Electrical characterization and functional properties of extract gum ( *Tnigoneola Foenum graecum L.* ) from Fenugreek seeds.

B B Al-Shammari¹, R M Al-Ali¹ and A A Al-Sahi¹

¹Department of food Sciences, College of Agriculture, University of Basrah, Iraq.

Email: bashra_bader@yahoo.com

Abstract. Gum was extracted from fenugreek seeds *Tnigoneola Foenum graecum L.*, and characterized by several techniques method such DSC and Thermo gravimetric TGA analysis, Spectroscopic techniques such as Fourier-Transformed FT-IR, X-ray powdered diffraction spectrometry and Nuclear Magnetic Resonance. The results indicated FT-IR spectra of the studied gums showed the presence of the same functional groups expect arabic gum contain COO group. The main bands observed were assigned to O-H (3406.26 – 3412.08) group from hydroxyl group, C-H aliphatic (2856.58 – 2926.01), C=O (1429.25 – 1668.43) group, COO (1735.43) group that are present only in the structure of arabic gum and C-O (1014.56 – 1155.36). Thermo chemical properties of fenugreek seeds gum was determined and compared with arabic gum and acacia gum by DSC and TGA. The results are showed that fenugreek seeds gum and acacia gum were degraded at 300°C. The mass loss being of 33% and 45%, while Arabic gum was degraded at 303°C with weight loss 53% analyzing. The X-ray powdered diffraction revealed that fenugreek seeds gum had one weak peak at the scattering angle 2θ at 5.32 in height 60.15 cts which the X-ray diffract gram of the arabic gum and acacia gum showed presence weak peaks at the scattering angle 2θ at 20.94 and 19.04 in height 47.78 and 52.84 cts respectively. Analysis of gum by the H-NMR spectra indicate fenugreek seeds gum is a galactomannan type polysaccharide which contain mannose and galactose, which arabic gum and acacia gum were had the same sugars. All these result showed that fenugreek gum is useful in food industry.

1. Introduction
Gums and mucilages are hydrophilic carbohydrate substances, They are high molecular weight, generally consist of monosaccharide units joined by glycosidic bonds [1]. Mucilage is normal product of metabolism made without injury to the plant [2]. Natural gums characterize a group gum which swell to procedure highly viscous solutions or distributions in aqueous medium [3]. They are considered advantageous compared to semi synthetic and synthetic due to their non – toxic, biocompatible, low cost and availability. emollient and less irritant issues [4]. Plants seeds are a traditional and olden source of gums. greatest seeds have starches, Embryonic plant is use as the main reserve food stored but many contain other gum are considered a useful source of commercial hydrocolloids with certain functional properties [5]. Fenugreek *Tnigoneola Foenum graecum L.* belong to the:Lequminoella family. Fenugreek is an annual legume plant, fenugreek seeds have various food application as flavour component and seasoning, the ride seeds are used in medicine such treating colic flatulence, dysentery, dyspepsia, chronic cough, drops, enlargement of liver and spleen and...
diabetes [6; 7]. Fenugreek seeds contain a high percentage of gum. Although it does not dissolve in water and forms a viscous tacky mass when exposed to fluids, fenugreek seeds gum was extracted from the endosperm of fenugreek seeds [8; 9]. Fenugreek seeds gum is composed of mainly galactomannan. The galactomannan are polysaccharides composed of linear β- (1-4) – D- mannann back bone with varying amounts of single D-galactose units attached to the main backbone by α- (1-6) glycosides bond. Fenugreek seeds gum swell up and produce high viscosity gum at low concentration, fenugreek seeds gum is used in various food industries such as gravies, baking, chocolate, soups, bread making and ice cream because of its functional properties and using in pharmaceutical applications as suspending, gelling agent, emulsifier and binding. The characterization of gums establishing their suitability in food industry [6; 10; 9]. The aim of this study was to characterize fenugreek seeds gum using several analytical method such as Fourier Transform Infrared (FTIR) Spectra, X-ray powdered X-ray powdered diffraction spectrometry, Thermal analysis such as DSC and TGA and proton NMR.

2. Material and methods

2.1. Seed samples collection
The seeds were get from local market of Basrah city. The seeds were grinding using a coffee grinder. Arabic gum and Acacia were obtained from the chemical BDH company, England. All the other solvent reagents and chemical used were analytical grade.

2.2. Extraction of gum
The gum was extracted according to the method described by [11]. The fenugreek powder was soaked in distilled water within a ratio (1:10 w/v), then kept under shaking for 4h at 40 Cº. The viscous solution was filtered through muslin to remove the fibers. Ethanol (99%) was added in the ratio 1:1 to precipitate out the gum present. The gum was dried in oven at temperature 40-45 Cº. The dried gum was stored in airtight container.

2.3. Characterization of gums.

2.3.1. Fourier transforms analysis (FTIR)
The FT-IR Spectrum of The gums was Recorded in an FTIR spectrometer (Shimadzu. Japan) using potassium bromide disc prepared from powdered gums mixed with dry KBr in Chemistry Department, college of Education for Pure Science, University of Basrah.

2.3.2. Thermo gravimetric Analysis (TGA) and Differential Scanning Calorimetric (DSC)
Thermal Analysis Thermo gravimetric Analysis (TGA) and Differential Scanning Calorimetric (DSC) were used to study The changes occurring during the heating. Thermo gravimetric measurement TGA and DSC of gums using TGA (Shimadzu. Japanan) was Recorded in Amir kabeer / Mahasr / Iran, The dried gum was placed in aluminum pan and the temperature was held at 30 for 1 minutes before heating up of 10 C per minutes under a nitrogen atmosphere purged gas at flow rate of 80 ml / min.

2.3.3. X-Ray Diffraction Analysis.
X-ray diffraction patterns of The gums were Recorded in Physical Department / College of Education for Pure Science / University of Basrah was carried out on XRD System, X per T-pro pw 3050 (X-PerT-Pro. Germany) in ambient condition using Cu kα radiation.

2.3.4. Nuclear magnetic Resonance (NMR)
NMR Spectra of proton was Recorded in Chemistry Department / College Science / Tehran university by using a Bruker AVANCE 400 MHZ Spectrometer and a Bruker AVANCE 500 MHZ spectrometer (Agllent . America).
3. Results and Discussion

3.1. Fourier Transform Infrared (FTIR) Spectra

The FTIR spectra of fenugreek seeds gum, arabic gum and acacia gum were recorded to compare the differences in their chemical structure and observed characteristic IR wave number. From the spectrum of three gums were shown in Table 1 and figures 1, 2 and 3. A broad bands at 3412.08 - 3406.29 cm\(^{-1}\) attributed to the O-H group stretching of gums and water involved in hydrogen bonding while bands at 2856.58, 2926.01 cm\(^{-1}\) were assigned to C-H stretching modes of methylene group of sugar. In the same spectrum, the bands at 1626.69, 1616.35, 1668.43 cm\(^{-1}\) were due to the presence of C=O group stretching in the gums. In addition to that, a band observed at 1735.93 cm\(^{-1}\) was assigned to COO that can be found in the constituent of galacturonic acid in arabic gum.

| Samples       | Frequency (cm\(^{-1}\)) | Intensity     | Functional groups |
|---------------|------------------------|---------------|-------------------|
| Fenugreek gum | 3410.15                | Strong/broad  | OH                |
| Arab gum      | 3412.08                | Strong/broad  |                   |
| Acacia gum    | 3406.26                | Strong/broad  |                   |
| Fenugreek gum | 2926.01 - 2856.58      | Strong        | CH                |
| Arab gum      |                        |               |                   |
| Acacia gum    | 2910-58                | Strong        |                   |
| Fenugreek gum | 1735.93                | Strong        | COO               |
| Arab gum      | 1626.69                | Strong        | C=O               |
| Acacia gum    | 1616.35                | Strong        |                   |

Table 1. FTIR spectral peak assignment for gums
| Gum Type    | Wavenumber Range | Intensity   | Functional Group |
|-------------|------------------|-------------|------------------|
| Acacia gum  | 1668.43-1429.25  | Strong      |                  |
| Fenugreek gum| 1155.36-1026.13  | Medium weak | C-O              |
| Arab gum    | 1033.85-1014.56  | Medium weak |                  |
| Acacia gum  | 1064.71-1029.99  | Medium weak |                  |
| Fenugreek gum| 677.01-435.91   | Medium weak | C-H out of plane |
| Arab gum    | 970.19-493.27   | Medium weak |                  |
| Acacia gum  | 808.17-709.80   | Medium weak |                  |

**Figure 1.** FT-IR spectra of fenugreek seed gum
The characteristic band at 1155.36 - 1014.56 cm⁻¹ attributed to the C-O. The fingerprint region of fenugreek spectra consists of four characteristic peaks between 677.01 - 435.91 cm⁻¹ which the fingerprint region of Arabic gum and Acacia gum spectra contain fifty and two characteristic peaks between 970.19 - 493.27 cm⁻¹ and 808.17 - 709.80 cm⁻¹. The results showed FTIR Spectra of the studied gums showed the presence of the same functional groups in the three gums. The FTIR Spectra of fenugreek seeds gum was slightly different from that Arabic gum. The results were agreed with [12] who indicated the presence OH group and alkane C-H in fenugreek seeds gum also agreed with [13] who illustrated the presence OH group, alkane C-H and COO in Acacia gum (Acacia senegal, Acacia mellifera, Acacia seyal, and Acacia tortilis).

**Figure 2.** FT-IR spectra of Arabic gum.
3.2. X-Ray Diffraction (XRD) spectra.

The XRD Spectra of the different gums were shown in Figures 4, 5, 6. Fenugreek seeds gum had one weak peak was observed at the scattering angle (2θ) at 5.32° in height 60.15 cts which confined semi-crystalline nature of this gum.

Figure 3. FT-IR spectra of Acacia gum

Figure 4. XRD spectra of Fenugreek seeds gum.

Figure 5. XRD spectra of Arabic gum.

Which the X-ray diffractogram of the Arabic gum and Acacia gum showed presence of numerous halves with weak peaks at the scattering angle 2θ at 20.94 and 19.04 in height 47.78 and 52.84 cts confirming its almost complete amorphous nature.
The results indicate fenugreek seeds gum was semi-crystalline compared to the Arabic gum and Acacia gum were amorphous, possibly due to intramolecular interactions. Therefore fenugreek seeds gum partially soluble in water while Arabic gum and Acacia gum completely soluble. Present results are in accordance with the results reported by [14] who reported the increased crystalline behavior of the fenugreek seeds gum.

3.3. Differential Scanning calorimetric (DSC).
Differential Scanning Calorimetric (DSC) gives insights into the physical and chemical changes that present during thermal [15]. Figure 7 showed DSC Thermo gram of Fenugreek seeds gum, it was observed that a sharp endothermic peak appear at temperature 300 C. While arabic gum and acacia gum had same the endothermic peak at temperature 300 C is shown in figures 8 and 9. The endothermic peaks are due to the loss of water content in these gums.

3.4. Thermo gravimetric Analysis (TGA)
The results of thermo gravimetric Analysis carried out on the fenugreek seeds gum, Arabic gum and Acacia gum are shown in figures 4, 5 and 6. The thermal decomposition process for fenugreek seeds gum can be observed in Figure 7. The phase decomposition takes place in the 30-700°C temperature range with maximum degradation rate at 300°C. The mass loss being of 33% analyzing,
The thermal decomposition process of Arabic gum and Acacia gum were given in figure 5 and 6. The thermal decomposition process take place between temperature of 30-700°C with maximum degradation rate at 300 °C and weight loss of 53% and 45% . The results indicate The gum extracted from fenugreek seeds had good thermal stability .
3.5 Analysis NMR

Table 2 and figure 10 showed signals in the H-NMR spectrum of fenugreek seeds gum, signals at chemical shift equal to 2.318 – 2.486 ppm assigned to OH or CH of mannose While Signals arise at 3.330 -3.352ppm in the fenugreek gum were due to the presence of CH₂ group of galactose.

Table 2. Characteristics of solid stale H-NMR spectra of gum Table
| Chemical shift ppm | Assignment |
|--------------------|------------|
| fenugreek gum      |            |
| 1.084–1.076        | \( \text{CH}_3 \) |
| Arabic gum         |            |
| 1.19               | \( \text{CH}_2 \) |
| Acacia gum         |            |
| 3.95 – 3.20        | \( \text{COO} \) |
|                    | 3.73 – 3.85|
|                    |            |
|                    | 4.03       |
|                    |            |
|                    | 2.318–2.485|
|                    | \( \text{CH or OH} \) |
|                    | 3.352–3.330|

**Figure 10.** H-NMR spectrum of Fenugreek seeds gum

Figure 11 showed the H-NMR spectrum of Arabic gum, signals at chemical shift at 1.084–1.076 assigned to the methyl group of rhamnose sugar. The chemical shift at 3.20 – 3.95 ppm indicates the \( \text{CH}_2 \) group of galactose. The peak at 4.03 ppm due to the presence of an acetyl group.
Figure 11. H NMR spectrum of Arabic gum

While table 2 and Figure 9 showed the H- NMR spectrum of Acacia gum, a signals at chemical shift 1.19 ppm assigned to the presence of the methyl group of rhamnose sugar. Signals arise at 3.73 -3.85 ppm due to the presence of CH₂ group of galactose

Figure 12. H NMR spectrum of Acacia gum

Figures (10, 11, 12) spectra showed exhibit the typical bands and peak characteristic of gums. The H- NMR spectra of gums indicated presence of certain sugars composition

The results agreed [13] who showed the H - NMR spectra of Acacia gum (Acacia senegal, Acacia mellifera, Acacia seyal and Acacia tortilis). contain 1.4 ppm assigned to the presence of the methyl group belong to rhamnose sugar, Signals arise at 3.3 -3.8 ppm belong to the presence of CH₂ group of galactose

4. Conclusions
This study characterized the gum was extracted from fenugreek seeds, FT-IR spectrum showed band characteristics of O-H, C-H, C=O, COO groups that are presence in the structure of gums. The XRD results as established that fenugreek seeds gum showed has weak peak which show a semi-crystalline compared Arabic gum and acacia gum were low crystallinity. Thermo chemical properties of fenugreek seeds gum was determined DSC and TGA showed that fenugreek seeds gum was stable at high temperatures and this indicate that the gum can withstand high temperature in food industry such baking. The H- NMR spectroscopy revealed that fenugreek seeds gum is a galactomannan type polysaccharide which contain mannose and galactose While Arabic gum and acacia gum had the same sugars such rhamnose, arabinose, galactose and glucuronic acid, The study showed some light on understanding the gum potential applications in the food industry.

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