gum all during the day time. A small axe was used to break the outer bark. Tapping was carried out by inserting an axe underneath the bark which was then pulled back until the bark broke horizontally to give two broken ends. The cut was made about 4.0 cm wide. The bark was then carefully peeled along the length of the wounded trunk. Gum droplets formed were 0.5-1.5 cm in diameter for IG gum and 2.0–3.0 cm for CZ gum. They were dried and hardened on exposure to atmosphere.

3.3 Purification of the Gum

The procedure adapted for the purification of the gum was that of Femi – Oyewo *et al.,* (2003) but with some modifications. The gums were dried in an oven (BS Size 3, Gallenkamp) at 40^{o}C for 2 hours and latter the size reduced using a blender (Model. MJ-176 NR, Matsushita Electric Industrial Co., Ltd. Osaka, Japan). It was hydrated in double strength chloroform (Sigma-Aldrich, Germany) water for five days with intermittent stirring to ensure complete dissolution of the gum and then strain through a 75μm sieve to obtain particulate free slurry which was allowed to sediment. Thereafter, the gum was precipitated from the slurry using absolute ethanol (Sigma-Aldrich, Germany) filtered and dried flakes formed as precipitate. The precipitate latter dried in the oven at 40°C for 48 hours. The dried flake was then pulverized using a blender and stored in an air tight container.

3.4 Physicochemical Analysis

3.4.1 Determination of pH and Conductivity

The pH and conductivity were determined according to the method of ASTM D3838-80 with slight modification as follows; 1.0g of gum sample was weighed and transferred into 250 ml beaker and 100 ml of distilled water was added and stirred for 1 hour. Samples were allowed to stabilize and the pH was measured using an electronic pH/Conductivity meter (Jenway 430 model). Electrical conductivity (EC) of the gums was latter measured using same samples (Toles *et al*., 1998).

3.4.2 Determination of Solubility in Various Solvents

The solubilities of the gums were determined in cold, hot distilled water and ethanol. One gram (1.0g) of the gum sample was added to 50 ml of each of the above mentioned solvents and left overnight. 25 ml of the clear supernatants were taken in small pre-weighted evaporating

dishes and heated to dryness over a digital thermostatic water bath. The weights of the residue with reference to the volume of the solutions were determined using a digital top loading balance (Model.XP-3000) and expressed as the percentage solubility of the gums in the solvents (Carter 2005).

3.4.3 Density

Density measurements were carried out at 35^0C using 25cm^3 density bottles. For each gum sample, densities of the samples in aqueous solution were determined. Clean, dry density bottle was weighed (M_0) on a digital top loading balance. The bottle was then filled with distilled water and reweighed again (M_1). Another weighing (M_2) was carried out with the gum solution by replacing distilled water in the density bottle. Relative density of gum solution was evaluated as:

$$\frac{M2-M0}{M1-M0} \qquad (3.1)$$

3.4.4 Determination of the Viscosities of the Gum Mucilage

A Cannon Ubbelohde capillary viscometer (Cannon Instruments, model I-71) was used. The gum solution was prepared by dispersing 1.00g of the gum sample in 100 ml of distilled water at room temperature. Twelve milliter (12 ml) of the prepared sample was transferred into the viscometer. The viscometer was immersed in a thermostated viscometer bath, calibrated using a thermometer with a precision of 0.01 K, to equilibrate for 10 minutes at 25^0C. After equilibration, the sample was pumped into the bulb and allowed to flow past the lower mark on the viscometer under the influence of gravitational force. The time of flow of the sample from

the upper through the lower mark was noted and recorded in seconds. Triplicate measurements were made and the average values reported. Serial dilution was performed *in situ* to obtain other concentrations (0.8, 0.6, 0.4 and 0.2%w/v). The relative viscosity was calculated using the equation 3.1

$$(\eta)_{\text{relative viscosity}} = \frac{T - T_o}{T_o} \qquad 3.2$$

Where T is the flow time of the gum mucilage in seconds and T_o is the flow time of solvent (water) in seconds.

$[\eta]$ is defined by the following relationships:

Relative viscosity: $(\eta_{rel}) = \frac{\eta_{solution}}{\eta_{solvent}}$ (3.3)

Specific viscosity: $(\eta_{sp}) = \eta_{rel} - 1$ (3.4)

Intrinsic/reduce viscosity: $(\eta_{red}) = \frac{\eta_{sp}}{C}$ (3.5)

3.5 Effects of Temperature, Concentration and Salt Changes on Viscosities of Gums

The viscometer was used as described above. Concentrations of each of the gums (1% w/v) were prepared and their viscosities at temperature range 30 - 70^0C were determined. Also another 1 %w/v concentration of each gums at different concentrations of KCl, $AlCl_3$, KBr i.e,

0.2, 0.4, 0.6, 0.8 and 1.0M of the salt solutions was prepared. Viscosity values of the pure gums at different concentrations of the salts were measured.

3.6 Heavy Metals Analysis

Concentrations of manganese, nickel, copper, magnesium, calcium, iron, lead, cadmium and zinc were determined using a UNICAMM 969 atomic absorption spectrophotometer. Calibration curves for each element were prepared and the concentrations of the elements were extrapolated from their respective plots.

3.7 SEM Analysis

Scanning electron microscope was used for the analysis of the surface morphology of each of the gum. Secondly the SEM machine was used for the determination of elements present at different point of the micrograph and finally the fibre and pores were measured, fibre and pores histogram were extrapolated. Area and diameter of each fibre were also analyzed.

3.7.1 Sample Preparation and Analysis

The sample housing was cleaned with a damp wipe, and a SEM substrate (stub) was prepared for receiving a filter by applying a double-sided, sticky conductive carbon pad to the stub surface. The underside of the stub was labeled with the sample number using a permanent marker. The filter was removed and placed on the stub, and the stub was placed in a vacuum evaporator for carbon coating. A thin layer of carbon was evaporated onto the surface of the sample at a vacuum of 5.0×10^{-5} torr. The filter was then removed and placed in a clean polycarbonate storage box for transfer. Examination in the SEM involved the use of variable voltages due to the beam-sensitive nature of the Teflon filter. Morphological examination was conducted at 5 KV, and EDS spectrum collection was conducted at 15 KV. During the analysis

of the sample, randomly located areas were selected for higher magnification scanning. Specific areas were scanned for particulate matter. Upon locating a particle the size and morphology were recorded, and a spectrum is collected with the EDS. The procedure was repeated for a minimum of 100 particles encountered, regardless of size, morphology or chemistry. Representative micrographs and spectra were stored digitally and later transferred to project digital file systems.

The PM2.5 filters were examined by microscopy by first evaporating a thin layer of carbon onto the surface of the sample. The carbon-coated filters were stored for subsequent examination by SEM and EDS. Particles on the sample filter were found using SEM; upon locating a particle, its size and morphology were recorded. The spectrum of the particle was then obtained using EDS. The EDS spectrum was used to determine the elemental composition of the particle. This procedure was repeated for a minimum of 100 particles per filter.

3.8 FTIR Analysis

FTIR analyses of the gums were carried out using Schimadzu FTIR-8400S Fourier transform infrared spectrophotometer. The sample was prepared using KBr and the analysis was done by scanning the sample through a wave number range of 400 to 4000cm^{-1}.

3.9 GC-MS Analysis

Gas chromatography mass spectroscopy (GC-MS) analysis was carried out on a GC Clarus 500 Perkin Elmer system comprising an AOC-20i auto-sampler and gas chromatograph interfaced to a mass spectrometer (GC-MS) instrument employing the following conditions: column Elite-1 fused silica capillary column (30 x 0.25 mm ID x 1µM df, composed of 100% Dimethylvpoly diloxane), operating in electron impact mode at 70eV; helium (99.999%) was used as carrier gas at a constant flow of 1 ml/min and an injection volume of 0.5µI was employed (split ratio of 10:1) injector temperature 250ºC; ion-source temperature 280 ºC. The oven

temperature was programmed from 110 ºC (isothermal for 2 min), with an increase of 10 ºC/min, to 200ºC, then 5ºC/min to 280ºC, ending with a 9 min isothermal at 280ºC. Mass spectra were taken at 70 eV, at scan interval of 0.5 seconds and fragments from 40 to 450 Da. Total GC running time were 24min.

Interpretation on mass spectrum GC-MS was conducted using the database of National Institute Standard and Technology (NIST 2008) having more than 62,000 patterns. The spectrum of unknown component was compared with the spectrum of the known components stored in the NIST library. The name, molecular weight and structure of the components of the test materials were suggested. Concentrations of the identified compounds were determined through area and height normalization.

CHAPTER FOUR

4.0 RESULTS

4.1 Physicochemical Properties

The result of physicochemical parameters of *Irviginia gabonensis (IG), Celtis zenkeri* (CZ) and *Acacia senegal* (AS) gum exudates are shown in Plate 4.1a-b and Table 4.1. The AS gum and CZ gum were found with higher solubility values (AS = 9.8, ZC = 8.2 and IG = 6.6) in water compared to IG gum, while IG gum has the highest UV maximum absorption and density values.

4.2 Effects of Temperature, Concentration and Addition of Electrolytes on the Viscosity of the Gum Solutions

Effects of temperature on the viscosity of the gums are shown in Figures 4.1a-b. The viscosity values were found to decrease with increase in temperature. Increases in concentration of the gums were also found to increase the viscosity of the gums as shown in Figures 4.2a-b. Addition of electrolytes such as KCl, KBr and $AlCl_3$ were found to increase the viscosity of the gums solution as shown in Figures 4.3a-c.

4.3 Metals Concentration

The mean concentration of metals are shown in Table 4.2 manganase, iron, zinc, lead, magnesium, cadmium, calcium and nickel were found to be present in *Irviginia gabonensis*, *Celtis zenkeri* and AS with the exception of copper which was not detected in AS gum

4.4 Surface Morphology

The results for the surface morphology analysis are shown in Figures 4.4a-c and 4.5a-c for *Irviginia gabonensis* and *Celtis zenkeri* respectively. The Figures generally show the shapes and dimension of the particles in addition to the amorphous nature of the gums particles which are clearly indicated.





Plate 4.1a: Processed and Unprocessed IG Gum (*Irviginia gabonensis* gum exudates)



Plate 4.1b: Processed and Unprocessed CZ Gum (*Celtis zenkeri* gum exudates)

Table.4.1: Physicochemical parameters of *Irviginia gabonensis* (IG), *Celtis zenkeri* (CZ) and *Acacia senegal* gum (AS)

Physicochemical parameters	*Irvingia gabonensis* (IG) Gum	*Celtis zenkeri* (CZ) Gum	*Acacia senegal* (AS)Gum
Colour	Brownish	Brownish yellow	Yellow
Odour	odourless	odourless	odourless
Taste	tasteless	tasteless	tasteless
Solubility (%) @ 30°C , (a) water	6.6 ± 0.1	8.2 ± 0.1	9.8 ± 0.0
(b) ethanol	0	0	0
pH@27°C,	5.42	4.61	5.01
UV Max. Absorption (nm)	366	276	200
Turbidity (FAU)	79 ± 1	68 ± 0	25 ± 0
Conductivity (µS/cm)	123.6 ± 1.4	155.0 ± 1.0	185.0 ± 0.0
Salinity (psu)	0.2 ± 0.1	0.1 ± 0.0	0.1 ± 0.0
Density (g/cm^3)	1.36 ± 0.00	1.34 ± 0.00	1.35 ± 0.00
Total Dissolve Solid (mg/l)	92.0 ± 0.2	72.0 ± 0.1	87.0 ± 0.1

N=3

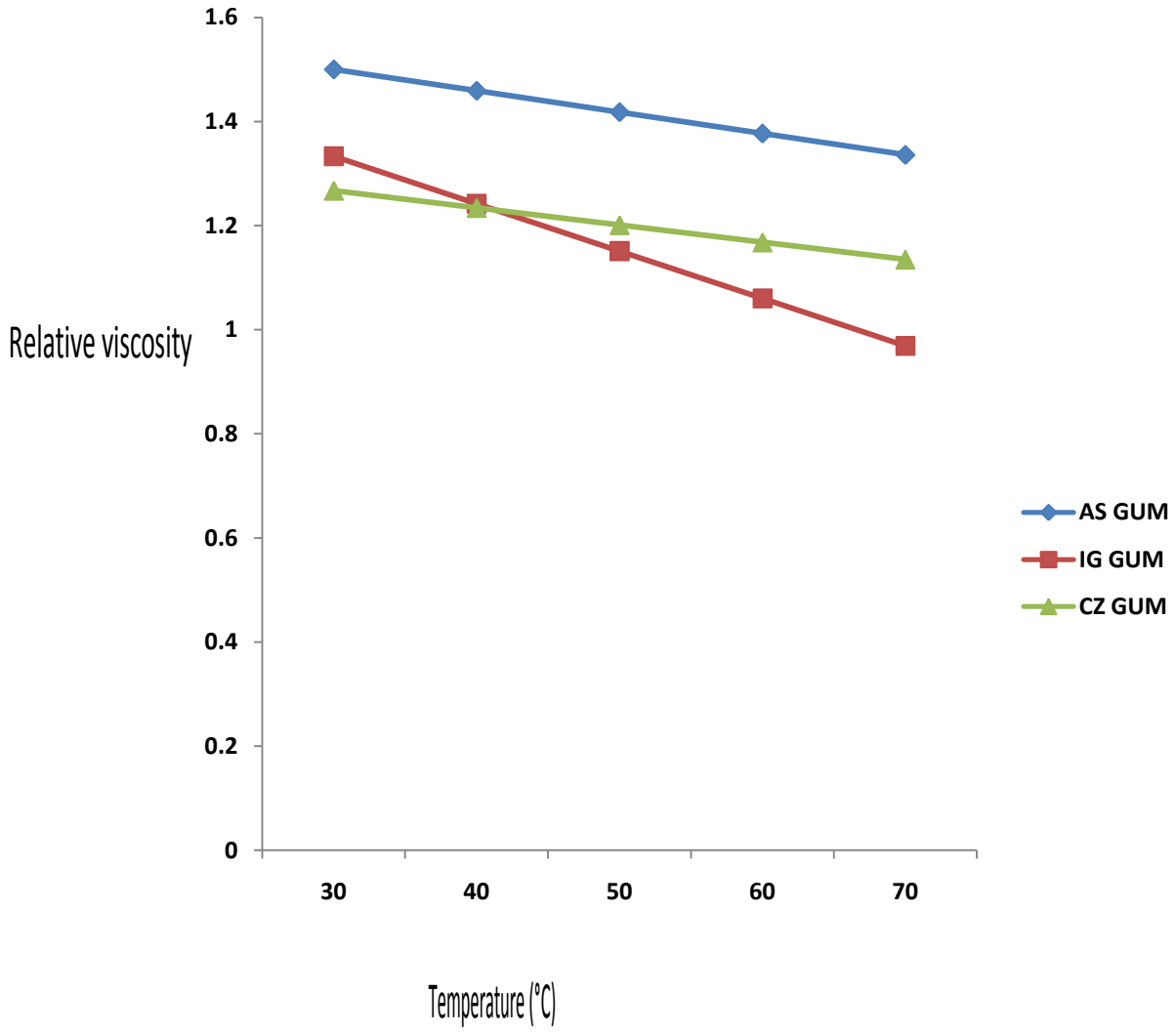


Figure 4.1a: Effect of Temperature on Relative Viscosity of the Gums

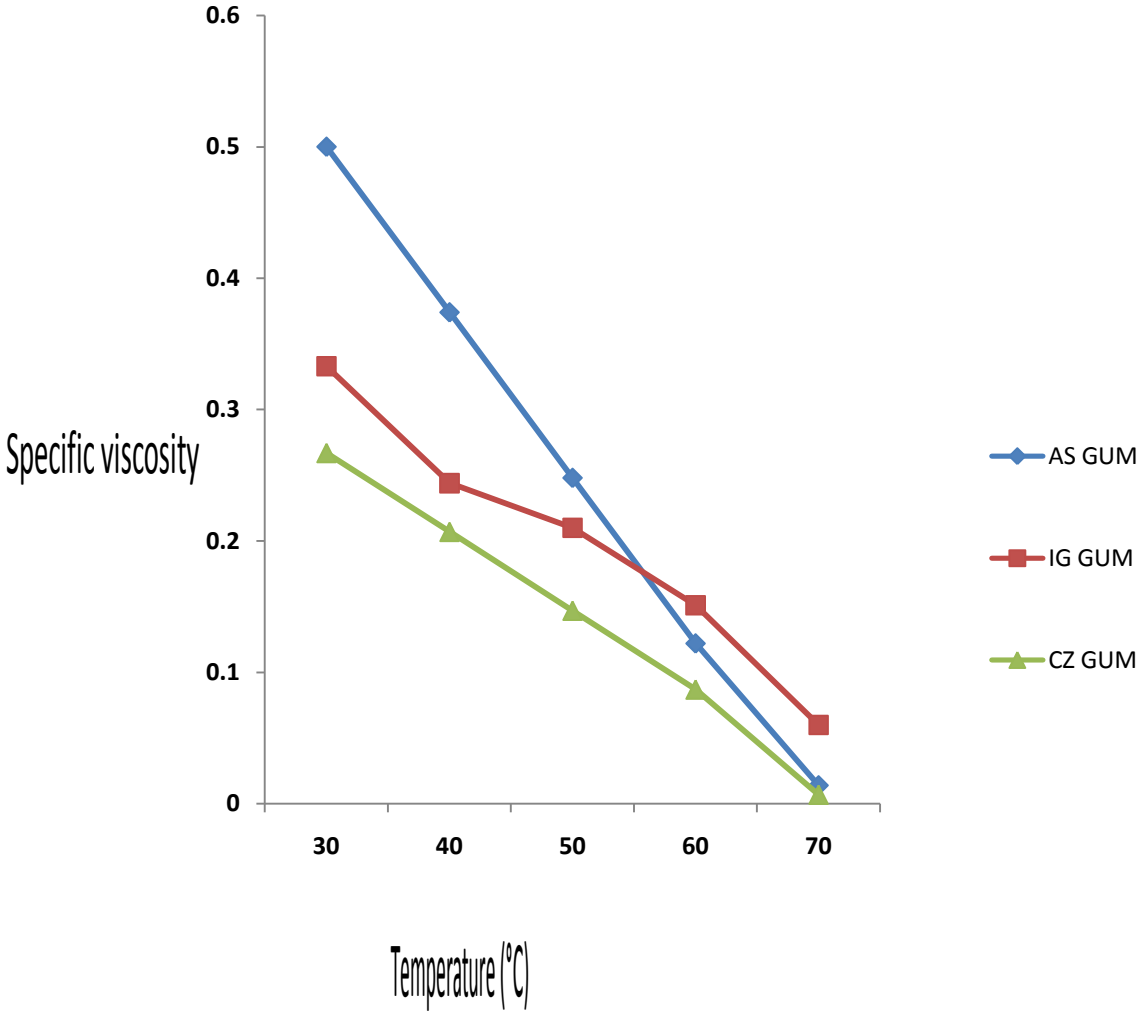


Figure 4.1b: Effect of Temperature on Specific Viscosity of the Gums

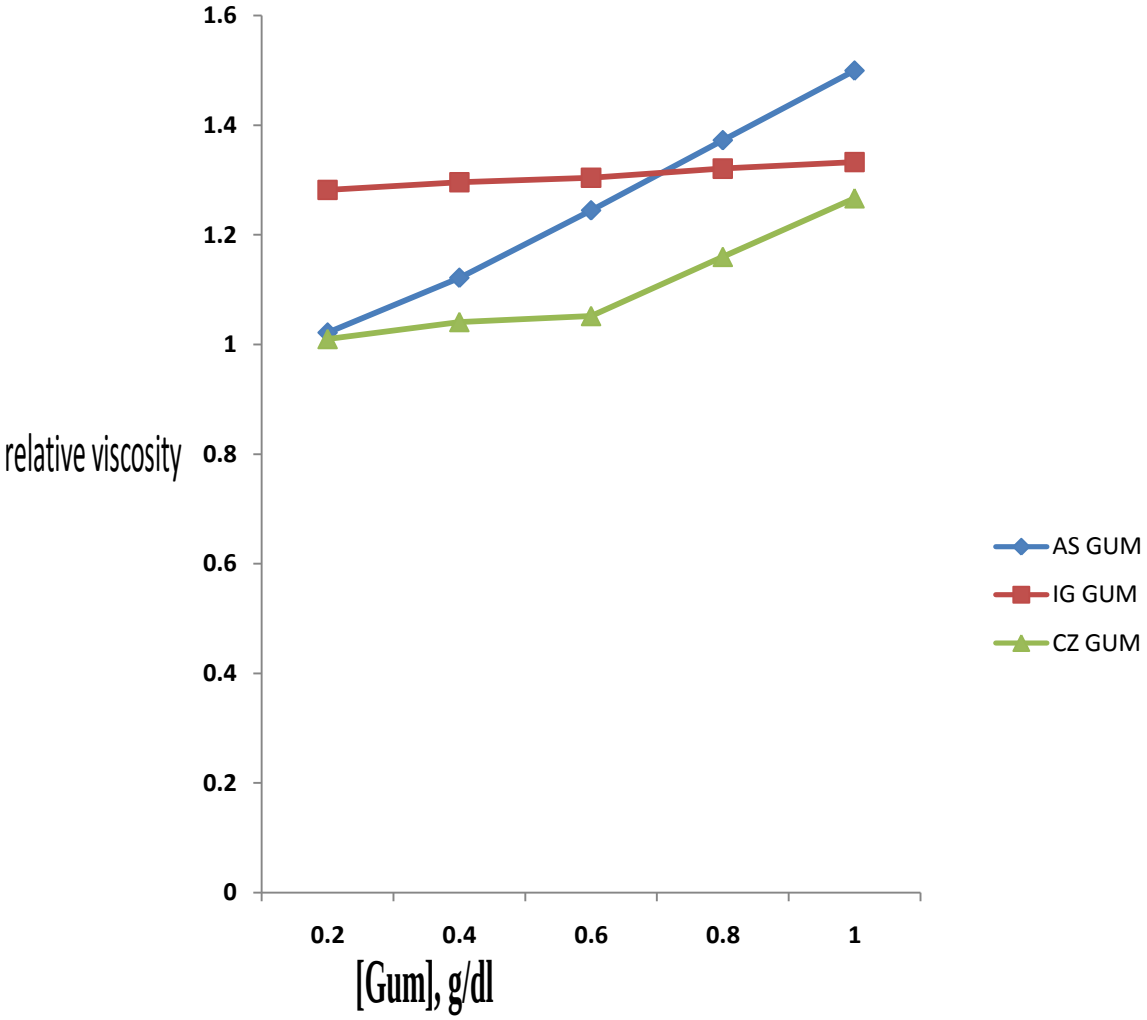


Figure 4.2a: Effect of Concentration on Relative Viscosity of the Gums

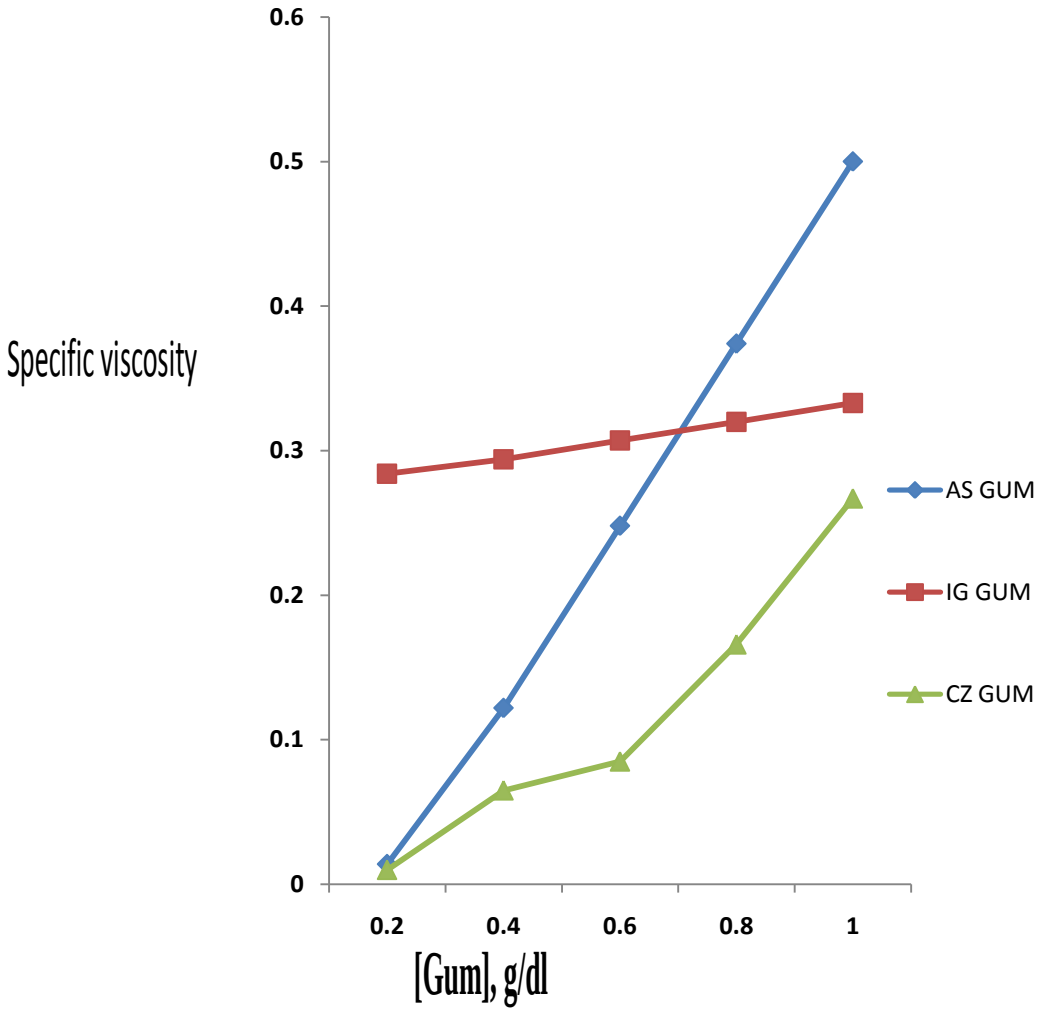


Figure 4.2b: Effect of Concentration on Specific Viscosity of the Gums

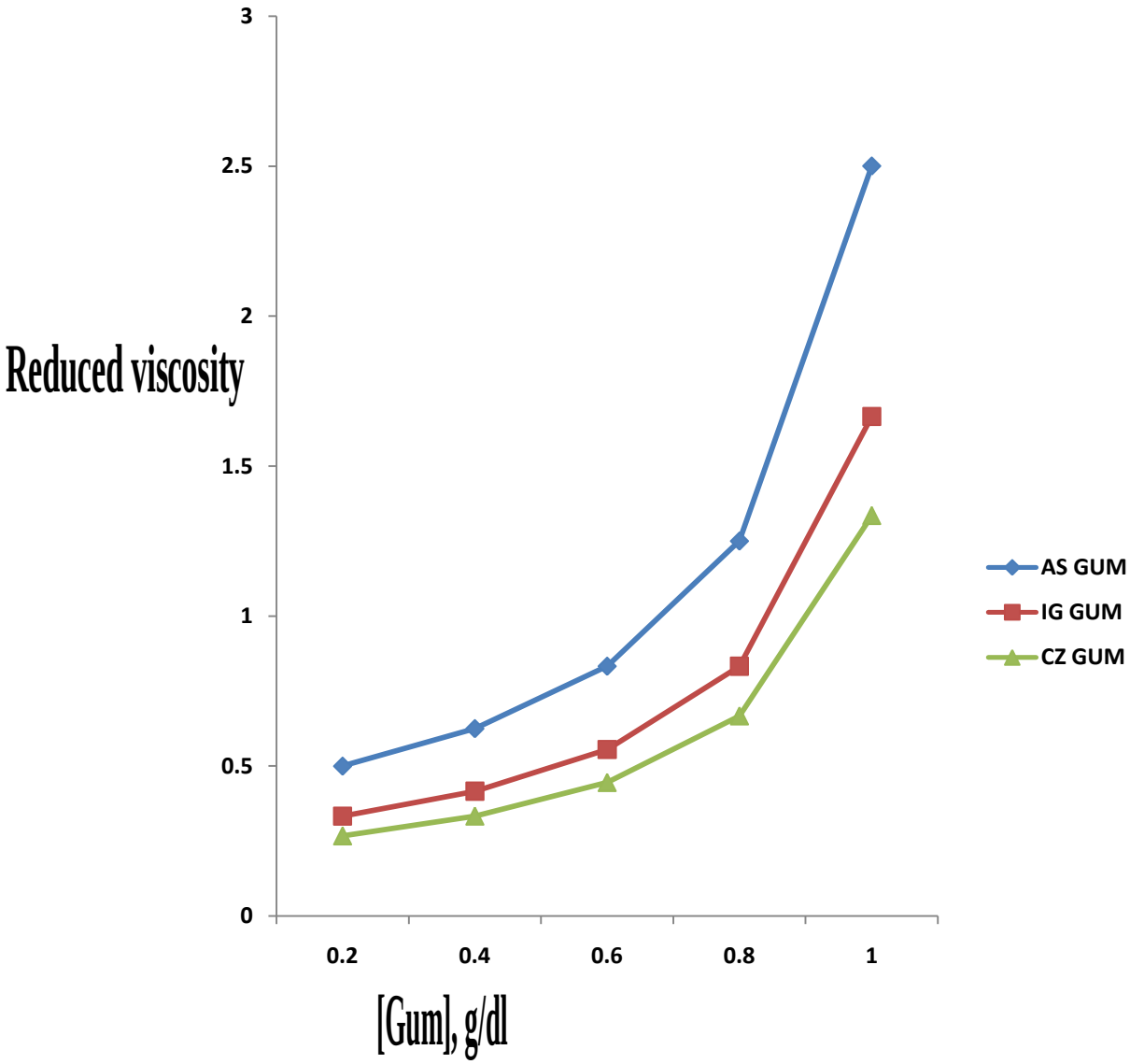


Figure 4.2c: Effects of Concentration on Reduced Viscosity of the Gums

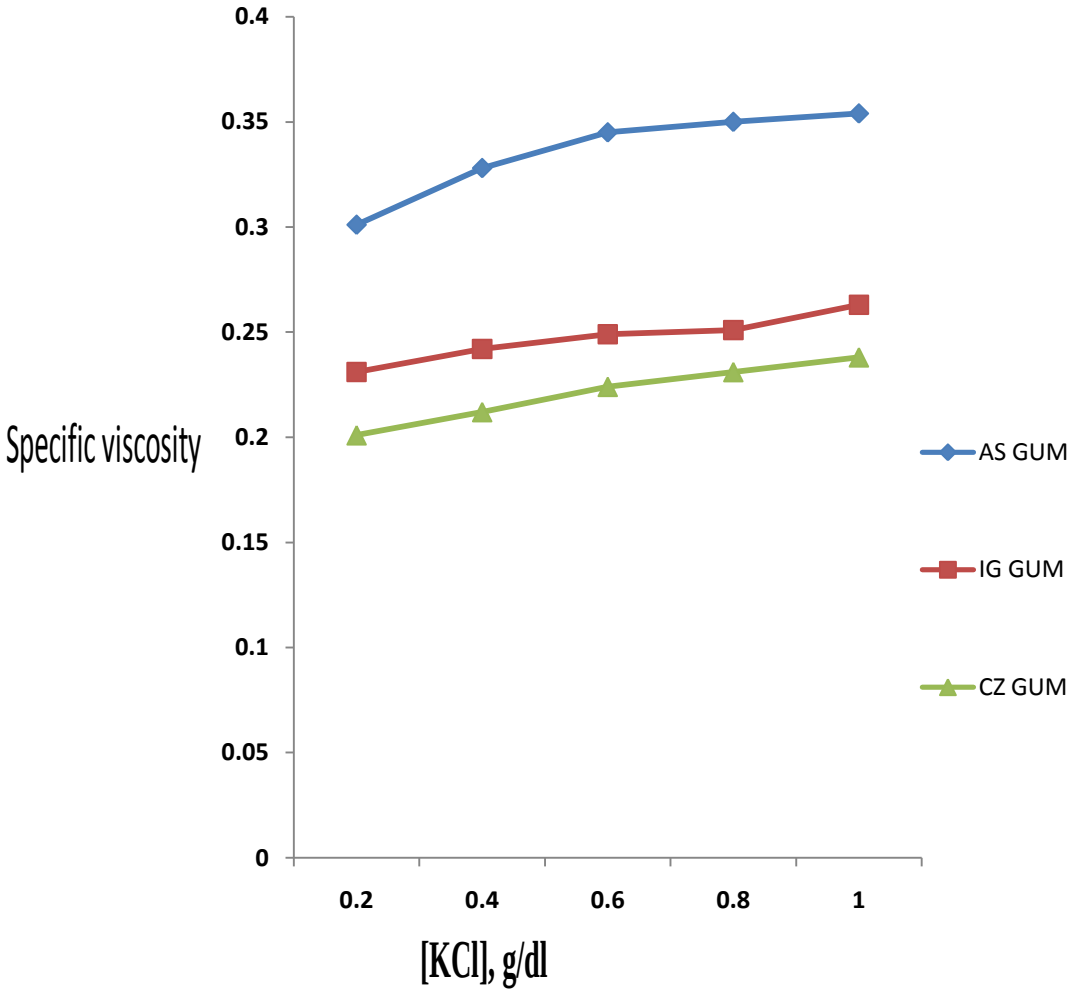


Figure 4.3a: Effect of Potassium Chloride (KCl) Concentrations on Specific Viscosity

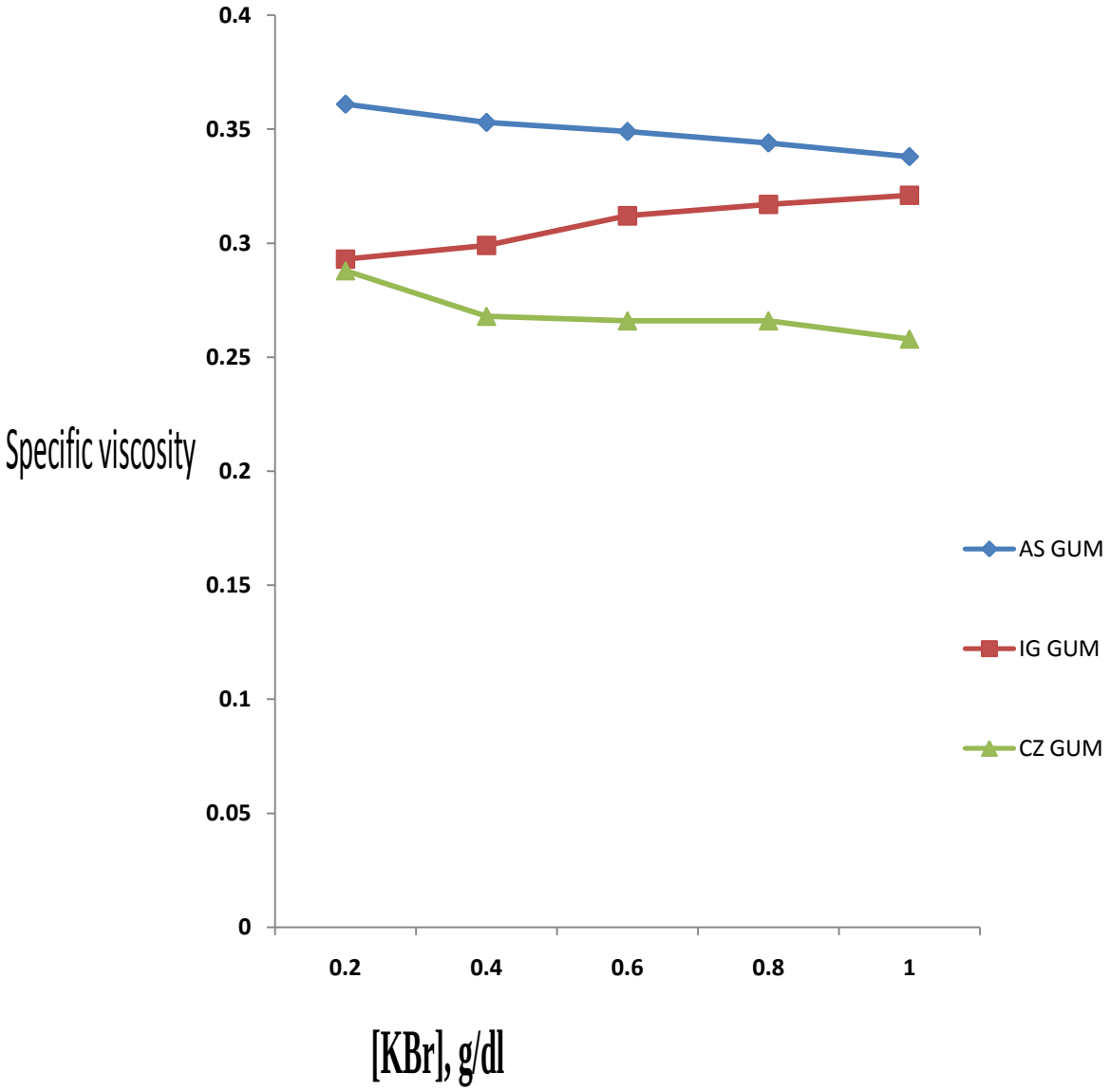


Figure 4.3b: Effect of Potassium Bromide (KBr) Concentrations on Specific Viscosity

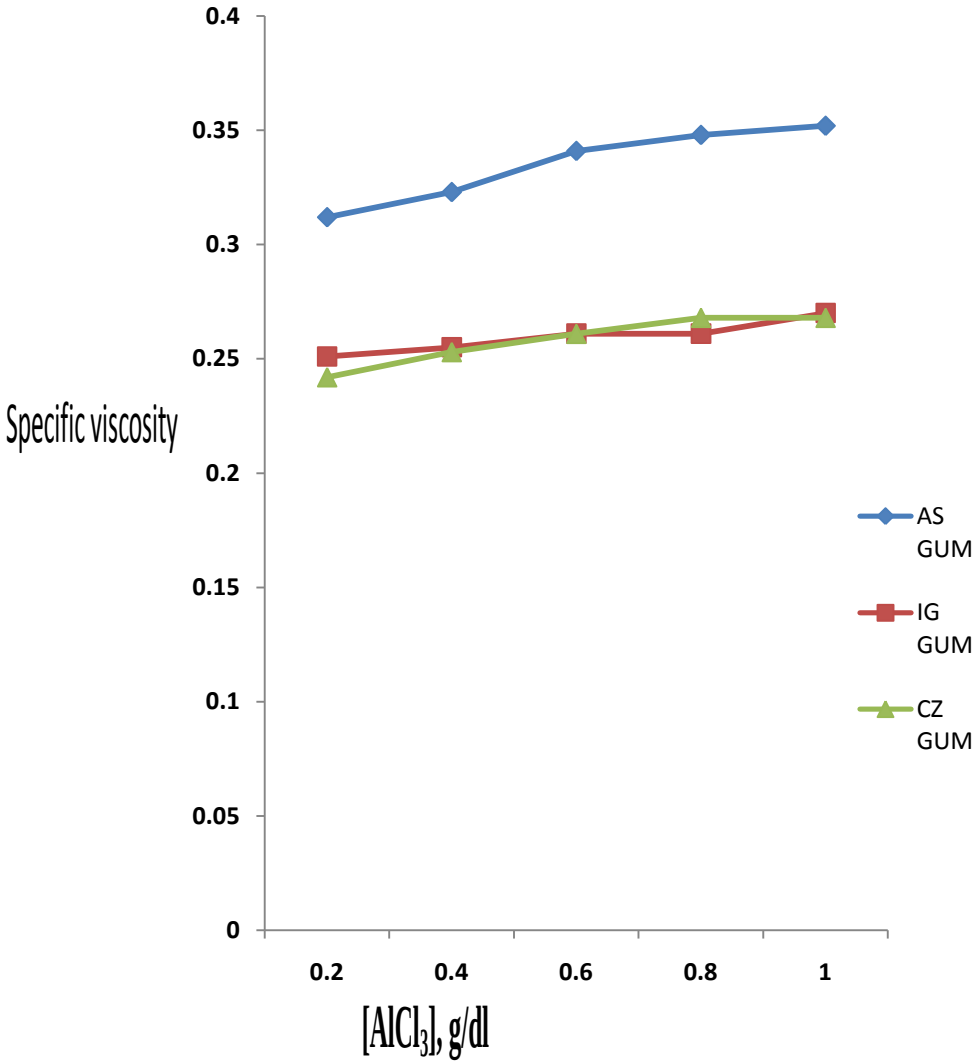


Figure 4.3c: Effect of Aluminium Chloride ($AlCl_3$) Concentrations on Specific Viscosity

Table 4.2: Mean Concentrations of some metals in *Irvingia gabonensis (IG), Celtis zenkeri (CZ) and Acacia Senegal (AS)* gums

Metals	[IG gum], mg/kg ± SD	[*CZ* gum], mg/kg ± SD	[AS gum], mg/kg ± SD
Mn	146.40 ± 0.20	213.2 ± 0.06	21.20 ± 0.21
Fe	311.10 ± 0.10	375.40 ± 0.20	1431.02 ±0.04
Zn	41.05 ± 0.05	10.64 ± 0.04	45.04 ± 0.12
Pb	63.20 ± 0.20	15.20 ± 0.15	19.52 ± 0.37
Mg	2274.20 ± 0.15	2285.30 ± 0.01	834.21 ± 0.17
Cd	1.00 ± 0.00	1.60 ± 0.20	0.10 ± 0.00
Ca	2315.90 ± 0.10	652.20 ± 0.12	132.05 ± 0.43
Cu	16.40 ± 0.20	16.60 ± 0.00	ND
Ni	48.30 ± 0.10	79.70 ± 0.01	11.00 ± 0.11

ND= Not detected

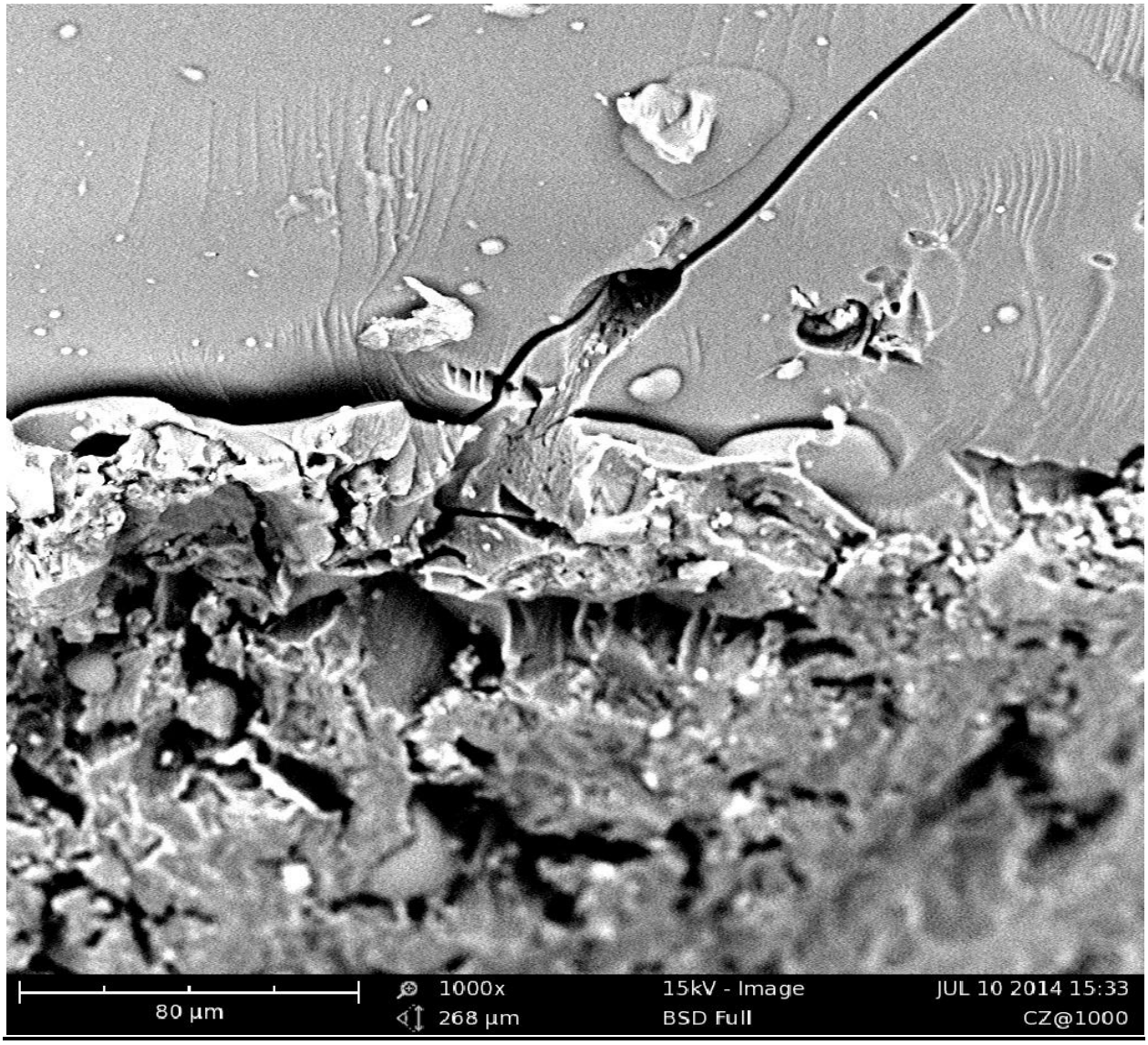


Figure.4.4a: Surface Morphology of CZ Gum (At 1000X Magnification)

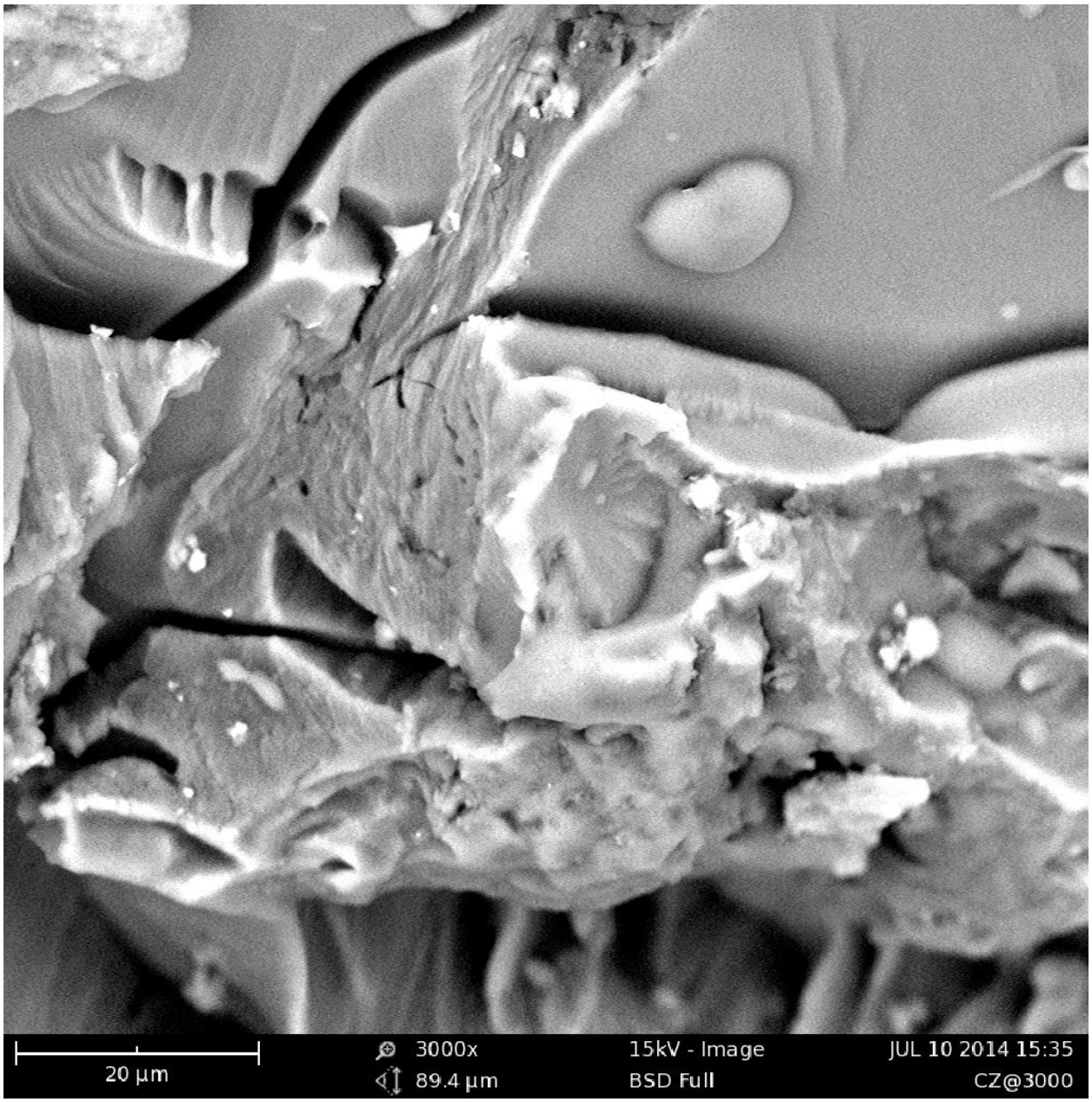


Figure 4.4b: Surface Morphology of CZ Gum (At 3000X Magnification)

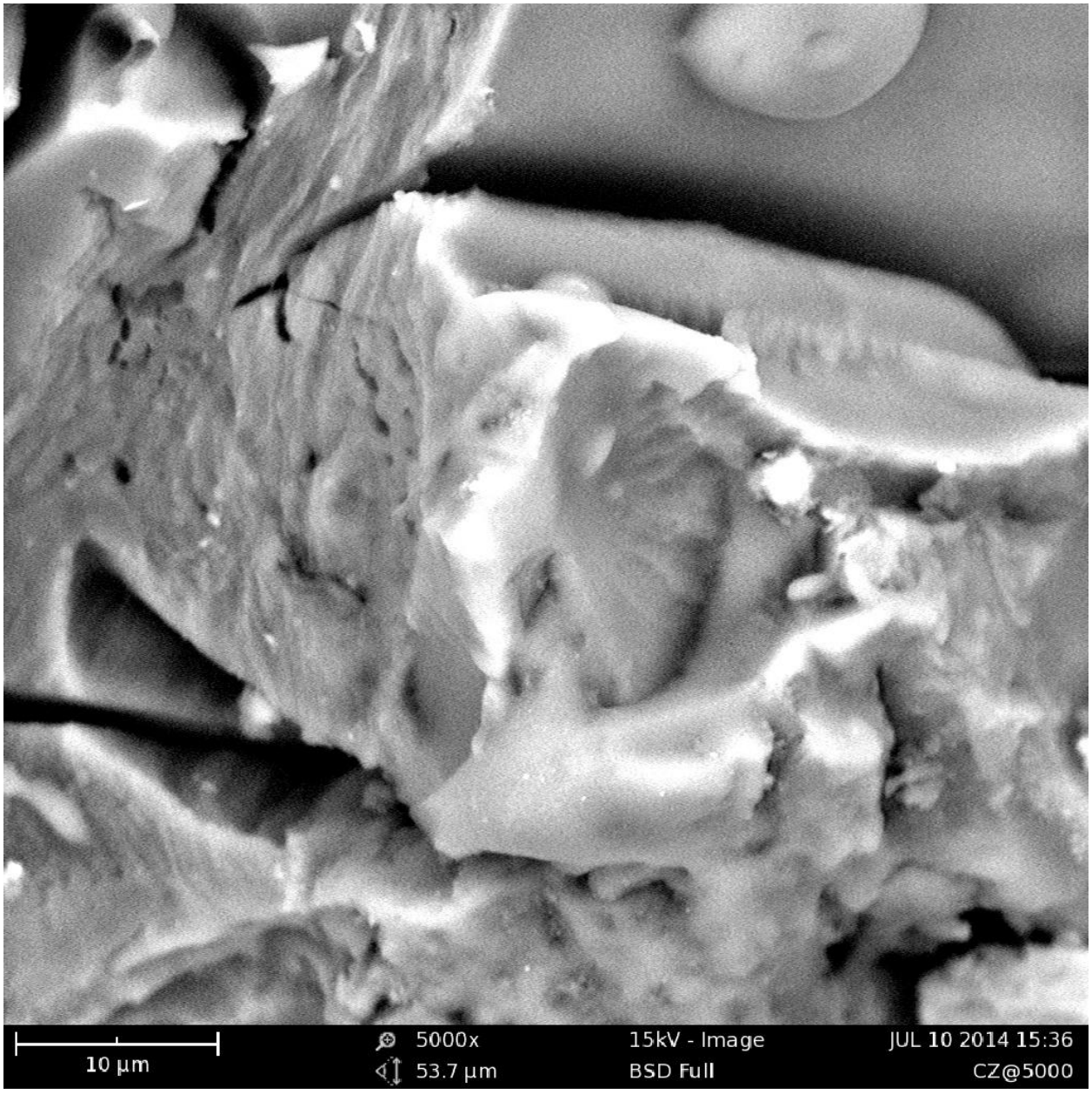


Figure 4.4c: Surface Morphology of CZ Gum (At 5000X Magnification)

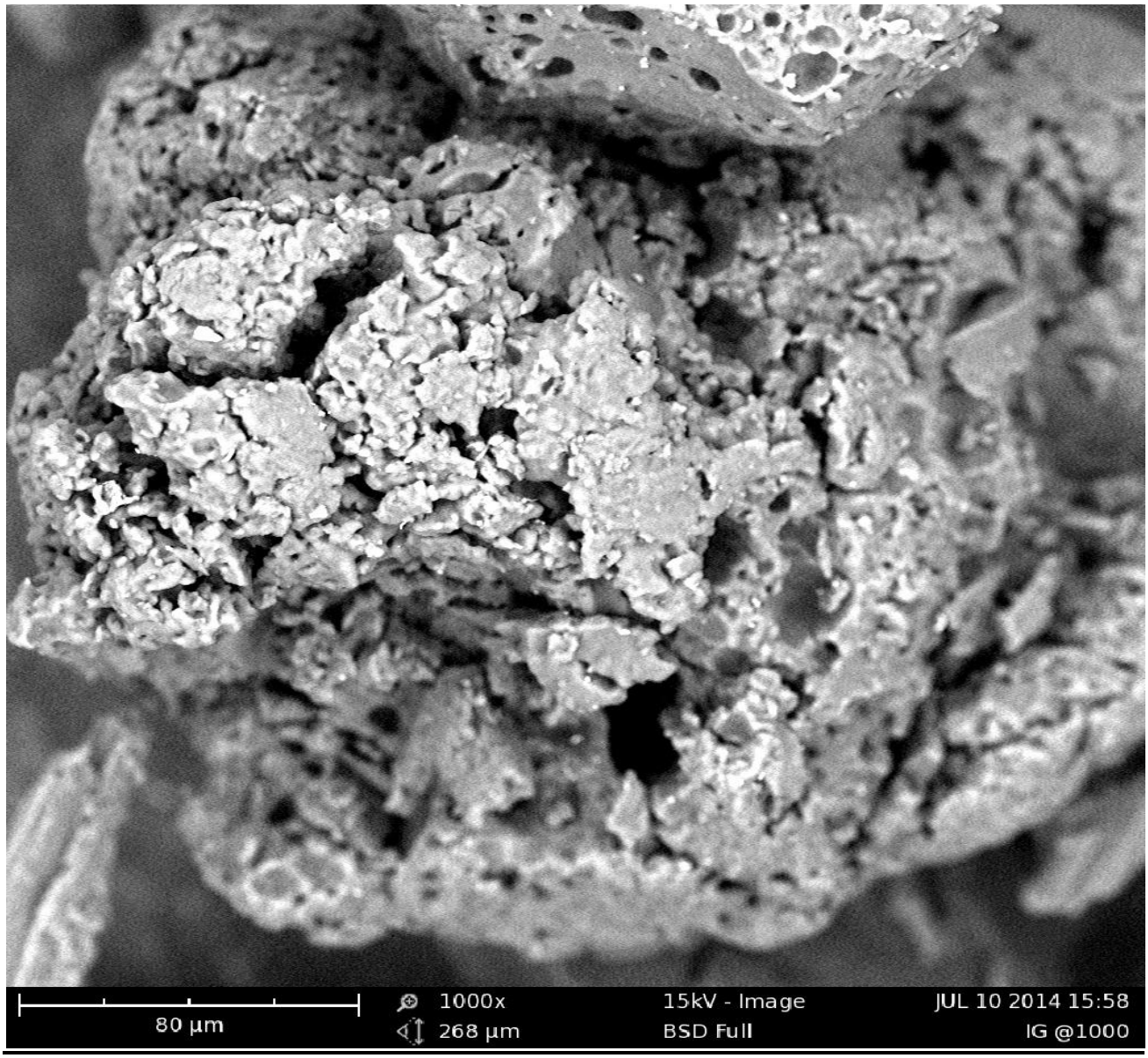


Figure 4.5a: Surface Morphology of IG Gum (At 1000X Magnification)

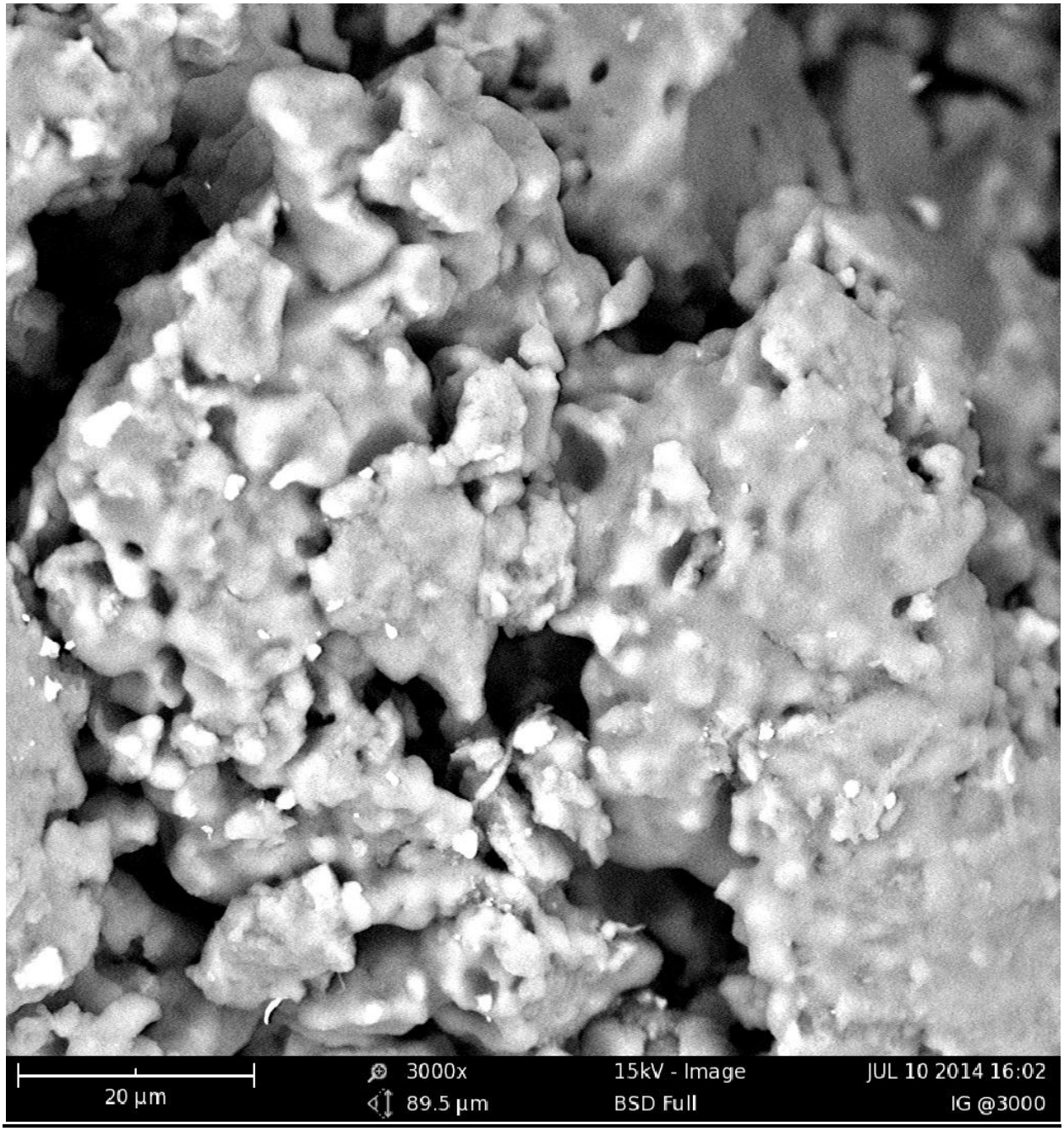


Figure 4.5b: Surface Morphology of IG Gum (At 3000X Magnification)

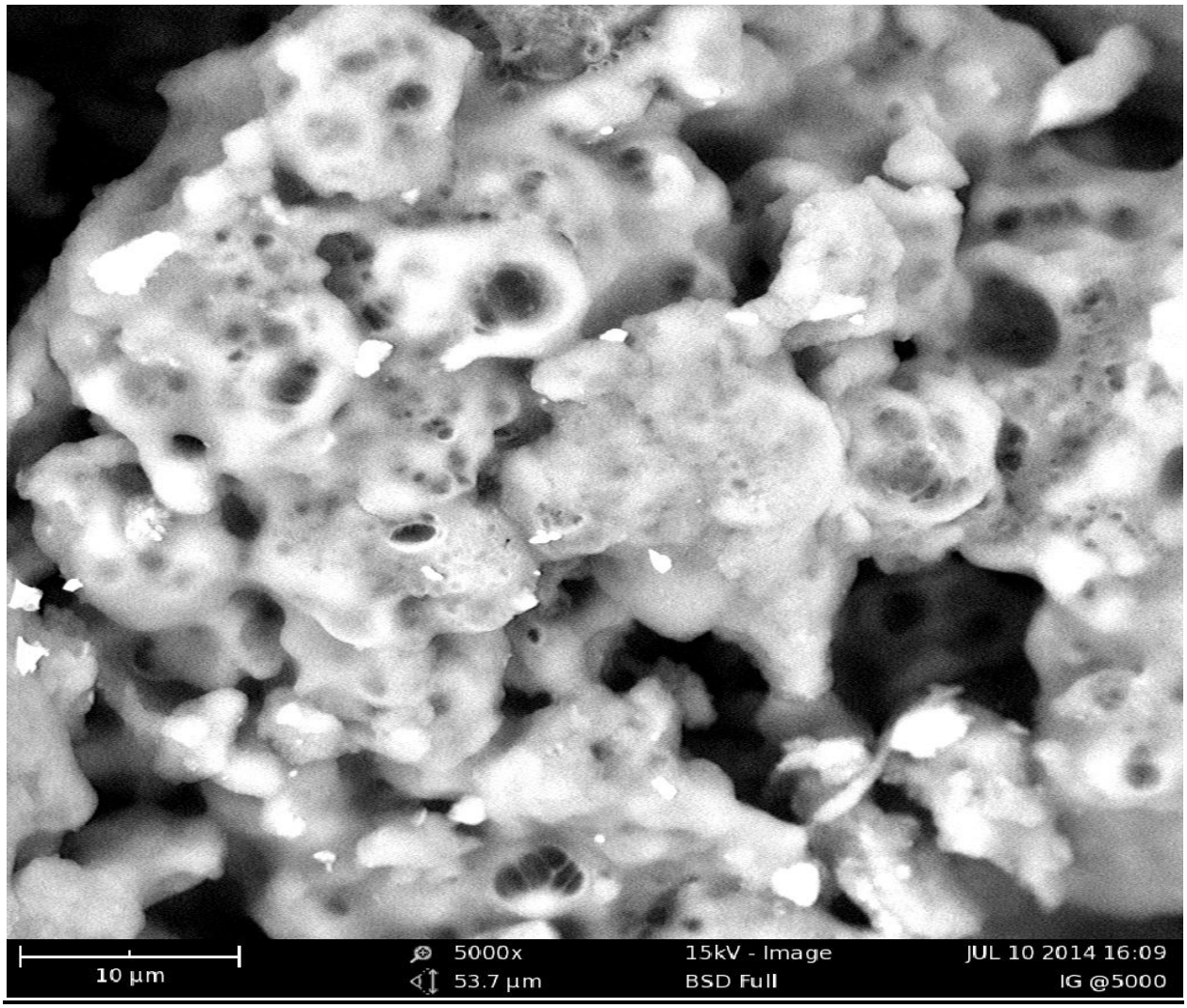


Figure 4.5c: Surface Morphology of IG Gum (At 5000X Magnification)

4.5 SEM Elemental Analyses

The results for the Scanning electron microscope (SEM) elemental analysis of the gum samples are shown in Figures 4.6 and 4.7 for *Irviginia gabonensis* and *Celtis zenkeri* gum respectively. Six different points in the micrograph were analyzed and the results presented in Figures 4.6a-f and 4.7a-f for *Irviginia gabonensis* and *Celtis zenkeri* gum respectively.

4.6 Fibre and Pore Measurement

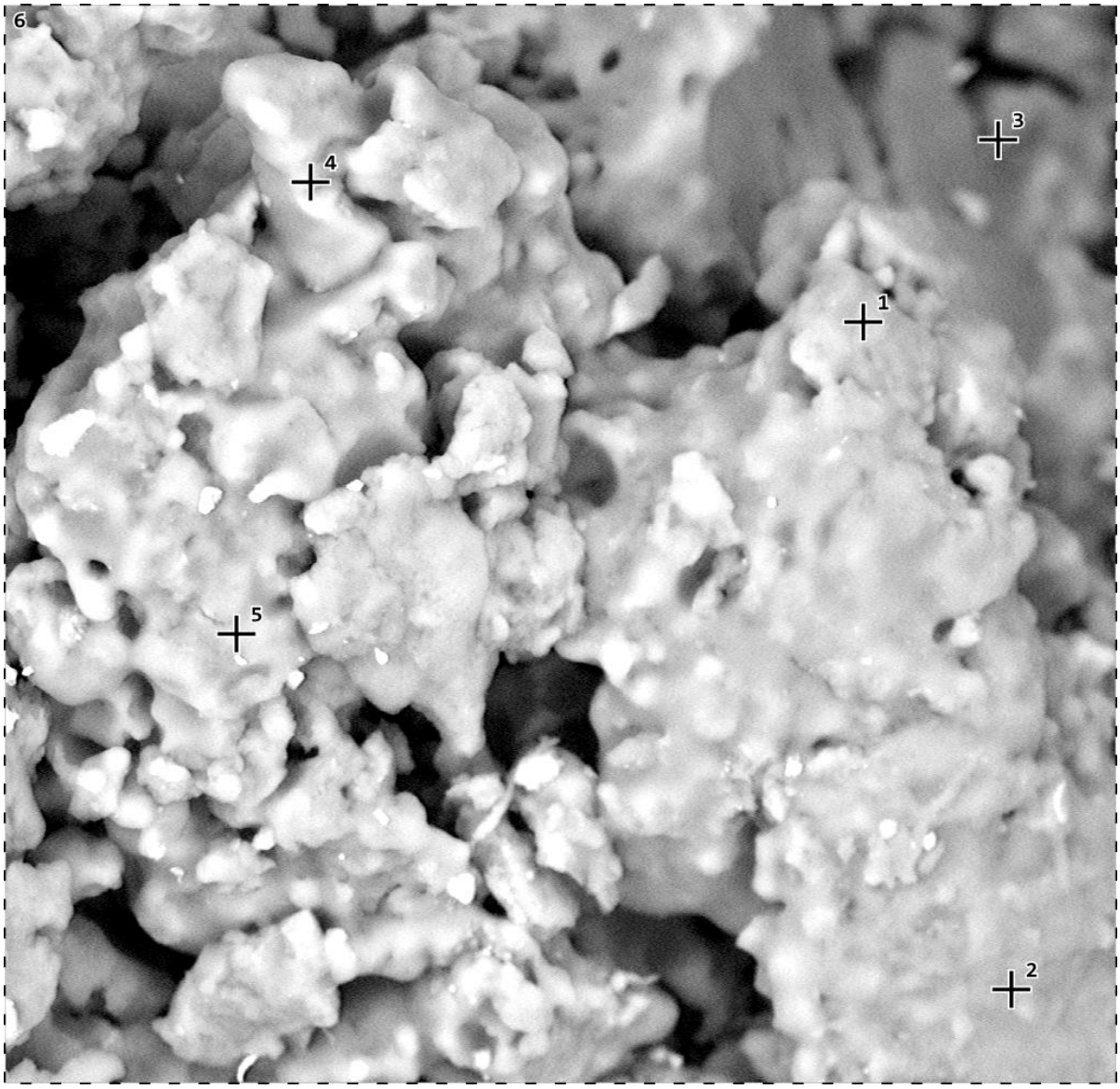
The results of the fibre and pores measurement using scanning electron microscope are shown in Figures 4.8a-d and 4.9a-d for *Irviginia gabonensis* and *Celtis zenkeri* respectively. It can be seen that the fibre length of CZ gum (Figure 4.8c) is far greater than that of IG gum (Figure 4.9c), while the pores area is $0.71\mu m^2$ (Figure 4.8d) and $0.02\mu m^2$ (Figure 4.9d) respectively.

4.7 FTIR

The results of the FTIR spectrum of *Irviginia gabonensis gum* and *Celtis zenkeri gum* are shown in Figures 4.10 and 4.11 respectively. The analyses of the spectrum are presented in Tables 4.3 and 4.4 for *Irviginia gabonensis* and *Celtis zenkeri* gum respectively.

4.8 GC-MS

The results of the GCMS analysis of the gum exudates are presented in Figures 4.12 and 4.13 for *Irviginia gabonensis* gum and *Celtis zenkeri* gum respectively. The spectrums of each line detected are shown in Figures 4.12a-h and 4.13a-e for *Irviginia gabonensis* and *Celtis zenkeri* gum respectively. The analytical parameters deduced from the GCMS spectrums are presented in Tables 4.5 and 4.6 for *Irviginia gabonensis* and *Celtis zenkeri* gum respectively.



At 3000X Magnification

Figure 4.6: Scanning Electron Micrograph of *Irviginia gabonensis* Indicating Spots of Elements Analysis

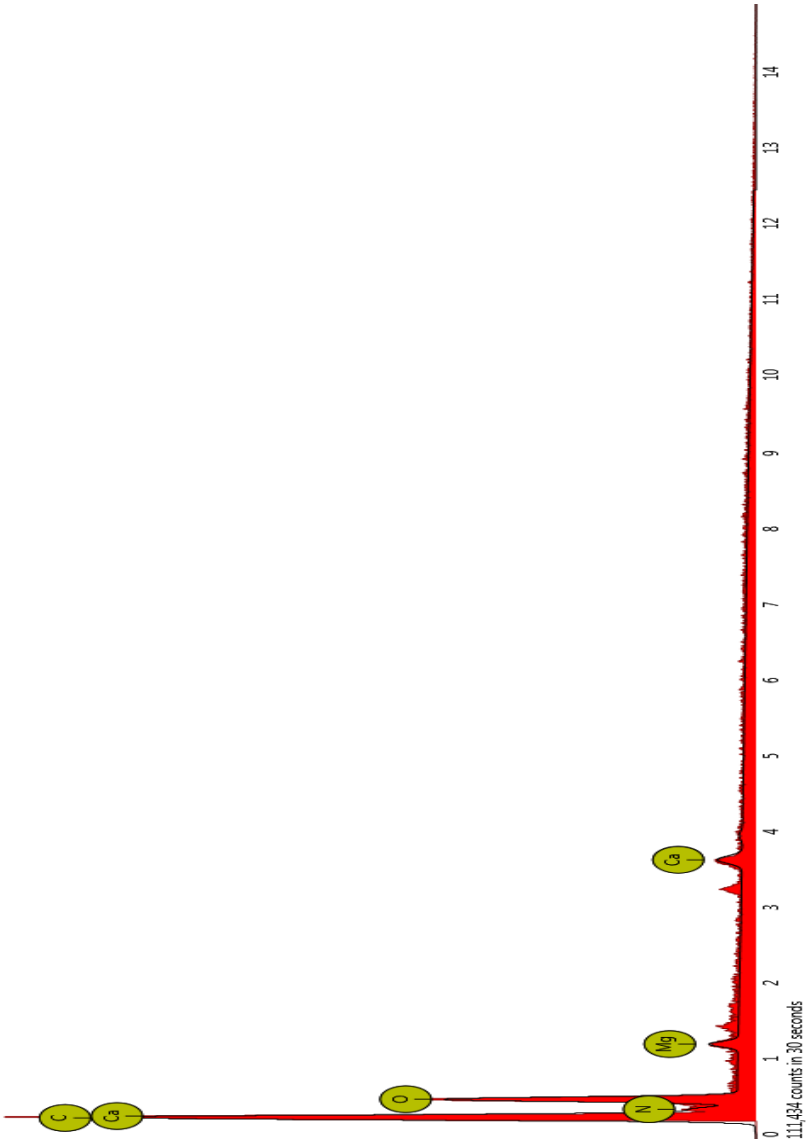


Figure 4.6a: SPOT 1 Elemental Analysis of *Irviginia gabonensis*

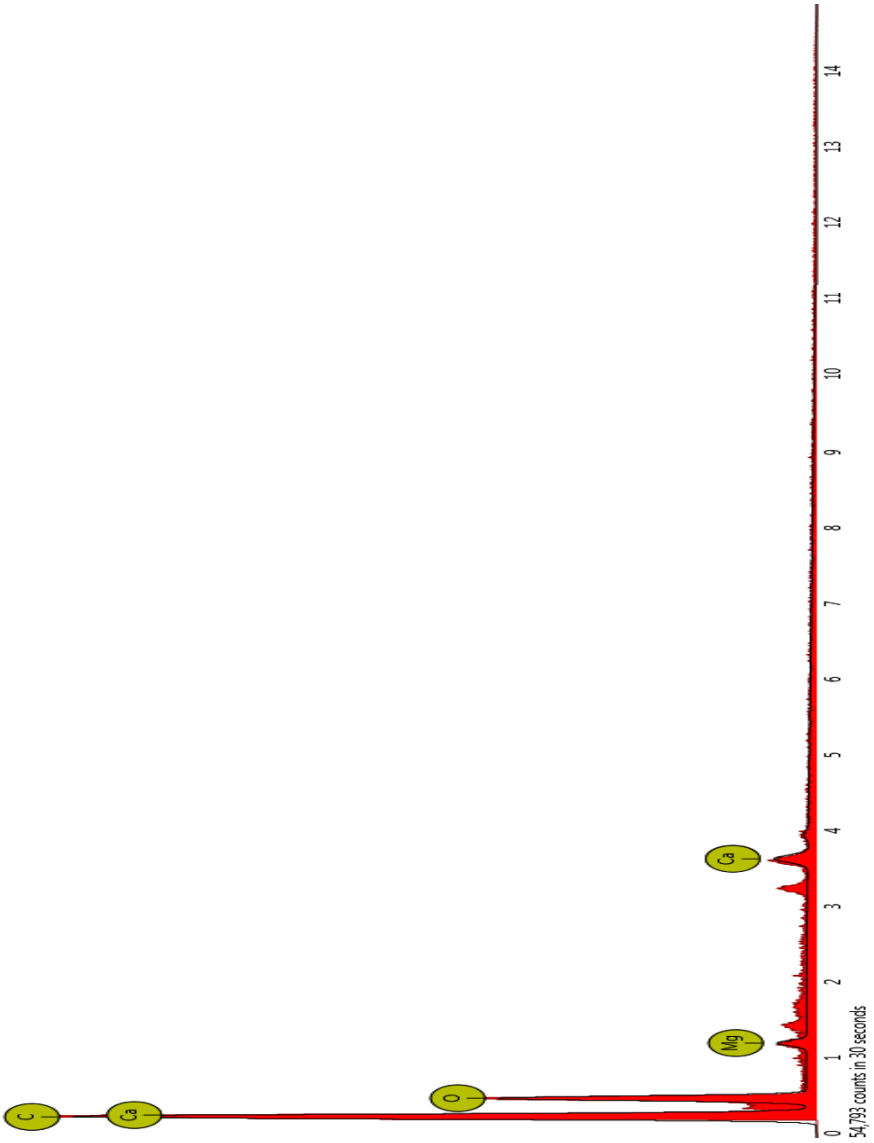


Figure 4.6b: SPOT 2 Elemental Analysis of *Irviginia gabonensis*

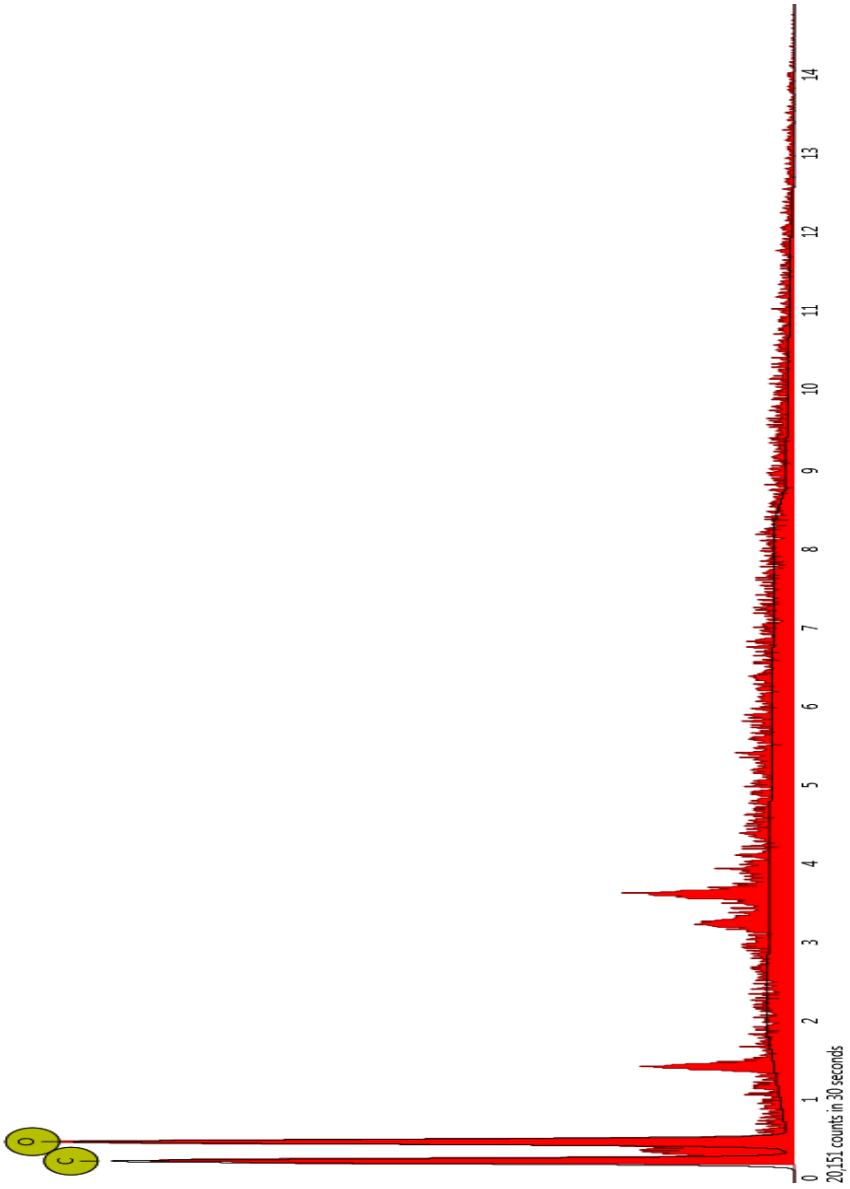


Figure 4.6c: SPOT 3 Elemental Analysis of *Irviginia gabonensis*

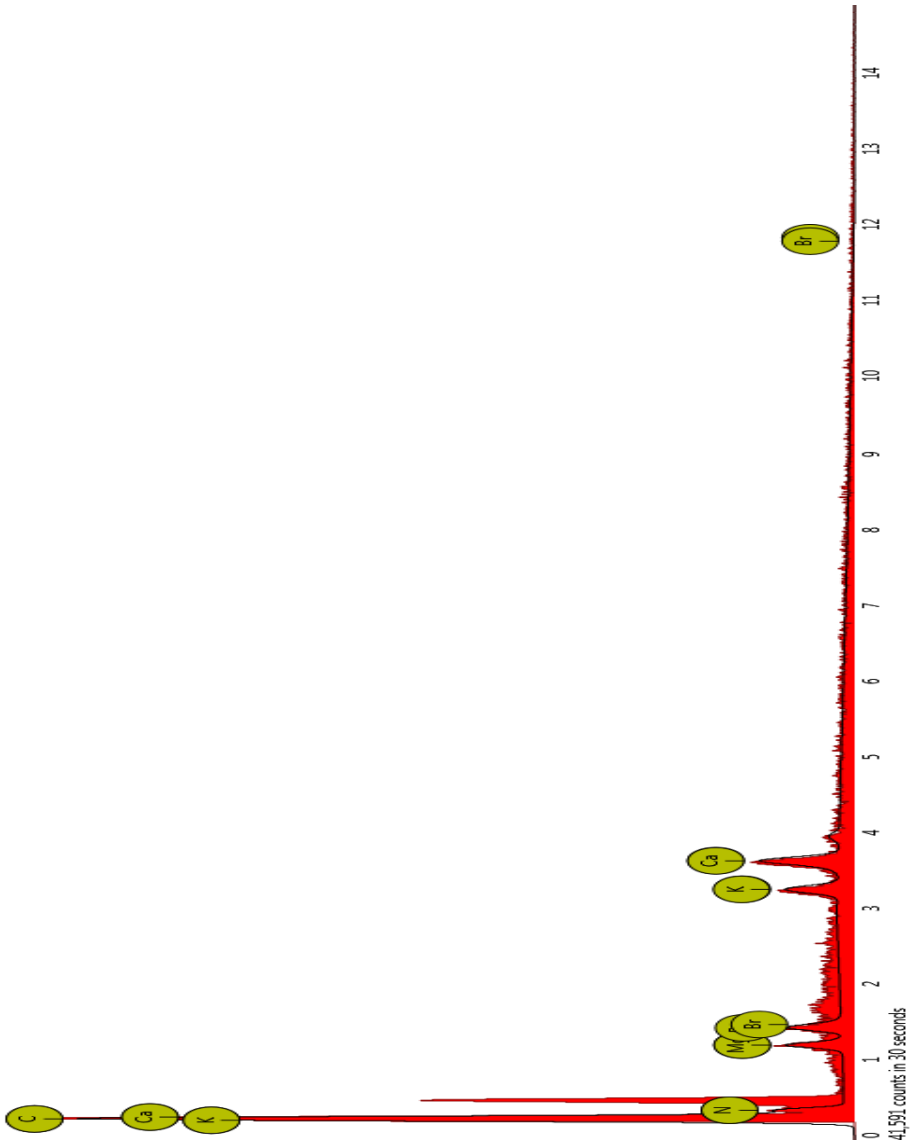


Figure 4.6d: SPOT 4 Elemental Analysis of *Irviginia gabonensis*

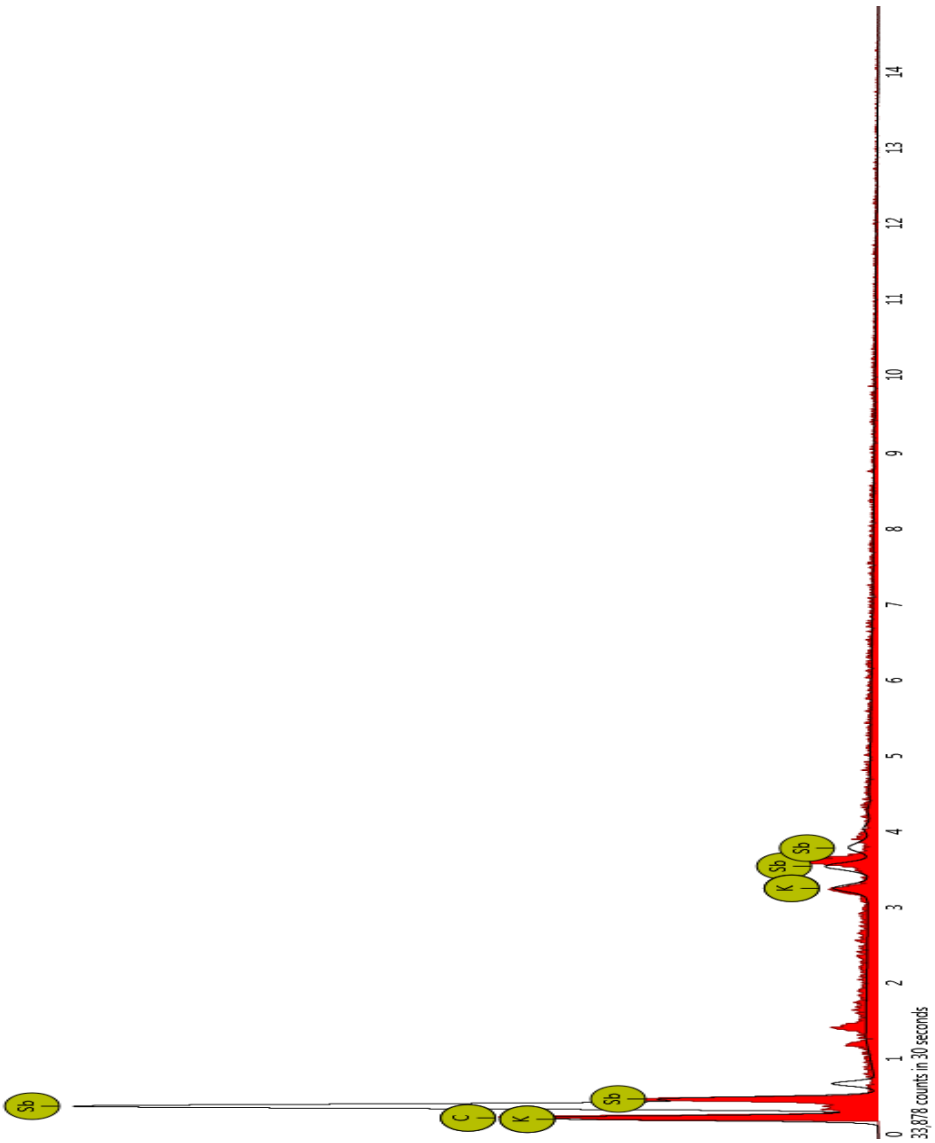


Figure 4.6e: SPOT 5 Elemental Analysis of *Irviginia gabonensis*

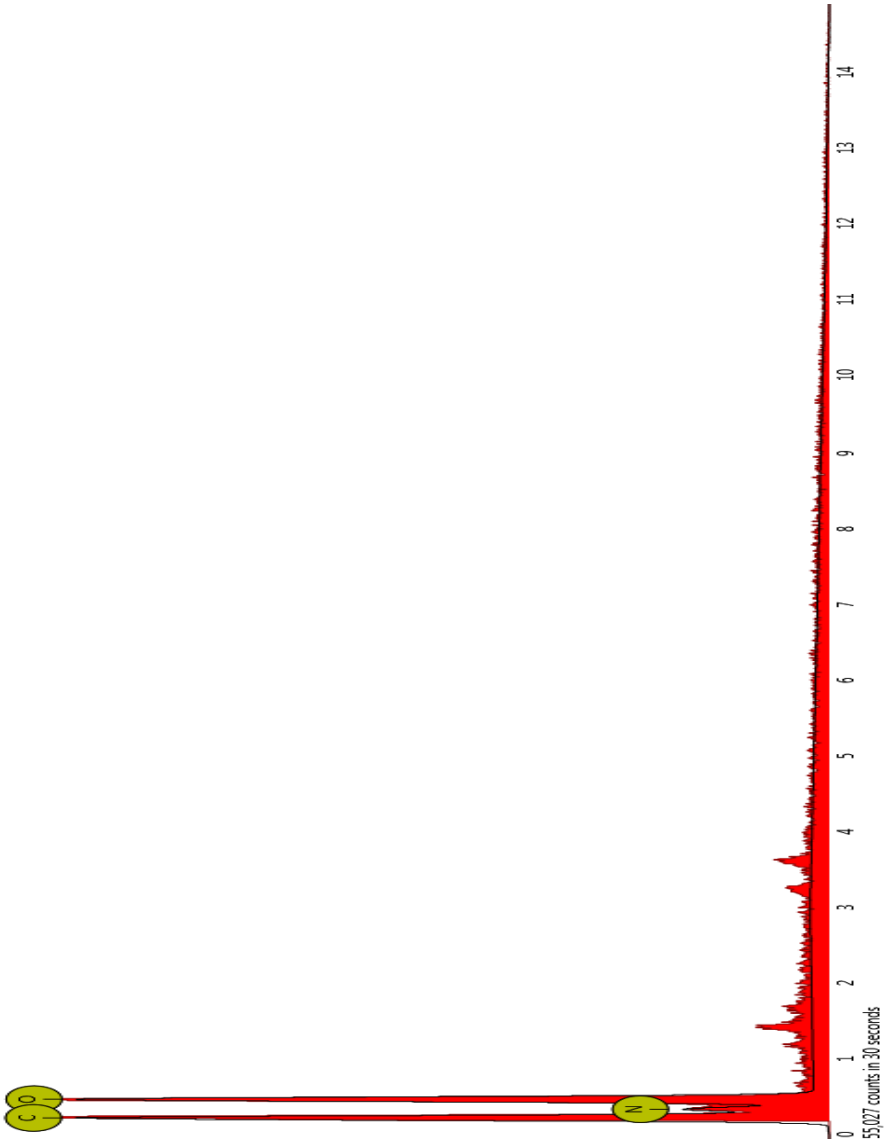
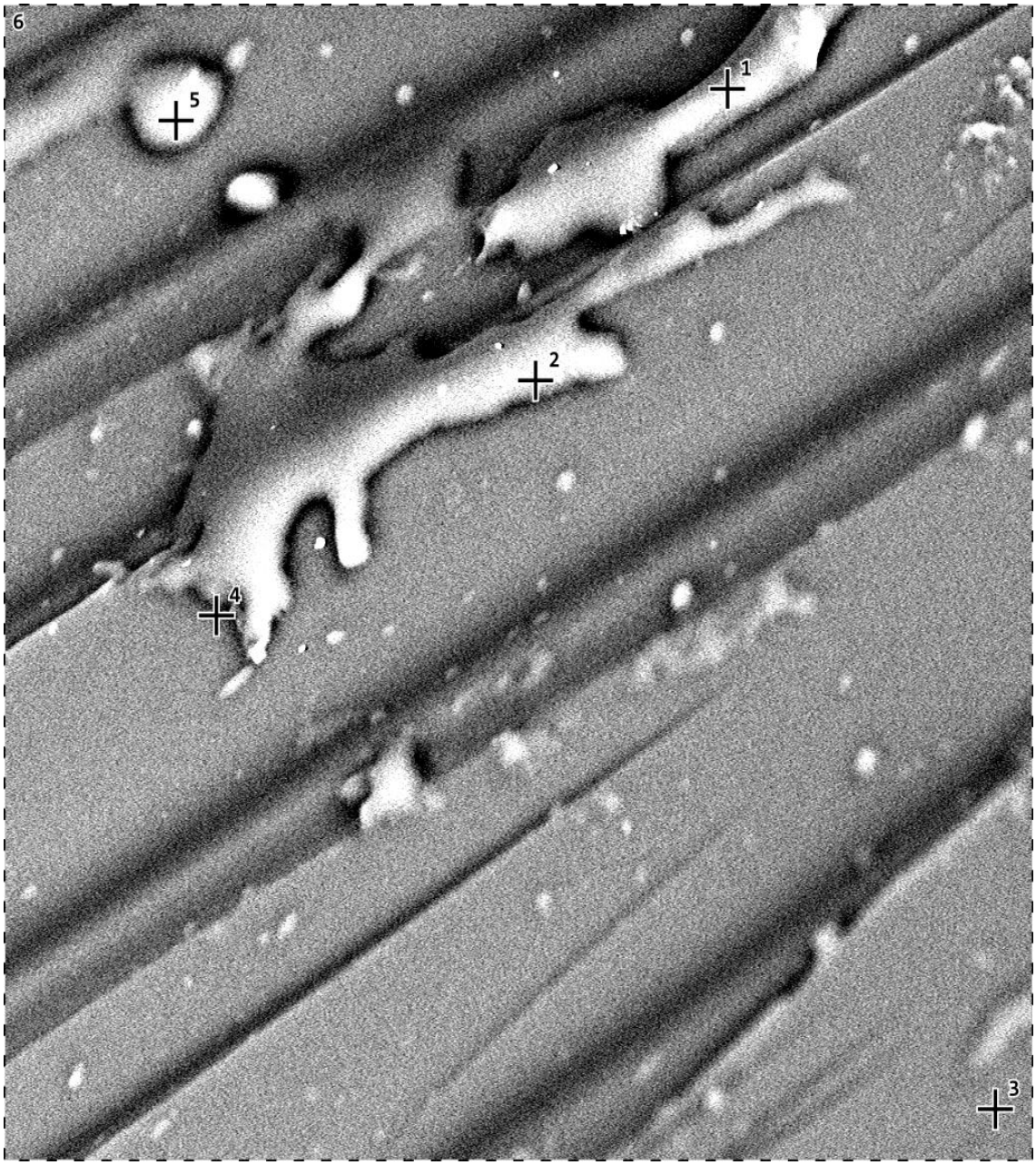


Figure 4.6f: SPOT 6 Elemental Analysis of *Irviginia gabonensis*



At 3000X Magnification

Figure 4.7: Scanning Electron Micrograph of *Celtis zenkeri* Indicating Spots of Elements Analysis

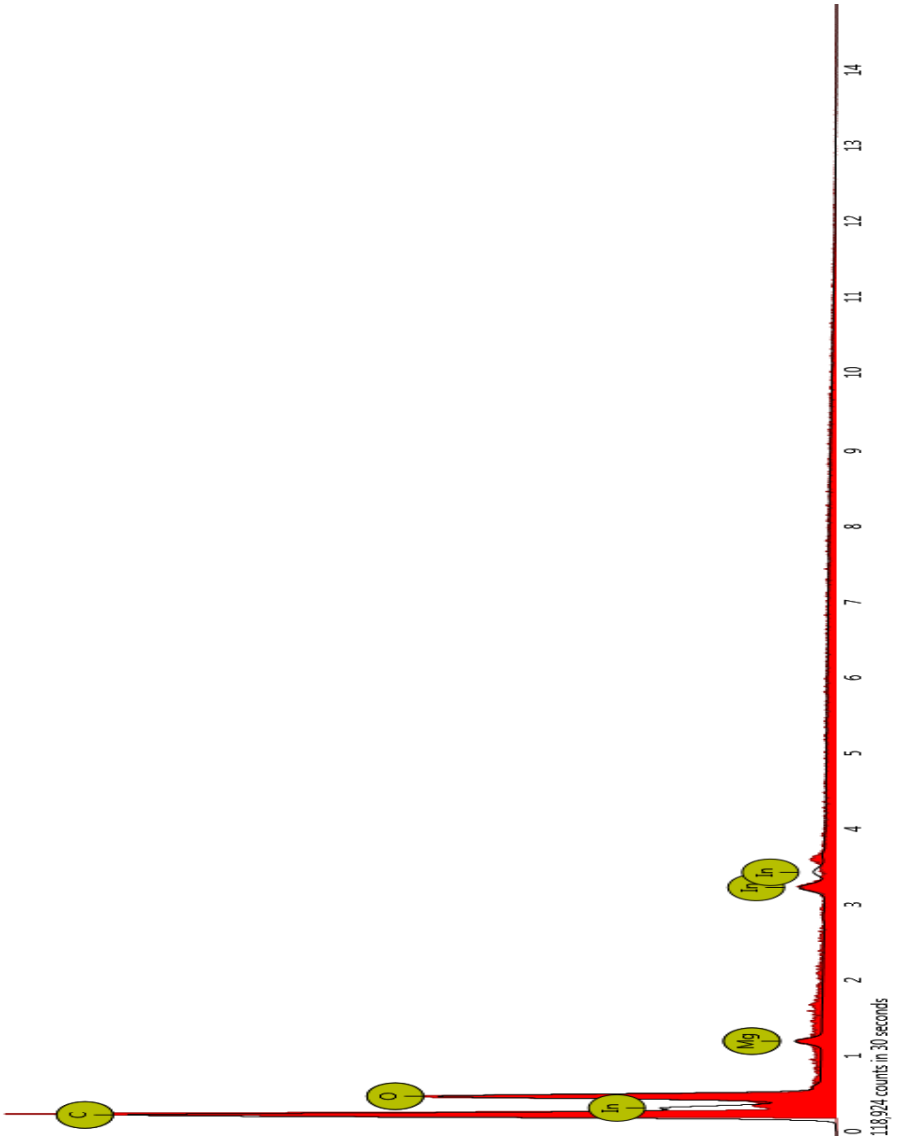


Figure 4.7a: SPOT 1 Elemental Analysis of *Celtis zenkeri*

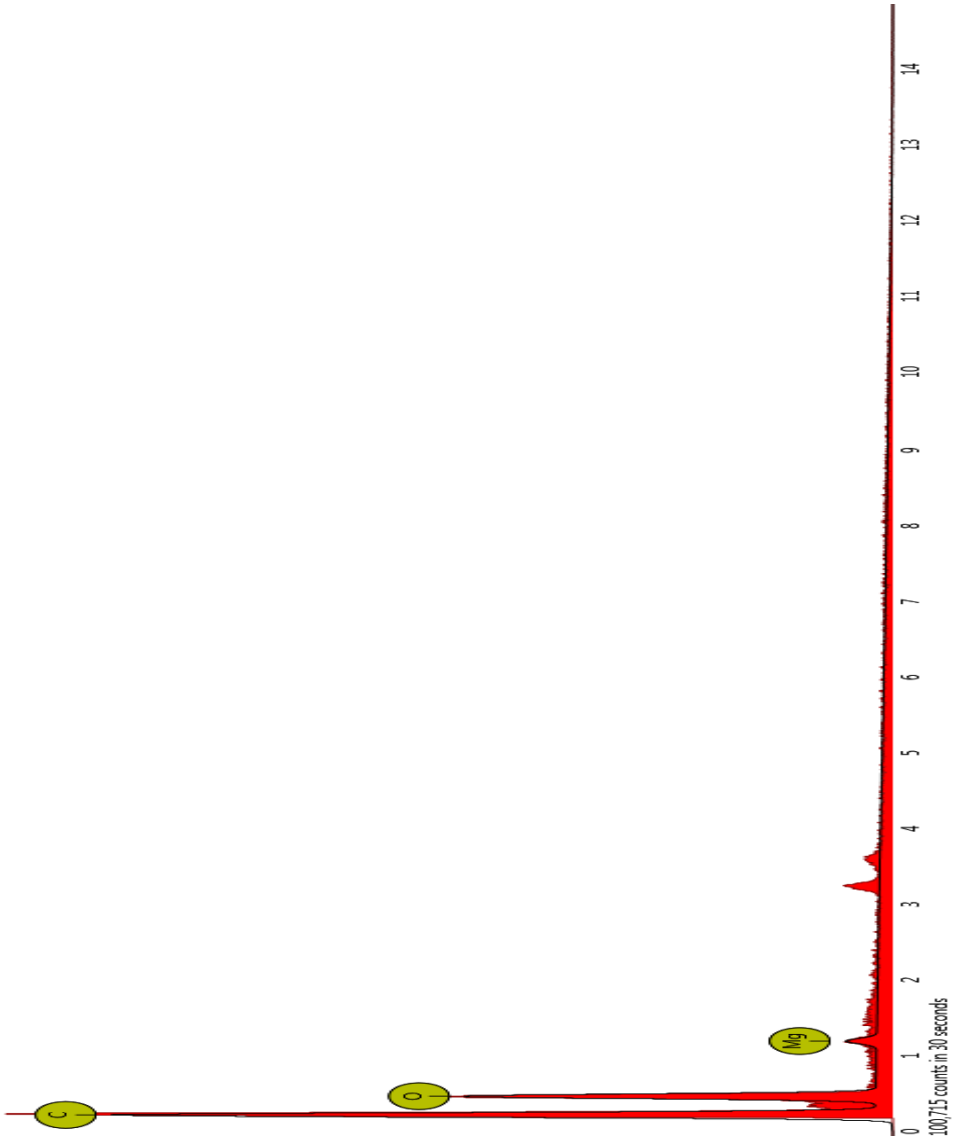


Figure 4.7b: SPOT 2 Elemental Analysis of *Celtis zenkeri*

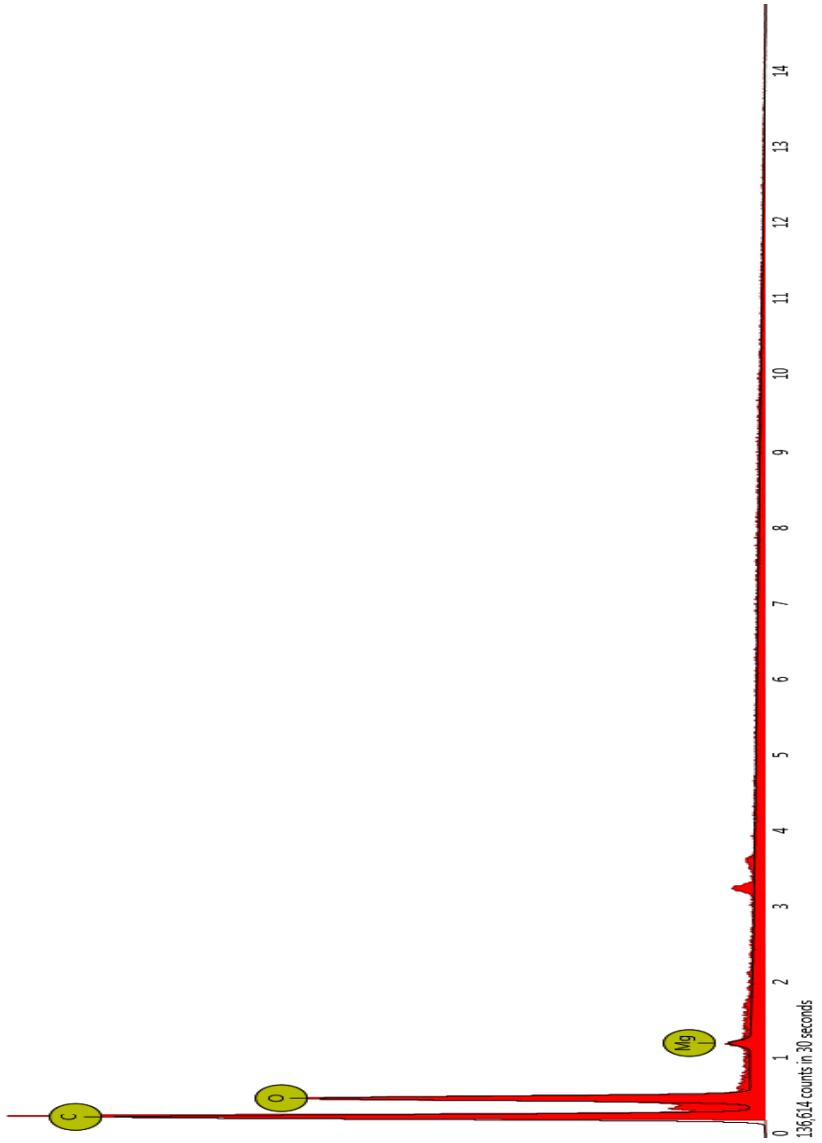


Figure 4.7c: SPOT 3 Elemental Analysis of *Celtis zenkeri*

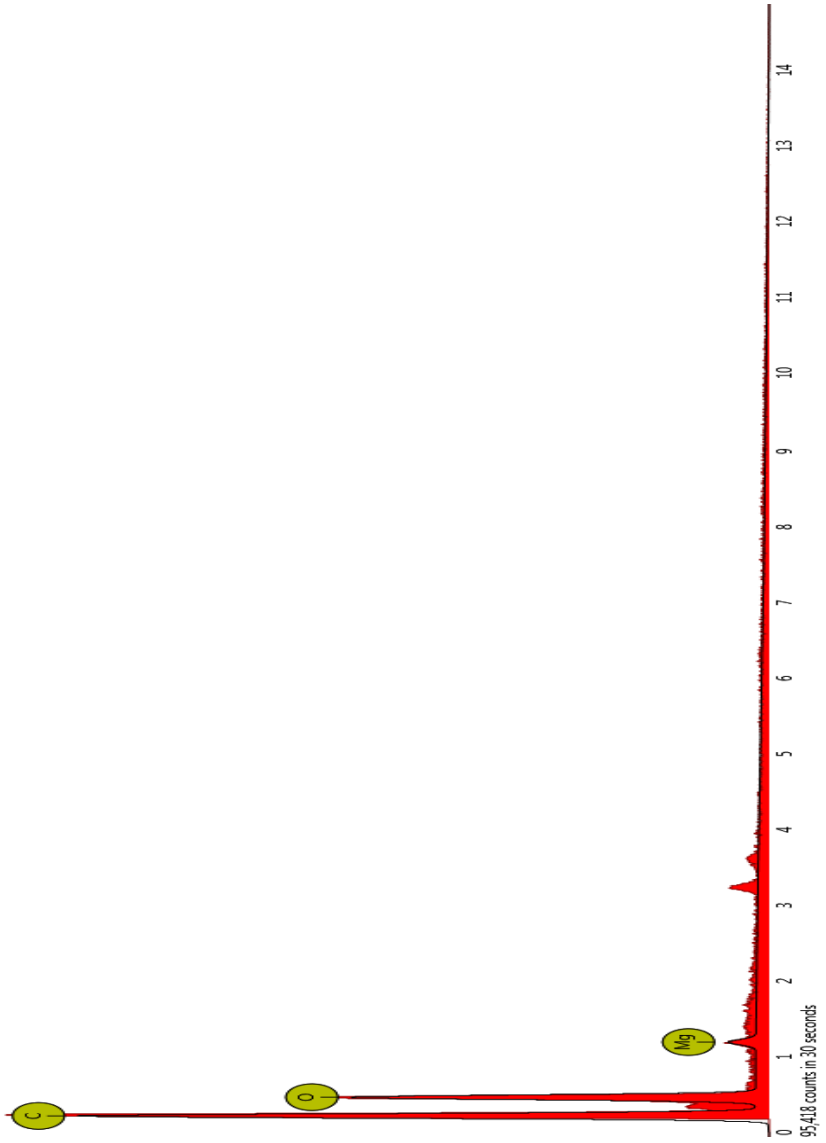


Figure 4.7d: SPOT 4 Elemental Analysis of *Celtis zenkeri*

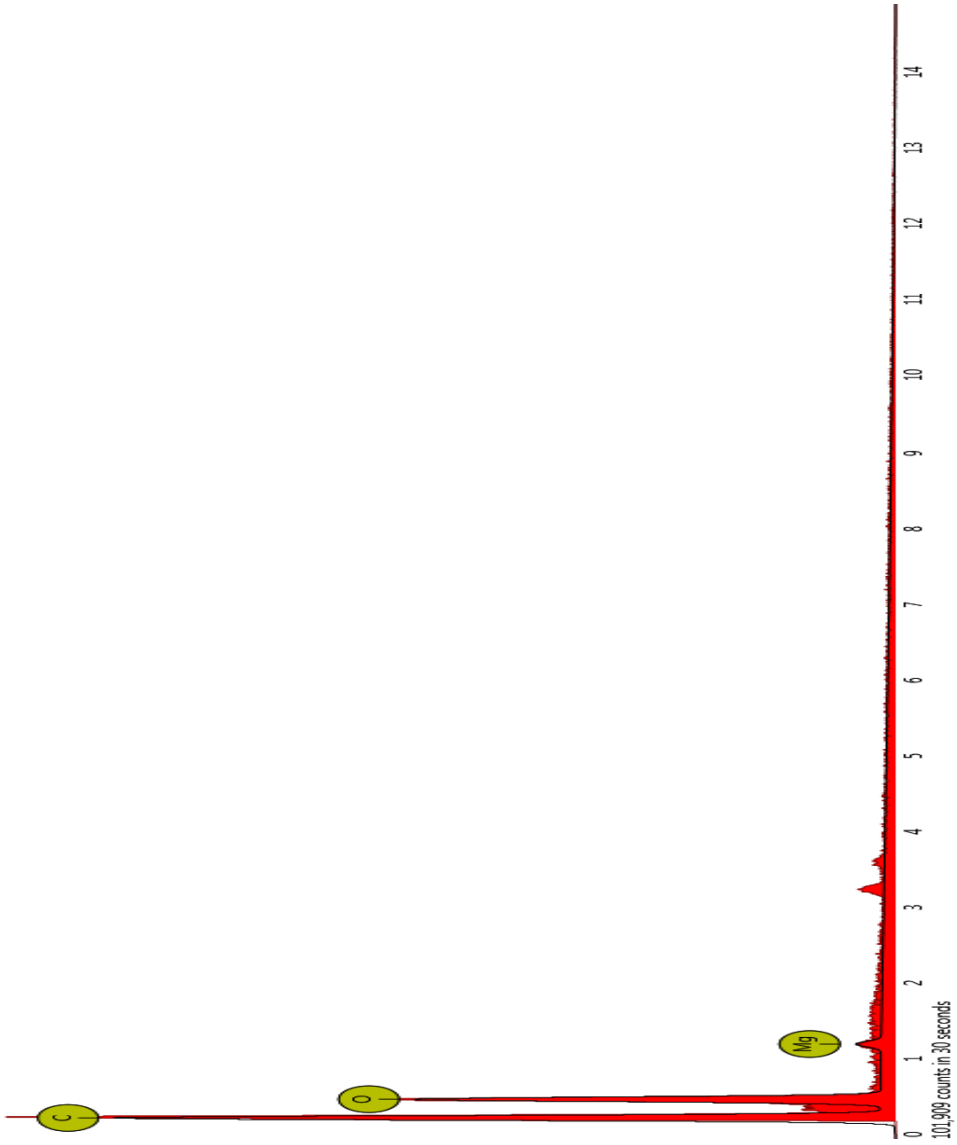


Figure 4.7e: SPOT 5 Elemental Analysis of *Celtis zenkeri*

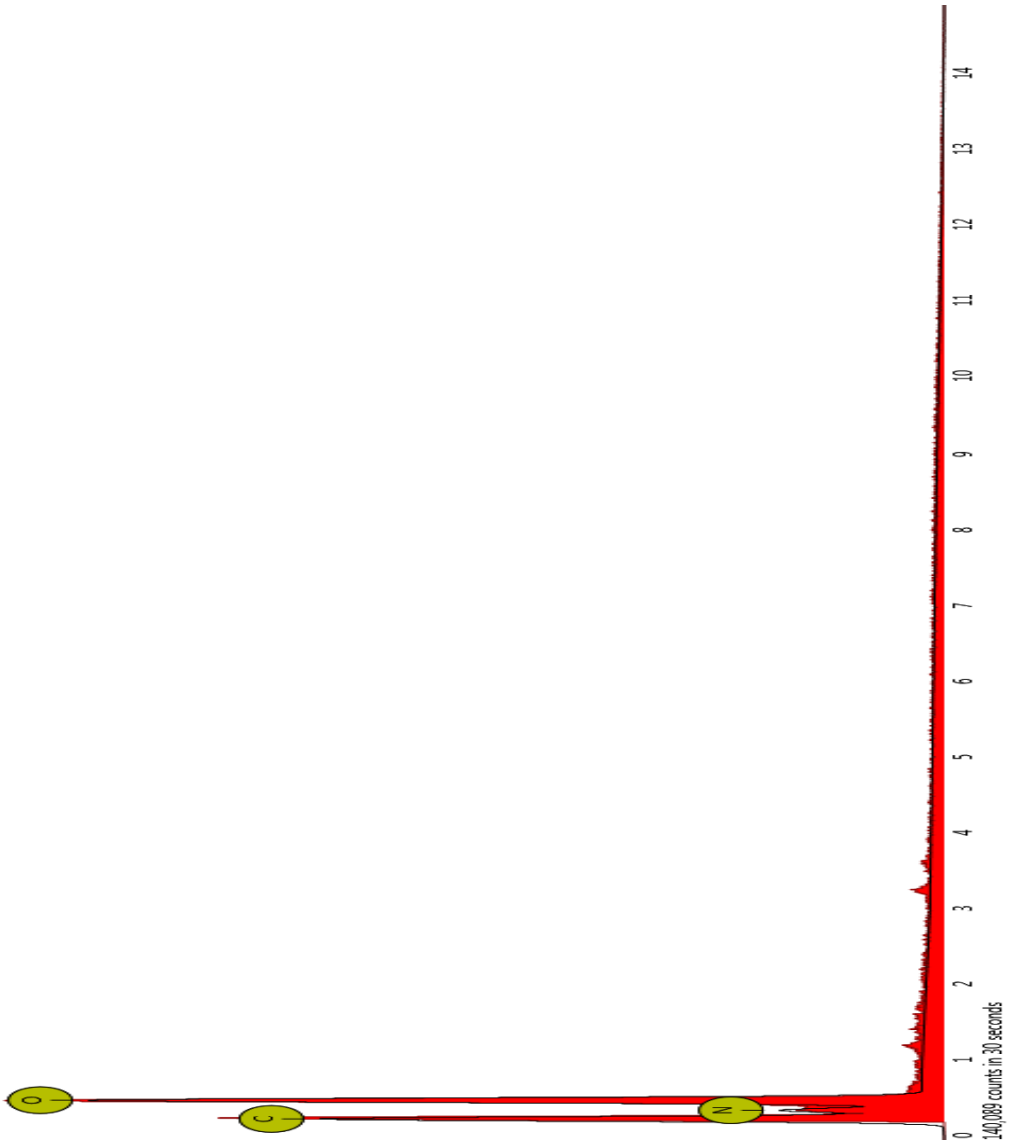


Figure 4.7f: SPOT 6 Elemental Analysis of *Celtis zenkeri*

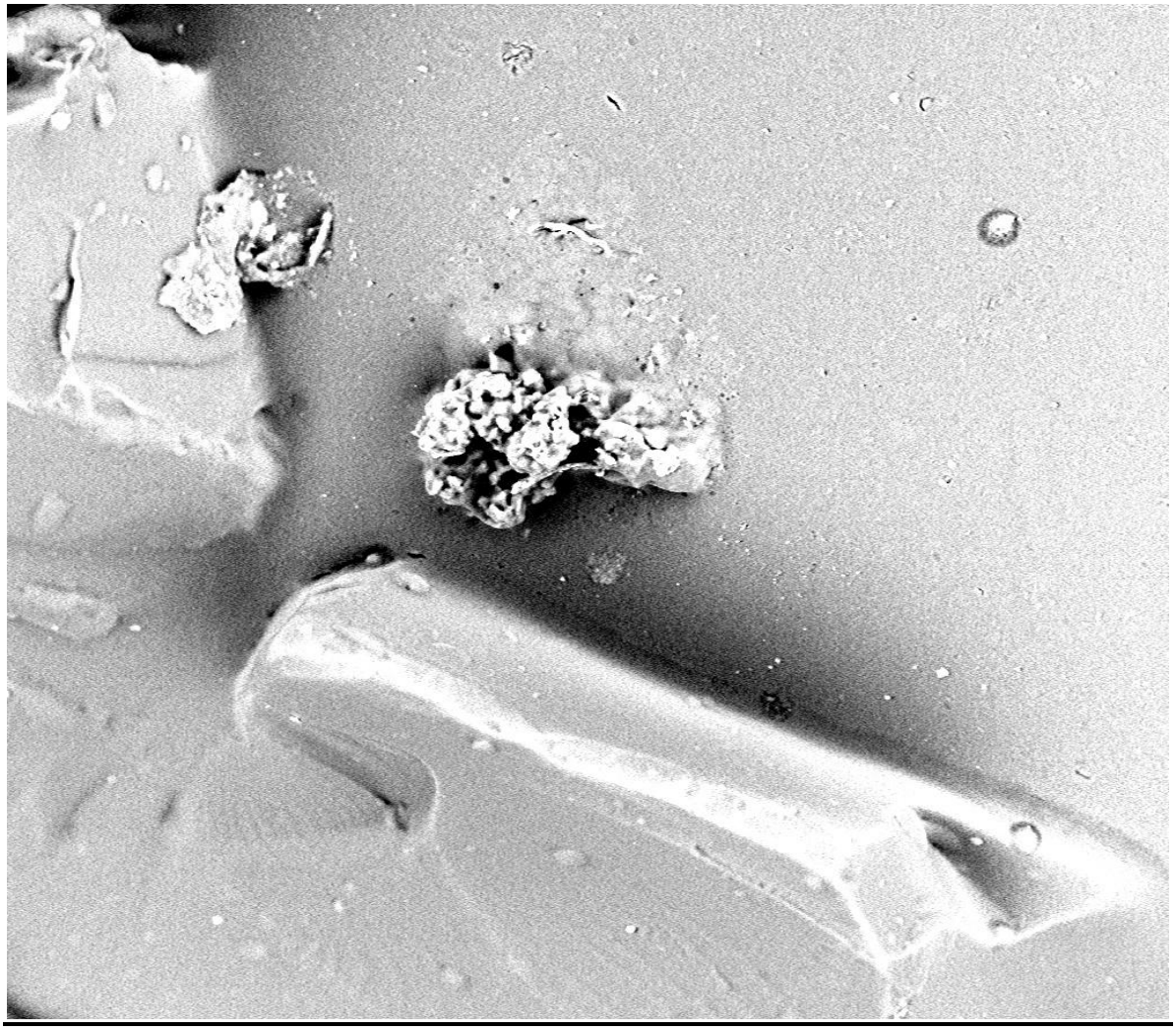


Figure 4.8a: *Celtis zenkeri* Fibre and Pore Measurement Surface Area of Analysis

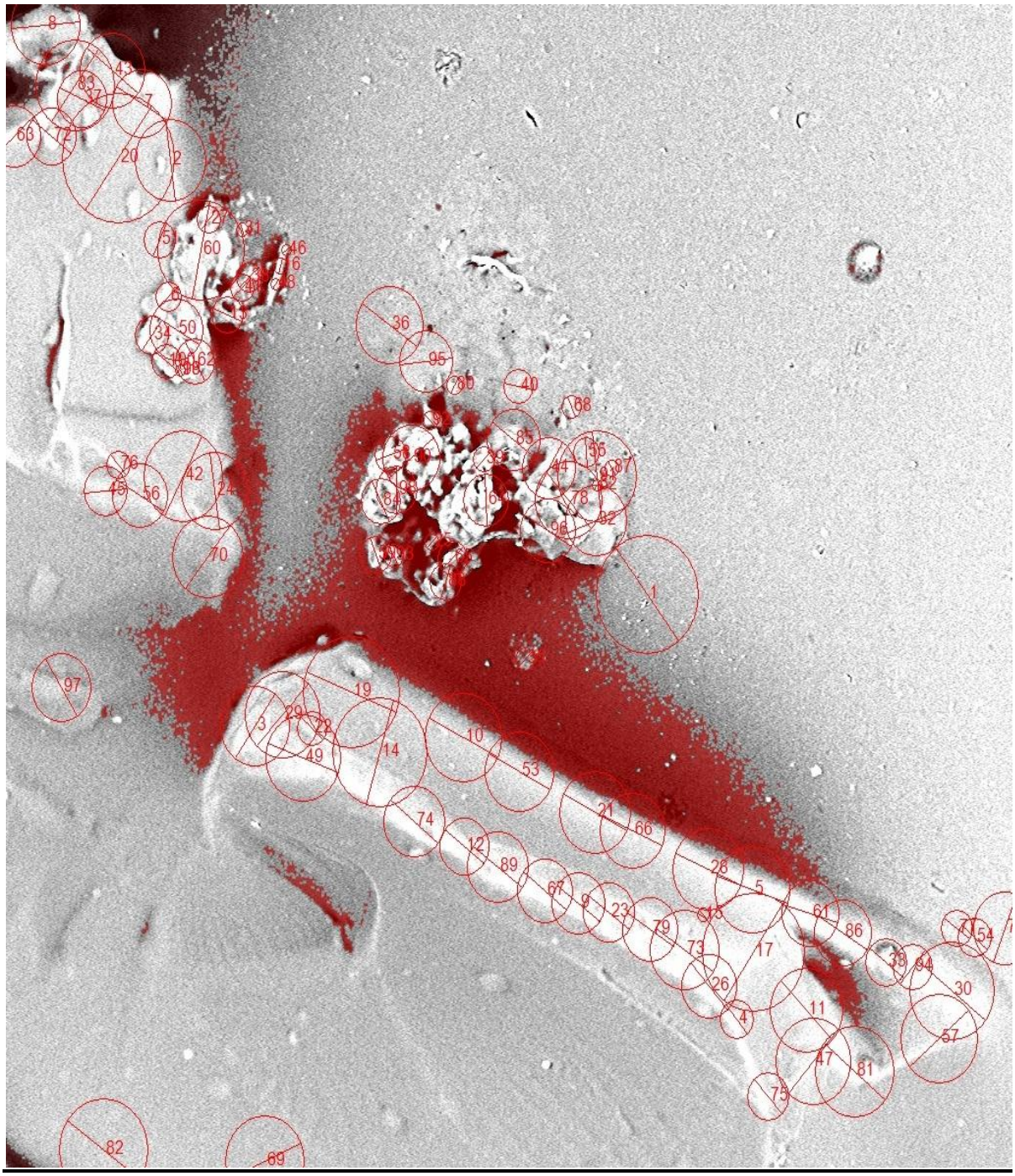


Figure 4.8b: Fibre and Pore Measurement *Celtis zenkeri* (CZ gum)

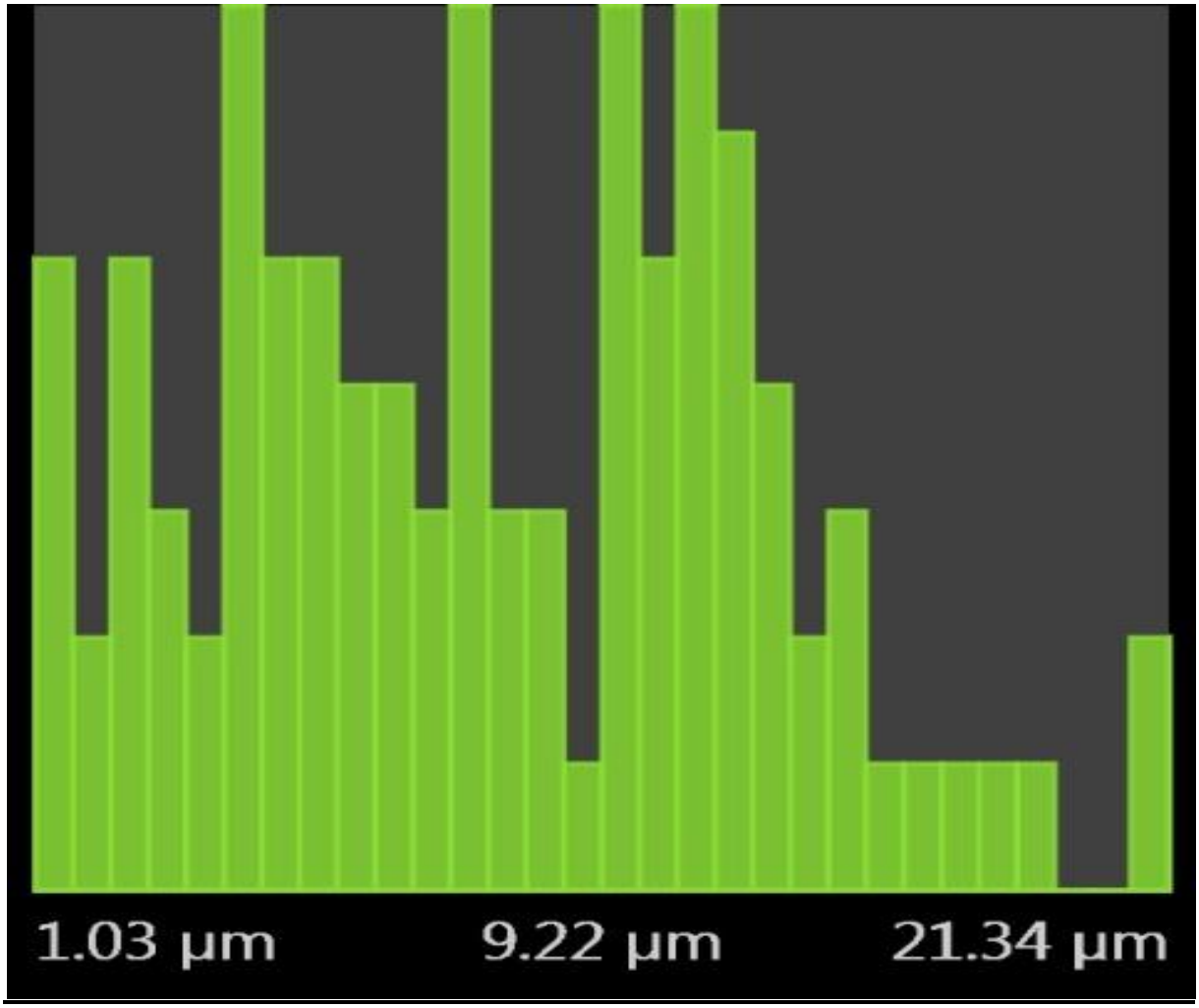


Figure 4.8c: Fibre Histogram of *Celtis zenkeri*

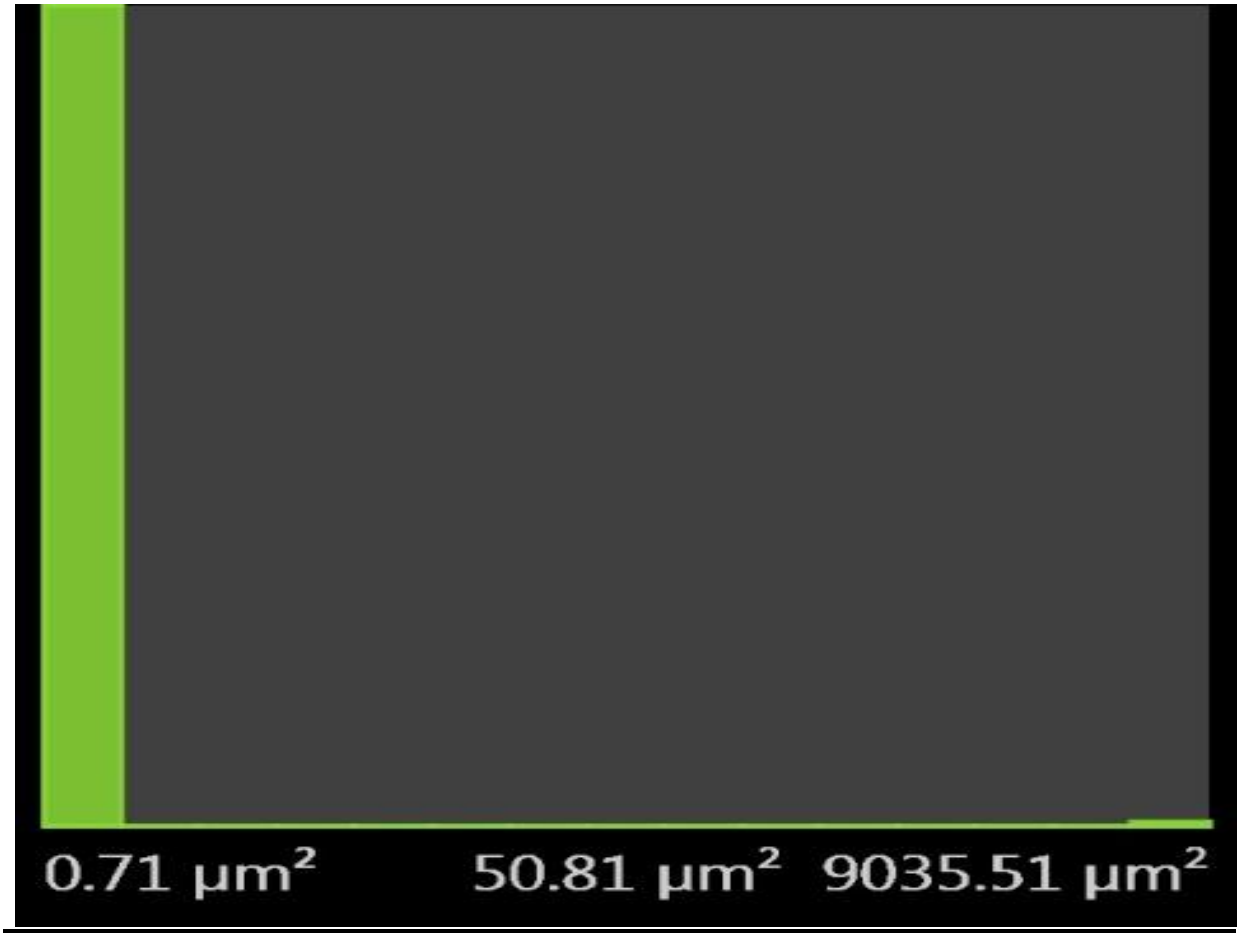


Figure 4.8d: Pores Histogram of *Celtis zenkeri*

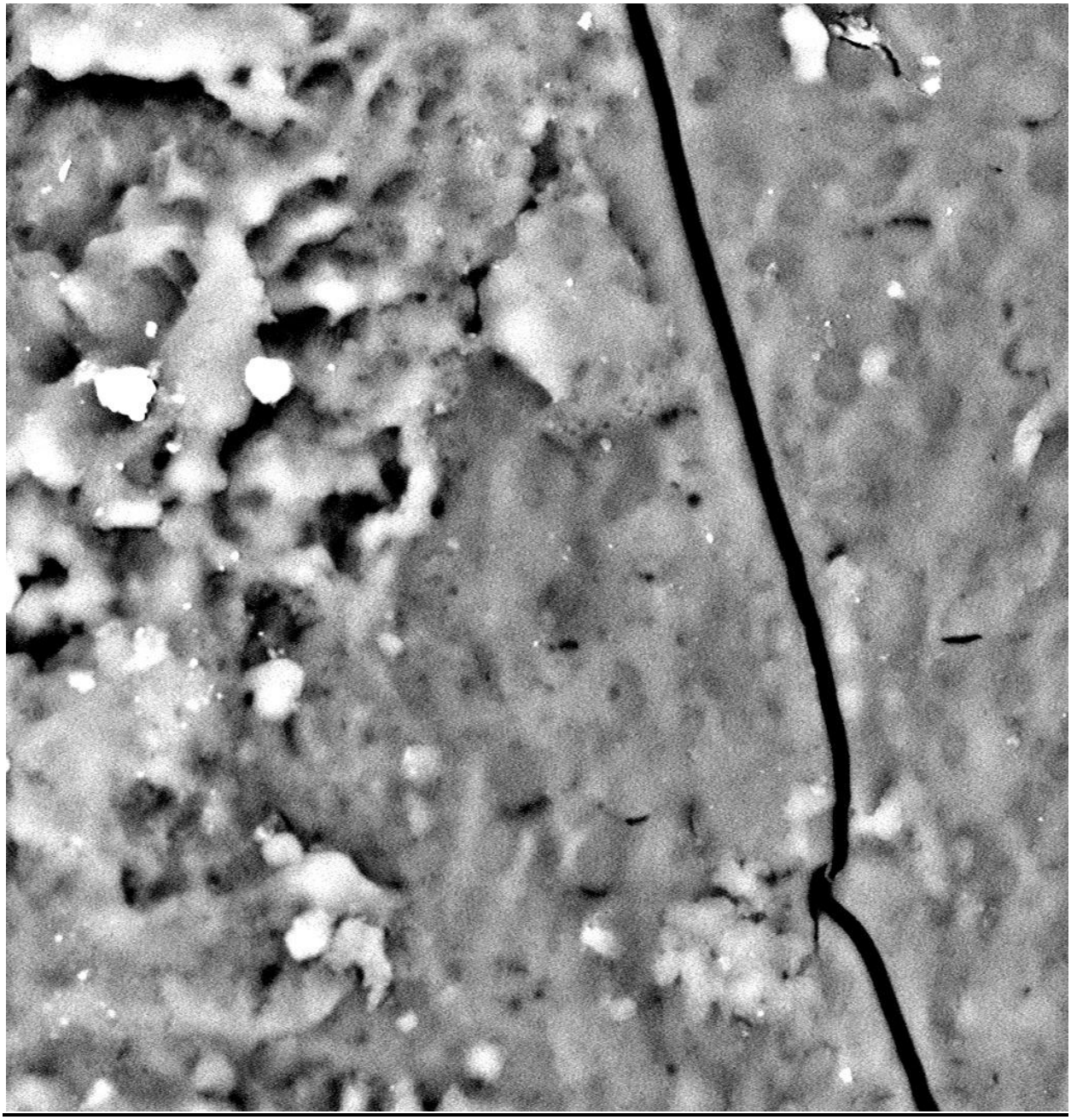


Figure 4.9a: ***Irviginia gabonensis*** **Fibre and Pore Measurement Surface Area of Analysis**

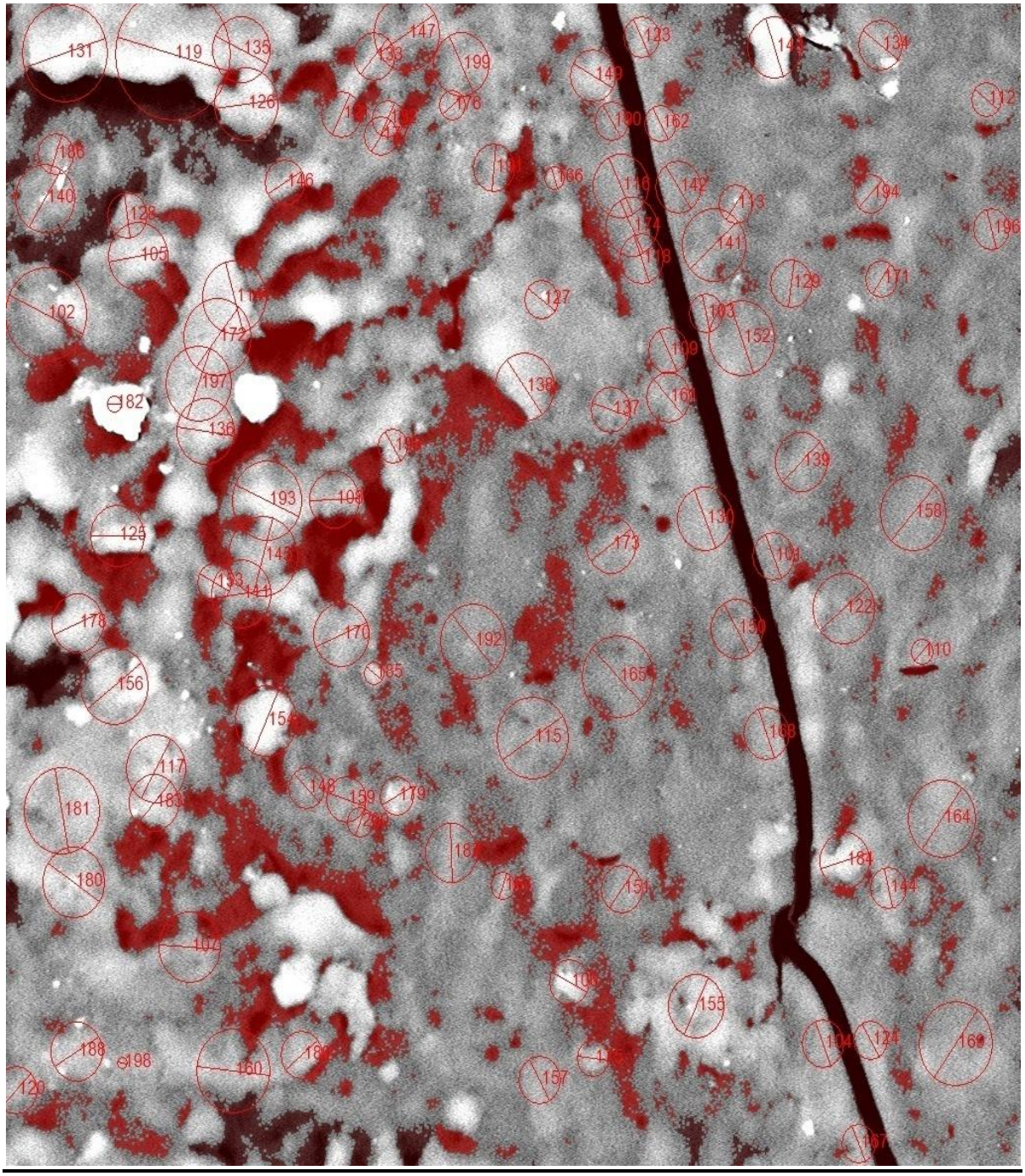


Figure 4.9b: Fibre and Pores Measurement of *Irvingia gabonensis* (IG Gum)

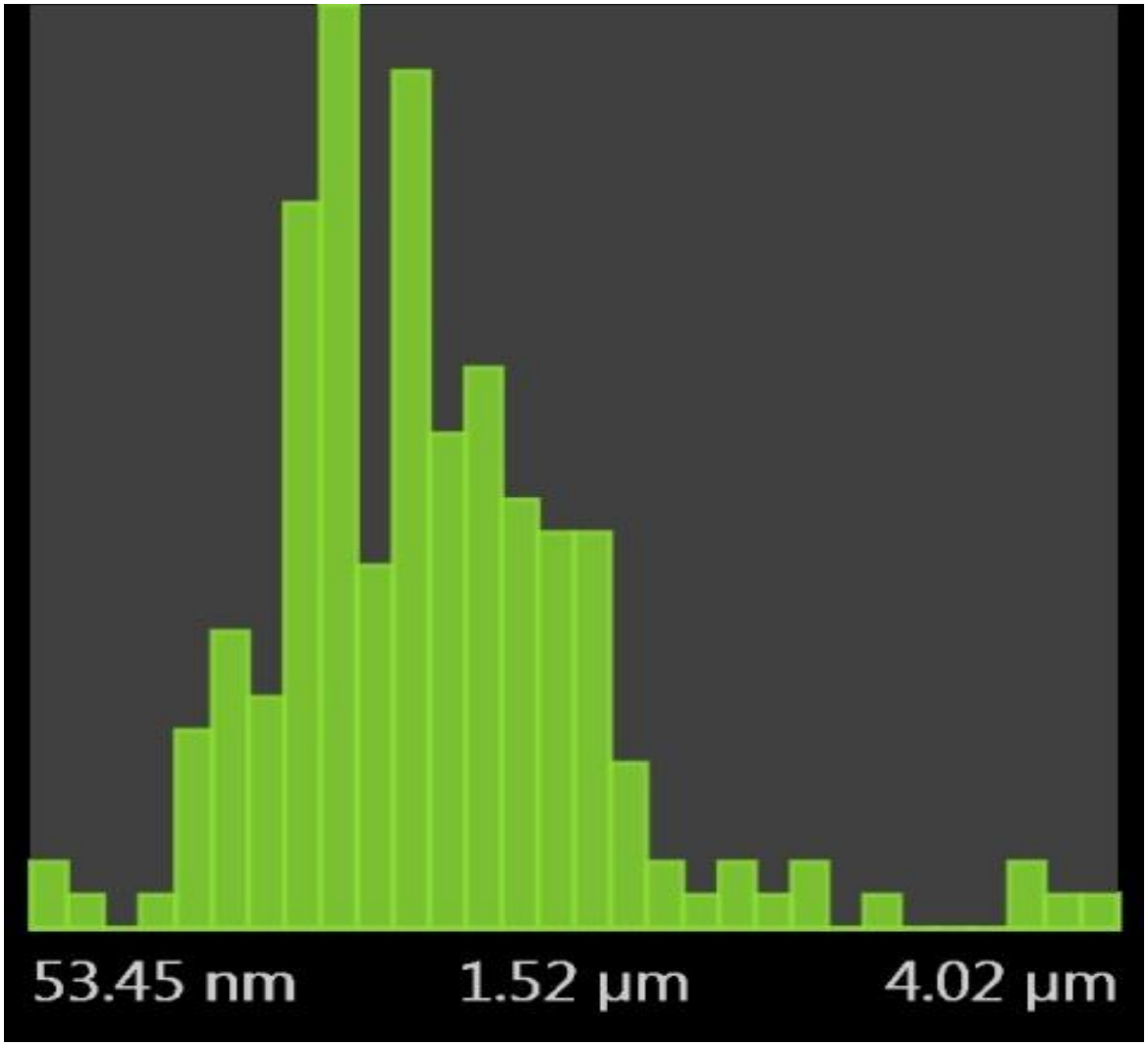


Figure 4.9c: Fibre Histogram of *Irviginia gabonensis*

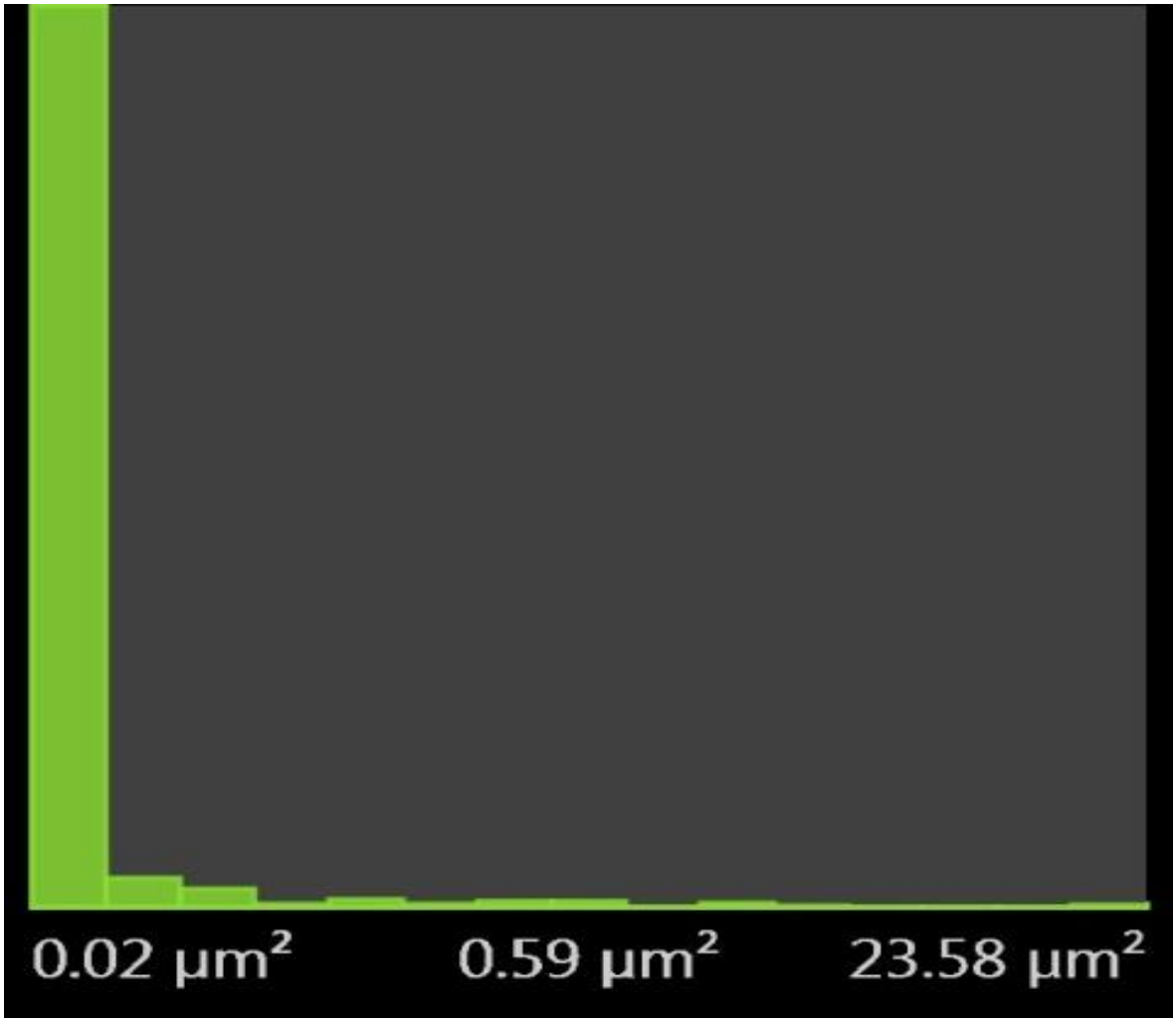


Figure 4.9d: **Pore Histogram of *Irviginia gabonensis***

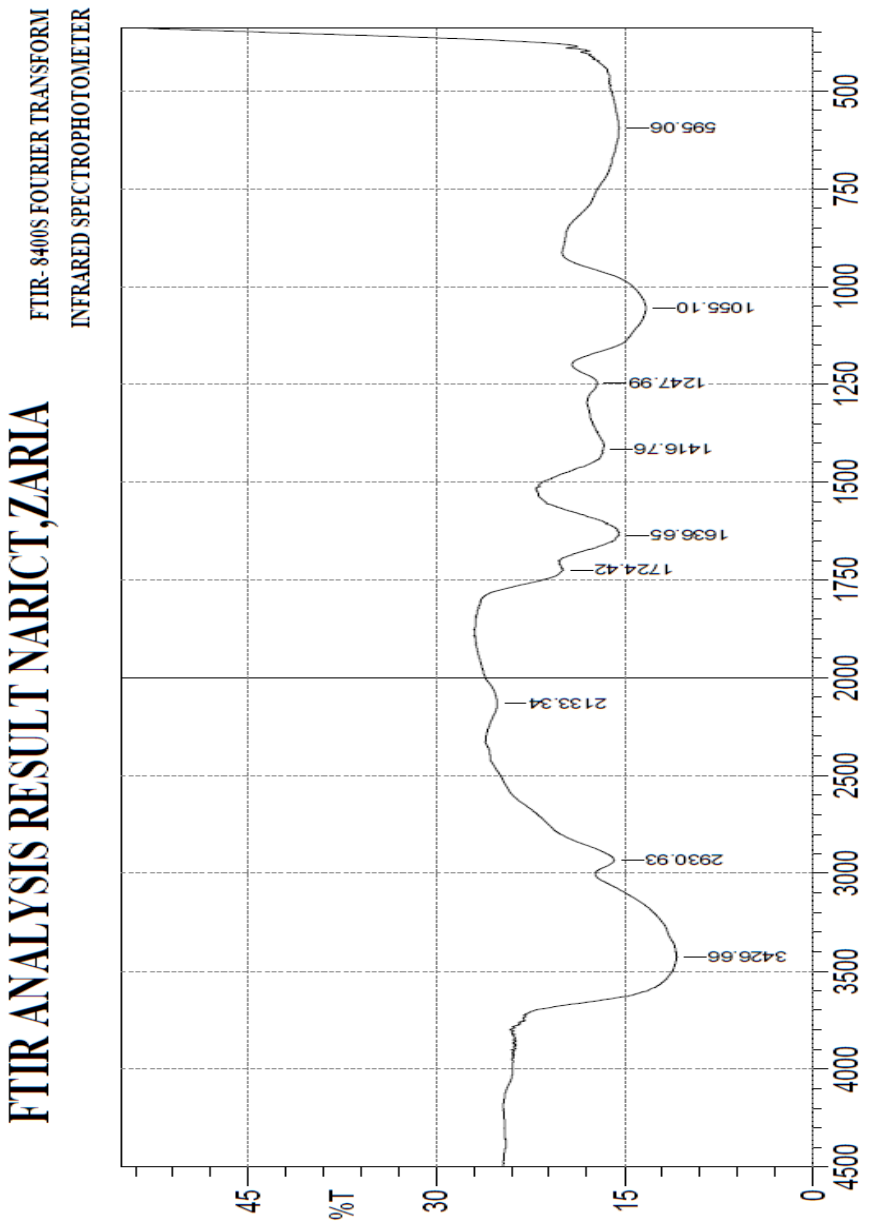


Figure 4.10: **FTIR Spectrum of *Irviginia gabonensis* Gum**

Table 4.3: Peak, Frequency and Assignment of FTIR Absorption Bands by *Irvingia gabonensis (IG)* Gum

S/no	Peak	Intensity	Area	Assignment/Functional Group
1	595.06	15.397	8.616	C-I Stretch/ Iodo derivative
2	1055.1	13.269	231.762	S=O Stretch/ Sulfoxide
3	1247.99	17.166	71.445	C-O Stretch/ Methylene acetal
4	1416.76	16.693	5.993	C-N Stretch/ Primary amide
5	1636.65	15.376	10.128	C=C Stretch/ Terminal olefin
6	1724.42	19.871	6.74	C=O Stretch/ Ketone
7	2133.34	25.119	15.04	N≡N Stretch/ Azides
8	2930.93	15.823	402.274	C-H Asymmetric Stretch/ Methylene
9	3426.66	10.815	7.451	O-H Stretch/ intermolecular bonded OH

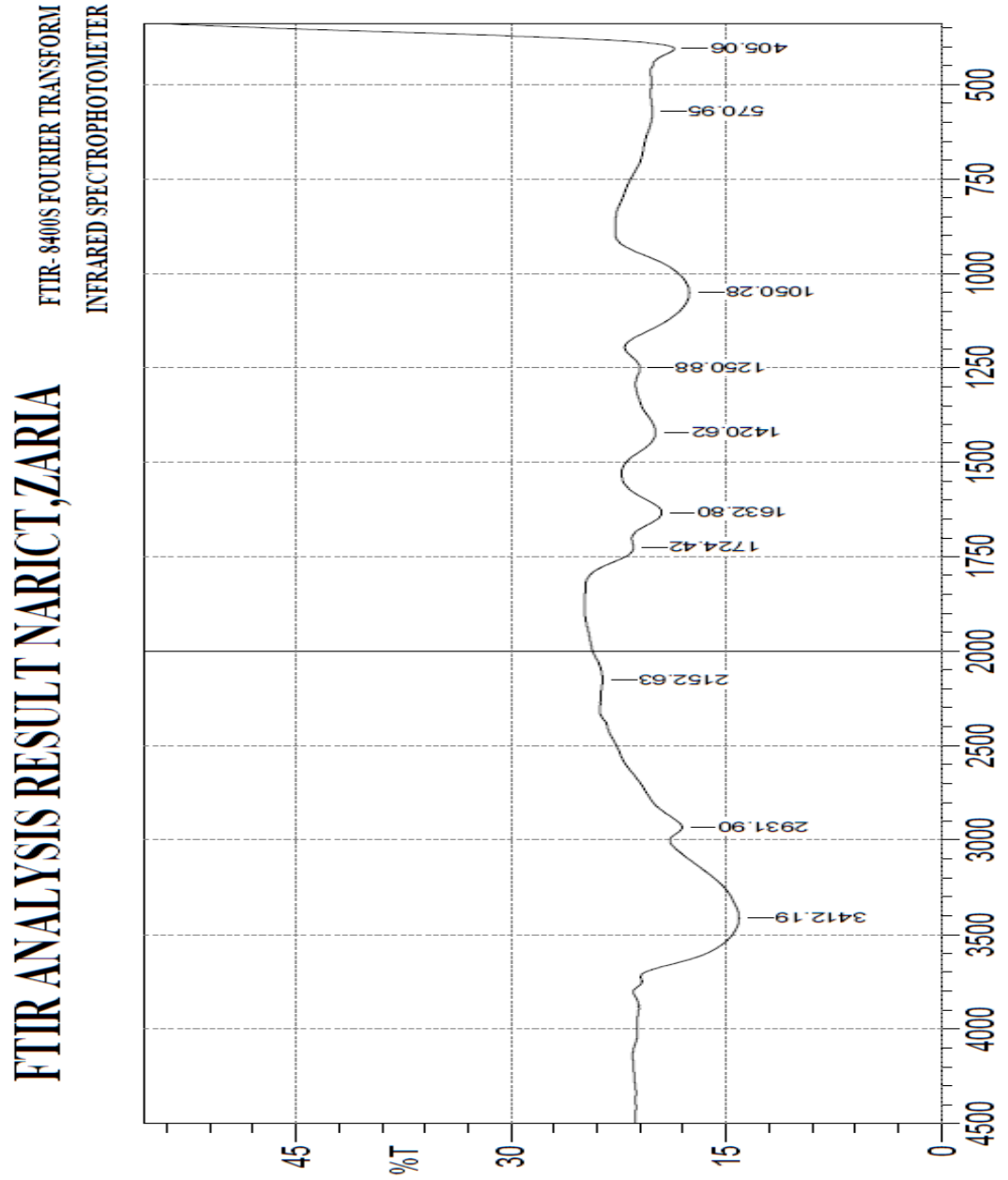


Figure 4.11: FTIR Spectrum of C*eltis zenkeri* Gum

Table 4.4 Peak, Frequency and Assignment of FTIR Absorption Bands by *Celtis zenkeri (*CZ*)* Gum

S/no	Peak	Intensity	Area	Assignment/Functional Group
1	405.06	18.618	62.004	
2	570.95	20.17	246.244	C-I Stretch/ Iodo derivative
3	1050.28	17.561	216.278	S=O Stretching/ Sulfoxide
4	1250.88	21.031	65.279	C-O Stretch/ Methylene acetal
5	1420.62	19.921	162.52	C-H Bending/ Inplane bend/ C=CH2
6	1632.8	19.511	113.562	C=C Stretch/ Terminal olefin
7	1724.42	21.481	77.657	C=O Stretch/ Ketone
8	2152.63	23.639	223.099	N≡N Stretch/ Azide
9	2931.9	18.087	463.328	C-H Asymmetric Stretch/ Methylene
10	3412.19	14.098	247.129	O-H Stretch/ Intermolecular bonded OH

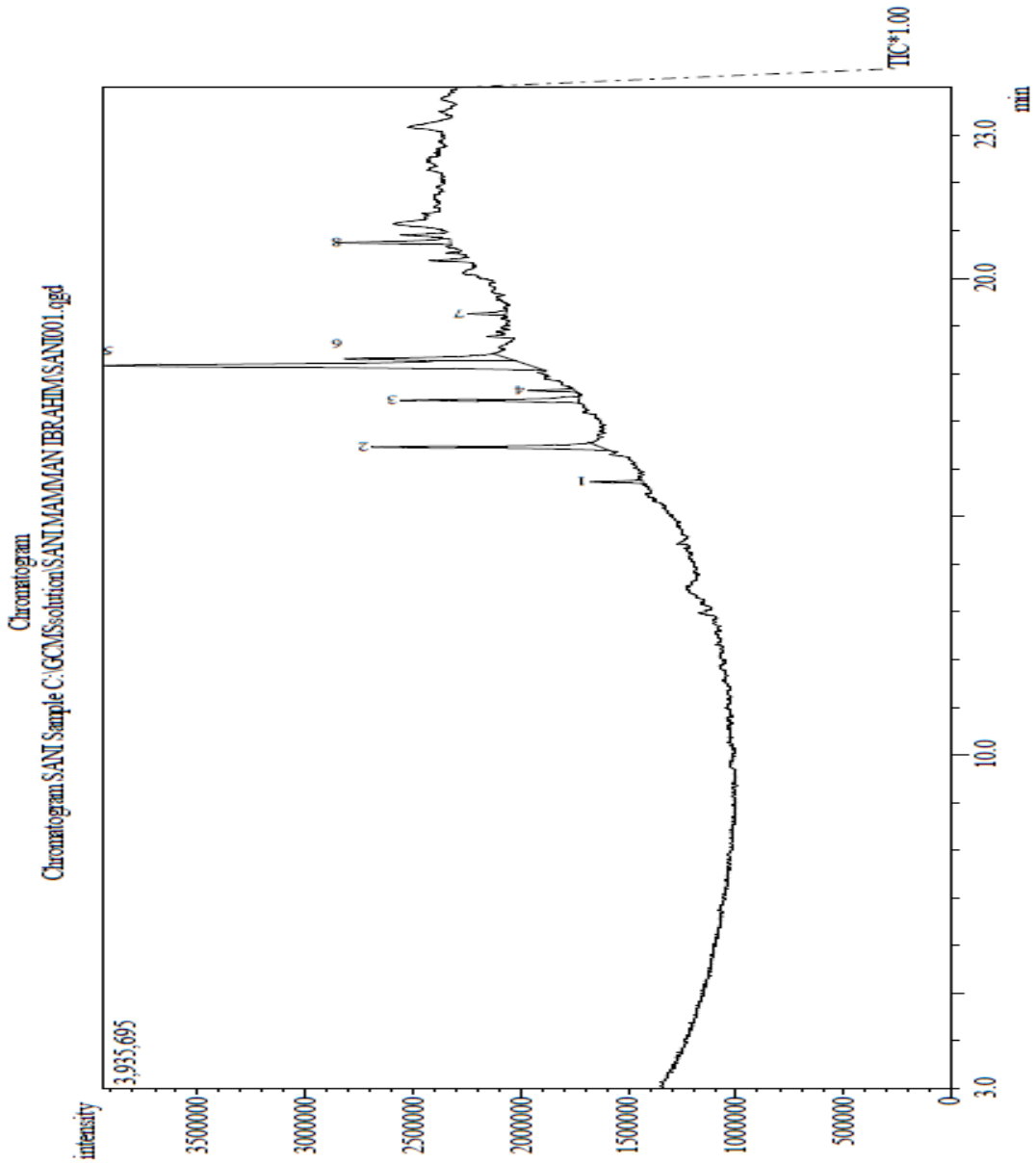


Figure 4.12: Chromatograph of *Irviginia gabonensis* Gum

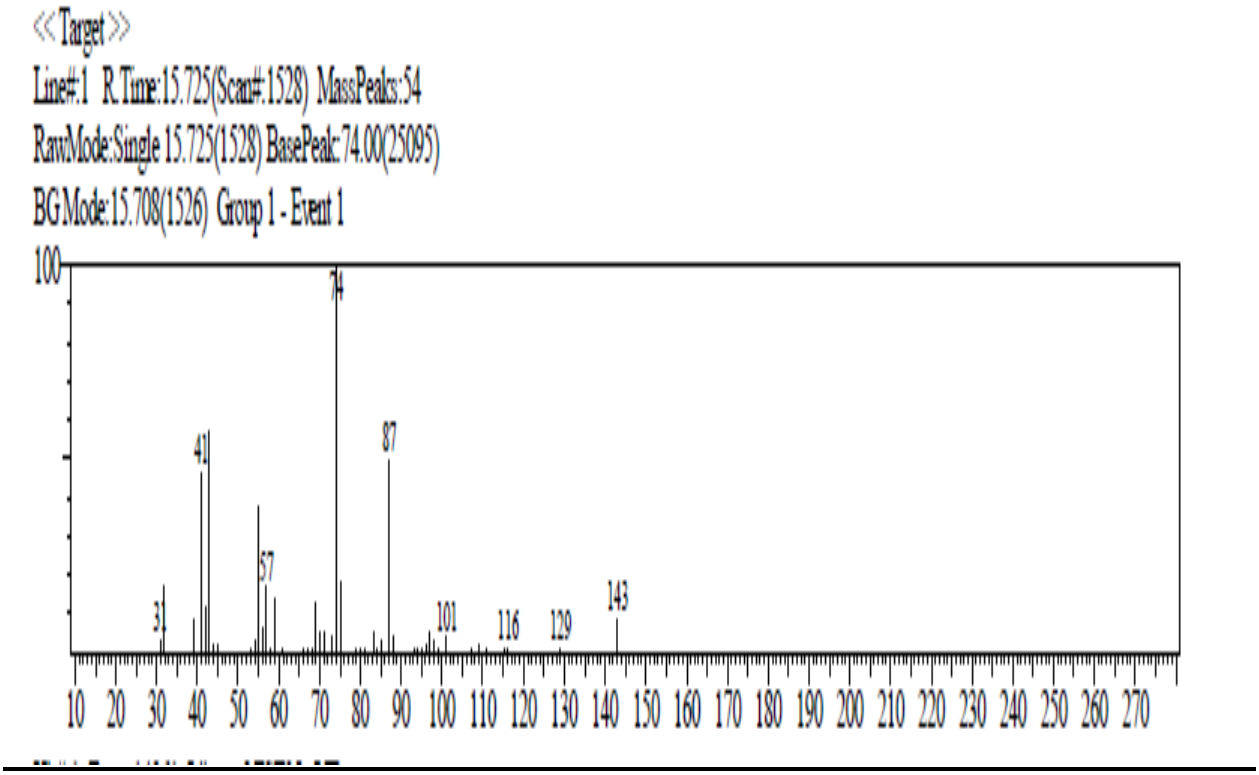


Figure 4.12a: Line 1 Spectrum of *Irviginia gabonensis* Gum

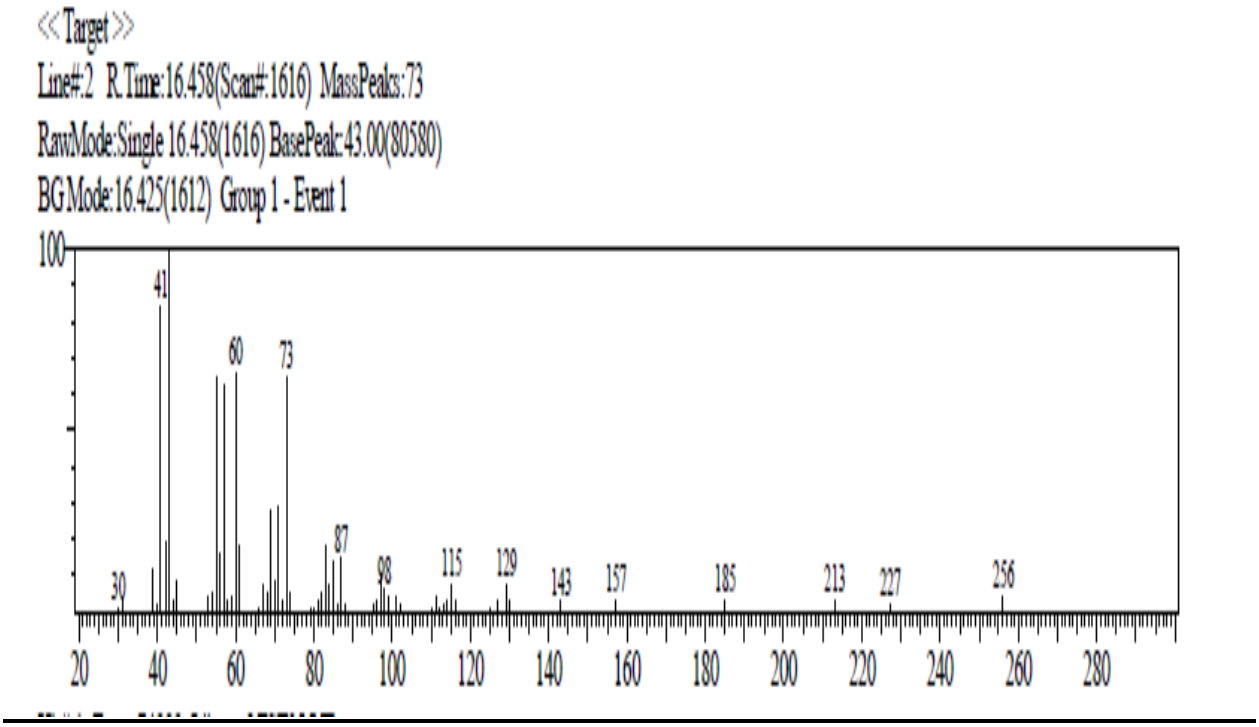


Figure 4.12b: Line 2 Spectrum of *Irviginia gabonensis* Gum

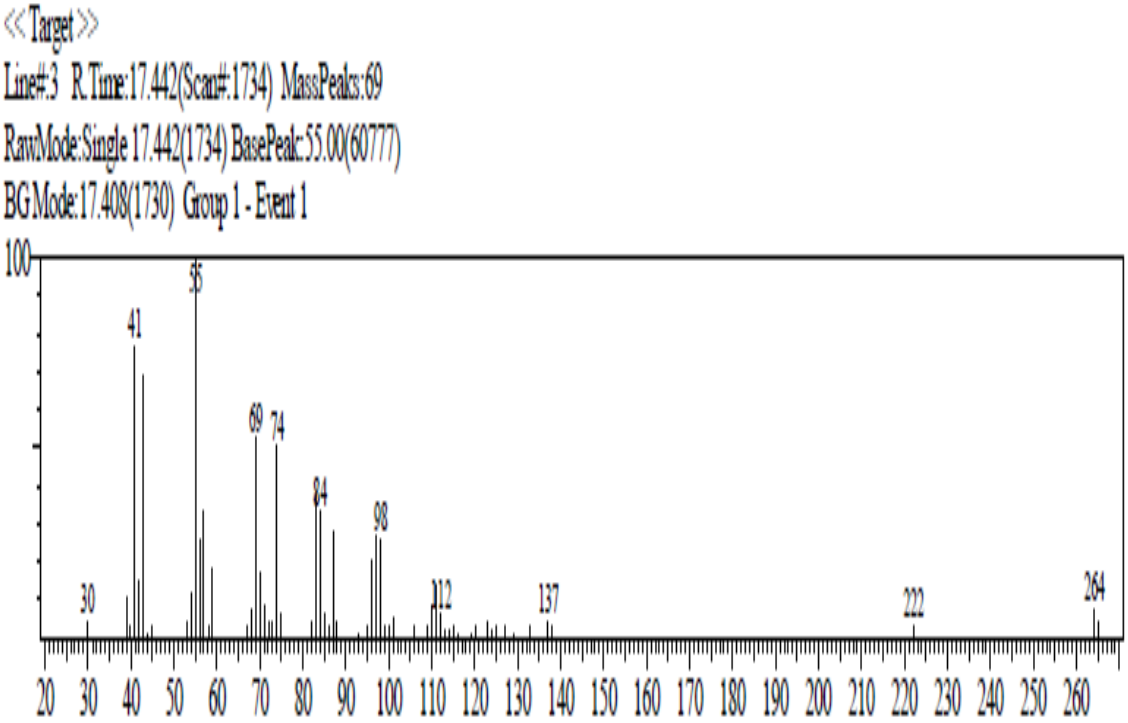


Figure 4.12c: Line 3 Spectrum of *Irviginia gabonensis* Gum

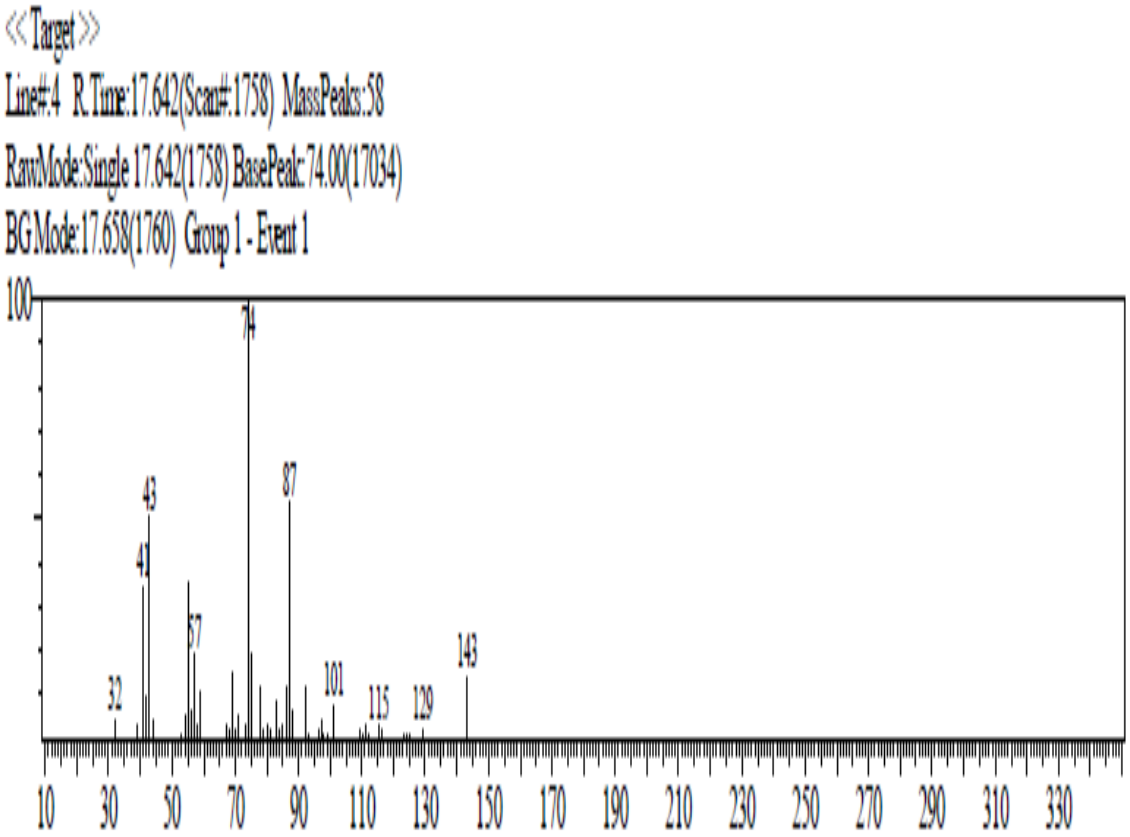


Figure 4.12d: **Line 4 Spectrum of *Irviginia gabonensis* Gum**

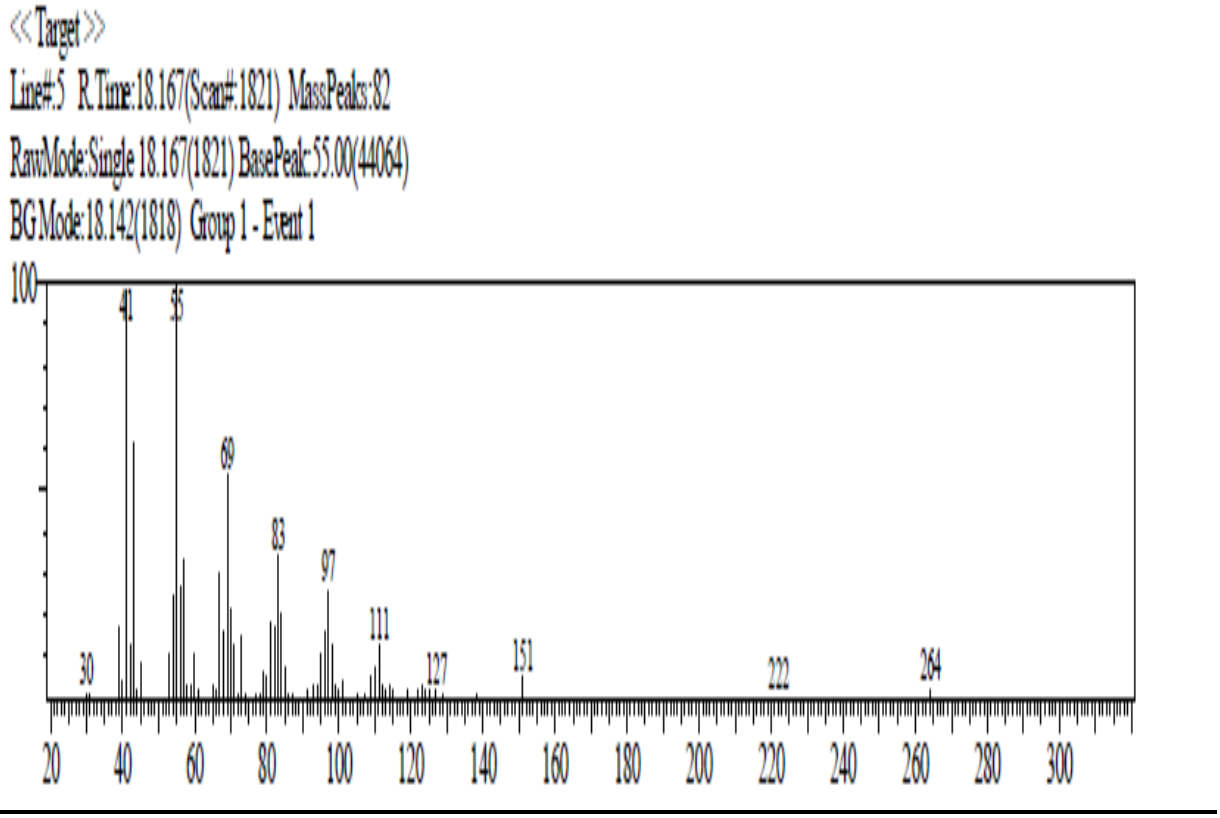


Figure 4.12e: Line 5 Spectrum of *Irviginia gabonensis* Gum

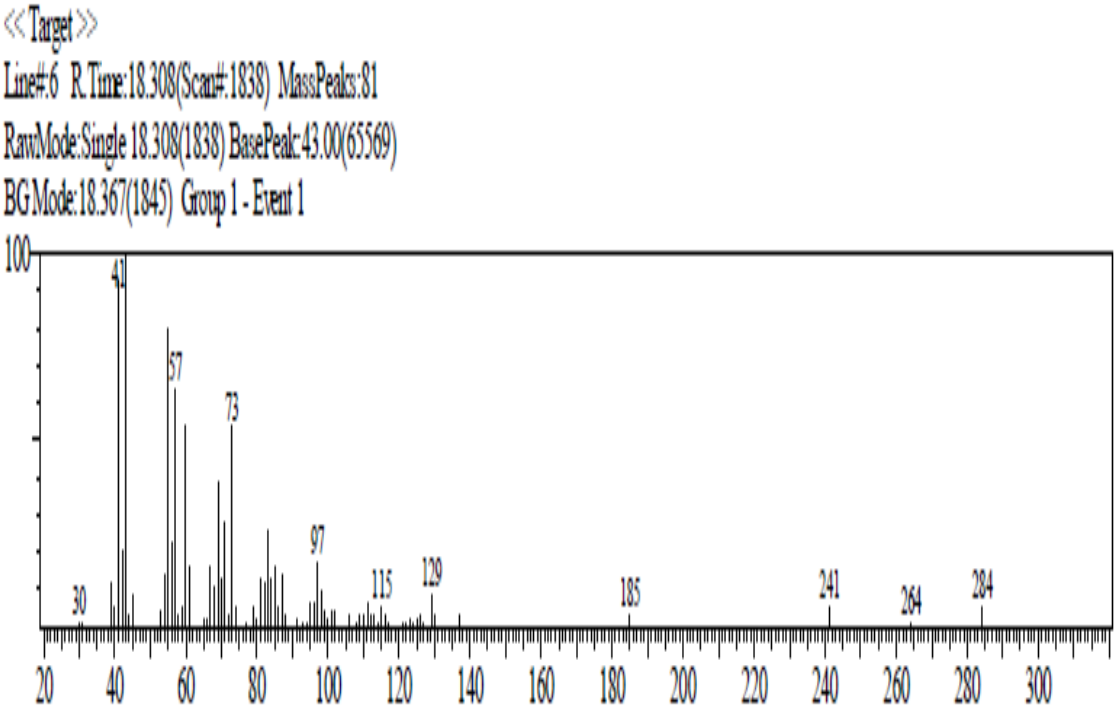


Figure 4.12f: **Line 6 Spectrum of *Irviginia gabonensis* Gum**

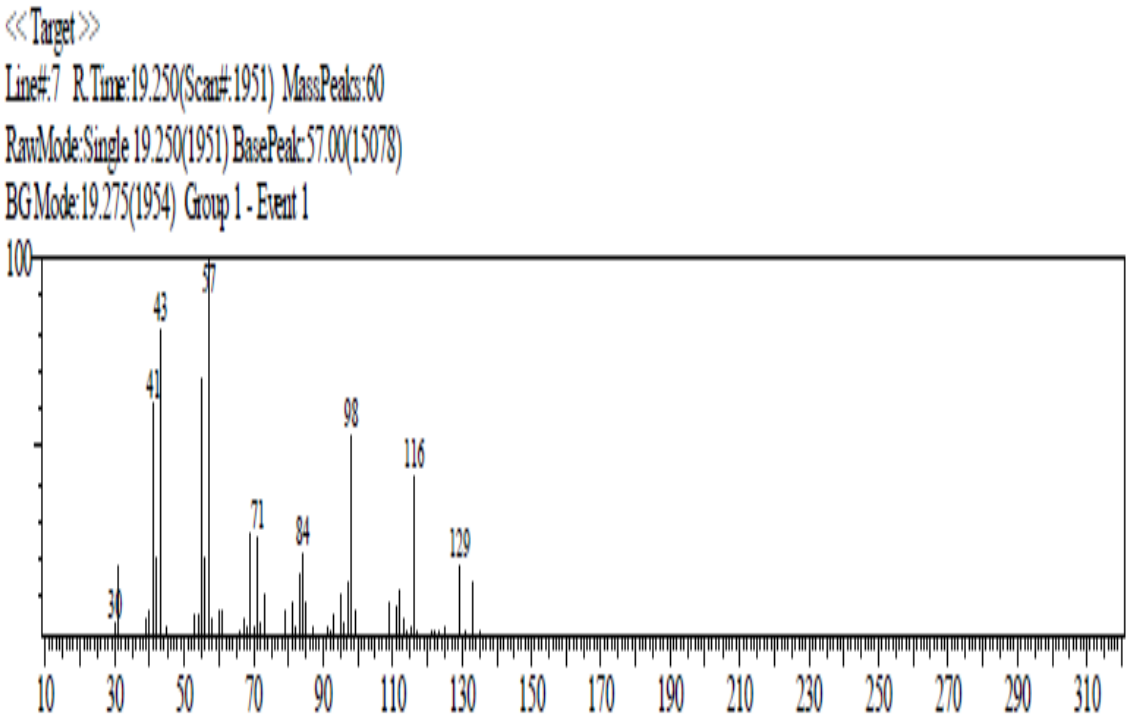


Figure 4.12g: Line 7 Spectrum of *Irviginia gabonensis* Gum

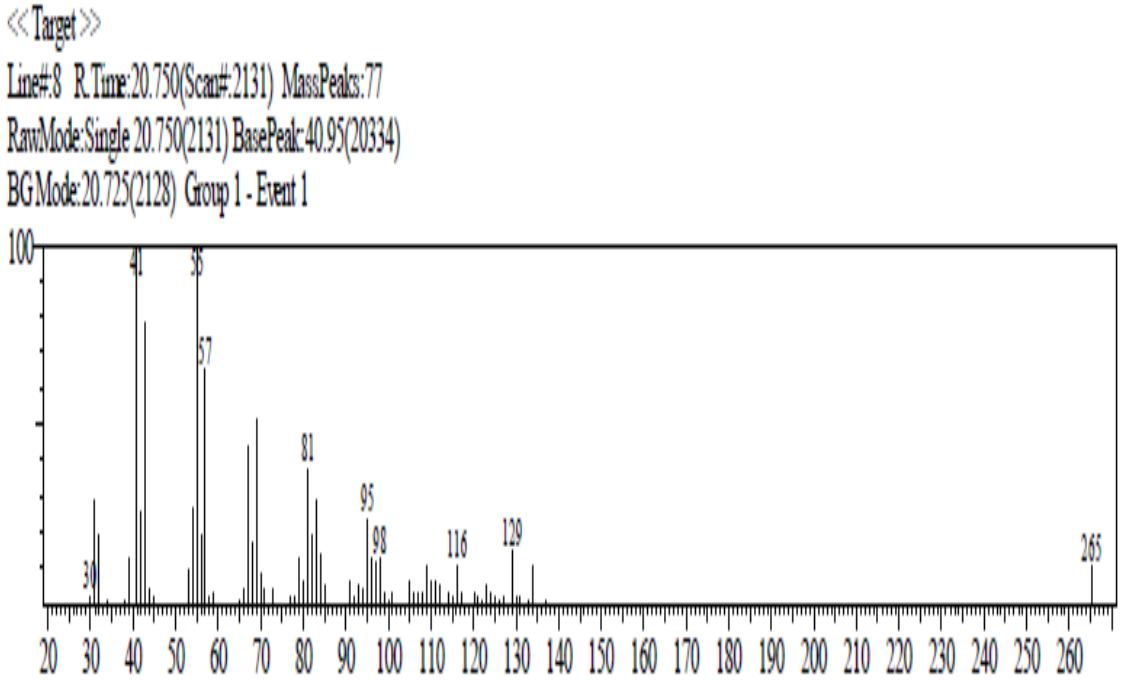


Figure 4.12h: Line 8 Spectrum of *Irviginia gabonensis* Gum

Table 4.5: Analytical Parameters Deduced from GCMS Spectra of *Irviginia gabonensis* Gum Exudates

Line no.	Mass peak	Base peak	Retention time	% Concentration
1	54	74.00	15.725	2.28
2	73	43.00	16.454	15.47
3	69	55.00	17.44	11
4	58	74.00	17.644	2.01
5	82	55.00	18.165	47.09
6	81	43.00	18.306	13.21
7	60	57.00	19.251	2
8	77	40.95	20.743	6.93

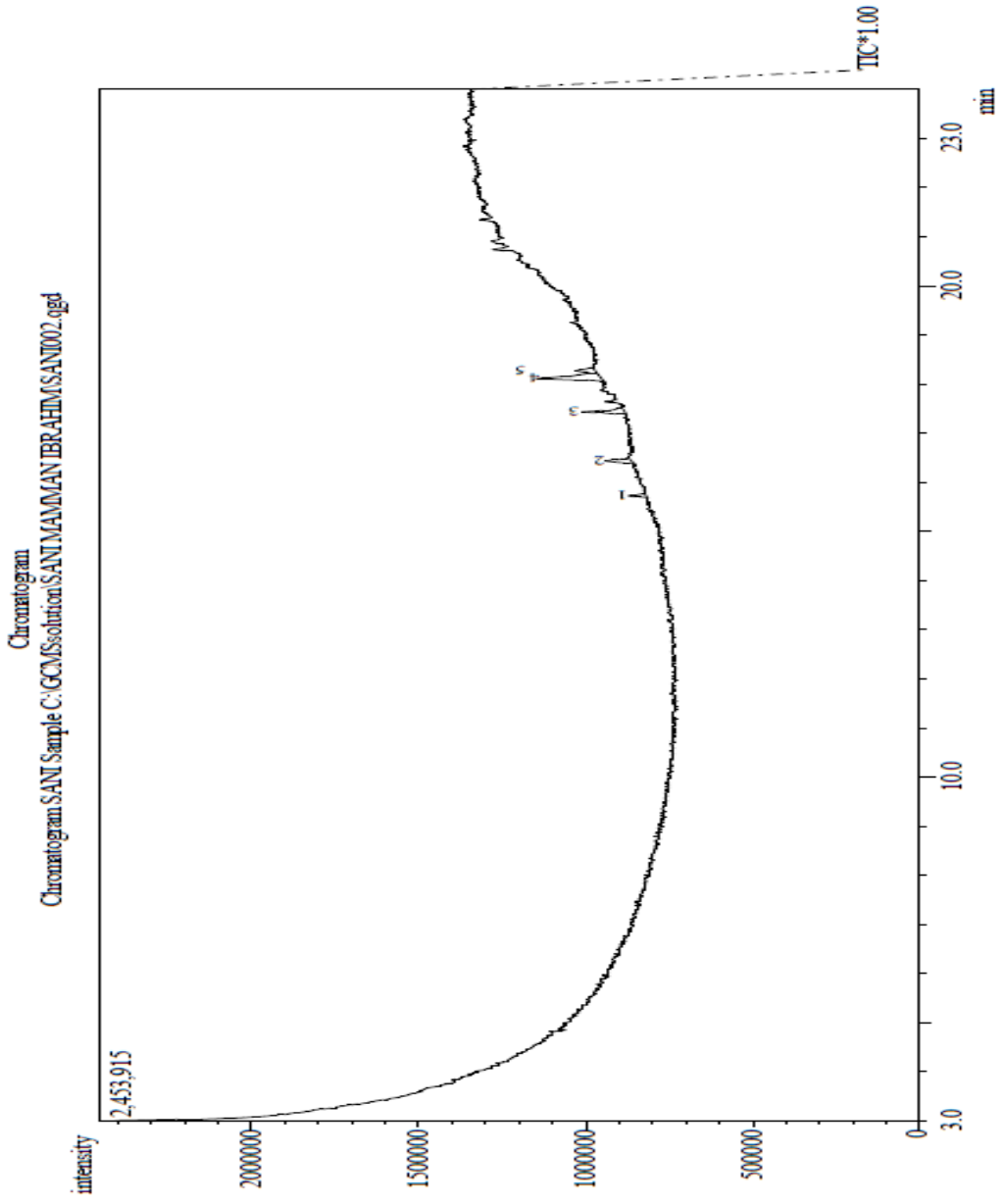


Figure 4.13: Chromatograph of *Celtis zenkeri* Gum

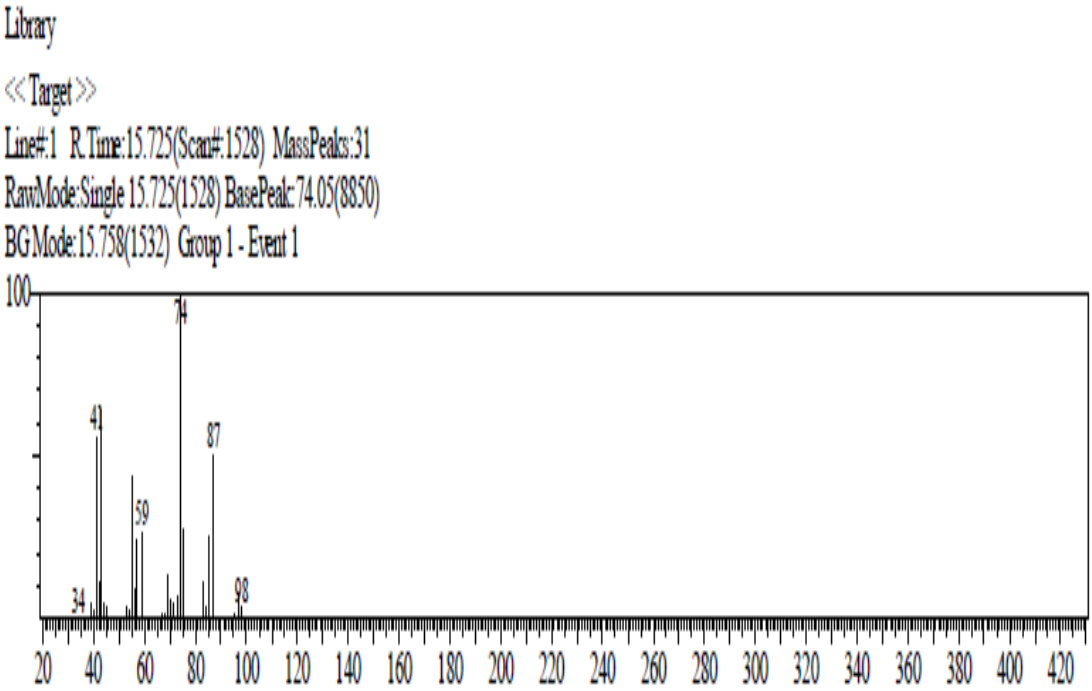


Figure 4.13a: Line 1 Spectrum of *Celtis zenkeri* Gum

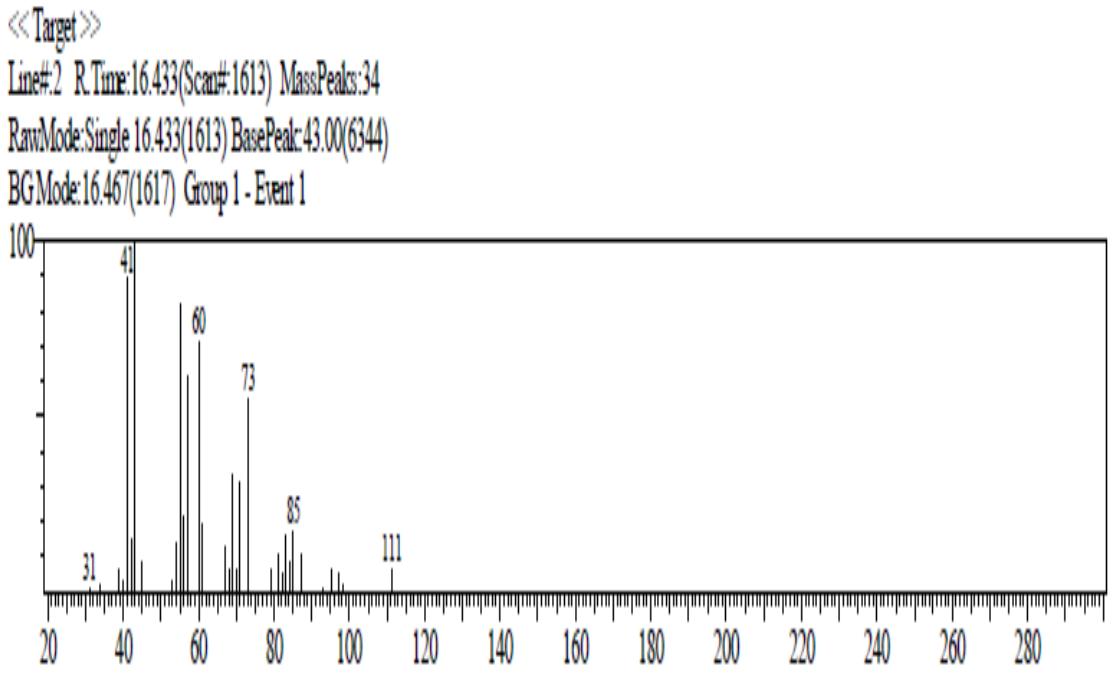


Figure 4.13b: Line 2 Spectrum of *Celtis zenkeri* Gum

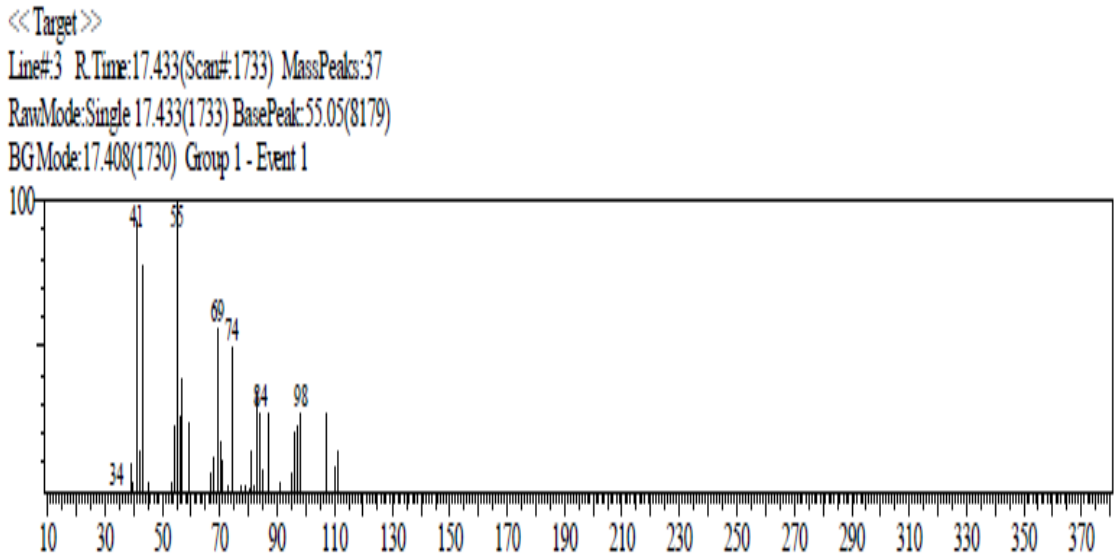


Figure 4.13c: Line 3 Spectrum of *Celtis zenkeri* Gum

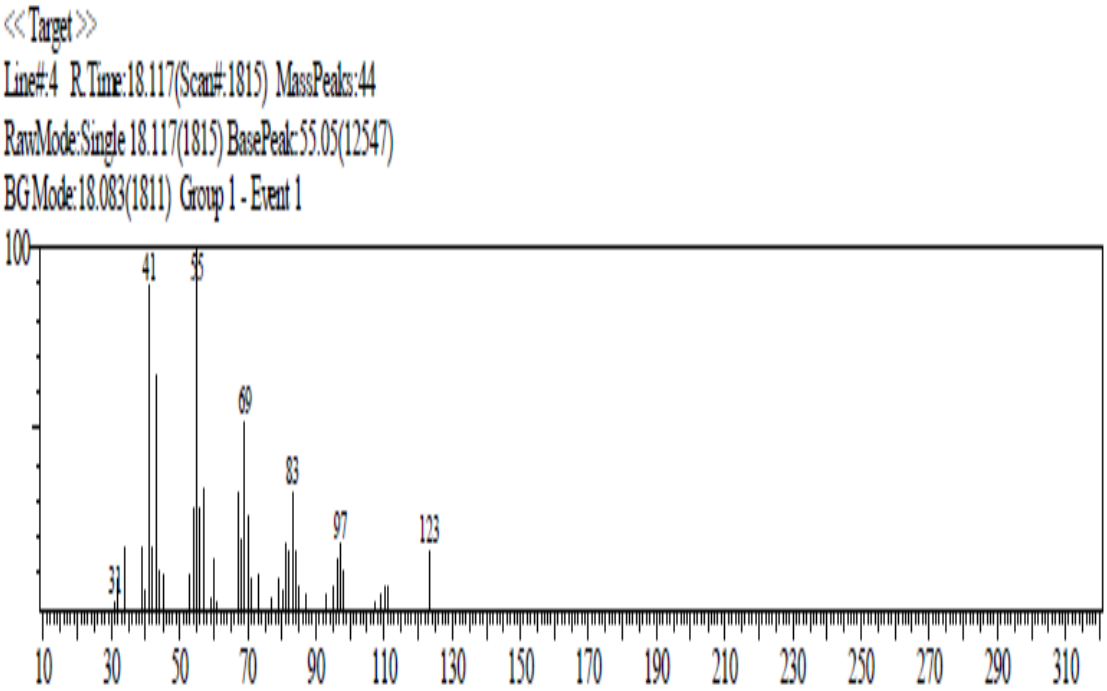


Figure 4.13d: Line 4 Spectrum of *Celtis zenkeri* Gum

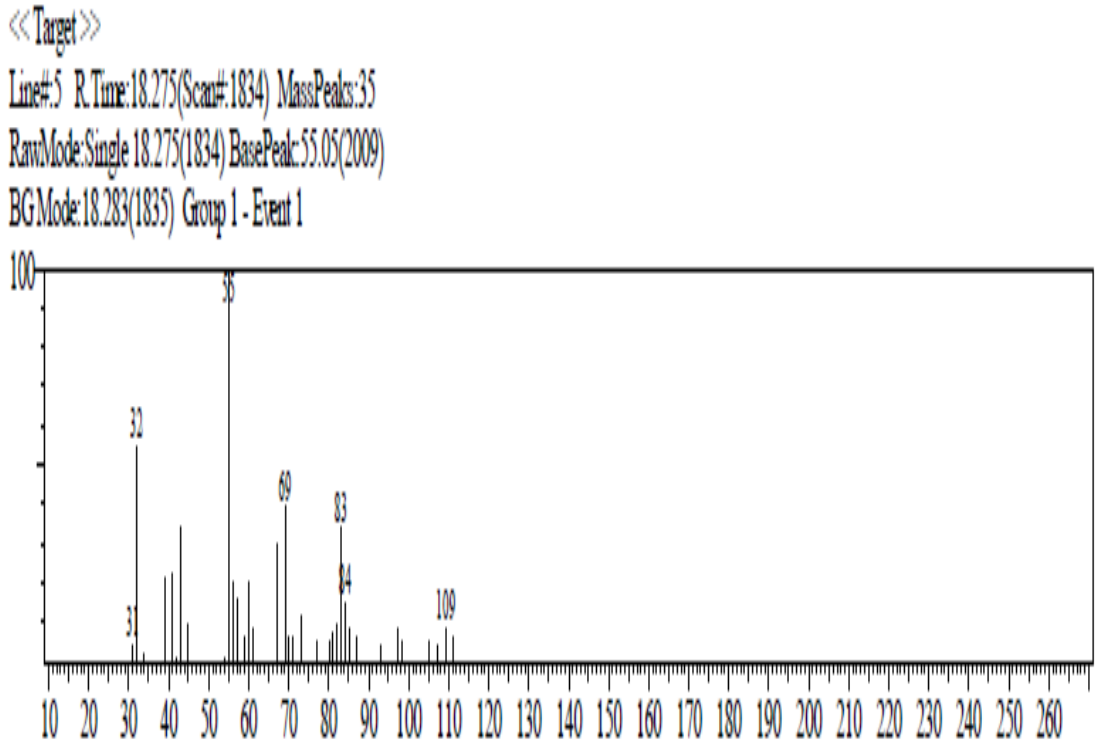


Figure 4.13e: Line 5 Spectrum of *Celtis zenkeri* Gum

Table 4.6: Analytical Parameters Deduced from GCMS Spectra of *Celtis zenkeri* Gum Exudates

Line no.	Mass peak	Base peak	Retention time	% Concentration
1	31	74.05	15.723	6.71
2	34	43.00	16.43	13.36
3	37	55.05	17.434	21.87
4	44	55.05	18.111	47.46
5	35	55.05	18.267	10.61

CHAPTER FIVE

5.0 DISCUSSION OF RESULTS

5.1 Physicochemical Parameters

Physicochemical parameters of *Irviginia gabonensis* (IG), *Celtis zenkeri* (CZ) *and Acacia senegal (*AS*)* gum exudates are presented in Table 4.1. The results obtained revealed that the colour of IG, CZ and AS gums (Plates 4.1a-b) were brownish, brownish yellow and yellow respectively. Also, the gums displayed odourless and tasteless characteristics. Upon purification, the yields for the gums were relatively high ranging from 86.60 % for CZ gum to 90.50 % for CZ gum.

At the measured room temperature (27 ^{0}C), the pH values of IG, CZ and AS gums were 5.42, 4.61 and 5.01 respectively. This indicates that the gums are acidic and the degree of acidity tends to vary from one gum to another. All the gums studied were found to be soluble in water but insoluble in ethanol. This also indicates that the gums are ionic. As a rule, ionic compounds are soluble in water and other solvents that have high dielectric constants. The solubility of IG, CZ and AS gums in water was found to increase with increase in temperature. The observed increase in solubility with temperature indicate that the heat given off in dissolving the gum is less than the heat required to break the gum apart. The net dissolution reaction is endothermic (energy required). Therefore, addition of more heat facilitated the dissolution of the gum by providing energy to break bonds within the gums. On the other hand, chloroform and acetone are non-polar solvents and as expected, non-polar compounds are soluble in non-polar solvents and vice versa. It has been found that the solubility of some ionic compounds vary in this manner with temperature due to changes in properties and structure of liquid water. An increase in temperature can increase the degree of solute-solvent interaction resulting in an increase in solubility.

The measured conductivities of IG, CZ and AS gums (123.6, 155 and185 µS/cm respectively) were relatively high and comparable with those of ionic compounds (Rouxel, 2011). However, the three gums were found to exhibit low salinity values indicating that the conductivity of the gums may not be primarily due to the presence of ions but due to movement of charges within the colloidal system (Zadeh *et al*., 2007).

Measured values of turbidity for IG, CZ and AS gums were 79, 68 and 25 FAU respectively. These results indicate that the ability of the studied gums to scatter light follows the order AS>IG>CZ. It is interesting to note that CZ gum is slightly turbid because of weak light scattering while IG is highly turbid because of strong light scattering. Turbidity is related to light scattering according to Dror *et al*. (2006) and according to Pablyana *et al*. (2007), increasing values of turbidity implies higher amount of insoluble contents in the polysaccharides while Yadav *et al*. (2008) also related turbidity as a property that increases with increasing emulsifying property of a polymer. The densities of the three gums indicate that the aggregate of particles or mass per volume of IG gum is higher than that of the AS gum, while that of the CZ gum is lower than the AS gum.

5.2. Effect of Increase in Temperature and Concentration of the Gum on Viscosity Value

Figures 4.1 and 4.2 present plots for the variation of relative and specific viscosity with variable temperatures and concentrations for IG, AS and CZ gums. It is evident from the Figures (4.1a and 4.1b) that the relative and specific viscosity of the gums tend to increase with increase in concentration of the gum but the viscosity decreased with increase in temperature of the gum (Figures 4.2a and 4.2b). The trend observed for the gums can be explained as follows. At low temperature values, the electrostatic repulsions between the gum particles is low but at higher temperature the repulsive force is high thereby reducing the viscosity, while on the other hand

the increase in viscosity due to increase in concentration of the gums is as a result of increase in association but at lower concentration there is decrease in association due to possible formation of hydrogen bonds, Van der Waals and other weak forces. Therefore increase in concentration of the pure gums led to an increase in their specific viscosity, with *Acacia senegal* gum (AS gum) having the highest value of specific viscosity value and *Celtis zenkeri* gum (CZ gum) being the least.

5.3 Effects of Electrolytes on the Viscosity of the Gum

Exudate gums are acid polysaccharides containing various metal ions as neutralized cations. From the results of metal composition of the studied gums (Table 4.2), the major constituents of the studied gums are Mn, Fe, Zn, Mg, Ca, Cu, Ni, Pb and Cd. It has been found that due to their metal content, exudate gums behave as polyelectrolyte (De Paula *et al*., 2001). Therefore, the viscosity of the gums can be affected by the addition of other electrolytes. In this study, effects of KCl, KBr and $AlCl_3$ on the viscosity of IG, CZ and AS gums were investigated. Figures 4.3a, 4.3b and 4.3c show the variation of viscosity in IG, CZ and AS gums with concentrations in the presence of 0.1 M of KCl, KBr and $AlCl_3$. The electrolytes (KCl, KBr and $AlCl_3$) were found to increase the viscosities of IG, CZ and AS gums such that the magnitude of increase is directly proportional to increase in the charge of the ions (i.e. $Al^{3+}>K^{+}$). The order observed for the effect of K^{+} and Al^{3+} on viscosity of IG, CZ and AS gums can be explained as follows. The KCl and $AlCl_3$ increase the viscosity of IG, CZ and AS gums because the electrolytes (KCl and $AlCl_3$) have more the steric capability of gelling the gums compared to KBr. This capability is less in KBr hence; KCl exhibited the greatest potential to increase the viscosity of the gums, followed by $AlCl_3$. The decrease found in AS and CZ gums in KBr according to De Paula *et al*. (2001) can be attributed to existence of less intermolecular

interaction due to the screening charges and contraction of the macromolecule in the presence of counter ion. It has been found that aluminium has a tendency of establishing strong interaction with macromolecules through intermolecular cross-linking effect. On the other hand, the strength of intermolecular cross-linking effect is lower in K^+ than in Al^{3+}. This explains the order observed for decrease in viscosity in terms of AS and CZ gums. Ahmad *et al*. (1994) found that in dilute solutions, the turbidity of dilute polyelectrolyte increases with increase in the charge of the ions present. In this work, the variation of the viscosities of the studied gums with increasing charge can therefore be attributed to their effect on turbidity. It is also significant to state that the affinity between each of the studied gums and their counter ions depends on the ratio (charge/ionic ratio). Generally, ions with higher charge will have a stronger affinity for the molecular chain of the gum. The charge to ionic radius ratio of K^+ and Al^{3+} are 0.66 and 4.41 respectively hence the expected order for the interaction of the studied gums with metal ions is $Al^{3+}>K^+$, which is in agreement with the findings of this study.

In all the studied gums, it was found that KCl and $AlCl_3$ increased the viscosity of the gum solution by their tendency to form a porous framework, they have the ability to trap many organic compounds in its so-called clathrates (the organic guest), molecules are held in channels formed by interpenetrating helices.

5.4 Metals Composition

Mean values for concentrations of manganese, iron, zinc, lead, magnesium, cadmium, calcium, copper and nickel ions in IG and CZ gums are presented in Table 4.2. The results revealed very high concentrations of magnesium (compared to other metals) in all the studied gums. Calcium is a major element needed by humans for healthy bone and teeth growth. This suggests that if the toxicity of these gums is established, their food value may be harnessed. The

trend observed for the decrease in calcium content of the studied gums is IG >CZ. This shows that IG gum has more calcium contents compare to CZ gum. In comparison, concentrations of calcium in the studied gums are 2315.9mg/kg and 652.20mg/kg which are greater than the range of values (132.05mg/kg – 133.48mg/kg) reported by Yusuf (2011) for *Acacia senegal, Acacia sieberiana and Acacia nilotica* gums.

Significant concentrations of iron were also found in the studied gums. These were 311.10 mg/kg and 375.40 mg/kg in IG and CZ gums respectively. Iron is an essential element needed by humans in protein synthesis and in the formation of some blood cells. Next to iron in terms of importance is zinc. Zinc is an essential mineral that stimulates the activity of about 100 enzymes in the body. It also supports healthy immune system and it is necessary in the synthesis of DNA, essential for wound healing and supports the healthy growth and development of the body during adolescence, childhood and pregnancy. In this study, concentrations of zinc in the studied gums were 41.05 mg/kg and 10.64 mg/kg for IG and CZ gums respectively. But the concentrations were below that of *Acacia senegal* which is the reference gum.

Manganese is another essential element that was found at average concentrations of 146.40 mg/kg and 213.20 mg/kg for IG and CZ gums respectively. Nutritional roles of manganese cannot be overemphasized. It enables the body to utilize vitamins C, B1, biotin as well as choline. It is used in the manufacture of fat, sex hormones and breast milk in females. It is thought to also help neutralize free radicals as well as being of assistance in preventing diabetes and is needed for normal nerve function. Manganese is also indicated in stimulating growth of the connective tissues and is also believed to be of importance in brain functioning.

The concentration of magnesium found in IG gum (2274.20 mg/kg) and in CZ gum (2285.30 mg/kg) are higher compare to the value in *Acacia Senegal* (834.21mg/kg). Magnesium

in humans serves several important functions including contraction and relaxation of muscles, formation of some enzymes, production and transport of energy and in the production of protein; by comparison the study gums will provide these essential element more than the *Acacia senegal*. On the other hand, concentrations of copper in the plant gums were relatively low compared to other essential metals. However, copper is a trace element needed by the body in minute concentration. Copper is required in the formation of haemoglobin, red blood cells as well as bones. Copper helps in the formation of elastin as well as collagen and is therefore necessary for wound healing. Deficiency of copper may lead to increased blood fat levels. Copper is also necessary for the manufacture of the neurotransmitter noradrenaline and for the pigmentation of human's hair and from the analysis copper was not detected in *Acacia senegal*. The concentrations of some heavy metals such as nickel, lead and cadmium were higher in the studied gums compared to *Acacia senegal* gum as reported by Yusuf (2011), hence the heavy metals in the studied gums were above permissible limit set by World Health Organization and Food and Agricultural Organization of the United Nations (FAO and WHO 2012).

5.5 Surface Morphology

Scanning electron microscopy (SEM) is used to record the images of the surface of materials/specimens at a desired position to obtain topographic/morphological picture with better resolution and depth of focus compared to an ordinary optical microscope. In addition it can also be used for elemental, fibre and pores measurement. In application to materials science of polymers and rubbers, SEM study commonly aims at visualization of phase morphology, surface and cross-sectional topography, surface molecular order and elucidation of failure mechanism (Setua *et al*., 2010).

Figures 4.4a, 4.4b and 4.4c present the scanning electron micrographs of *Celtis zenkeri* (CZ) gum at 1000X, 3000X and 5000X magnifications respectively. The figures generally revealed that the particles in the gum have irregular shapes and dimensions which appear porous. At 1000X magnification, the micrograph clearly revealed the existence of some cavities which could enhance its adsorption capacity. Among the studied gums, CZ gum was found to exhibit the highest water swelling capacity, highest solubility in water suggesting that the gum is a hydrogel (Vinod and Sashidhar, 2009). At other magnification such as 3000X and 5000X the amorphous nature of the gum particles are clearly indicative.

Figures 4.5a, 4.5b and 4.5c present the scanning electron micrographs of *Irvingia gabonensis* (IG) gum at 1000X, 3000X and 5000X magnifications respectively. The Figures revealed that the gum consists of aggregates of irregular shapes and dimensions. Existence of molecular slaps or cavities can also be seen at the right side of the micrographs. SEM diagrams for IG gums are presented in Figures 4.6a-c. At a magnification of 1000X, IG gum is seen to have a rough structure with irregularly spaced pores and network (Figure 4.6a). At higher magnifications (3000 and 5000X), it can be seen that each pores contains particles with irregular shapes and dimensions.

5.6 Elemental Analysis

The results for the Scanning electron microscope (SEM) elemental analysis of the gum samples are shown in Figures 4.6 and 4.7 for *Irviginia gabonensis* and *Celtis zenkeri* gum respectively. Six different points in the micrographs were analyzed and the results presented in Figures 4.6a-f and 4.7a-f for *Irviginia gabonensis* and *Celtis zenkeri* gum respectively.

A close examination of the proximate composition of the studied gum reveals that the two gums are dominantly rich in carbohydrate. The high carbohydrate contents of plant gums have been supported by other research findings (Ahmed *et al*., 2009). For example, Amid *et al.,* (2012) found that several studies on some plant-based gums (mainly plant gum exudates and seed gums) have resulted in the identification of valuable natural sources of complex carbohydrate polymers that promote the desired quality, stability, texture and appearance. The plant gum exudates and seed gums are the complex polysaccharides/carbohydrate polymers commonly used as dietary fibres, thickening agents, foaming agents, film, emulsifiers, stabilizers and drug delivery agents. Anderson *et al.,* (1990) have also reported high concentrations of sugar in eleven species of *Acacia* gum exudates.

The importance of nitrogen (hence protein) in gum which is clearly seen in Figures 4.6 and 4.7 cannot be overemphasized. The immune responses, which are important in providing evidence for the safety of food additives, are customarily accredited to the proteinaceous component of food (Akinhanmi *et al*., 2008; Youssef *et al*., 2009). According to Pablyana *et al*. (2007), the presence of protein in polysaccharides can induce inflammatory response to tissue and the response may have a vital role to play in its pharmacological applications.

5.7 Fibre and Pore Measurement

The results of fibre and pores measurements using scanning electron microscope were shown in Figures 4.8a-d and 4.9a-d for *Irviginia gabonensis* and *Celtis zenkeri* respectively, it can be seen that the fibre length of CZ gum (Figure 4.8c) is far greater than that of IG gum (Figure 4.9c), while the pore areas were 0.71µm^2 (Figure 4.8d) and 0.02µm^2 (Figure 4.9d) respectively.

Fibre contacts influence the characteristic dimensions of pores, For a given network porosity, the product of the specific surface and fibre perimeter exhibits a one-to-one relationship with the mean pore height divided by the fibre perimeter; this relationship varies with porosity to yield a unique surface characterizing the relationship among these variables according to Kallmes and Corte (1960) and Miles (1964). Both specific surface area and pore height increase with porosity where as specific surface area is only weakly dependent on porosity, at a porosity greater than 0.9, pore height exhibits its greatest sensitivity to porosity in that region. We can therefore say that when seeking to maximize specific surface area and pore dimensions, primary consideration should be given to the selection of fibre geometries to maximize specific surface area allowing pore height to be subsequently controlled by targeting network porosity. From the histograms of fibre and pores measurements of the studied gums, it can be seen that the fibre length of CZ gum is far greater than that of IG gum reading, given the range of 1.03 µm to 21.34µm for CZ gum while IG gum has a range of 53.45nm to 4.02µm fibre lengths respectively. While for the pores area measurement it can be seen that CZ gums has larger area than IG gum given as 0.71 μm^2 and 0.02 μm^2 (Figures 4.8d and 4.9d) for CZ and IG gums respectively, which also indicate vast differences in pore areas of the studied gums.

5.8 FTIR Studied Peaks

FTIR spectrum of *Irvingia gabonensis* (*IG)* gum and *Celtis zenkeri (CZ)* gums are presented in Figures 4.10 and 4.11 respectively. Frequencies and peaks of FTIR adsorption deduced from the spectra are presented in Tables 4.3 and 4.4.

The FTIR spectrum of IG gum (Figure. 4.10) gives bands and peaks that are characteristics of polysaccharides. The broad band occurring at 3423 cm^{-1} is due to the presence of hydroxyl group (i.e. OH stretch). The peak obtained at 2919 cm^{-1} is due to stretching modes of

C-H bonds of methyl group (CH_3). According to Nep and Conway (2010), natural gums usually contain fractions of sugar acid units which will usually impart a weakly anionic character to the gum macromolecule. Absorption bands around 1623 cm^{-1} and 1421 cm^{-1} are typical of carboxylate groups of the galacturonic acid residues (Figueiro *et al*., 2004). The medium absorption at 1421 cm^{-1} is due to C-C stretch in ring due to the presence of aromatics. The strong C-O stretch absorption at 1060 cm^{-1} is attributed to the presence of carboxylic acid, alcohol and ester. Finally, the weak vibration at 610 cm^{-1} represents stretch due to alkyl halides.

FTIR spectrum of CZ gum is shown in Figure 4.11 and the spectrum displayed strong OH vibrations at 3449 cm^{-1}, 3342 cm^{-1}, CH stretches at 3177 cm^{-1} and 3090 cm^{-1} (Gilani *et al*., 2011). Symmetric CH stretch was observed at 2939 cm^{-1} while OH stretch due to (-COOH) functional groups appeared at 1639 and 1430 cm^{-1}(Vinod and Sashidhar, 2010). C=O stretch due to acetyl group was observed at 1246 cm^{-1} (Xiaodong and Marek, 2007). C-O stretches were found at 1139 cm^{-1} and 1045 cm^{-1} while C-H bending vibrations due to alkynes were found at 797 cm^{-1} and 629 cm^{-1}.

Similarities were observed in the following peaks of FTIR for the studied gums (IG and CZ) and that of the reference gum (AS); peak at 479.33 cm^{-1} (C-C Bending), 591.2 cm^{-1} (CO_2 Bending), 1057.03 cm^{-1} (C-O Stretch), 1268.24 cm^{-1} (C-H Wag), 1424.48 cm^{-1} (C-H in plane bending), 1639.55 cm^{-1} (C=C Stretch), 2272.22 cm^{-1} (C-D), 2386.99 cm^{-1} (C=O Asymmetric stretch of CO_2) but C-C Bending at 479.33 cm^{-1} peaks was not observed at IG gum FTIR Peaks indicating the absence of C-C bending.

5.9 GCMS Studied Peaks

Spectra obtained from gas chromatography mass spectrophotometry (GCMS) of IG and CZ gums are shown in Figures 4.12 and 4.13. Area under the GCMS spectrum corresponds to

the concentration of the most abundant species, area and height normalization were carried out on each spectrum and the results obtained, (along with other spectra data) for IG and CZ gums are presented in Tables 4.6 and 4.7 respectively. The labeling in each chemical compound corresponded to the line number in the respective GCMS spectrum.

Results obtained from GCMS spectrum of *Irvingia gabonensis* (IG) gum (Figure 4.12) indicated the presence of some compounds (Figures 4.12a-f) at Lines 1, 2, 3, 4, 5, 6, 7 and 8 with percentage concentrations of 2.28%, 15.47%, 11%, 2.01%, 47.09%, 13.21%, 2.0% and 6.93% respectively. The GCMS spectrum of *Celtis zenkeri* (CZ) gum (Figure 4.13) revealed the presence of some compounds at Line 1, 2, 3, 4 and 5 with percentage concentration of 6.71 %, 13.36%, 21.87%, 47.46% and 10.61% respectively. The National Institute of Standards and Technology (NIST) library used could not identify the compounds positively.

CHAPTER SIX

6.0 CONCLUSION AND RECOMMENDATIONS

6.1 Conclusion

Physicochemical analysis of *Irvingia gabonensis* (*IG)* and *Celtis zenkeri* (*CZ)* gums revealed that the gums are ionic, mildly acidic (5.42 and 4.61pH respectively) and are soluble in hot and cold water but insoluble in ethanol. The conductivity of the gums was comparable to those of ionic compounds. The gums exhibited Tyndall effect due to their total solid contents. Strong relationship was found between the turbidity and wavelength of maximum absorption, while turbidity had a direct relation with the amount of light scattered thereby relating the absorbance with the turbidity value. The higher the absorbance the higher the turbidity and the higher the amount of light scattered. The gums are not saline but with higher conductivity value indicating that the conductivity may not be primarily due to the presence of electrolytes but rather due to movements of charges/ions.

IG and *CZ* gums are rich in macro elements such as calcium (2315 and 652.2mg/kg), magnesium (2274 and 2285mg/kg), iron (311 and 375mg/kg), zinc (41 and 10.6mg/kg), manganese (146 and 213mg/kg) and copper (16.4 and 16.6mg/kg respectively) which are very essential for growth and development of the human body, deficiency in any of the mentioned elements will lead to one illness or the other. Trace elements such as cadmium (1.0 and 1.6mg/kg), nickel (48.3 and 79.7mg/kg) and lead (63.2 and 15.2mg/kg) for IG and CZ gums were found at values above the recommendation of World Health Organization (WHO, 2012) permissible limit which may be due to the nature of the soil where these plants are grown. The FTIR analysis indicated the presence of functional groups such as ether, ketone, terminal olefin and intermolecularly bonded OH that are typically found in most plant gums while Gas

Chromatography Mass Spectroscopy (GCMS) analysis of the gums indicated the presence of some unidentified compounds at different percentage concentrations.

Rheological studies revealed that the viscosities of the gums decrease with increase in temperature, whereas increase in concentration of the gums and addition of electrolytes increases the viscosity of the gum solutions. The viscosity value was found to decrease with addition of potassium bromide (KBr) solution; this indicates that potassium bromide is acting as a counter ion by decreasing the rate of association of the gums particle. But on the other hand potassium chloride and aluminium chloride on addition increases the viscosity value of the gums.

The surface morphology of the gums have been adequately studied using scanning electron microscope at different magnification (1000X, 3000X and 5000X) and the results obtained have given insights into the possibility of the high value of adsorption, viscosity, turbidity and other surface properties of the gums.

The plant gum exudates obtained from the *Irvingia gabonensis* and *Celtis zenkeri* can be very useful in food additives, pharmaceutical and industrial applications due to high content of essential elements and a viscosity values that resist changes with slight change in temperature and concentration.

6.2 Recommendation

Investigations into the antimicrobial and toxicological tests on the gums is recommended, since the gums appear to possess properties closely related to the gums used in foodstuffs, pharmaceutical and other industrial applications although priority should be given to the high concentrations of lead in *Irvingia gabonensis* gum.

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APPENDIX I

Relative Viscosity: $\eta_{rel} = \frac{\eta_{solution}}{\eta_{solvent}}$

$$\eta_r = \frac{\eta}{\eta_0}$$

Gums	Ƞrel @30®C, 1.0g	Ƞrel @30®C, 0.8g	Ƞrel @30®C, 0.6g	Ƞrel @30®C, 0.4g	Ƞrel @30C, 0.2g
AS	1.579	1.480	1.398	1.131	1.072
CZ	1.072	1.050	1.036	1.023	1.022
IG	1.462	1.421	1.149	1.100	1.032

Gums	ηrel @30®C, 1.0g	ηrel @40®C, 1.0g	ηrel @50®C, 1.0g	ηrel @60®C, 1.0g	ηrel @70C, 1.0g
AS	1.579	1.548	1.529	1.466	1.416
CZ	1.072	1.036	1.000	0.964	0.941
IG	1.462	1.407	1.140	1.100	1.100

Specific Viscosity: $\eta_{sp} = \eta_{rel} - 1$

Gums	Ŋsp @30®C, 1.0g	Ŋspl @30®C, 0.8g	Ŋsp @30®C, 0.6g	Ŋsp @30®C, 0.4g	Ŋsp @30C, 0.2g
AS	0.579	0.480	0.398	0.131	0.072
CZ	0.072	0.050	0.036	0.023	0.022
IG	0.462	0.421	0.149	0.100	0.032

Gums	Ƞsp @30®C, 1.0g	Ƞspl @40®C, 1.0g	Ƞsp @50®C, 1.0g	Ƞsp @60®C, 1.0g	Ƞsp @70C, 1.0g
AS	0.579	0.548	0.529	0.466	0.416
CZ	0.072	0.036	0.000	-0.036	-0.059
IG	0.462	0.407	0.140	0.100	0.100

APPENDIX II

Gums	Ƞsp @28°C, 0.2gKCl	Ƞspl @28°C, 0.4gKCl	Ƞsp @28°C, 0.6gKCl	Ƞsp @28°C, 0.8gKCl	Ƞsp @28°C, 1.0gKCl
AS	0.367	0.376	0.380	0.385	0.394
CZ	0.054	0.059	0.072	0.086	0.095
IG	0.371	0.380	0.398	0.416	0.439

Gums	Ŋsp @28°C, 0.2gKBr	Ŋspl @28°C, 0.4gKBr	Ŋsp @28°C, 0.6gKBr	Ŋsp @28°C, 0.8gKBr	Ŋsp @28°C, 1.0gKBr
AS	0.416	0.407	0.389	0.376	0.357
CZ	0.009	0.027	0.036	0.041	0.041
IG	0.466	0.448	0.443	0.430	0.416

Gums	Ŋsp @28°C, 0.2g$AlCl_3$	Ŋspl @28°C, 0.4g $AlCl_3$	Ŋsp @28°C, 0.6g $AlCl_3$	Ŋsp @28°C, 0.8g $AlCl_3$	Ŋsp @28°C, 1.0g $AlCl_3$
AS	0.367	0.385	0.403	0.421	0.439
CZ	0.041	0.054	0.068	0.072	0.081
IG	0.538	0.561	0.579	0.602	0.620

$$\eta_{red} = \frac{\eta_{sp}}{c}$$

Gums	ηred @28°C, 0.2g	ηred @28°C, 0.4g	ηred @28°C, 0.6g	ηsred @28°C, 0.8g	ηred @28°C, 1.0g
AS	0.579	0.600	0.664	0.328	0.362
CZ	0.072	0.062	0.060	0.057	0.111
IG	0.462	0.526	0.249	0.249	0.158