

استهلال

قال تعالى :

بسم الله الرحمن الرحيم

﴿ الْحَمْدُ لِلَّهِ الَّذِي أَنْزَلَ عَلَى عَبْدِهِ الْكِتَابَ وَلَمْ يَجْعَلْ لَهُ عِوَجًا (1) قَيِّمًا لِيُنذِرَ بَأْسًا شَدِيدًا مِمَّنْ لَدُنْهُ وَيُبَشِّرَ الْمُؤْمِنِينَ الَّذِينَ يَعْمَلُونَ الصَّالِحَاتِ أَنَّ لَهُمْ أَجْرًا حَسَنًا () ﴾

سورة الكهف الآيات 1-2

صدق الله العظيم

Dedication

I dedicate this work

to my parents,

husband,

brothers and sisters.

Acknowledgment

Praise to Alla Almighty, who gave me health and strength to complete this research.

My gratitude and appreciation are due to my supervisor Professor Mohamed El Mubarak Osman whose supervision resulted in this study. My thanks are due to my co-supervisor Dr. Elfatih Ahmed Hassan who supported me with endless efforts.

My deep thanks go to Professor Saphwan Al-Assaf - Chester university, UK - Seif Aldawla Ibrahim - Sinnar university. staff of the faculty of science Sudan University of Science and Technology, Jameel alla Jomaa Khartoum University faculty of forest, Dr Ahamed Ali- Faculty of Agriculture Shampat Center Laboratory Khartoum university, Alwaleed Ali - Nopic company, staff of Research and Industrial Consultancy Center, Mr. Zokaa Shmseldeen Ahamed- International Standardizations Organization, Fadl Elseed Jobara Saudi Arabia for technical support.

Thanks and appreciation are due to Professor Nour Eldayem Osman the Vice-Chancellor of Elimam Elmahadi University for financial and moral support.

Abstract

Forty five samples of the gum from *Azadirachta indica* gum were collected from three locations (15 samples from each location) Khartoum, White Nile and Northern Kordofan states. from Sudan, during seasons 2014-2016; Three composite samples were prepared from the fifteen samples of each location by mixing. Chemical and Physicochemical properties were studied amino acids profile, molecular weight and molecular weight distribution, rheological behavior and emulsification properties of *Azadirachta indica* gum.

The results show the ranges for moisture content 11.2 - 12.2%, Ash content 3.0 - 3.30%, pH 4.2 - 4.8, Specific optical rotation -65.5 - -67, color Gardner (0.1 - 0.9), nitrogen content 4.8- 5.2%, hence protein% 30.2-31.3%, tannin .04 - .010%, acid equivalent weight 1543 - 1708, total uronic acid% 11.3 - 12.5% and intrinsic viscosity 24 - 36 cm³g⁻¹. The calorific value was 4.3 kJ/mole. Acid hydrolysis revealed a sugar content Arabinose 14-22%, Galactose 17- 24%, Xylose 3 -4% and Rhamnose 2 -4%. The Cationic composition showed that Mg 57.1-38.9, Na 24.5-13.9, Ca12.6-9.6, K11.9-6.2, Cu 0.11-0.16, Zn 0.06-0.08, and P0.02-0.04mg/g. The amino acid results shows the presence of seventeen amino acids namely, Asparagine, Threonine, Serine, Glutamine, Proline, Glycine, Cysteine, Alanine, Valine, Methionine, Isoleucine, Leucine, Tyrosine, Phenylalanine, Histidine, Lysine, and Arginine. Number average molecular weight (Mn) was range in the 6.4×10⁵-12×10⁵ g/mol . The molecular weight determined by gel permeation chromatography (GPC-MALLS) for the three location composite (Khartoum, White Nile and Northern Kordofan states) respectively. The samples have Molar masses of 4.8×10⁵ , 4.00 ×10⁵ g/mole,

and 3.6×10^5 g/mole. With a radius of gyration (Rg) in the range of (25.6 – 36.6 – 66), respectively. *Azadirachta indica* gum samples (composite) collected from Khartoum location show an Arabinogalactan protein (AGP) with total molecular weight 3.8×10^5 mass% 1.34, and Rg 63.7 . The second and third fractions correspond to an (Arabinogalactan AG + Glycoprotein GP) have molecular weight of 3.292×10^5 g/mole and mass recovery of 98.13. GPC-MALLS for neem gum samples collected from White Nile shows an AGP with a molecular weight of 4.00×10^5 , mass% 1.77, and Rg 43.2 The second and third fractions are (AG +GP) have a molecular weight of 3.193×10^5 g/mole , mass% 98.66, GPC-MALLS for *Azadirachta indica* gum samples collected from Northern Kordofan location shows an AGP with a molecular weight of 3.8×10^5 , mass%.87, and Rg 52.2 The second and third fractions are (AG +GP) have a molecular weight of 3.292×10^5 g/mole , mass% 99.13. The rheological behavior of *Azadirachta indica* gum show a Newtonian behavior All emulsions show Low droplets size, exhibit a typical bimodal droplet size distribution with a pronounced shoulder reflecting best formity and stable emulsions.

The gum emulsion shows first grade emulsification properties. *Azadirachta indica* gum shows insignificant differences (were observed) samples collected from the three different locations from Sudan.

المستخلص

تم جمع خمس وأربعون عينة من صمغ ازدراختا انديكا من ثلاث مناطق (15 عينة من كل منطقة) من ولايات الخرطوم، النيل الأبيض وشمال كردفان - السودان خلال موسم 2014 -2016. تم عمل ثلاث مخلوطات للخمسة عشر عينة لكل منطقة. تمت دراسة الخواص الكيميائية الفيزيوكيميائية والأحماض الامينية ، الوزن الجزيئي وتوزيعات الكتلة الجزيئية، الريولوجي والخواص الاستحلابية لصمغ أزدراختا إنديكا.

النتائج أوضحت أن متوسط محتوى الرطوبة 12.2 – 11.2% محتوى الرماد 3.0 – 3.3%، درجة الحموضة 4.8 – 4.2، الدوران الضوئي 65.5 – 67-، درجة اللون 0.1 – 0.9، محتوى النيتروجين 4.8 – 5.2% ، محتوى البروتين 30.2 – 31.3%، محتوى التانين 0.10 – 0.04. % الوزن المكافئ للحامض 1708 - 1543 حامض اليورنيك الكلي 12.5 – 11.3%، اللزوجة 36 – 24 سم³/جرام¹ والقيمة السعيرية ~ 3.4 كيلو جول/مول. التحلل الحمضي متبوعا بقياسات كروماتوغرافيا السائل ذات الأداء العالي كشفت ان محتوى السكر كالاتي: ارابينوز 14 – 22% ، جلاكتوز 17 – 22%، زيلوس 3 – 4% ورامنوز 2 – 4% . تمت دراسة العناصر للعينات الثلاث وتدرج قيم العناصر الماغنيسيوم 38.9 – 57.1 ، الصوديوم 13 – 124.5 ، الكالسيوم 9.6 – 12.6 ، البوتاسيوم 6.2 – 11.9 النحاس 0.11 – 0.19 ، الخارصين 0.06 – 0.08 والفسفور 0.02 – 0.04 ملم/جرام. أظهرت دراسة محتوى الأحماض الامينية ان صمغ أزدراختا إنديكا يحتوي علي سبعة عشر من الاحماض الامينية كل من اسبارجين، ثيرنين، سيرين، جلتوماين، برولين، جلايسين، الانين، سيوسين، فالين، ميثولين، ايسولايسين، ليوسين، تيروسين، فانيلائين، هستدين، لوسين وارجنين وجد ان متوسط الرقم الجزيئي لصمغ أزدراختا إنديكا 12.0×10^5 ، 10.8×10^5 و 6.4×10^5 جرام/مول لمناطق الخرطوم، النيل الأبيض وكردفان علي التوالي ووجد Rg يساوي 66، 25.6 و 36.6 علي التوالي. تم تقدير الوزن الجزيئي عن طرق كروماتوجرافيا الاستبعاد بالجل للثلاث مناطق الخرطوم ، النيل الأبيض وشمال كردفان علي التوالي وجد انه يساوي 4.8×10^5 جرام/مول ، 4.00×10^5 جرام/مول و 3.6×10^5 جرام/مول. أظهرت كروماتوجرافيا الاستبعاد بالجل لعينات منطقة الخرطوم ثلاثة أجزاء: الارابينوجالاكتان بروتين، بوزن جزيئي 3.8×10^5 ، وكتلة % 1.34، و 63.7 Rg. والجزء الثاني والثالث (الارابينوجالاكتان جلايكو بروتين) بوزن جزيئي 3.292×10^5 جرام/مول، وكتلة % 99.13. أظهرت كروماتوجرافيا الاستبعاد بالجل لعينات منطقة النيل الأبيض أيضا ثلاثة أجزاء اربينوجالاكتان بروتين، بوزن جزيئي 4.00×10^5 ، وكتلة % 1.77، و 43.2 Rg. والجزء الثاني والثالث (ارابينوجالاكتان و جلايكوبروتين) بوزن جزيئي 3.193×10^5 جرام/مول، وكتلة % 98.66. كما أظهرت كروماتوجرافيا الاستبعاد بالجل لعينات

منطقة شمال كردفان أيضا ثلاثة أجزاء اربينوجالاكتان بروتين، بوزن جزيئي 3.8×10^5 ، وكتلة %87، و 52.2 Rg. والجزء الثاني والثالث (اربينوجالاكتان و جلايكوبروتين) بوزن جزيئي 3.292×10^5 جرام/مول، وكتلة %99.13. دراسة الريولوجي لصبغ النيم أظهرت خصائص نيوتونية كل المستحلبات أظهرت أقطار صغيرة للقطرات وبتوزيعات مختلفة، الشيء الذي يفسر تجانس و ثبات المستحلبات. كما اظهر الدرجة الأولى في الخصائص الإستحلابية. صبغ أزدراختا إنديكا اظهر مع الملاحظة من النتائج المدروسة ان هنالك اختلافات ضئيلة بين العينات التي تم جمعها من ثلاث مناطق مختلفة في السودان .

Table of Contents

Content	Page No
استهلال	I
Dedication	II
Acknowledgment	III
Abstract (English version)	IV
المستخلص	V
List of abbreviations	VI
List of Tables	VII
List of Figures	VIII
Chapter One :Introduction and Literature Review	
1.1 Introduction	1
1.2 Gum Arabic	3
1.3 Definition of gum	3
1.4 Types of gum	4
1.5 The gum belt of Sudan	5
1.6 Theories of gum formation	6
1.7 Properties of gum	7
1.8 Application of gum	7
1.8.1 Gum in food industry	8
1.8.1.1 Confectionery	8
1.8.1.2 Flavours	8
1.8.1.3 Bakery	8
1.8.1.4 Beverages	9
1.8.2 Gums in non food application	9
1.8.2.1 Pharmaceuticals	9
1.8.2.2 Inks and other industries	9
1.9 Sudan gum producing trees	9
1.10 Gum collection in Sudan	10
1.11 Other non <i>acacia</i>	11
1.12 <i>Azadirachta indica</i> trees	12

1.12.1 Scientific classification	12
1.12.2 Uses of neem trees	14
1.12.3 Distrpuation	17
1.12.4 Neem in Sudan	17
1.12.5 Chemistry of neem	18
1.12.6 The chemicals classified	19
1.12.7 Application of neem trees	20
1.12.8 Medical application of neem	20
1.12.9 <i>Azdirachta indica</i> gum (neem gum)	21
1.12.10 Origin	23
1.12.11 General Description	23
1.12.12 Chemical characteristics	23
1.12.13 Application of neem gum	25
1.13 Physicochemical properties	26
1.13.1. Solubility	26
1.13.2. Colour	26
1.13.3. Shape	26
1.13.4 Moisture	27
1.13.5 Ash	27
1.13.6 pH	27
1.13.7 Specific optical rotation	27
1.13.8 Nitrogen and protein	28
1.13.9 Number average molecular weight	28
1.13.9.1 Osmotic pressure	28
1.13.10 Equivalent weight and total uronic acid	30
1.13.11 Tannin Content	30
1.13.12 Viscosity	31
1.13.13 Calorific value	32
1.13.14 The Rheology of neem gum	33
1.13.14.1 Introduction	33
1.13.14.2 Stress and strain of the gum	34
1.13.14.3 Viscosity and elasticity of the gum	35
1.13.14.4 The visco elasticity	36

1.13.14.5	Kinematic and dynamic viscosity	36
1.13.14.6	Viscous and elastic modulus	37
1.13.14.7	Structural effect of the gum molecule	38
1.13.14.8	The goal of the scientist, engineer or technician on rheology	42
1.13.15	Molecular weight distribution of neem gum	42
1.13.15.1	Rayleigh Scattered light and Molar mass	45
1.13.15.2	Scattered light and Molar mass	45
1.13.15.3	Gel Permeation Chromatography instrumentation	46
1.14	Emulsification properties of <i>azdirachta indica</i> gum	47
1.14.1	Definition of emulsion	47
1.14.2	British pharmacopoeia (Bp) definition of oral emulsion	47
1.14.3	The primary and Secondary emulsion	48
1.14.4	Theories of Emulsification	48
1.14.5	The emulsifying agents	48
1.14.6	Monomolecular adsorption	49
1.14.7	Solid particle adsorption	50
1.14.8	The factors affecting the choice of emulsion type	50
1.14.9	Emulsions preparation methods	51
1.14.9.1	Continental or dry gum method	51
1.14.9.2	English or wet gum method	52
1.14.9.3	Bottle or forces Bottle method	52
1.14.9.4	Control emulsion type during for mutations	52
1.14.10	Instability mechanisms of Emulsions	53
1.14.11	Phase Inversion	53
1.14.12.	Application of emulsions	53
1.15	Objectives	54
Chapter Two: Materials and Methods		
2.1	Materials	55
2.2	Preparation of Samples	57
2.3	Analytical Methods	58
2.3.1	Determination of moisture content	58
2.3.2	Determination of total ash content	58
2.3.3	pH measurement	58

2.3.4	Specific optical rotation	59
2.3.5	Viscosity measurements	59
2.3.6	Nitrogen and protein Content	60
2.3.7	Acid Equivalent weight	61
2.3.8	Total Uronic acid	61
2.3.9	Determination of sugar composition	61
2.3.9.1	Sample preparation	61
2.3.9.2	Method	62
2.3.10	Determination of Total Polyphenol (Tannin %)	62
2.3.11	Calorific Value	63
2.3.12	Determination of Cationic Composition	63
2.3.13	Amino acids composition	64
2.3.13.1	Chromatographic analysis	64
2.3.14	Number Average Molecular Weight by Osmotic pressure	64
2.3.15	Molecular weight and Molecular weight distribution	65
2.3.15.1	Sample preparation	65
2.3.15.2	Gel Permeation Chromatography Multi angle laser Light scattering	65
2.4.	Rheological Measurement	66
2.5.	Emulsification properties of the gum	66
2.5.1	Emulsion preparation	66
2.5.2	Droplet size analysis	67
Chapter Three: Results and Discussion		
3.1	Moisture content	69
3.2	Ash content	71
3.3	pH value	71
3.4	Nitrogen and protein content	71
3.5	Specific optical rotation	71
3.6	The Intrinsic Viscosity	72
3.7	Equivalent weight and Uronic Acid	72
3.8	Cations composition	73
3.9	Amino Acid Composition	74
3.10	Sugar Composition	75

3.11 Colour Gardner and tannin content	76
3.12 Calorific value	78
3.13 Number average molecular weight by osmometric	78
3.14 Molecular weight and Molecular weight distribution	79
3.15 Dynamic rheology	84
3.15.1 Shear flow viscosity	84
3.15.3 Dynamic rheology behavior	86
3.16 The Emulsification properties of the <i>Azadirachta indica</i> gum	88
3.16.1 Span%	89
Conclusion	94
Recommendation Further work	95
References	96

List of Abbreviations

GPC	Gel Permeation Chromatography
MALLS	Multi angle laser light scattering
JECFA	The joint Expert Committee of Food additives of the FAO/WHO
FAO	Food and Agriculture Organization of the United Nation
WHO	World Health Organization
ESI	Emulsion Stability Index
AGP	Arabino Galactan protein
AG	Arabino Galactan
GP	Glycoprotein
LS	Light scattering
RI	Refractive Index
UNCTAD	United Nation Conference on Tread and Developedment
VAM	Vesicular –arbuscular - mycorrhiza

List of Tables

Table 1.1 Classification of gums	2
Table 1.2 Sudan gum producing trees	10
Table 1.3 Mathematical models for flow behavior	37
Table 1.4 Process shear, shear rate, and applications	42
Table 1.5 The emulsifying agents(49
Table 1.6 The Multimolecular adsorption examples	50
Table 1.7 The differential between oil in water and water in oil	51
Table 2.1 Samples code, location and date collection of neem gum(2014)	55
Table 2.2 Samples code, location and date collection of neem gum(2015)	56
Table 2.3 Samples code, location and date collection of neem gum(2016)	56
Table 3.1 Physicochemical properties –Khartoum state	70
Table 3.2 Physicochemical properties – White Nile state	70
Table 3.3 Physicochemical properties – Northern Kordofan state	70
Table 3.4 The Cations composition of neem gum	73
Table 3.5 The Amino acid composition	74
Table 3.6 Sugar Composition	75
Table 3.7 Colour Gardena and tannin value	77
Table 3.8 calorific Value	78
Table 3.9 Molecular weight parameters determined by GPC .MALLS	81
Table 3.10 The Emulsification characters of <i>A.indica</i> gum (Khartoum)	90
Table 3.11 The Emulsification characters of <i>A.indica</i> gum (W. Nile)	91
Table 3.12 The Emulsification characters of <i>A.indica</i> gum (N. Kordofan)	91

List of Figure

Fig. 1.1 Sudan Gum belt	6
Fig. 1.2 karaya gum sample	11
Fig. 1.3 Neem tree	14
Fig. 1.4 Azadarachtin	19
Fig. 1.5 Neem gum (trunk)	22
Fig. 1.6 Neem gum	22
Fig. 1.7 Chemical characteristic	25
Fig. 1.8 Perkin Elmer Lambda 40 UV/vis Spectroscopy	31
Fig. 1.9 Calorimeter IKA C1System and the accessories bags, and Benzoic	32
Fig.1.10 Flow Curves are normally use for the graphical description of flow behavior	34
Fig.1.11 The elastic modulus G', and the viscous modulus G''	38
Fig. 1.12 A plot of $\log \eta_0$ vs. $\log C$	39
Fig. 1.13 Schematic of pree Vs. analyte and the	43
Fig.1.14 A typical GPC Instrument including Auto sampler, column, pump, RI detector and UV-vs-detector .	47
Fig.1.15 Schematic diagram of most common instability mechanisms	53
Fig .2.1 Neem gum sample	57
Fig.2.2 Homogenizer	66
Fig. 2.3 poly TRON (PT2100)	67
Fig. 2.4 Mastersizer 3000 instrument	68
Fig.3.1 Intrinsic viscosity (η/c) Variation with concentration	72
Fig. 3.2 Number average molecular weight	79
Fig. 3.3 Gel permeation chromatography (GPC-MALLS) analysis	83
Fig. 3.4 Molar mass of neem gum	83
Fig. 3.5 CWF of neem gum	84
fig.3.6 Dynamic Rheology	86
Fig. 3.7 The effects of frequency on G' and G''	87
Fig.3.8 Emulsion particle	89
Fig. 3.9 The span% of neem gum from three locations	92
Figs.3.10 The emulation particle size profile of <i>A.indica</i>	93