



Sudan University of Science and Technology
College of Graduate Studies



Physicochemical Characterization of Gamma Irradiated *Acacia seyal* Gum

توصيف الخصائص الفيزيوكيميائية لصمغ الطلح المعالج بأشعة قاما

**A Thesis Submitted in Partial Fulfillment of the Requirements of the
Degree of MSc., in Chemistry**

By

Samah Mohamed Salih Osman (BSc., Honours, chemistry)

Supervisor:

Dr. EssaEsmail Mohammad Ahmad

Oct2020

Dedication

To my parents, brothers, and sister for their constructive support and for being a source of encouragement and moral support.

To my husband Abd Almotalb Mohamed Noor for his understanding, support, and encouragement during the period of this study.

Acknowledgement

First of all thanks to Almighty Allah for the infinite help and persistent supply with patience to accomplish this work successfully. I would like to thank my supervisor Dr. Essa Esmail Mohammad Ahmad, for his suggestions, assistance, patience and understanding throughout this research work.

My thanks are extending to the members of the Department of Chemistry at Sudan University of Science and Technology as well as to Sudan Atomic Energy Commission for technical support.

Abstract

The effects of *A. seyal* gum aqueous solutions varying gamma radiation doses (10, 30 and 50 KGy) on some physicochemical properties of. The crude and gamma irradiated samples were characterized using pH measurements, specific optical rotation, intrinsic viscosity and FTIR spectroscopy. The results show that, the pH values of the irradiated samples were decreased with the increase in radiation dose regardless of the gum concentration. The optical rotation of the crude and gamma irradiated *A. seyal* gum samples does not show specific trend with increase in radiation doses. Intrinsic viscosities of the gamma-irradiated *A. seyal* gum samples have shown interesting trend with regard to gum concentration and gamma radiation doses. Finally FTIR spectra showed slight differences between raw and γ -irradiated samples.

مستخلص البحث

هدفت هذه الدراسة الي التحقق من اثر جرعات مختلفه من اشعة قاما 10,30,50 كيلو جراي علي بعض الخصائص الفيزيوكيميائية لصبغ السيال. تم تشخيص العينات بقياس رقم الحموضه والدوران النوعي واللزوجه الضمنيوك ذلك بأستخدام مطيافية الاشعه تحت الحمراء . قيم الاس الهيدروجيني للعينات المشععه اظهرت انخفاض بزيادة الجرعات بغض النظر عن تركيز الصمغ, بالاضافه الي ذلك فإن قيم الدوران النوعي لم تظهر نسق محدد بزيادة الجرعه. بينت قياسات اللزوجه الضمنيه منحا جدير بالإهتمام فيما يخص كل من تركيز الصمغ واشعة قاما, أخيرا اظهرت مطيافية الاشعه تحت الحمراء فروقات ضئيله بين العينه الخام والعيته المشععه.

Table of contents

Contents	Page
Dedication	i
Acknowledgment	ii
Abstract	iii
مستخلص البحث	Iv
Table of contents	V
List of Tables	Vii
List of Figures	Vii
Chapter one	
Introduction and literature review	
1.1 Acacia gums	1
1.2 Physical Properties of gums	2
1.2.1 Solubility	3
1.2.2 Emulsifying properties	3
1.2.3 Viscosity	3
1.2.4 Effect of Heat	3
1.2.5 Sensory properties	4
1.3 Chemical properties of gums	4
1.4 Applications of Gums	5
1.4.1 Confectionery	5
1.4.2 Bakery products	6
1.4.3 Flavors and beverages	6
1.4.4 Other Food Applications	6
1.4.5 Pharmaceuticals	6
1.4.6 Cosmetics	7
1.4.7 Printing and paints	7

1.5. <i>Acacia seyal</i> var. <i>seyal</i> tree	7
1.5.1 Botanical Classification of <i>Acacia seyal</i> Tree	7
1.5.2 Description	7
1.5.3 Distributional	8
1.5.4 Habitat	8
1.5.5 Physical, chemical and structural characteristics of <i>Acacia seyal</i> gum	8
1.6. Effect of radiation on physicochemical characteristics of polysaccharides	9
1.7 Previous studies	9
1.8 Objective	11
Chapter two Materials and methods	
2.1 Sample collection and pretreatments	12
2.2 Gamma irradiation of the aqueous solutions of <i>A. seyal</i> gum	12
2.3 FT-IR measurements	12
2.4 pH measurements	12
2.5 Specific optical rotation	13
2.6 Intrinsic viscosity	13
Chapter three Results and discussion	
3.1 FT-IR measurements	15
3.2 pH measurements	17
3.3 Intrinsic viscosities of crude and γ -irradiated <i>A. seyal</i> gum samples	19
3.4 Specific optical rotation	21
3.5 Conclusion	23
References	24

List of Tables

Title	No
Table 1.1: Physico-chemical characteristics and nutritional data for <i>A. senegal</i> and <i>A. seyal</i> gums (typical value)	4
Table 3.1: pH of raw and γ -irradiated aqueous solutions of <i>Acacia seyal</i> gum of 10%, 30% and 50 (w/v) samples	17
Table 3.2: Intrinsic viscosities of crude and gamma irradiated samples	21
Table 3.3: Specific optical rotation of unirradiated and γ -irradiated samples	22

List of Figures

Title	No
Figure 3.1: FTIR spectrum of a crude <i>A. seyal</i> gum	16
Figure 3.2: FTIR spectrum of <i>A. seyal</i> gum irradiated with 10 KGY	16
Figure 3.3: FTIR spectrum of <i>A. seyal</i> gum irradiated with 30 KGY	16
Figure 3.4: FTIR spectrum of <i>A. seyal</i> gum irradiated with 50 KGY	17
Figure 3.5: Reduced viscosities of the crude <i>A. seyal</i> gum and γ -irradiated samples against concentrations	

Chapter One

Introduction and Literature Review

Chapter One

Introduction and literature review

1.1 Acacia gum

Acacia gums are exuded from acacia trees mainly from *Acacia senegal* and *Acacia seyal*. The main gum acacia producing countries are Sudan, Nigeria, Chad and Senegal. Sudan is considered to be the world's largest producer of gum acacia. Gum acacia is the oldest and best known of all the polysaccharide plant exudates [Caius, 1939].

Acacia gum arabic is defined by FAO/WHO Joint Expert committee on food Additives (JECFA) as: "Gum arabic is dried exudates obtained from the stems and branches of *Acacia senegal* (L.) Willdenow or *Acacia seyal* (fam. leguminosae)". Acacia gum is defined by the European pharmacopoeia 6.8 as: "Air hardened, gummy exudates flowing naturally from or obtained by incision of the trunk and branches of *Acacia senegal* L. Willdenow [Idris and Haddad, 2011].

United States pharmacopoeia Official Monograph for NF26 (USP 31) defines gum acacia as: "Acacia is the dried gummy exudates from the stems and branches of *Acacia senegal* Willdenow or of other related African species of Acacia". The Japanese Official monograph for part II/ powdered Acacia (JP XIV) defines gum acacia as: "Acacia is the secretions obtained from the stem and branches of *Acacia senegal* Willdenow or other species of the same genus (leguminosae)" [Idris and Haddad, 2011].

Acacia gums are a highly heterogeneous complex polysaccharides consisting of galactose, arabinose, rhamnose, glucuronic acid and 4-O-methylglucuronic acid [Stepgen, 1995; Osman *et al.*, 1993].

The gum belt agro-ecosystem refers to a broad band stretching across Sahelian regions of Africa and the Middle East situated between latitude

10° and 14° North. It starts from Mauritania in the West, through Senegal and Mali, Burkina Faso, Niger, Northern Nigeria to Sudan, Eritrea, Ethiopia, Kenya, Somalia and Northern Uganda in the East. It is also found in the Middle East, Yemen, India and Pakistan.

Sudan is the world's biggest producer of gum arabic, it is also the main source of gum in international trade. The gum belt falls in central Sudan roughly between latitudes 10° and 14° North, with two areas outside these borders found in the north east (Faw, Gedaref and Kassala) and in the south east along the Blue Nile/Upper Nile border [Abdel Nour, 1999]. It spans the traditional rainfed agricultural areas of western and central Sudan that include Kordofan Al Kubra 49.3% (N. Kordofan, W. Kordofan and S. Kordofan), Darfur Al Kubra 24.4 % (W. Darfur, N. Darfur and S. Darfur), Kassala region 23.4% (Kassala and Gedaref) and White and Blue Nile region 2.9% (White Nile, Sennar, Blue Nile).

Outside Africa, India produces small amounts of gum, similar in quality to gum (Talha), but a proportion of its exports of gum arabic consists either of re-exports of African gum or locally produced gum *ghatti* (from *Anogeissus latifolia*) misclassified as gum arabic.

1.2 Physical properties of gums

The physical properties and appearance of natural gums are of greatest significance in determining their marketability and end use. This differs with different botanical sources. There is a considerable dissimilarity in gum from the same species collected from plants grown under different climatic conditions or even from the same plant in different season. Physical properties are also affected by the age of Trees and treatment of the gum after collection by, for example, washing, drying, sun-bleaching and storage temperatures (Glicksman, 1969).

1.2.1 Solubility

Gum acacia is unique among natural hydrocolloids in that it is highly soluble in hot and cold water. Most gum cannot be dissolved in water at concentrations higher than 5% due to their viscosity, but gum acacia can yield solutions up to 50% concentration due to the high degree of branching within the gum structure and therefore small hydrodynamic volume [Williams, 1990; Street, 1983].

1.2.2 Emulsifying properties

Acacia gum from *A.senegal* is a very effective emulsifying and stabilizing agent and has found widespread use in the preparation of varied oil-in-water beverage emulsions. It is not less than the gold standard of emulsifiers used in beverages [Matthias, 2010].

1.2.3 Viscosity

Most gums form viscous solutions at low concentrations (>5%). However, at such relatively low concentrations, gum acacia yields solutions that are essentially Newtonian in behavior and have very low viscosities compared to other polysaccharides of similar molecular mass [Williams, 1990].

The viscosity of acacia gums solutions decreases with the addition of electrolytes and this is explained by a reduction in the effective volume due to the suppression of the electrostatic charge. Solution of acaciagums are slightly acidic (typical pH 4.5) and at this pH, the gum is at its maximum viscosity. Gum acacia is stable over a wide range of pH from 3.0 to 9.0 [Angelo, 2006].

1.2.4 Effect of heat

Prolonged heating causes the thermal destruction of acacia gums. It results in the denaturation and precipitation of the proteins from the high molecular weight AGP and GP complexes and this causes a reduction in the emulsification capacity and solution viscosity [Randall, 1989].

1.2.5 Sensory properties

Senegal gum is generally odourless, colourless and tasteless [Imeson, 1992] while *A. seyal* gum is slightly dark in colour.

Table (1.1):Physico-chemical characteristics and nutritional data for *A. senegal* and *A. seyal* gums (typical value)[Idris and Haddad, 2011]

Parameter	<i>A. senegal</i> gum	<i>A. seyal</i> gum
Appearance	Amber, transparent,hard nodules	Red/brown fragile fragments
Nutritional value (Kcal/g)	1.7	1.7
Total fat (%)	0	0
Complex carbohydrates (%)	95	95
Soluble dietary fibre(%)	>85	>85
Protein (%)	~2	~0.8
Tannins (%)	0	0.11
Potassium (ppm)	8500	2000
Magnesium (ppm)	1400	1200
Calcium(ppm)	9000	11000
Viscosity (25% w\v soln, cps)	100	80
Emulsion capacity	High	Low
Specific optical rotation (°)	-30	+50
pH	4.5	4.4
Ash (%)	3.2	2.7

1.3Chemical properties of gums

Gums are composed of carbon, hydrogen, oxygen, small quantities of mineral matter and sometimes a little nitrogen [Howes, 1949]. The pure gum may also contain small quantities of tannin.

The chemical composition of the three main exudate gums is complex and varies to some extent, depending on their source and age. Therefore, it is not possible to provide defined structural formulas for these biopolymers [Verbeken *et al.*, 2003]. Gum Arabic is recognized by many

researchers that Gum Arabic consists of mainly three fractions [Yael, 2006].

i) The major fraction is a highly branched polysaccharide consisting of galactose backbone with linked branches of arabinose and rhamnose, which terminate in glucuronic acid found in nature as magnesium, potassium and calcium salt.

ii) A smaller fraction is a higher molecular weight arabinogalactan-protein complex (GAGP-GA glycoprotein) in which arabinogalactan chains are covalently linked to a protein chain through serine and hydroxyproline groups. The attached arabinogalactan in the complex contains glucuronic acid.

iii) The smallest fraction having the highest protein content is a glycoprotein which differs in its amino acids composition.

1.4 Applications

Acacia gum enjoys a remarkable diversity of application and this is mainly due to its desirable physicochemical properties and functions as reported earlier. The functions of gum acacia include emulsifier, formulation aid, stabilizer, thickener, surface finishing agent, processing aid, firming agent, texturizer, adhesive, plasticizer, soluble fibre and prebiotic source, and many others [Idris and Haddad, 2011].

1.4.1 Confectionery

Acacia gum has been widely used in the confectionery industry for many centuries. This is due to its ability to prevent sugar crystallization, modify texture, emulsify and keep fatty components evenly distributed. It can also act as a boundary film in glazing system [Adamson, 1974; Langwill, 1939].

1.4.2 Bakery products

Gum acacia is used for its comparatively low water-absorption properties. In addition, it has favorable adhesive properties for use in glazes and toppings and imparts smoothness when use as an emulsion stabilizer [Glickman, 1983].

1.4.3 Flavors and beverages

Gum acacia has been used extensively for many years in the flavor and beverage industry due to its unique emulsifying, stabilizing, low viscosity and acid stability properties.

1.4.4 Other food applications

Acacia gum is used as a base for the preparation of spray dried colour oleoresins such as annatto, paprika and turmeric. It can also be used to prepare and stabilize liquid colour emulsions. Moreover, it is designed as a convenient means of adding soluble fibre to high fibre/low fat food products ranging from yoghurts to cakes. Acacia gum can also be used with antioxidant, fat and lactose to stabilize Oil-soluble vitamin A by spray drying them in an emulsion to give an encapsulated powder retaining 85% of its vitamin activity after 12 months storage at room temperature. A coating of gum acacia will help protect unstable oils and flavors from the development of rancidity and off-tastes [Glickman, 1983].

1.4.5 Pharmaceuticals

Acacia gum has been used successfully in a variety of pharmaceutical products because of its many function properties such as a binder, adhesive and glaze for pharmaceutical table [Tame-Said, 1997]. In demulcent syrups, it is used for its soothing and protective action, as a suspending agent and as an emulsifying agent. Acacia gum is also used as one of the main ingredients in medicated cough drops and lozenges.

1.4.6 Cosmetics

In cosmetics, acacia gum has a variety of roles as a result of its excellent functions such as a stabilizer. It also imparts spreading properties, gives a protecting smooth feel. It is used as a binding agent for cake material and an adhesive in facial masks. Gum acacia is used in the formulation of mascara, facial moisturizer, other moisturizers, anti-aging creams, body wash/cleaner, liquid hand soaps, hair spray, eyeliner, lipsticks and others [Whistler, 1993].

1.4.7 Printing and paints

Used in lithography, [FAO, Rome, 1995] inks and water colors.

1.5. Acacia seyal var. seyal tree

1.5.1 Botanical Classification of *Acacia seyal tree* [Acacia nilotica, 2016]

Family: *Leguminous*

Sub Family: *Mimosoideae*

Genus: *Acacia*

Species: *seyal var. fistula, seyal var. seyal*

Vernacular surnames: *talha*

1.5.2 Description

Acacia seyal is a small to medium-sized tree, growing to 17 m tall and 60 cm in diameter at breast height; crown is umbrella shaped, resembling that of *A. tortilis var. raddiana*, *var. tortilis* and *var. spirocarper*. A characteristic feature of the tree is its rust-coloured powdery bark; *A. seyal var. fistula* has whitish bark. Large, straight spines occur on the branches, and smaller, curved thorns are present near the tips of the branches.

1.5.3 Distributional range (native)

Africa-Northern Africa: Egypt; Northeast Tropical Africa: Chad, Ethiopia, Somalia, Sudan; East Tropical Africa: Kenya, Tanzania, Uganda; West- Central Tropical Africa: Cameroon, Central African Republic; West Tropical Africa: Cote D'Ivoire, Mali, Mauritania, Niger, Nigeria, Senegal; South Tropical Africa: Malawi, Mozambique, Zambia. Asia, Temperate-Arabian Peninsula: Saudi Arabia, Yemen [USDA, ARS, National Genetic Resources Program, 2008].

1.5.4Habitat

Dark cracking clay. Found often on higher slopes of the rivers and valleys in addition to the hard clay plains of Central Sudan. Also in clay depression areas where water is accumulating. It is distributed all over the Sudan. More than 70% of the Sudanese gum production comes from *Acacia seyal*, which is prevalent in the southwestern part of the country and in the Nile region. These trees are not tapped and only natural exudates are collected and sold as talha gum.

1.5.5Physical, chemical and structural characteristics of *Acacia seyal* gum

In comparison with *A. senegal* and depending on the source the glycan components of *A. seyal* contains a greater proportion of L-arabinose relative to the D-galactose. The gum from *A. seyal* also contains significantly more 4-O-methyl-D-glucuronic acid but less L-rhamnose and unsubstituted D-glucuronic acid than that from *A. senegal*. The gum is inferior to hashab and is reported to be structurally different to *A. senegal* gum. With specific optical rotation of +41 to +61, a nitrogen content of 0.14% (w/v) and a tannin content of 1.9%. As such, gum talha did not satisfy the 1990 specification for food grade gum Arabic [Siddeg, 2003].

1.6Effect of ionizing radiation on physicochemical characteristics of polysaccharides

Recently decontamination of gum Arabic was tried [Serag, 2007] by some researches using ionizing radiation. Although it is a well known tool in sterilization, ionizing radiation has some effects on the physical and chemical properties of the material. Up to now it is a challenge to obtain the optimum radiation dose which does the job of sterilization or enhancement of the properties without any side effects on the gum Arabic. Gamma radiation is known to induce polymerization [Tsuyoshi, 2006; Al-Assaf, 2007], and hence change of the molecular weight of gum Arabic in its aqueous phase. In the solid phase on the other hand the change of properties is dependent on the amount of the radiation dose used.

Many interactions cause the effects of radiation on gum Arabic in a way or another i.e., hydrogen bonds may be altered so that water molecules degrade producing hydroxyl groups. Water radiolysis could on the otherhand produce hydrogen and hydrogen peroxide [Phillips, 1972].

1.7 Previous studies

Dong, *et al.*, [2003] assessed the effect of irradiation on the degradation of alginate. The aqueous solution of alginate was irradiated by ^{60}Co gamma rays in the dose range of 10 to 500 KGy. The irradiation-induced changes in the viscosity, molecular weight, color, monomer composition were measured. The molecular weight of raw alginate was reduced from 300000 to 25000 when irradiated at 100 kGy. The degradation rate decreased and the chain breaks per molecule increased with increasing irradiation dose. The viscosity of irradiated alginate solution reached a near minimum as low as at 10 KGy. No appreciable color changes were observed in the samples irradiated at up to 100 KGy, but intense browning occurred beyond 200 KGy. The ^{13}C NMR spectra showed that homopolymeric blocks increased and the M/G ratio decreased with irradiation.

Considering both the level of degradation and the color change of alginate, the optimum irradiation dose was found to be 100 kGy.

Alginates were irradiated as solids or in aqueous solution with ^{60}Co gamma rays in the dose range of 20 to 500 kGy to investigate the effect of radiation on alginates showed by [Naotsugu *et al.*, \[2000\]](#). Degradation was observed both in the solid state and solution. The degradation in solution was remarkably greater than that in the solid. For example, the molecular weight of alginate in 1% (w/v) solution decreased from 6×10^5 for 0 kGy to 8×10^3 for 20 kGy irradiation while the equivalent degradation by solid irradiation required 500 kGy. Degradation G-values were 1.9 for solid and 55 for solution, respectively. The free radicals from irradiated water must be responsible for the degradation in solution. The degradation was accompanied by a color change to deep brown for highly degraded alginate. Little color change was observed on irradiation in the presence of oxygen. UV spectra showed a distinct absorption peak at 265 nm for colored alginates, increasing with dose. The fact that discoloration of colored alginate was caused on exposure to ozone suggests a formation of double bond in the pyranose ring.

Radiation degradation of cellulose was studied by [Leonhard *et al.*, \[1985\]](#). The results of the degradation of gamma and electron treated wheat straw are reported. Complex methods of treatment (e.g. radiation influence and influence of lyes) are taken into consideration. In vitro experiments with radiation treated straw show that the digestibility can be increased from 20 % up to about 80 %. A high pressure liquid chromatography method was used to analyze the hydrolysates. The contents of certain species of carbohydrates in the hydrolysates in dependence on the applied dose are given.

1.8. Objective

The aim of this study was to investigate the effect of different gamma radiation doses on the physicochemical characteristics of the aqueous solutions of *A. seyalgum* at different concentrations.

Chapter Two

Materials and methods

Chapter Two

Materials and methods

2.1 Sample collection and pretreatments

The gum sample (*Acacia seyalvarseyal*) used in this study was collected from their native growing regions (Blue Nile State, Sudan). It was hand cleaned to insure freedom from sand, dust and bark impurities. The sample was ground using a mortar and pestle and kept in plastic bags for the next steps.

2.2 Gamma irradiation of the aqueous solutions of *Acacia seyal* gum

A. seyal gum was dissolved in distilled water to prepare solutions of various concentrations (10, 30, and 50% (w/v)). The gum solutions were irradiated with different doses of gamma radiation (10, 30 and 50 KGy). The radiation source used was Cobalt-60 Gamma cell 220 (Nordion-Science Advancing Health, Canada) at the laboratories of the Sudan Atomic Energy Commission, Khartoum-Sudan. The samples were dried at ambient conditions and sealed in polyethylene bags for further analyses.

2.3 FT-IR measurement

The infrared spectra of crude and gamma irradiated gum samples were recorded using a Shimadzu-Fourier transform infrared spectrometer (FTIR-4100, JASCO) in the range between 4000 and 400 Cm^{-1} . Few milligrams (2mg) of each sample were mixed thoroughly with 200 mg of spectroscopic grade KBr, pressed into pellets and the FTIR spectra of the crude and irradiated samples were obtained.

2.4 pH measurements

The pH values of the crude and gamma irradiated gum samples were determined by a Jenway pH-meter 3510, which was previously calibrated using standard buffer solutions (pH 4, 7 & 10). 1% aqueous solution of each gum sample was prepared and the pH electrode (combination

electrode) was immersed in the sample, left for few minutes and the pH value was recorded at room temperature.

2.5 Specific optical rotation

The specific optical rotation was determined according to FAO (1991). 1.0% (w/v) solutions of crude and gamma irradiated samples were prepared and filtered to be highly pure. Optical rotation was measured using an optical activity Polarimeter (ATAGO Company Ltd., Japan Model: POLAX-2L). The tube was filled with the test solution and the specific optical rotation was measured at room temperature. The following equation was used for calculation.

$$\text{Specific rotation } [\alpha] = \frac{\alpha \times 100}{C \times L} \text{ dm}^{-1} \text{ ml g}^{-1}$$

Where,

α = Observed optical rotation

C = Concentration of the solution (g/ml)

L = Length of the Polarimeter tube (dm)

2.6 Intrinsic viscosity

The viscosities of the solutions of crude and gamma irradiated samples were determined using an Ostwald viscometer. A series of dilute aqueous solutions (1, 2, 3, 4% w/v) of each gum sample was prepared using 1M NaCl as a solvent and the time of flow between two calibration marks of the viscometer was measured. The experiment was repeated three times and a comparative method was used to calculate the viscosity of each solution according to the following equations:

$$\text{Relative viscosity: } \eta_{\text{rel}} = \frac{\eta}{\eta_0} = \frac{t}{t_0} \dots \dots \dots (2.1)$$

Where:

η = Solution viscosity

η_0 = Solvent viscosity

t = Flow time of solution

t_0 = Flow time of solvent

Specific viscosity: $\eta_{sp} = \frac{\eta - \eta_0}{\eta_0} = \frac{t - t_0}{t_0} = \eta_{rel} - 1 \dots \dots \dots (2.2)$

Reduced viscosity: $\eta_{red} = \frac{\eta_{sp}}{c} = \frac{\eta_{rel} - 1}{c} \dots \dots \dots (2.3)$

Inherent viscosity: $\eta_{inh} = \frac{\ln \eta_{rel}}{c} \dots \dots \dots (2.4)$

Intrinsic viscosity: $\lim_{c \rightarrow 0} \frac{\eta_{sp}}{c} \dots \dots \dots (2.5)$

Chapter Three

Results and Discussion

Chapter Three

Results and discussion

3.1 FTIR analysis

FT-IR analysis was performed to examine the influence of gamma radiation on the structural features of the gum molecule. Figures 3.1 to 3.4 display the FTIR spectrum of the crude and gamma irradiated samples. Figure 3.1, shows the FTIR spectrum of the crude A. seyal gum. As can be observed from the figure, the intense broad band at 3424 cm^{-1} is attributed to -OH group stretching vibration, while the sharp peak at 2930 cm^{-1} is due to the stretching vibration of -CH group (sp^3 -hybridized). In addition, the sharp intense peak at 1632 cm^{-1} could be resulted from the bending vibration of -OH group or possibly to the absorption bands of the water molecules. The broad intense area lie between 1422 and 1072 cm^{-1} which include a number of weak absorption and overlapped peaks are due to the bending vibrations of -CH , stretching vibration of -C-C and stretching vibration of -C-O groups. Similar results were reported by Daoubet *al.*, [2016]. On the other hand, the FT-IR spectra of gamma irradiated samples, Figures 3.2 to 3.4, have shown almost identical absorption peaks to the crude sample with only exception is the presence of a weak band between 1700 to 1750 cm^{-1} which is attributed to the stretching vibration of carbonyl group of carboxylic acid functional group.

Figure (3.1): FT IR spectrum of a crude *A.seyal* gum

Figure(3.2): FT IR spectrum of *A. seyal* gum irradiated with 10 KGy

Figure(3.3): FT IR spectrum of *A. seyal* gum irradiated with 30 KGy

Figure(3.4): FT IR spectrum of A. seyal gum irradiated with 50 KGy

3.2 pH measurements

The pH measurements of the aqueous solutions of raw and γ -irradiated samples having different concentrations (10%, 30% and 50%) were carried out to examine the influence of different γ -radiation doses on the physicochemical characteristics of the samples. As it is evident from the table, all irradiated samples have higher pH values compared to crude sample. Moreover, the pH values of the irradiated samples decrease with the increase in radiation dose regardless of the gum concentration.

Table (3.1): pH of raw and γ -irradiated aqueous solutions of A. seyalgum

Concentration (w/v)	Doses of γ -radiation (KGy)		
	10	30	50
10%	4.45	4.43	4.41
30%	4.76	4.73	4.53
50%	4.56	4.43	4.39
The raw gum sample	4.34		

The pH value of the irradiated sample decreases with the increase in gamma radiation dose could be due to increase in content of carboxyl groups or formation of degradation products such as carboxylic acids. The FT-IR spectra of the irradiated samples have demonstrated the presence of a small shoulder-peak around $1700\text{-}1750\text{ cm}^{-1}$ which supports the above interpretation. These findings are in agreement with the previous study by [Singh and Sharma \[2013\]](#).

In another study, [Singh *et al.*, \[2010\]](#) have reported that the pH values of gamma irradiated samples of potato starches decrease due to the increase in carboxyl content of the samples as a result of radiation.

It is worth noting that generally the concentrated solutions of carbohydrate materials are influenced differently from the diluted ones as exposed to radiation. The difference between concentrated and diluted solutions can be explained in terms of the radiation chemistry of carbohydrates [[Al-Assaf *et al.*, 1999](#)) [Katayama, *et al.*; 2006](#)]. In dilute solutions, the initiating chemical process is the radiolysis of water, and the most reactive species produced is the OH radical. It has the ability to abstract hydrogen atom from a carbohydrate to give a reactive-free radical with extreme efficiency. The carbohydrate-free radical formed is unstable, and has little probability of encountering another radical in a dilute solution [[Al-Assaf *et al.*, 1999](#)], and so gum molecule degrades since the abstraction process is random. In a concentrated solution, the carbohydrate interacts with radiation by an additional mechanism. The energy absorbed is proportional to the electron fraction of the solute and solvent. Thus, a greater proportion of the radiation energy will be deposited in the polysaccharide at the higher concentrations, which is known as direct action, as opposed to the indirect action of the water radiolysis process followed by -OH radical attack. With such an initially large molecule and low viscosity such as gum Arabic, there need only to

be a small number of such radical-radical steps to yield the higher molecular weight products we have observe

3.3. Viscosities of crude and γ -irradiated *A. seyal* gum samples

The viscosities of the aqueous solutions of crude and gamma irradiated *Acacia seyal* samples were determined using Ostwald viscometer and the results were presented in Table 2.2 and Figure 3.5. As it is seen from the results (Figure 3.5 and Table 2.2), the intrinsic viscosity of the gamma-irradiated *A. seyal* gum decreased continuously with the increase in irradiation dose.

The results were in agreement with the previous studies of the effects of irradiation on pasting viscosities of various starches [Bao, 2002; Bao, 2001; MacArthur, 1984; Sokhey, 1993; Bachman, 1997]. The findings of these studies have shown that the pasting viscosity of the gamma-irradiated rice flour decreased continuously with the increase in irradiation dose, and may be due to the degradation of the gum molecule as a result of gamma radiation which was clearly observed in pH measurements and FTIR analysis. Irradiation in general leads to a significant decrease in the viscosity of dispersions.

The effects of electron beam irradiation (10, 15, 20, and 25 KGy) on the physico-chemical properties of sago starch have been detailed by Pimpa and others (2007a). They reported an increase in the gel strength at 10 and 15 kGy, with a decrease observed at higher doses. Solubility, redness, yellowness, and free acidity were enhanced, while swelling power, peak viscosity, intrinsic viscosity, molecular weight, and degree of polymerization decreased along with the increase in irradiation dose.

Recently, Lee and others (2008a) have evaluated the effects of gamma irradiation (20 kGy) on the reduction of viscosity and for the enhancement of solid content of four cereal porridges (cereals, wheat, rice, waxy rice). They reported an increase in the viscosity of cereal

porridges along with an increase of solid contents, with minimal changes in the starch digestibility. They also reported that the viscosity of cereal porridges showed a stronger correlation to the setback viscosity of cereal flours than the maximum viscosity during gelatinization. Radiation processing produced a decrease in the viscosity of rigid cereal porridges, which turned into semi-liquid consistencies. This has been attributed to the radiolysis of the starch gel due to the action of the radicals produced by gamma irradiation. The increase in the solid contents of all porridges after irradiation (high in the waxy rice) was attributed to the fact that waxy rice can form a very weak gel during gelation and retrogradation [BeMiller and Whistler 1996].

Abu and others (2005) studied the functional properties of cowpea (*Vigna unguiculata* L. Walp) flours gamma-irradiated at 2, 10, and 50 kGy. Some of the starch-related functional properties of cowpea flours and pastes, like the swelling index, and gel strength and viscosity, were found to be significantly reduced at all the doses of irradiation and these effects were dose dependent, which has been attributed to radiation-induced degradation of the starch. The radiation-induced decrease in the swelling and gel strength has been attributed to the decreases in viscosity owing to starch degradation. These researchers also reported that the degradation of starch in flours and pastes might have inhibited the ability of the starch granules to trap water and swell during gelatinization, contributing to the decrease upon irradiation.

Table (3.2): Intrinsic viscosities of crude and gamma irradiated samples

Sample	Intrinsic viscosity
Crude gum	13.2
50% gum solution-30 KGy	10
30% gum solution-30 KGy	14.4
50% gum solution-50 KGy	15.8
50% gum solution-10 KGy	22.2
30% gum solution-50 KGy	24
30% gum solution-10 KGy	28

3.4 Specific optical rotation of crude and gamma irradiated samples

Table 3.2 shows the optical rotation of the crude and gamma irradiated samples. As evident from the table, the optical rotation of the samples does not follow a specific trend as it increased at 10 KGy and decreased with further increase in gamma radiation doses 30 and 50 KGy. However, it is important to note that the optical rotation values of the irradiated samples are all higher than the crude one. Based on the literature survey, significant differences in the values of the optical rotation of *A. seyal* gum were reported by many authors. These results agree with JECA for specification of Talha gum. Elfatih[2013] has reported a range of optical rotation for *A. seyal* gum samples between +40 and +54.

Table (3.3): The specific optical rotation of crude and gamma irradiated *A. seyal* gum samples.

Sample	Optical rotation (°)
Crude sample	+45
10KGy irradiated sample (10%)	+55
30 KGy irradiated sample (10 %)	+50
50 KGy irradiated sample (10 %)	+50

Conclusions

Modification of A. seyal gum was performed using varying doses of gamma radiation (10, 30 and 50 KGy) and various aqueous concentrations (10, 30 and 50% w/v). Significant increase in intrinsic viscosities of the irradiated samples was noticed which indicate both grafting as well as crosslinking of the gum molecules. In addition, degradation of the gum molecule was also noticed as the pH measurements and FTIR were both shown indications of such processes.

References

Abdel Nour, H. O., (1999): Gum arabic in Sudan: production and socio-economic aspects. In: Medicinal, Culinary and Aromatic Plants in the Near East. Food and Agriculture Organization of The United Nations.

Acacia nilotica (gum arabic tree)". Invasive species compendium. Centre for Agriculture and Biosciences International. Retrieved 24 January 2016.

Al-Assaf, S., 2007. Application of ionization radiations to produce new polysaccharide .Nuclear Instruments and Methods in Physical Research, 2007.

Al-Assaf, S., Clare, L., Hawkins, C. L., Parsons, B. J., Davies, M. J., & Phillips, G. O. (1999).Derivatives using electron paramagnetic resonance spectroscopy.*Carbohydrate Polymers*, **38**, 17–22.

Angelo, L.L.D., (2006). In: Gum and Stabilizers for the food Industry 13, eds.P.A. Williams and G.O. Phillips, Royal society of Chemistry, Cambrige, UK, P. 421.

Bachman, S.,Gambus, H., Nowotna, A., 1997.Effect of gamma radiation on some physicochemical properties of triticale starch.*Pol. J. Food Nutr. Sci.***6**, 31–39.

Bao, J.S., Corke, H., 2002. Pasting properties of g-irradiated rice starches as affected by pH.*J. Agri Food Chem.***50**, 336–341.

Bao, J.S., Shu, Q.Y., Xia, Y.W., Bergman, C., McClung, A., 2001.Effects of gamma irradiation on aspects of milled rice (*Oryza sativa*) end-use quality.*J. Food Qual.***24**, 327–336.

- Caius, J.F., and Randha, K.S., Bombay Nat, J.,1939. Hist. Soc., 41, 261.
- Daoub, R.M., Elmubarak, A.H., Misran, M., Hassan, E.A. and Osman, M.E., 2016. Characterization and functional properties of some natural *Acacia* gums. *Journal of the Saudi Society of Agricultural Sciences*.
- Dong WookLee, Won SeokChol,(2003).Myung Woo Byun Hyun Jin Park, Yong-Man Y. U. and Chong M. Lee, *J.Agric. Food Chem.*, **51**, 4819.
- Glicksman, M., 1983. in Food Hydrocolloids, Volume 2, ed. Glicksman, CRC Press, Boca Raton, USA, P. 7.
- Gums, Resins and Latexes of Plant Origin (Non-Wood Forest Products 6), FAO, Rome, 1995.
- Howes, F. N. (1949); Vegetable Gums and Resins; D.Sc. WALTHAM, MASS, U.S.A.; vol. 20.
- Idris, OHM and Haddad, GM. Gum arabic's (Gum acacia's) journey from tree to end use. In: Kennedy, JF, Phillips, GO, and Williams, PA (Eds.). 2011. Gum Arabic. RSC publishing, UK.
- Imeson, A., 1992. in Thickening and Gelling Agents for food, ed. A. Imeson, Chapman and Hall, London, UK, P. 66.
- Katayama, T, Nakauma, M, Todoriki, S, Phillips, GO and Tada, M. 2006. Radiation-induced polymerization of gum arabic (*Acacia senegal*) in aqueous solution. *Food hydrocolloids*, **20**(7):983-989.
- langwill, K.E., 1939. Confect. Manuf, 19, 37.

Leonhardt J., Arnold G., Baer M., Langguth H., Gey M. and Hgbert S.,(1985)*Radi.Phys. Chem.*, **25**, 899.

MacArthur, L.A., D'Appolonia, B.L., 1984.Gamma radiation of wheat. II. Effects of low-dosage radiations on starch properties. *Cereal Chem.*61, 321–326.

Matthias, S., 2010. in Gums and stabilizers or the food industry 15, eds. P.A. Williams and G.O. Phillips, Royal society of Chemistry, Cambridge, UK, P. 257.

Naima N. A. siddeg .2003.Characterization, Fractionation and functional studies on some Acacia Gums, PhD thesis, University of Khartoum.
NaotsuguNagasawa, Hiroshi Mitomo, Fumio Yoshii, TamikazuKume, *Polym. Degrad. and Stab.*, 69, 279 (2000).

Omar B. Ibrahima, Mohame. E. Osman, Elfatih A. Hassanb.(2013); *.Acacia Seyal characterization and fractionation, Journal of ChmicalActa*,1:129-135.

Osman, M.E., menzies, A.R., Martin, B.A., Williams, P.A., Phillips, G.O., and Baldwin, T.C., 1993 *Carbohydr. Res.*, **246**, 303.

Phillips, G.O., 1972. Effect of ionization radiation on carbohydrates system.*Radiation Research Reviews*, **3**: 335-351.

Randall, R.C., Phillips, G.O., and Williams, P.A., (1989). in Food Colloids, eds. RD. Bee, P.J. Richmond and J. Mingins, Royal society of Chemistry, Cambridge, UK, P. 386.

Serag, F.Z., M.Y. Bothaina, D. Omar, S.E. Mohamed, (2007). National Center for Radiation Research and Technology (NCRRT), Egypt. Decontamination of gum Arabic with γ -rays or electron beams and effects of these treatments on the material. *Applied radiation and isotopes*, **65**: 26-31.

Singh, B and Sharma, V. (2013). Influence of gamma radiation on the physico-chemical and rheological properties of *Sterculia* gum polysaccharides. *Radiation Physics and Chemistry* **92**:112-120.

Sokhey, A.S., Chinnaswamy, R., (1993). Chemical and molecular properties of irradiated starch extrudates. *Cereal Chem.* **70**, 260–268.

Sopade, P.A., Halley, B.J., Cichero, J.A.Y., Ward, L.C., Hui, L.S., Teo, K.H., (2008). Rheological characterization of food thickeners marketed in Australia in various media for the management of dysphagia. II. Milk as a dispersing medium, *J. Food Eng.* **84**, 553–562.

Stephen, AM and Churms, SC. (1995). Food polysaccharides and their Applications, ed. A.M. stepgen, Marcel Dekker, New York, USA, p.337.

Street, C.A., and Anderson, D.M.W., 1983. *Talanta*, 30, 887.

Tame-Said, J.I., 1997. World Patent WO **9**,719,668.

The Market for Gum Arabic, A.D. Adamson and J.M.K. Bell, Tropical products Institute, London, UK, 1974, G-87.

Tsuyoshi, K., N. Makoto, T. Setsuko, O.G. Phillips, T. Mikiro, (2006). Radiation-induced polymerization of gum Arabic (Acacia Senegal) in aqueous solution. *Food Hydrocolloids*, **20**: 983- 989.

Verbeken, D.; Dierckx, S. and Dewettinck. (2003). Exudate gums: Occurrence, production, and applications. *Applied Microbiology and Biotechnology*, Vol.**63**, No. 1, (November 2003), pp. 10–21, ISSN: 0175-7598.)

Whistler, R.L., (1993). in Industrial Gums: Polysaccharides and their Derivatives, eds. R.L. Whistler and J.N. BeMiller, Academic Press, San Diego, USA, P. 318.

Williams, P.A., Phillips, G.O., and Randall, R.C., (1990). In: Gum and Stabilizers for the food Industry 5, (eds). G.O. Phillips, D.J. Wedlock and P.A. Williams, IRL press, Oxford, UK, P.25.

Williams, P.A., Phillips, G.O., and Stephen, A.M., 1990. *Food Hydrocolloids*, **4**, 305.

Yael, D., C. Yachin and Y. Rachel, (2006). Structure of gum Arabic in aqueous solution. *J. Polymer Sci.*, **44**: 3265-3271. DOI: 10.1002/polb.20970.

Appendix A