A STUDY OF GUAR SEED AND GUAR GUM PROPERTIES

(*Cyamopsis tetragonolabous*)

By

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DEDICATION

To my lovely brother Dr. Mussadag

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ABSTRACT

Guar seed (*Cyamopsis tetragonolobus*) components of three genotypes (HFG53, HFG182 and HFG363), are hull (13.4-14.1%), germ (43.3-44.2%) and endosperm (36.0-40%). The proximate composition of guar seed in mean values is moisture (11.13 ± 0.01%), crude protein (29.10±0.01%), crude fat (1.58±0.01%), crude fibre (9.01±0.01%) and carbohydrates by difference (45.70±0.05%).

The endosperm analysis showed mean values for moisture (6.18±0.03%), ash(1.35±0.03%), crude protein (4.41±0.0%), crude fat (0.30±0.0%), crude fibre (1.59±0.01%) and carbohydrates or gum (86.38±0.04%). The mechanically purified guar gum fractions shows ash (0.67±0.041%), protein (3.85±.029%), fat (0.17±0.007%) and fibre (1.36±0.006%) for the overs (0.5 mm). For the throughs (0.5 mm), the results are ash (2 37±0.041%), protein (7.13±0.029%), fat (0.41±0.007%) and fibre (8.15±0.006%).

The micro- and macro-elements quantities of the endosperm of the three genotypes are as follows: Zn (29-44 mg/kg), Fe (52-112 mg/kg), Cu (2.6-3.8 mg/kg), Pb (0.34-0.38 mg/kg) and As (0.24 mg/kg); Na (0.1-0.5%), K (0.70-0.95%), Ca (0.30-0.37%) and Mg (0.11%), respectively. The micro- and macro-elements of germ and hull are also reported in this study.

The effects of concentration, temperature and dispersibility or hydration rate of guar gum were measuered by Ostwald, Redwood and Brookfield viscosity methods, respectively. The Ostwald relative viscosity of guar gum behave Newtonian up to 0.5 mg/ml. The relative viscosity linear curves have high coefficient of

correlation (r=0.87, 0.82-1.05, and 0.99) for gum of endosperm, (overs) and (throughs), respectively.

Redwood measures kinematic viscosity of guar gum for the three genotypes at varying temperatures 40-80°C. Heat stability of HFG53 is the best among the three genotypes guar gum of endosperms and overs. The high contamination in throughs gum lowered the heat stability of gum of the three genotypes. Highest kinematic viscosities curves were shown by endosperm. This method is a useful technique for measuring guar gum stability. Brookfield method shows a high rate of dispersibility for HFG363 followed by HFG182 and HFG53. The comparative study of the effect of purification on guar gum viscosities measured by Ostwald within the three genotypes show high coefficient of correlation (r=0.87-1.02). Redwood kinematic viscosities within genotypes gives high stability for endosperm followed by overs and throughs. The influence of salt (sodium chloride) concentration 1.0, 1.5 and 2.0% on heat stability of commercial guar gum (200 mesh and 80 mesh) show high viscosities. The salt effect on 80 mesh is less significant, compared to 200 mesh. Sugar influence in heated guar gum solution 0.5% (200 mesh) gives a high viscosity increase than 80 mesh with 5, 10 and 15% added sugar. The effect of combined salt-sugar on commercial guar (200 mesh) gives increased viscosity than the control. The best heat stability produced is given by the combination of low salt-sugar. The salt-sugar effect on guar gum (80 mesh) is non-uniform. Good quality guar gum grades gives better heat stability in combination with salt, sugar or combined salt-sugar.

بسم الله الرحمن الرحيم

خلاصة الأطروحة

أختيرت ثلاث عينات من بذور القوار:HFG53، HFG182 وHFG363 لهذه الدراسة ووجد أن بذرة القوار تتكون من ثلاثة أجزاء هى القشرة (13.4-14.1٪)، الجنين (43.3-44.2٪) والسويداء أو المادة النشوية (36-40٪). كانت نتائج التحليل التقريبى لبذرة القوار كمايلى: الرطوبة (0.01+11.13٪)، الرماد (3.25-3.78٪)، البروتين (0.01+29.1٪)، الدهون (0.01+1.58٪)، الألياف (0.01+9.01٪)، النشويات (0.05+45.7٪). أما بالنسبة للمادة النشويه التى فصلت يدويا فكانت نتائج التحليل كالآتى: الرطوبه (0.03+6.18٪)، الرماد (0.03+1.35٪)، البروتين (4.41٪)، الدهون (0.3٪)، الألياف (0.01+1.59٪)، النشويات (0.04+86.38٪).

تم فصل الماده النشويه ميكانيكيا وبعد غربلتها نتج عن ذلك عينتان (أعلى الغربال وأسفله) وتم تحليلها كمايلى:الرماد (0.04+0.67٪)، البروتين (0.03+3.85٪)، الدهون (0.007+0.17٪)، الألياف (0.006+1.36٪) للعينه أعلى الغربال، أما العينه أسفل الغربال فكانت نتائجها كمايلى: الرماد (04.+2.37٪)، البروتين (03.+7.13٪)، الدهون (0.007+0.41٪)، الألياف (0.006+8.15٪).

نسبة لأهمية المعادن من الناحية الغذائية فقد أختير بعض منها للدراسة فى كل من القشرة، الجنين والسويداء (المادة النشوية) وكانت نتائج الأخيرة كالآتى:الزنك (29-44 ملم/كجم)، الحديد (52-112 ملم/كجم)، النحاس (2.6-3.8 ملم/كجم)، الرصاص (0.34-0.38 ملم/كجم)، الآرثنك (0.24 ملم/كجم)، الصوديوم (0.1-0.5٪)، البوتاسيوم (0.7-0.95٪)، الكالسيوم (0.3-0.37٪)، المغنسيوم (0.11٪).

تم فحص تأثير التركيز، درجة الحرارة ودرجة الذوبان على لزوجة صمغ القوار بإستخدام طريقة (Ostwald)، (Redwood) و (Brookfield) على التوالى. أثبتت طريقة (Ostwald) أن لزوجة صمغ القوار تزداد طرديا مابين التركيزين (0.1-0.5 ملم/مل)، وكان معامل الإرتباط (0.87، 0.82-1.05 و0.99) لكل من السويداء والصمغ أعلى

الغربال والصمغ أسفل الغربال على التوالى. أما طريقة (Redwood) أثبتت أن العينه HFG53 ذات أفضل ثبات حرارى بين درجتى حرارة (40-80 درجة مئوية)، بينما كان ترتيب درجة الذوبان فى العينات الثلاث على النحو التالى:HFG363، HFG182 وHFG53 بإستخدام طريقة (Brookfield).

بمقارنة طرق تنقية صمغ القوار الثلاثة:(اليدويه)، الميكانيكية (أسفل وأعلى الغربال) كان معامل الإرتباط عاليا فى طريقة (Ostwald) للثلاث عينات، (0.87-1.02) أما فى طريقة (Redwood) فكانت العينة التى تم تنقيتها يدويا ذات أعلى لزوجه تلتها العينة أعلى الغربال. تمت دراسة تأثير ملح الطعام والسكر على لزوجة صمغ القوار، لوحظ أن إضافة كل من الملح (1.0، 1.5 و2.0٪) للصمغ يزيد من لزوجته عند إضافة السكر (5، 10، 15) أدى إلى زيادة فى لزوجة صمغ القوار بإرتفاع درجة الحرارة. كما لوحظ أن تأثير كل من ملح الطعام والسكر كان واضحا فى العينه (200 mesh) أكثر منه فى العينه (80 mesh). كما وأن إضافة الملح والسكر لمحلول صمغ القوار زاد من لزوجة الصمغ مقارنة بالمحلول غير المعامل. كانت العينه (200 mesh) ذات ثبات حرارى عالى بينما أعطت العينه (80 mesh) تأثير غير منتظم.

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CHAPTER ONE

INTRODUCTION

Guar gum is found in the seed of two annual leguminous plants (*Cyamopsis tetragonolobus* and *C. psoraloides*). The seeds are contained in pods 2.54 cm to 5.08 cm long.

In India and Pakistan, this crop has been grown for centuries as a food for both humans and animals. Guar gum production is carried out by a series of crushing, sifting and grinding steps to separate the seeds from the pod and then to separate the valuable gum from the seeds.

The gum is contained within a portion of the seed known as the endosperm which is 35 to 42% of the weight of the seed. The endosperm is ground into powder by the usual mechanical processing technique that does not produce completely pure endosperm. Therefore, the gum is not perfectly pure but contains small amount of hull and germ.

Since the whole seed is edible, this contamination dilutes slightly the amount of gum quality, but does not harm its suitability as a food additives.

Guar gum is a cold water swelling carbohydrate polymer. In its produced commercial form the rate of thickening and the final viscosity reflect the process of the product, including the particle size of the powder. Guar gum is made up of the galactomannan units with the chemical structure consisting of 6-D-galactopyranosyl repeating units and 4-D-mannopyranosyl units.

Guar plant is an annual shrub, the old regions of production are India and Pakistan, but recently it has been introduced into United States of America. In the Sudan, guar plant was unknown till recently when this crop was found as a wild plant in the Red Sea mountains and Arashekol mountains of White Nile State. Agronomical trials were conducted at the Agricultural Research Corporation (ARC) on guar genotypes which were brought from Tanzania. These genotypes were successfully grown commercially by Guar Gum Company established at Singa 1996.

The advent of guar gum production in Sudan will pressurize gum Arabic to better commercial production and quality in order to compete in the national and international markets. The good news are that Gum Arabic Company is the largest shareholder in Guar Gum Company. Guar gum is used in many industries such as food industries, baked products, icing, dairy products, meat products, beverage and soft drinks. The non-food industries are paper industry, mining, oil-well drilling, textile, pharmaceutical and explosives.

Objectives of this study will be basically centered on the quality of gum produced from the three genotypes HFG53, HFG182 and HFG363. The investigation will be divided as follows:

1. To study the physical and chemical properties of guar seeds and gum.
2. Purification techniques of guar gum.

3. The rheological characteristics of guar gum in water solution and the effect of salt and sugar on commercial guar gums.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction:

A gum may be generally defined as any water soluble polysaccharide that is extractable from land or marine plants or from microorganisms and that possesses the ability to contribute viscosity or gelling ability to their dispersions. This definition, however, excludes starches and pectins in this group. Common gums from plant include the seed galactomannans of guar and locust bean; the plant exudates, gum Arabic and gum tragacanth; and the sea weed derived gums, agar, carrageenan, and algin.

These polysaccharides gums are used widely for thickening foods. The great number of polysaccharides present in foods perform some useful service based on their molecular structure, their size, and their secondary forces, mainly those of hydrogen bonding.

The bulk of dietary polysaccharides are insoluble and indigestible, mainly cellulose and hemicellulose components of cell walls from vegetables, fruits and seeds. These are inert structures that give physical compactness, crispness, and good mouthfeel to many foods. The remaining polysaccharides in foods are water-soluble or water dispersible and serve numerous divers roles, such as providing hardness, crispness, compactness, thickening quality, viscosity, adhesiveness, gel-forming ability and mouth feel.

They permit food to be designed into shapes and structures that may be brittle or soft, swollable or gellable or completely soluble. The literature discussed in this study is based on the plant gums roles in the food systems especially that of guar gum newly introduced into the food industry and other industries, (Whistler and Hymowitz, 1979).

Guar gum production in Sudan is a very recent development in 1996. Its application in the food and non-food industries was unknown in the country. The well known gum in food industries has been gum arabic which has been known for centuries in Sudan. It is brief citation in this work is necessary since guar gum is entering into competition in the local and external markets with gum arabic.

2.2 Seed Gums:

2.2.1 Guar Gum:

Guar gum or guaran, is the endosperm polysaccharide of the seed of *Cyamopsis tetragonolobus*, family leguminosae, that grows naturally in India and Pakistan and has been introduced as cash crop in the United States in 1905 (Whistler and Hymowitz, 1979).

Sudan has introduced guar as a cash crop in 1996 to supply the only guaran producing industry established in Singa (Sennar State) 365 km South of Khartoum.

Guaran is a galactomannan, containing a backbone of (1-4). B-D-manopyranosyl units with every second unit bearing a (1-6) B-D-galactopyranosyl unit (Goldstein *et al.*, 1973).

The polymer is relatively large, with a molecular weight of about 220,000 daltons. Guaran hydrates rapidly in cold water to give a highly viscous, thixotropic solution. A 1.0% dispersion has a viscosity of about 6000 cps, depending on temperature, strength, and the presence of other food components.

Dissolution of the gum is hastened by heating a dispersion, but the gum is degraded at very high temperatures. Because guar gum can produce solutions of high viscosity, it is usually used at a concentration of 1.0% or less in foods. Since the gum is neutral, its solution viscosity is little affected by changes in pH. Salts have little effect on its solution viscosity, but large amount of sucrose may reduce viscosity and delay attainment of maximum viscosity (Whistler and Daniel, 1985).

Guaran exhibit viscosity synergism with wheat starch and some other gums. It is used in cheeses, in which it eliminates syneresis, and in ice-cream, in which it contributes body, chewiness, and resistance to heat shock. In baked goods it promotes long shelf life; and in pastry icing it lessens absorption of water by sucrose. Guaran is also used in meat products, such as sausage, to improve casing stuffing. When used in dressings and sauces at a level of 0.2 - 0.8%, guaran increases viscosity and contributes to a pleasant mouth feel (Whistler and Daniel, 1985).

2.2.2 Locust Bean Gum:

Another plant seed galactomannan, is derived from the carob seed *Ceratonia siliqua*. The plant grows predominantly in the Near East and Mediterranean areas. It consist of a D-mannopyranosyl back bone with attached D-galactopyranosyl units, the two components existing in a ratio of 4:1, respectively (Rol, 1973). However, its D-galactopyranosyl units are not uniformly distributed, leaving long stretches of the mannan chain devoid of D-galactopyranosyl units. This leads to unique synergistic properties, especially with seaweed polymer carragenan, where the two cross-link to form a gel.

Locust bean gum is employed in frozen desserts to bind water, and provide body, smoothness, and chewiness. In soft cheese manufacture it speeds curd formation and reduces the loss of solids. In composite meat products, such as salami, bologna and sausages, it acts as binder (Whistler and Daniel, 1985).

2.3 Exudate Gums:

2.3.1 Gum Arabic:

Among the plant exudates polysaccharide, gum Arabic is the oldest and best known. It is produced as tear-drop-shaped globules exuding from bark wounds of Acacia trees (Glicksman and Sand, 1973).

Production of the gum is stimulated by intentional removal of bark.

Gum Arabic in Sudan:

Gum arabic is an oldest, national dominated commodity in the Sudan. Sudan is the leading country in the trading and exportation of gum Arabic, it represents 70-85% of world gum export (Anon, 1996).

Production of gum arabic is facing stif competition from other gums including synthetic polysaccharide gums. Gum arabic is a complex heteroglycan with a molecular weight of 250,000-1,000,000 daltons. The polymer usually contains some minor amounts of L-arabinose and L-rhamnose. Gum arabic dissolves readily in water to produce solutions of low viscosity. It can be dissolved to an extend of 50% to form a high gel similar to that from starch.

At a concentration of less than 40% its solution exhibits Newtonian rheology; above 40% concentration the dispersions are pseudo plastic. High quality types of this gum form colourless, tasteless solutions (Whistler and Daniel, 1985). Due to the presence of ionic charges, the viscosity of solutions of gum Arabic change with changes in pH. Viscosity is low at low and high pHs and reaches a maximum at pH 6-8.

Gum arabic is incompatible with some food polymers, such as gelatin and sodium alginate, but is compatible with most other gums.

2.4 Guar:

Guar plant belongs to family Fabaceae or Leguminaceae. Common name, is guar, from "sankrist word" "go" or "gav". Local name is cluster bean. Latin name is *Cyamopsis tetragonolobus* or *C. psoraloides*.

2.4.1 History:

The early history of old-world legume guar is unknown. However, established records and circumstantial evidence indicates man cultivated guar in Indo-Pakistan sub continent for numerous generations, until recently guar has remained as a minor crop. Now it seems destined to assume a larger role among the domesticated plants that supply the food and needs of man.

The discovery that guar seed endosperm could be a source of useful industrial gum brought this little known crop world recognition and started on its way to major crop prominence (Hymowitiz and Matlock, 1963).

Guar gum has grown rapidly in industrial usage since its first industrial trail during world war II. Guar was first investigated as a source of gum in 1945 (Whistler, 1948).

2.4.2 Distribution:

Guar seed production is concentrated in India, Pakistan and United States. Most guar is produced in regions in which the average rainfall is 75 cm or less per year (Whistler and Hymowitiz, 1979).

2.4.3 Traditional Uses:

The traditional uses of guar in the Indo-Pakistan sub-continent are as follows:-

2.4.3.1 Human Consumption:

Immature pods are dried, salted and preserved for future use (Wiser, 1955, Huprikar and Sohanie, 1961). Immature pods are dried and fried like potato chips. Mature pods are also used as an emergency pulse in time of drought. Green pods are cooked like French fried beans (Murry, 1908).

2.4.3.2 Cattle Feed:

Plants were cut and fed as green forage (Murry, 1908). Beans are bailed in a large kettle and fed to cattle as high protein source (A dictionary of economic products of India, (1889-1896).

2.4.3.3 Medical Purposes:

Plants are ashed, then mixed with oil and used as poultice on cattle boils (Indraji, 1910). Leaves are eaten to cure night blindness (Indraji, 1910). Seeds are used as a chemothrapeutic agent against smallpox. Boiled guar seeds are used as poultices for the plague, enlarged liver, head swelling and on swelling due to broken bones (Roxburgh, 1814). Seeds are used as laxative (Chopra *et al.*, 1956).

2.4.4 Entry of Guar into Sudan:

Guar plant is found as a local crop in the areas of Red Sea mountains and Arashekol at White Nile State. Guar plant tolerates different types of soil ranging from sandy to heavy clay soil, but it is successfully grown in sandy and light clay soils of Sudan. In Sudan breeding and agronomical trails

were carried out with four genotype materials from Tanzania. The yield and quality of these genotypes were found to be compatible with Sudan climate (Anon, 1990).

2.4.5 Guar Plant:

The length of the plant reaches two feet with many branches which start above the soil level Figure (1). Guar plant has strong root system with many large nodules. It has opposite leaves, small white flowers. Guar capsule length is about 12 cm which contains 6-12 seeds, white to black in colour, it does not exceed 8 mm in length Figure (2).

Formation of capsule starts above the soil level 5-10 cm up to tip of the plant in a dense groups or clusters Figure (3). Guar takes about 120-160 days from sowing till maturity depending on the variety and humidity.

2.4.6 Guar Seed:

Surprisingly, although guar primarily is grown for its gum content, very little was known about the variability of gum content in the seed of Cyamopsis tetragonolobus. In his pioneering work, Anderson (1949) screened seeds of 163 species of legumes for sources of endosperm gums. He reported that guar seed contain about 50% endosperm and yielded approximately 42% gum.

According to Goldstein and Alter (1959) report, the three major components of guar seed are the seed coat (14-17%), endosperm (35-42%), and germ (43-47%). The germ contains most

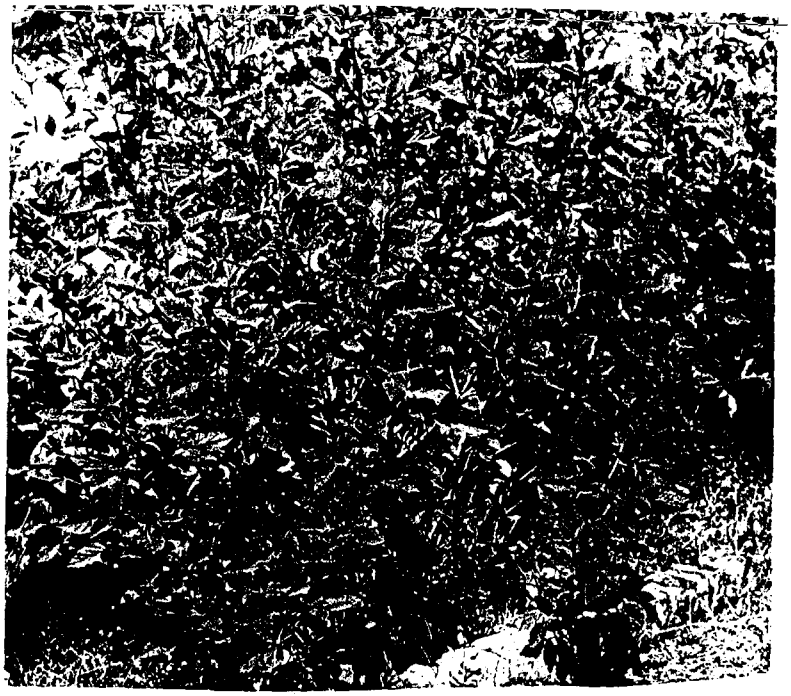

Fig. 1: Guar plant

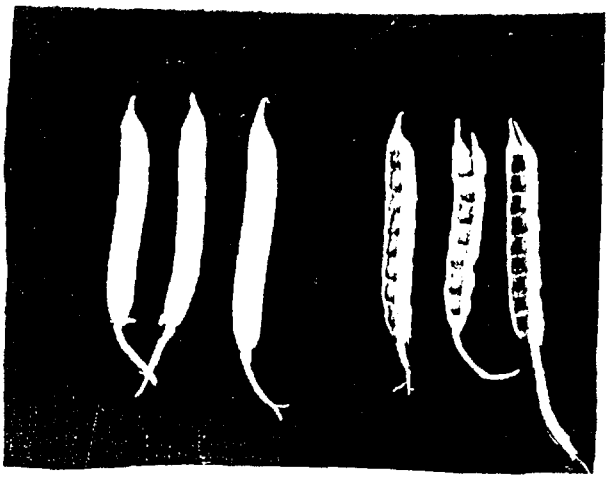

Fig. 2: Guar pods

Fig. 3: The formartion of guar pods

of the protein in the seed while the endosperm contains the galactomannan gum.

Menon *et al.* (1972) found that gum content of a whole seed, endosperm content, and percentage gum in endosperm varied from 19.1-34.1%, 38.0-49.9%, and 47-68%, respectively, in 86 guar selections grown in 1968 in India.

4.7 Guar Gum Production:

The guar seed is dicotyledonous, having a diameter of approximately 1/8 in (8 mm). The hull is loosened by water soaking and removed by grinding. The difference in hardness of the various seed components is utilized, and a purification is accomplished by multistages of grinding and sifting. Germ is removed by differential grinding. There are many types of hammer and roller mills, that can be employed because of the difference in hardness of the endosperm and germ.

After the endosperm is separated from the hull and germ, it is ground to a fine particle size and marketed as guar gum (Whistler, 1973). The vitreous parts of the seed, or "splits" as they are sometimes called, are ground to a suitable mesh size for specific uses in industry without further purification; except that the material may be treated to in- activate the enzymes (Smith and Montgomery, 1959).

2.4.7.1 Guar Gum Purification Method:

Commercial gum was purified by the usual method of precipitation with ethanol. A 1.0% aqueous solution of the gum was prepared by vigorous stirring. After standing for 2-hrs at

room temperature, the gum was precipitated by addition of 50% ethanol. The white amorphous mass so obtained was washed with several volumes of ethanol and finally the precipitated gum was washed with di-ethyl ether and dried at room temperature (Gupta *et al.*, 1978).

2.4.8 Guar Gum Properties:

2.4.8.1 Physical Properties:

One of the important properties of guaran is its ability to hydrate in cold water to produce high viscous solution. Guar gum has D-galacto-pyranosyl branch on every D-mannopyranosyl unit, the greater number of branches in guar gum is responsible for both its easier hydration and its different hydrogen-bonding activity as compared to that of locust bean gum (Whistler and Hymowitiz, 1979).

Hydration rates and water binding properties of guar depend on the processing techniques used in making the gum, and upon the final conditions of the solid materials particularly its particle size.

Guar gum when completely hydrated form a viscous, colloidal thixotropic dispersions. Solutions of guar gum of less than 1% are less thixoptropic than solutions of concentrations of 1% or high. Physical properties are important in determining the commercial values and the use of such a product.

Newtonian Flow:

Newtonian fluids exhibits direct proportionality between shear rate and shear stress. At any given temperature, those materials have viscosity that is independent of the rate of shear. In simple term, it take twice as much force to move the liquid twice as fast (Glicksman, 1969).

- **Non-Newtonian Flow:**

Non-Newtonian flow is characterized by the fact that the viscosity is not directly proportional to the rate of shear, but varies with rate of shear, in certain systems, is also dependent upon the parameter of time (Charm, 1960).

- **Pseudo plastic Flow:**

A pseudo plastic material flows more readily as it stirred or sheared. It is irreversible structural break down and shows a decreasing viscosity with an increasing rate of shear.

Thixotropic Flow:

Thixotropic is formally defined as "a reversible gel-sol--gel transition" and is caused by the building up of definite structure within the material. The gelled structure upon shaking or stirring become a sol, which if allowed to remain undistributed, become gelled again (Glicksman, 1969).

Guar gum viscosity increases linearly (Newtonian) with increases in concentration up to 0.5%. At higher concentrations guar solutions behave as non-Newtonian, thixotropic systems (Glicksman, 1969).

2.4.8.1.1 Viscosity of Guar Gum:

Viscosity of a liquid is its resistance to shearing, stirring or to flow through a capillary tube. Viscosity was considered as one of the most important analytical and commercial parameters, since it is a factor involving the size and shape of the macro- molecules (Anderson, 1969). Viscosity can be presented in many terms such as relative viscosity. inherent viscosity and intrinsic viscosity.

Viscosity is a commercial value of the gum. the higher the viscosity of the gum, the higher its commercial value. Gums are industrially demanded according to their viscosities. The molecules of the gum readily undergoing change both in shape and size, thus indicating that the forces of attraction between neighbouring sugar units are quite weak (Briggs and Haning, 1944). This behavior is typical of gums possessing a high branched chain structure and it should be compared with that of gums and mucilages like guar and locust bean gum whose molecules though branched, are essentially linear in character. The difference is illustrated by the fact that a 20 percent solution of gum arabic has the same viscosity of 0.5% solution of locust bean gum (Coumu, 1935).

Gums which give solutions that are sensitive to pH are usually characterized by the presence of carboxyl or sulphate groups. Neutral gums such as guar, forms solutions which are not dependent on pH. The viscosity does fall, however in the presence of strong alkali, but this may be due to destruction

of the protein which forms a complex with the carbohydrate polymer. Mechanical grinding of gums, a process usually practiced in commerce, leads to molecular brake down. This is revealed by the fact that solutions made from unground pieces of gum tragacanth are more viscous than those of the same concentration made from the powdered gum (Chambers, 1949).

Aqueous solution of some gums show a decrease in viscosity with time. This is true of guar and locust bean gums and it is probably due to enzymatic degradation (Gayezo and Otero, 1951). Ionization of soluble neutral polysaccharides remains roughly constant over a wide range of pH values and is greatly altered only in strong alkaline solutions. Therefore, these polysaccharides maintain their shape and absorb water essentially at pH near neutrality, and except in strongly alkaline conditions their solution viscosities vary only slightly with changes in pH. It is easy to state that neutral. linear and branched polysaccharides will differ greatly in their ease of dissolution and in their ability to affect viscosities. Linear molecules will dissolve more readily but will form solutions that at equal concentration with branched molecules of the same molecular weight, have much lower viscosity (Whistler, 1973).

Some polysaccharides are long chains with numerous very short branches. These molecules have many of the properties of both linear and highly branched molecules. Good examples are guar and locust bean gum. Guar gum was originally developed as

a replacement for locust bean gum. However, it soon becomes obvious that, although both gums are galactomannans, there are significant differences in their chemical composition and behaviour. Locust bean gum requires cooking at elevated temperatures to achieve its maximum viscosity, whereas guar gum will hydrate in cold water (Whistler and Hymowitiz, 1979).

The number of branches in guar is responsible for both its easier hydration and its different hydrogen-bonding activity as compared to that of locust bean gum (Ahmed and Whistler, 1950).

As with most other gums, the viscosity of guar is dependent on time, temperature, concentration, pH, ionic strength and the type of agitation. A 1.0% aqueous dispersion of good quality guar gum has a viscosity of 3000-6000 CPs.

The temperature influences the rate of hydration and development of maximum viscosity. Guar solutions prepared at higher temperatures reach maximum viscosity much faster than those at lower temperatures. However, the advantage of using heat to achieve faster hydration of the gum is partly off-set by the possible degradative effect of prolonged heat in certain processing conditions. In many cases, solutions of guar prepared by heating have a lower final viscosity than the same guar solution prepared with cold water and allowed to hydrate slowly. The maximum viscosities of guar gum dispersions are achieved at temperatures of about 25-40°C. The

factors influencing the rate of viscosity development are the increase in viscosity caused by short-term heating and the decrease in viscosity caused by the degradative effect of prolonged heat. In dilute solutions, the viscosity of guar gum increase linearly with concentration up to 0.5%. Thereafter guar solutions behave as non-Newtonian solutions mainly as a result of complex molecular interactions at higher concentration (Glicksman, 1969).

2.4.8.1.2 Effect of pH:

Guar gum is stable in solution over a wide pH range. Guar solutions have an almost constant viscosity over pH range of about 1.0-10.5. This stability is believed to be due to non ionic, uncharged nature of the molecule. While the pH does not affect the final viscosity, however the maximum hydration takes place at pH 8.0-9.0. Slowest hydration is at pH, above 10.0 and below 4.0. The preferred method of preparing a guar solution is to dissolve the gum at pH of fastest hydration rate and then to adjust the pH to desired value. Maximum viscosities achieved at both acid and alkaline pH's are the same despite the difference in hydration rates (Whistler and Hymowitiz, 1983).

2.4.8.1.3 Effect of Salt:

Since salt and sugar are probably the two most widely used ingredients in food other than water, their effect on guar has been extensively investigated (Carlson *et al.*, 1962).

The behaviour of guar in brine is essentially the same as in water. The hydration rate is not affected, although the final viscosity is somewhat increased by the presence of sodium chloride. This property has made it a very valuable component of oil-well drilling muds, where the capacity of maintain high viscosity in the presence of brine encountered during drilling operation is absolutely essential.

2.4.8.1.4 Effect of Sugar:

In the presence of sugar, there is a competition of the sugar and guar for the available water, and high percentages of sugar have a marked delaying action on the hydration of the gum. In addition, the viscosity of guar sugar solution decreases in direct proportion to the sugar concentration (Carlson and Ziegenfuss, 1965).

Viscosities of guar solutions containing sugar continue to increase for several days and this delay of hydration rate may be due to a reduction in the mobility of the water, proportional to the sugar concentration. In such systems, the full value of guar gum as a thickening and stabilizing agent is developed after about a week of storage. Sugar is effective in protecting guar gum against hydrolysis and loss of viscosity when heated or autoclaved.

The presence of 5-10 % sugar in the liquid offers maximum protection with maximum viscosity. Sugar also offers protection from the hydrolyzing effect at low pH's (down to pH 3.0) under cooking conditions (Carlson and Ziegenfuss, 1965).

2.4.8.1.5 Synergistic Effect:

Guar gum has shown a high degree of synergism when combined with other gums or starches (Carlson *et al.*, 1962). They have reported that the viscosity of a combination of guar and wheat starches cooked at high temperature is higher than the combined thickening capacity of the two individual ingredients. It is felt that the wheat starch, which has an excellent capacity to withstand thermal and mechanical abuse, it is tied to the guar gum by means of hydrogen d-bonding reaction.

2.4.8.1.6 Gelling Property:

Borate ion acts as a cross linking agent with hydrated guar gum to form cohesive, structured gels. The formation and strength of these gels are dependent on the pH, temperature, and concentrations of reactants. The optimum pH range for gel formation is 7.5-10.5. The solution-gel transformation is reversible; the gel can be liquefied by decreasing the pH below 7.0 or by heating.

Other polysaccharides with numerous adjacent hydroxyl groups in the cis position can form similar three-dimensional borate gels. Borated gels can also be liquefied by the addition of glycerol or manitol, capable of reaction with the borate ion.

Borate ion will inhibit the hydration of guar gum if it is present at the time the powdered gum is added to water. The minimum concentrations necessary to inhibit hydration are

dependent on pH. For example, with 1.0% of guar gum, 0.25-0.5% (based on guar weight) of borax (sodium tetraborate) is needed at pH 10.0-10.5, while at pH 7.5-8.0, 1.5-2.0% of borax is required. The complexing reaction is reversible and lowering the pH below 7.0 permits the gum to hydrate normally. This technique is often used to provide better mixing and easier dispersion (Whistler and Hymowitiz, 1979).

2.4.8.2 Chemical Properties of Guar Gum:

2.4.8.2.1 Chemical Structure:

Guar gum is a galactomannan. The endosperm of guar is guaran. It contains 34.6% D-galactopyranosyl units and 64.4% D-mannopyranosyl units. Guar gum forms of structure consisting of 6-D-galactopyranosyl repeating units in a polymer chain (Figure 4). In other words the polymer is a chain of 1-4 linked B-D-mannopyranosyl units with every second unit bearing on carbon 6 (C6) a single side chain consisting of -D-galact-opyranosyl group (Baker and whistler, 1975).

The ratio of D-galactose to D-mannose in guar gum is 1:2 (Heyne and Whistler, 1948).

2.4.8.2.2 Detection and identification of Gum and Mucilages:

The identification of a specimen of a gum or mucilage may be relatively simple if full use of the present knowledge of these substances and the available analytical techniques were understood. However it is sometimes found to be more conve-nient in industry, for one reason or another, to use a mixture

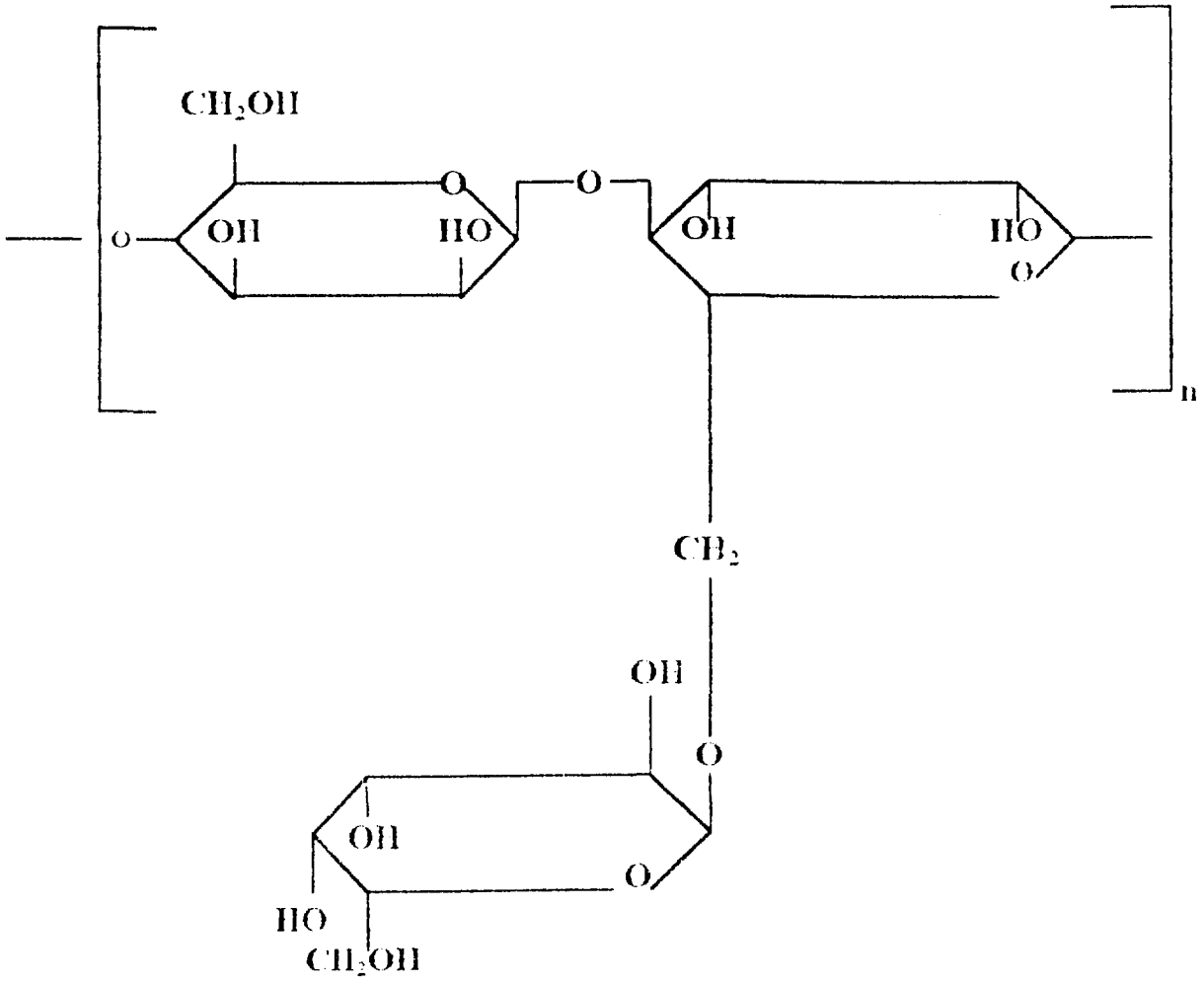

Fig. 4: Proposed structure of guaran

of two or more gums or even to add some other carbohydrate compounds, such as lactose. sucrose, starch or starch dextrin, in which cases the problem of identification is more complex and difficult.

In the native state, single specimen of gum may sometimes be recognized by their physical appearance such as size, shape, colour and brittleness. Commercial samples of gum cannot be recognized in this way since most of them are used in the powdered form. Some gums can be recognized by the way they dissolve or disperse in water.

In general, the natural gum exudates, such as gum Arabic dissolve more readily and completely, and give less viscous solutions than the root and seed mucilages. The physical appearance and the rate of settling precipitates formed when aqueous solutions of the gums are treated with alcohol may also be used as a preliminary test for the identification of gums (James and Smith, 1945, Scklubach and Lubbers, 1956).

In applying this technique for the identification of commercial gums in food products, it has been stated that locust been gum gives stringy, adherent precipitate. Karaya gum yields fine filaments, non-adherent particles while agar gives rise to a heavy flocculent precipitate which readily settles. The preliminary tests should also include measurement of the specific optical rotation in either water or dilute alkali. The specific rotation is also useful for identification purposes.

The more modern technique of infra-red analysis should also prove to be useful since the various components, sugar and glycosidic linkages can often be distinguished (Baker *et al.*, 1956).

The technique of deferential thermal analysis may well prove to be available analytical method in the study of gums and mucilages. With a mixture of gums or other polymer of gum-like character, it is useful to take advantage of certain specific reactions that gums and mucilages undergo with a series selected coagulating reagents.

Identification of gums may be effected in certain cases by the way they react with dyes (Trans, 1920; Jong and Kok, 1942). Gum arabic with one drop of Millon reagent, a white precipitate forms slowly. No precipitate is formed with an excess of the reagent. For locust bean gum. iodine solution added to a solution of gum produces a purplish colouration, and with sodium tetraborate a solution of the gum gels. This is not specific for locust bean gum, but for the galactomannan gums isolated from seeds of leguminous plants (Arni and Percival, 1951).

2.4.9 Guar Gum Applications:

2.4.9.1 Food Industry

- **Processed Cheese Products:**

Guar gum has found wide spread application in soft cheese products of various types, and guar as well as locust bean gum is approved as an optional ingredient by Food and Drug Administration in standard of identity for cheese products (Whistler and Hymowitiz, 1979).

In cold-pack cheese foods, it is allowed at levels up to 3.0% of the total weight (Federal Register, 1965). The use of guar in cold-pack products, in addition to eliminate syneresis or weeping, also gives improved products having more uniform texture and flavours due to its control of moisture distribution and migration (Klis, 1966).

In soft cheeses, the use of guar increases the yield of curd solids and produces curds of a soft, compact, tender texture with separated lipid whey.

- **Packed Goods:**

Stabilizers are often added to packaged cake mixes and guar gum offers several functional advantages in single-step mixing procedures, reduced butter mixing time, reduced crumbling in the finished cake, easier application of frostings and icings, and greater moisture-retention during prolonged shelf life. Guar gum also is used at concentrations in the range of 0.1-1.15% of the total weight of the dry ingredients.

The addition of guar solutions to doughs while kneading give increased yields and produce a dough of greater resiliency and a drier, less flabby appearance. Baked products have a better, softer texture as well as a longer shelf life.

In cake and biscuit doughs, the addition of guar gum yields softer, more moist products that are more readily removed form their pans and easily sliced without crumbling.

- **Pastry Icings:**

A common problem in packed iced bakery goods is the accumulation of moisture within the transparent wrapping. The moisture causes the icing to adhere to the wrapping and result in an unpleasant appearance. Addition of guar gum to icings eliminates this problem by absorbing the free water.

- **Meat Binder:**

The strong water-holding properties of guar gum in both hot and cold water make it effective as a binder and lubricant in the manufacture of sausage products and related stuffed meat products (Whistler and Hymowitiz, 1979).

- **Canned Meat products And Pet Foods:**

In the processing of canned meat products, the addition of guar gum to about 0.5% of the total weight including liquid offers processing advantages. Some of these are prevention of fat migration during storage, control of free water separation in the can during storage and reduction of the tendency for void development in the can (Whistler and Hymowtiz, 1979).

- **Dressings and sauces:**

Guar gum is often used as a thickener in salad dressings at about 0.2-0.8% of total weight. Its advantages are its cold-water dispersibility, its compatibility with highly acidic solutions, and its comparatively low cost on viscosity basis. It functions as an emulsion stabilizer by increasing the viscosity of the aqueous phase, thereby decreasing the separation rate of water and oil phases. Guar is useful as a

thickener in pickle and relish sauces when rapid cooling is used (Burrel, 1958).

- **Beverages:**

Guar is often used as a thickening or viscosity control agent in beverage levels of 0.25-0.75% guar, based on total weight of the product. Guar gum is useful because of its resistance to break down under the low pH conditions. In addition, since guar is soluble in cold water, it is easy to use in most beverage processing plants. It is economical to use and practical because of its high viscosity, fast hydration rate and extremely bland flavour. For most applications it is used at 0.1-0.15% of the weight of the beverage.

Blend of guar gum and carragenan are used in certain cocoa products such as chocolate syrups and powdered chocolate mixes as effective stabilizing and suspending agents (Whistler and Hymowtiz, 1979).

2.4.9.2 Non-food Industry:

- Tobacco:

In this process, finely-ground tobacco leaves and stems are mixed in slurry with guar gum and a cross-linking agent such as glycoxal. The mixture was then poured onto metal belt as a film of appropriate thickness, which after drying, is stripped off in a continuous operation (Whistler and Hymowitiz, 1979).

- **Cosmetics and Pharmaceutical:**

Guar gum is used to thicken various cosmetics and pharmaceuticals (Hutchins and Singiser, 1955, Chudzikowski, 1971). It is used as a binder and disintegrater for compressed tablets (Eatherton *et al.*, 1955)

- **Mining Industry:**

Guar gum is used in forth flotation of potash as an auxiliary reagent, depressing the gangue minerals, which might be clay, talc or shale (Atwood and Bourne, 1954). Guar gum is also used as a flocculent or settling agent to concentrate ores in the mining industry. In which, filtration operations are designed to remove suspended slimes or clay particles. These small particles tend to form compact filter cake that traps water and plugs the filter. The addition of guar gum to a pulp, results in flocculation of these small particles.

The large flocs no longer have a tendency to clog the filter screen and allow a faster flow of liquid through the filter cake. The result is faster filtration and a drier cake. Guar gum is approved by the US public Health Service for use in potable water treatment as coagulant aid in conjunction with such lime (Calcium oxide). It increases the size of floc initially formed by the coagulants, thereby increasing the rate of settling of solid impurities, reducing solids carry over to the filter and increasing periods between back washes (Whistler and Hymowitiz, 1979).

In industrial waters, guar gum flocculates clays, carbohydrates, hydroxides and silica when used alone or in conjunction with inorganic coagulants.

- **Oil-well Drilling:**

Water-soluble polymers have found abroad range of application in the production of petroleum. They serve one or more functions, such as water-loss control, viscosity control, flocculation, suspension, turbulent friction reduction or mobility control. Drilling fluids range form clear water through conventional clay suspensions to barite-loaded muds.

The wide range of properties required for these fluids has led to a proliferation of additives. Guar and Xanthan gums are tolerant of salt and widely used as additives (Whistler, 1973).

- **Explosives:**

In the production of water resistant ammonium stick explosive, guar gum is used as a binding agent. When the explosive stick is immersed in water, the gum in the outer wall swells rapidly and the resultant gel retards leaching of the salts. It is also used as a thickener and gelling agent for slurry explosives.

- **Paper Industry:**

The major use of galactomannan in paper making is in the wet end of the process. The pulping process, which designed to remove lignin and thereby produce a fibrous cellulosic pulp also remove a large part of the hemicelluloses normally

present in the wood. These hemicelluloses, which are mostly mannans and xylans, could contribute greatly to the hydration properties at the pulp and strength of the paper formed from the pulp.

Galactomannan replace or supplement the natural hemicelluloses in paper bonding. Advantages gained by addition of galactomannans to pulp include improved sheet formation with a more regular distribution of pulp fibers (less fiber bundle), increased fold strength, increase tensile strength, increased pick in printing grades; pick is a measure of force required to pull a fiber from the surface of a sheet), easier pulp hydration, improved finish, decreased porosity, increased flat crush of corrugating medium (this refers to the pressure required to crush corrugated flute), increased machine speed with maintenance of test results and increased retention of fines (Whistler, 1973).

- Textile Industry:

Guar gum can also be used in the textile industry as printing thickener for fabric and carpets, it aid in friction reduction, reduction, improve viscosity and control fluid loss (Whistler and Hymowitiz, 1979).

CHAPTER THREE

3. MATERIALS AND METHODS

3.1 Materials:

3.1.1 Guar Seeds:

Three guar genotypes were obtained form Gezira Research Station which are HFG53, HFG182 and HFG363 Figure (5). These genotypes are now produced in Sudan on commercial scale.

3.1.2 Preparation of Materials (Shown in Figure 6):

3.1.2.1 Manual Purification:

Guar seeds were sieved to remove broken seeds, soil particles and foreign-materials. The seeds were soaked in sufficient distilled water for about 10-12 hours. The seed was swollen due to imbibition. The outer layer (Hull) was removed easily and the medium layer (Endosperm) was opened into two to separate the inner portion (the Germ). These were oven dried at a temperature 100°C and ground (Fig. 6) (Bureng, 1996).

3.1.2.2 Mechanical Purification:

Guar seeds were purified mechanically, using stone mill, United Milling System, dehuller and Sifter (Figures 7 and 8) (Bureng, 1996).

3.1.2.3 Particle size Preparation:

This was done as shown in Figure (6).

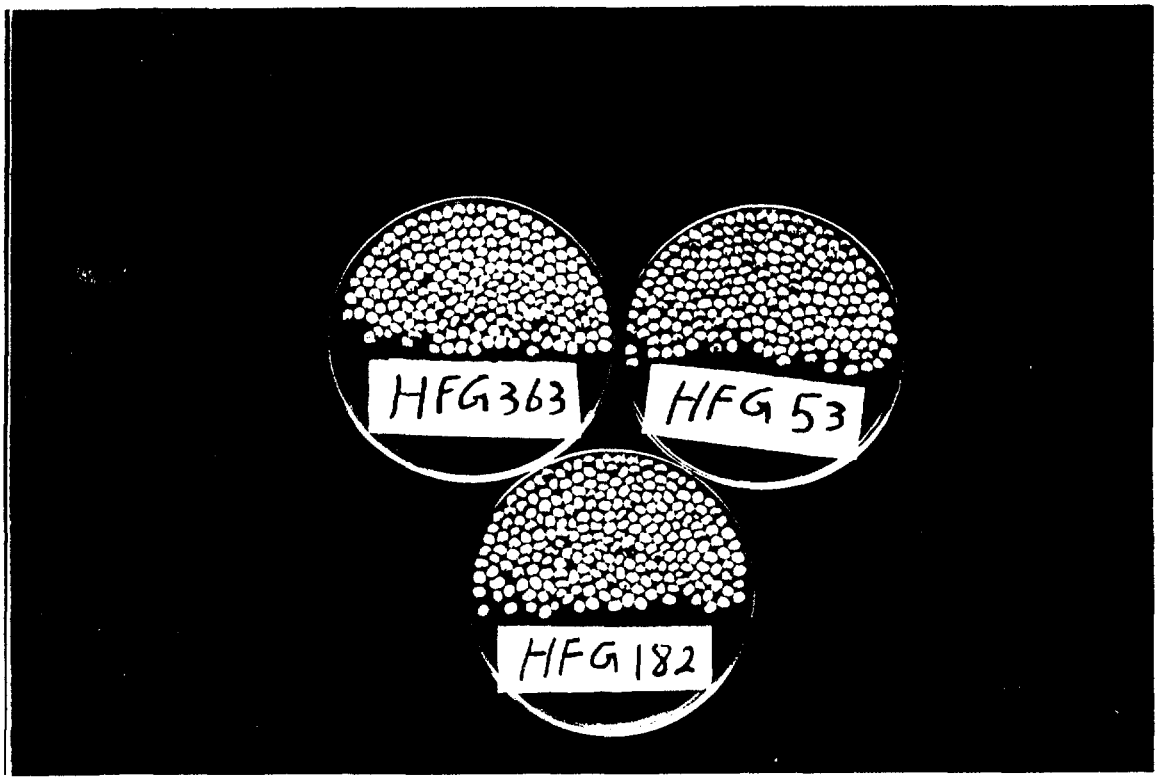

Fig. 5: Guar seed of three genotypes (HFG53, HFG182 and HFG363)

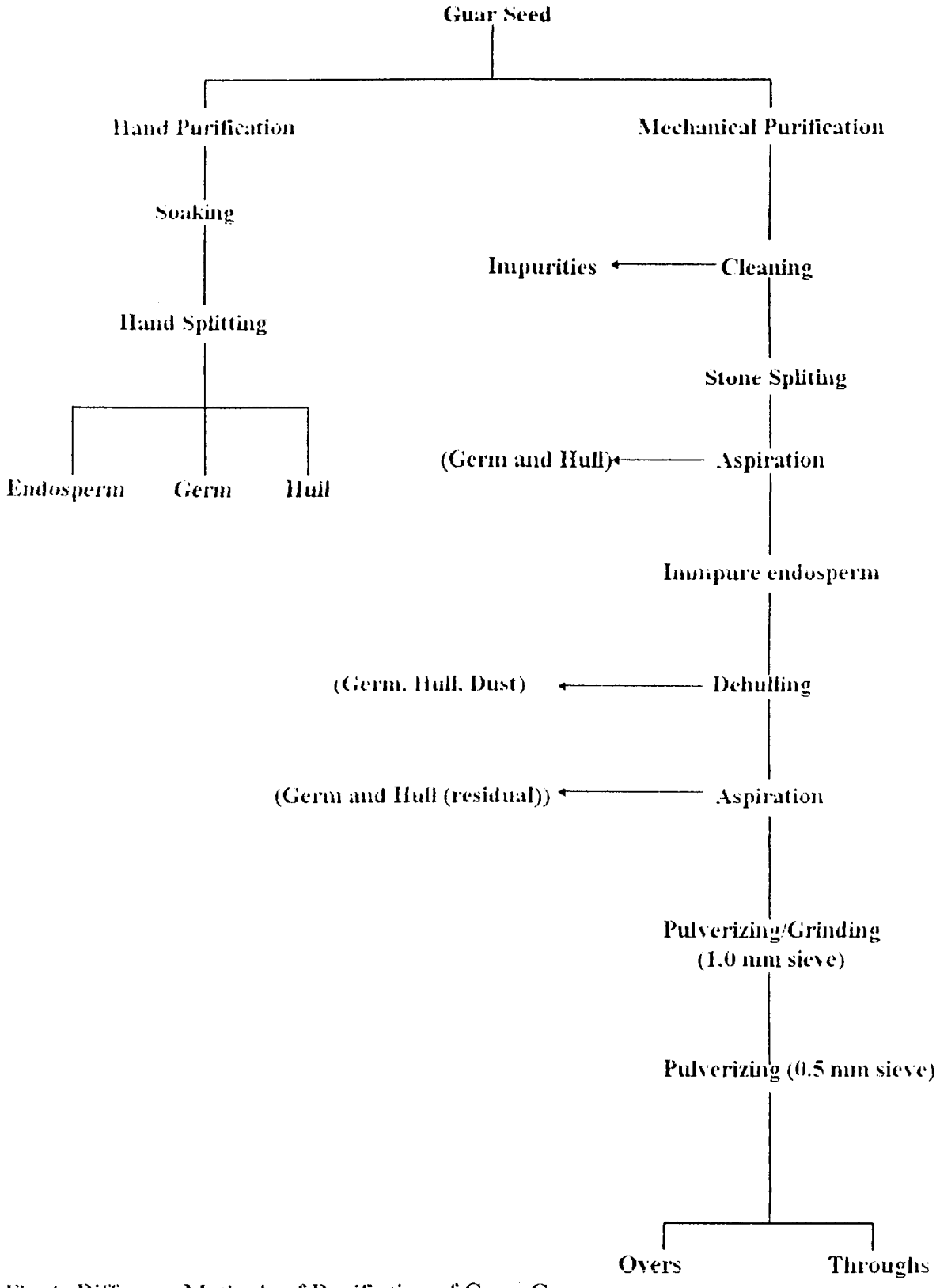

Fig. 6: Different Methods of Purifiation of Guar Gum.

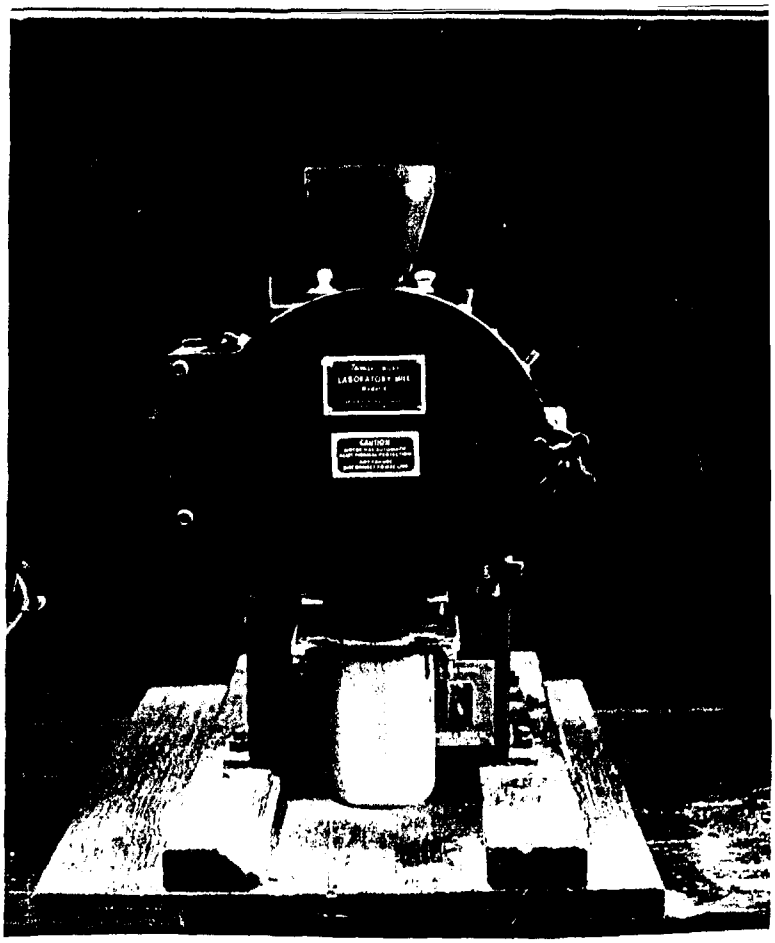

Fig. 7: Hammer mill (United Milling System)

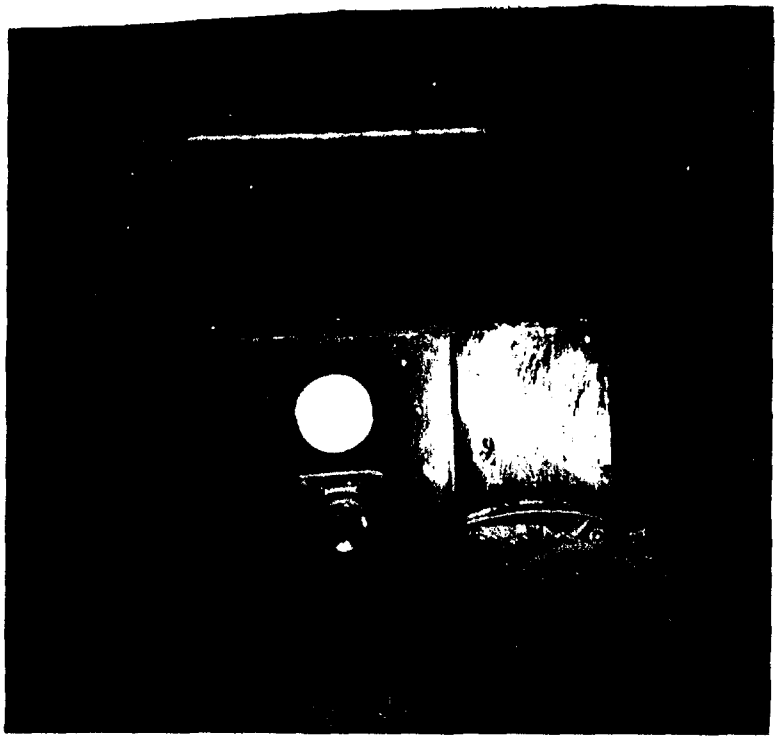

Fig. 8: Dehuller

3.2 Methods:

3.2.1 Physical Analytical Methods:

3.2.1.1 Germination Test:

Using capacity germinating method, hundred intact seeds were randomly taken, placed on a wet filter paper in a sterilized petri dish and left for about 24-48 hours. This was done in a triplicate for each genotype. The percentage of germination was obtained by calculation as follows:

$$\text{Germination\%} = \frac{\text{No of germinated seeds}}{\text{Total No of planted seeds}} \times 100$$

[Thesis, 1994]

3.2.1.2 Thousand Seed Weight:

It was determined according to AOAC (1984).

3.2.1.3 Hectoliter weight (gm/liter):

Using a test weight measuring device, the weight was measured and recorded in gm/l (ICARDA, 1986).

3.2.1.4 Black Seed Percent:

100 grams of intact guar seed were weighed. Black seeds were selected, weighed and then the black seeds percent was calculated as follows :-

$$\text{Black seeds\%} = \frac{\text{Weight of black seeds}}{\text{Total weight of seeds}} \times 100$$

3.2.1.5 Hard Seed Percentage:

According to Hulse method (1977) 100 seeds of guar were randomly taken, soaked in distilled water for about 12 hrs,

the hard seeds (unsoaked) were selected, calculated and then the hard seed percent was obtained.

$$\text{Hard seeds\%} = \frac{\text{Hard seeds}}{\text{Total No. of soaked seeds}} \times 100$$

3.2.1.6 Determination of Guar Seed Components:

Five grams seeds were weight, then soaked in 100 ml distilled water for about 10-12 hours and manually separated into hull, endosperm and germ. Then these were oven dried. Each part (hull, germ and endosperm) was weighed separately until constant weight was reached. The percentage of each was calculated as follows:

$$\text{Part\%} = \frac{X}{5} \times 100$$

where:

x = The weight of each part (Bureng, 1996).

3.2.2 Chemical Analytical Methods:

3.2.2.1 Moisture Content:

Moisture content was carried out at 105°C according to AOAC (1984). The moisture percent was obtained as follows:-

$$\text{Moisture\%} = \frac{(W1 - W2)}{W1} \times 100$$

Where:

W1 > Original weight of the sample.

W2 > Weight of the sample after drying.

3.2.2.2 Ash Content:

The percentage of ash was obtained by combusting the material at 550°C using muffle furnace to ash (white or grey) according to AOAC (1984). Ash percent was calculated as follows:-

$$\text{Ash\%} = \frac{\text{Weight of residue}}{\text{Weight of sample}} \times 100 \times \frac{(100)}{100 - M}$$

Where:

M = Moisture % of sample.

3.2.2.3 Crude Protein:

Crude protein was determined according to AOAC (1983) with some modifications using kjeltec system 1 stream lines.

The steps are as follows:-

- 2.0 gm were weighed.
- 7.0 ml of sulphuric acid concentrated (Analar grade) were added to the guar samples.
- 5.0 ml of hydrogen peroxide were added
- Digestion of the sample was carried out at 420°C for 20 - 45 min.
- After cooling the digested sample was diluted by adding 25 ml of distilled water.
- 25 ml of boric acid (4.%) were used in the receiver flask.
- The tube containing the sample was connected on the distilling unit 50 ml of NaOH (40%) was dispensed.
- Distillation was carried out for about 7-10 min.

- The receiver flask was then titrated against hydrochloric acid 0.1M. The crude protein percentage was calculated as follows:

$$\text{Crude protein\%} = \frac{(5- B) \times N \times 14 \times 6.25}{\text{Weight of sample} \times 1000} \times 100$$

Where:

5 = ml back titration of blank

B = ml back titration of sample

N = Normality of acid,14 molecular weight of nitrogen, 6.25 = protein factor for guar seed.

3.2.2.4. Crude Fat:

Crude fat was determined according to (AOAC, 1984) method.

The method is a brief modified soxhlet method but much faster. The main steps are as follows:

* 3.0 gm of sample were placed in an extraction thimble
* The sample was immersed in the solvent for 10 min.
* The sample was raised above solvent surface and continue extraction for 20 minutes.
* Solvent was removed by evaporation.
* The residue was dried and weighed.

Crude fat percentage was calculated on dry basis as follows:-

$$\text{Crude fat\%} = \frac{\text{Weight of fat (on dry bases)}}{\text{Weight of sample}} \times 100 \times \frac{(100)}{100-M}$$

Where:

M = Moisture % of the sample.

3.2.2.5 Crude Fiber:

Crude fiber was determined according to the (AACC, 1983) standard method. Crude fiber percentage was calculated on dry basis as follows:

$$\text{Crude fiber\%} = \frac{\text{loss in Weight on ignition}}{\text{Weight of sample}} \times 100 \times \frac{(100)}{100-M}$$

Where:

M = Moisture % of the sample.

3.2.2.6 Minerals Determination:

Minerals were extracted from ash according to the AOAC (1975) method. To the ash obtained about 5 ml of 5N HCl was added and the mixture was brought to boiling for 10 minutes to dissolve the minerals in the HCl. Then the mixture was filtered into a conical Flask. Magnesium, calcium, iron, lead Arsenic and Copper were determined using Atomic absorption spectrophotometer.

The minerals percentage were calculated as fallows:

$$\text{Mineral \%} \quad \frac{C1}{A1} = \frac{C2}{A2}$$

Where:

C1 = The concentration of standard solution

A 1 = The absorbence of the standard solution

C2 = The concentration of sample solution

A2 = The absorbence of the sample.

Potassium and sodium were determined using the Flame photometer, the percentage of which was calculated as follows:

$$\text{Mineral\%} = \frac{F.R \times D.F. \times 50 \times 100}{MW \times 5 \times SW \times 10^4}$$

Where:

F.R = Flame reading

D.F = Dilution factor

M.W = Molecular weight of the mineral

S.W = Sample weight

3.2.2.7 Qualitative Determination of Ammonia:

Ammonia was determined for the hull of guar seed according to AACC (1988) standard method.

3.2.4 Guar gum Rheology-Viscosities:

The methods of viscosities measurement of guar gum solutions were Ostwald, Redwood and Brookfield methods.

3.2.4.1 Ostwald Viscometer Method (Bs. IPCF. 71):

0.2 g of guar flour was taken, dissolved in 200 ml distilled water. One ml of 0.4 M KOH was added to guar solution. Different concentrations were taken, relative viscosities were obtained according to the following equation (Figure 9).

$$V = \frac{T - To}{To}$$

where:

T = Flow time of guar solution and KOH.

To = Flow time of KOH solution.

V = Relative Viscosity of guar solution.

3.2.4.2 Redwood Viscometer No 1 Method:

This method was established for measurement of oils viscosities at high temperature. But it has been modified in this study to suit the measurement of guar solution at varying temperatures from low to high(40-80).

The water bath was filled with heated water (nearly the test temperature) which was kept constant by the movement of the agitator. The sample was heated in a separate vessel and poured into the cylinder till the sample reached the edge of the hook. When the two temperatures (bath and sample) read exactly the same, ball valve was lifted an supported by the thermometer-bracket.

The time for 50 cm3 of sample solution to run out was noted and the result was expressed as "Redwood second" at temperature taken which should not vary more than 0.20°C during the out flow of the 50 cm3 of the solution (0.5%) (Figure 10).

Then the viscosity was calculated as follows:

$$V = 0.0026\ t - \frac{1.79}{t} \quad (\text{For } 34 < t < 100)$$

$$V = 0.00247\ t - \frac{0.5}{t} \quad (\text{For } t > 100)$$

where:

V = Kinamatic viscosity of guar solution in Centistoke. (Ratio of absoulte viscosity and density).

t = Flow time of guar solutions.

0.0026, 1.79, 0.00247 and 0.5 are factors.

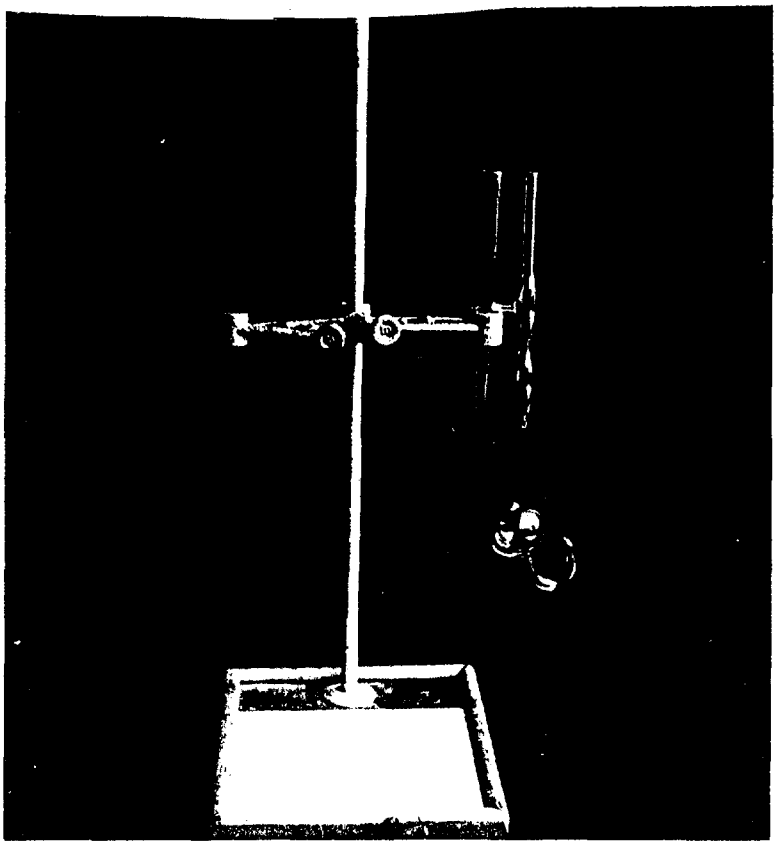

Fig. 9: Ostwald viscometer

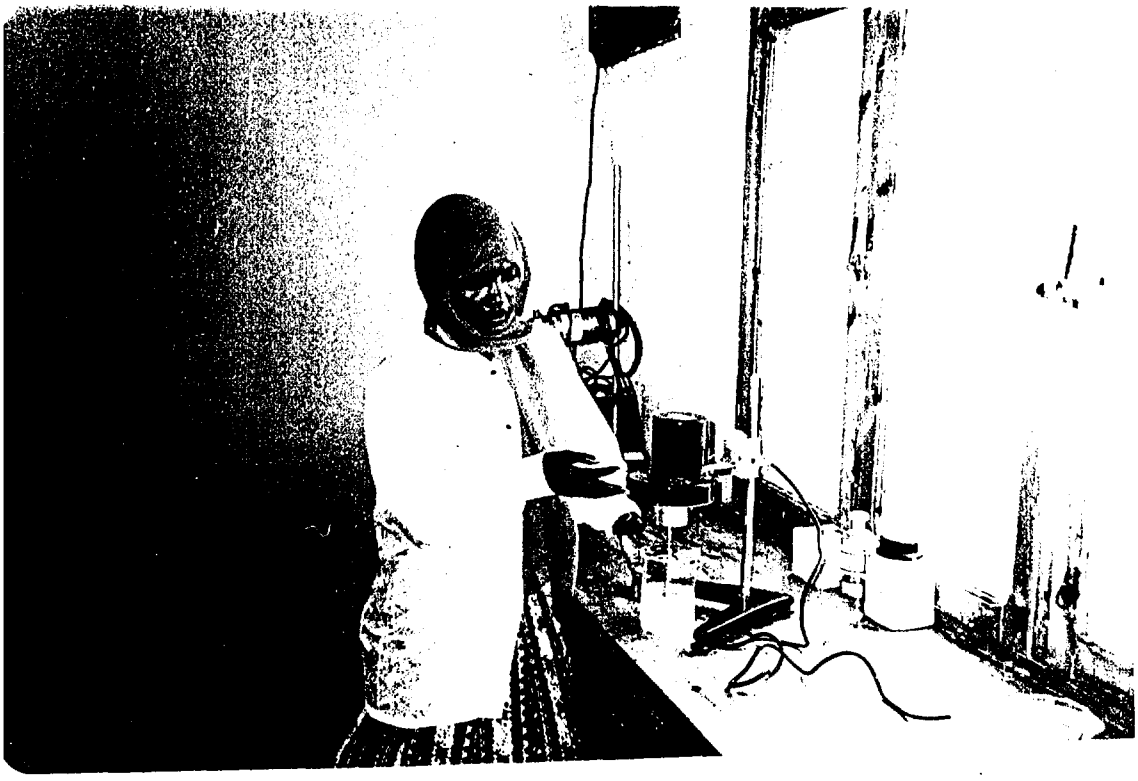

Fig. 11: Dehuller

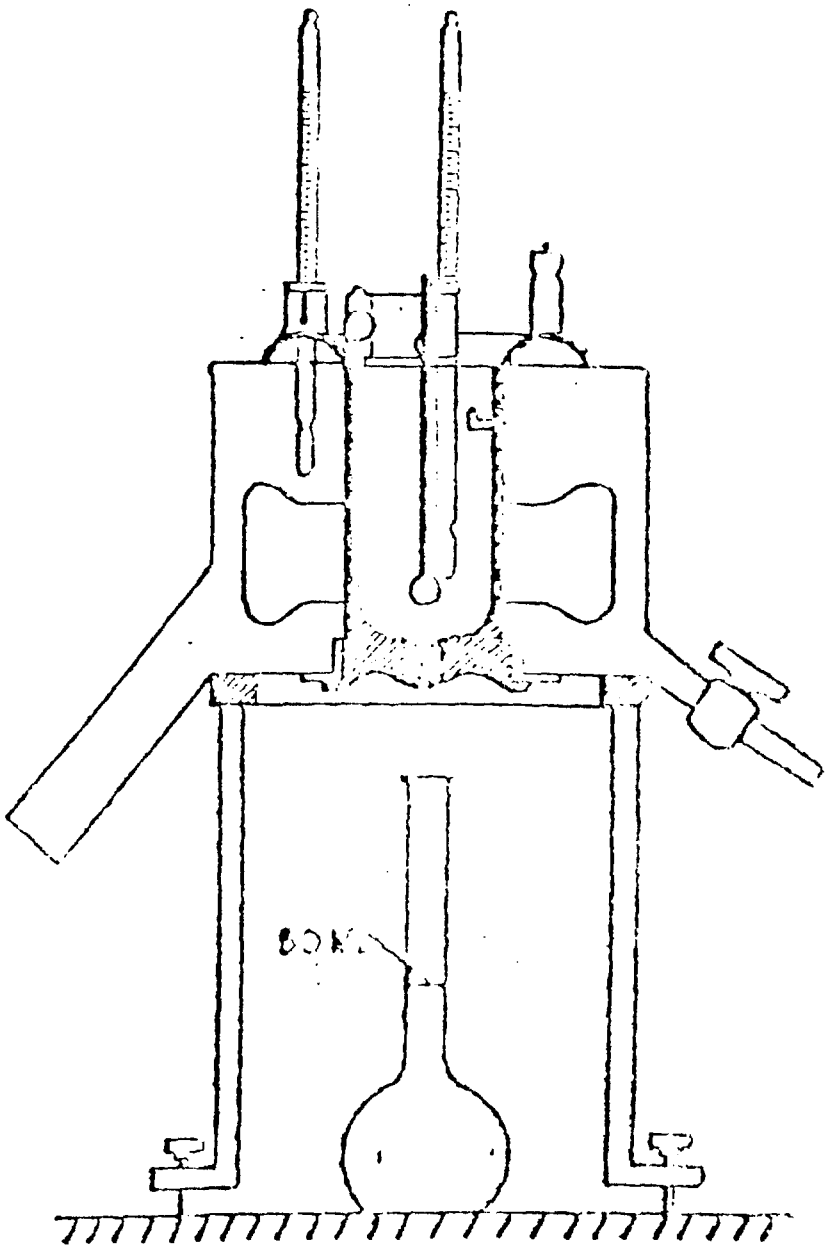

Fig. 10: Redwood viscometer

3.2.4.3 Using Brookfield Viscometer Method:

Guar gum viscosity was measured using a brookfield viscometer RV, spindle No 4, speed 20 RPM (Figure 11). Four gm of guar flour were weighed into a 600 ml beaker, 6 ml of methanol was added, the solution was stirred well. 400 ml of distilled water were added slowly with constant stirring.

The sample solution was allowed to stand for 15 min. Then the viscosity was recorded at different intervals, 30 min, one hour, two hours and three hours . The viscosity was calculated as follows:-

V = Reading x 100

where:

V = Viscosity of guar solution in CPs

100 = The factor according to spindle.

(Agrisale limited, 1990).

3.2.4.4 Preparation of Guar Gum Solution in Combination With Salt (sodium chloride) and Sugar:

0.5% of guar solution was prepared. The addition of salt and sugar was done as follows:

Solution (1) salt 1, 1.5 and 2%.

Solution (2) sugar 5, 10 and 15%.

Solution (3) salt/sugar 1.0/5, 1.0/10, 1.0/15%; 1.5/5, 1.5/10 and 1.5/15%; 2.0/5, 2.0/10 and 2.0/15%.

These solutions were prepared and their viscosities were determined using Redwood viscometer similar to that of guar gum solution detailed above in section 3.2.4.2.

3.2.5 **Method of Statistical Analysis:**

Replicates of each sample was analyzed chemically and physically using stastical analysis system. The analysis of variance was performed to examine the significant effect in all parameters measured. Least significant test was used to separate the means (Peterson, 1985).

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Physical Examination of Guar Seed:

The physical examination for the three guar genotypes (HFG53, HFG182 and HFG363) results are shown in Table 1.

4.1.1 Germination Percentage:

Germination test is a measure of seed soundness and purity. The germination percentage ranged between 80.5-95.5% (mean 91.0%). The HFG53 and HFG182 were not significantly different and shows high values. The germination percent is in the range reported by Thorne (1909) who stated that guar seed germination percentage, with an initial value of 98% can drop to 2% when submerged in water for 38 days.

4.1.2 Hard Seed Percentage:

The hard seeds can affect the granulation of the powder and purification of gum fractions. It ranged between 5.98-13.-6% (mean 8.58%). There was no significant difference between HFG53 and HFG182 genotypes.

4.1.3 Black Seed Percentage:

Black colouration was due to fungus attack when grown in humid areas as stated by Sinclair and Shurtl *et al.* (1975). The presence of black seeds reduces gum quality in color and increases microbial load, hence rapid deterioration in storage.

Table 1: Physical examination of guar seed for the three genotypes

Genotype	Germin-ation (%)	Hard seed (%)	Black seed (%)	1000-seed weight (g)	Hectoliter weight (g/l)
HFG53	95.50[a]	06.15[b]	01.80[b]	21.05[b]	849.40[b]
HFG182	93.00[a]	05.98[b]	13.80[a]	32.65[a]	857.85[a]
HFG363	84.50[b]	13.60[a]	2.00[b]	28.28[c]	830.30[c]
Mean	91.00	8.58	5.87	30.99	845.85
C.V%	1.10	1.74	1.39	0.20	0.01
SE±	1.00	0.15	0.08	0.06	0.09
Lsd	3.18	0.47	0.26	0.19	0.29

Means values with the same letters in each column are not significantly different (P≥0.05).

The values of black seed percentage were insignificantly different ($P \geq 0.05$) in both HFG53 and HFG363 genotypes. HFG182 showed the highest black seed percent (13.8%).

4.1.4 One Thousand Seed Weight:

The highest value of 1000-seed weight is shown by HFG182 genotype, while the lowest value showed by HFG53.

4.1.5 Hectolitre Weight:

It is the bulk density measurement that indicates the weight of seeds per unit volume (g/litre). The highest weight is shown by HFG182, while the lowest value was shown by HFG53. The variation in might have been due to seed weight.

4.2 Proximate Composition of Guar Seed:

The values of moisture content were significantly different in the three genotypes. It ranged from 9.5-12.4% (mean 11.13%). The highest value of moisture content is shown by HFG53 genotype whereas, the lowest value shown by HFG363 (Table 2).

4.2.1 Moisture Content:

Moisture content was greatly affected by relative humidity of the surrounding atmosphere at harvest and during storage. Since these seeds have been the in same environment, therefore, the variation in the moisture content may be due to retention capacity of the seed endosperm and initial moisture at the time of harvest or at the storage.

Table 2: **Proximate composition of guar seed (on dry matter basis)**

Genotype	Moist-ure (%)	Ash (%)	Crude protein (%)	Crude fat (%)	Crude fibre (%)	Carbo-hydrate (%)*
HFG53	12.40[a]	3.78[a]	28.17[c]	1.66[a]	9.20[b]	44.84[c]
HFG182	11.95[b]	3.25[c]	29.51[b]	1.68[a]	8.48[c]	45.14[b]
HFG363	09.05[c]	3.45[b]	29.62[a]	1.42[b]	9.37[a]	47.11[a]
Mean	11.13	3.49	29.1	1.58	9.01	45.70
C.V%	0.52	1.74	0.03	0.45	0.08	0.11
SE±	0.06	0.06	0.01	0.01	0.01	0.05
Lsd	0.18	0.19	0.03	0.02	0.02	0.15

- Means values with the same letters in each column are not significantly different (P≥0.05).

Carbohydrate by difference

4.2.2 Ash Content:

It is a measure of minerals quantities in the guar seed. Ash content percentages were significantly different in the three genotypes. It ranges between 3.25-3.78% (mean 3.49%). The highest value is shown by HFG53 while HFG182 shows the least value.

4.2.3 Crude Protein:

The crude protein percentage of the three genotypes showed significant differece (at$P \leq 0.05$ and Lsd .03) ranged from 28.17-29.62% (mean 29.1%).

HFG363 shows the highest value of crude protein while HFG53 showed the lowest crude protein value.

4.2.4 Crude Fat:

There was no significant difference between HFG53 and HFG182 genotypes at ($P \geq 0.05$ LSD.02) and they showed the high values of crude fat.

4.2.5 Crude Fibre:

There is a significant difference between the three genotypes. The highest value was given by HFG363 genotype while the lowest value was given by HFG182.

4.2.6 Carbohydrates by Difference:

There was a significant difference between the carbohydrates of the three genotypes. It ranged between 44-47.11% (mean 45.7%). HFG363 genotype has the highest value of carbohydrate while the lowest value is given by the HFG53 genotype.

4.3 Guar Seed Components:

4.3.1 Hull:

The hulls show significant difference in the three genotypes (P≤0.05) Table (3) The highest value shown by HFG53 (14.05%) whereas, the lowest percentage is shown by HFG363 (13.48%). Whistler and Hymowitiz (1979) reported that the percentage ranged from 14 to 17%.

4.3.2 Germ:

The germ of the three genotypes is significantly different (P≤ 0.05). HFG53 gave the highest value (44.24%), while the least value was found in HFG363 (43-37%) Table (3). This result is similar to that reported by Whistler and Hymowitiz (1979) which is 43-47%.

4.3.3 Endosperm:

For the endosperm in the three genotypes there was a significant difference (P≤0.05). The highest value was given by HFG53 (39.93%), while the lowest value was given by HFG363 (35.96%) (Table 3).

Whistler and Hymowitiz (1979) reported that the endosperm percentage ranged between 35 and 42%.

4.4 Proximate Composition of Guar Seed Components:

4.4.1 Hull Composition:

- **Moisture Content:**

The moisture content of the hull of the two genotypes is in significantly different in HFG53 and HFG363 (P≤0.05), but

Table 3: Guar seed components

Component	% Hull	% Germ	% Endosperm
PG53	14.05[a]	44.24[a]	39.93[a]
PG182	13.49[b]	43.68[b]	37.89[b]
PG363	13.48[b]	43.37[c]	35.96[c]
C.V%	0.42	0.02	0.17
S±	0.04	0.01	0.05
Sd	0.124	0.001	0.201

Means values with the same letters in each column are not significantly different (P≥0.05).

significant with HFG 182. The genotypes HFG 53 and HFG363 give the highest moisture value (5%) while the lowest value was 4.45% by HFG182 (Table 4A). Both values are less than the moisture content reported by Whistler and Hymowitiz (1979) which was 10%.Khartoum is a dry area with very low humidity.

- **Ash content:**

The hull ash content shows a significant difference in the three genotypes ($P \leq 0.05$). The highest value was given by HFG182 (4.0%) while the lowest value was given by HFG53 (2.30%) (Table 4a). The results obtained in this study was less than that reported by Whistler and Hymowitiz (1979) which was 4.6%.

- **Protein content:**

The hull shows a significant difference in the three genotypes ($P \leq 0.05$). HFG182 has the highest value (5.08%) whereas the least value was shown by HFG53 (4.62%) (Table 4a). The value of the protein content reported by Whistler and Hymowitiz was 5%.

- **Fat content:**

The fat content of the hull was significantly different ($P \leq 0.05$). The highest value of fat was given by the genotype HFG53 (0.07%), while HFG182 gave the lowest value (0.04%) (Table 4a). This result was less than the value reported by Whistler and Hymowitiz (1979) which was 0.3%.

Table 4a: **Proximate composition of the guar seed hull components**

Genotype	Moist-ure (%)	Ash (%)	Crude protein (%)	Crude fat (%)	Crude fibre (%)	Carbo-hydrate (%)*
HFG53	4.95[a]	2.30[c]	4.62[c]	0.07[a]	52.23[a]	34.54[c]
HFG182	4.45[b]	4.00[a]	5.08[a]	0.04[c]	47.07[c]	39.38[a]
HFG363	5.00[a]	3.45[b]	4.62[b]	0.06[b]	49.45[b]	37.48[b]
C.V%	1.70	1.26	0.12	0.014	0.01	0.21
SE±	0.04	0.03	0.00	0.00	0.01	0.06
Lsd	0.174	0.142	0.001	0.001	0.001	0.246

Means values with the same letters in each column are not significantly different (P≥0.05).

Carbohydrate by difference

- Fibre content:

There was a significant difference in the fiber content of the three genotypes (P≤0.05). The HFG53 has the highest value of fibre (52.23%), while the lowest value was recorded by HFG182 (47.07%) (Table 4a). The two levels were greater than that reported by Whistler and Hymowitiz (1979) who reported 36%.

- **Carbohydrates by difference:**

The carbohydrates by difference in the hull shows a significant difference in the three genotypes (P≤0.05). HFG182 has the highest value (39.38%), while the least value was given by HFG53 genotype (Table 4a).

4.4.2 Germ Composition:

- **Moisture:**

The moisture content of the germ showed insignificant difference (P≥0.05). The moisture content ranges from 5.0-5.-25% (Table 4a), which was lower than that reported by Whistler and Hymowitiz (1979) which was 10%.

- **Ash content:**

There was a significant difference (P≥0.05) in the ash content of the germ three genotypes. HFG53 genotype has the highest value (6.35%), while the least value was recorded shown by HFG182 (5.15%) (Table 4b). The results of this study was greater than that investigated by Whistler and Hymowitiz (1979) which was 4.6%.

Table 4b: **Proximate composition of the guar seed germ**

Genotype	Moist-ure (%)	Ash (%)	Crude protein (%)	Crude fat (%)	Crude fibre (%)	Carbo-hydrate (%)*
HFG53	5.25[a]	6.35[a]	51.43[b]	2.68[c]	3.09[a]	31.21[b]
HFG182	5.00[a]	5.15[c]	51.00[a]	2.99[b]	2.94[b]	32.48[a]
HFG363	5.00[a]	6.05[b]	51.30[c]	3.88[a]	2.73[c]	31.05[b]
C.V%	4.02	1.21	0.01	0.38	0.14	0.49
SE±	0.14	0.05	0.00	0.01	0.00	0.11
Lsd	0.65	0.225	0.001	0.001	0.001	0.493

- Means values with the same letters in each column are not significantly different (P≥0.05).

Carbohydrate by difference

- Protein content:

For germ protein, there was a significant difference between the three genotypes (P≤0.05). The highest value of protein was shown by HFG182 (51.43%) while the lowest value given by HFG363 genotype (51.3%) (Table 4b). Both values were lower than that reported by Whistler and Hymowitiz (1979) which was 55.3%.

- **Fat content:**

The germ was of greater fat content in the seed components. The fat content shows a significant difference in the three gnoteypes (P≤ 0.05). The highest value was recorded in HFG363 (3.88%) whereas the least value was recorded by HFG53 (2.68%) (Table 4b). This range was lower than that reported by Whistler and Hymowitiz (1979) which was 5%.

- **Fibre content:**

The fiber content of the germ was significantly different in the three genotypes (P≤ 0.05). HFG53 gave the highest value (3.09%) whereas, the least fiber content was obtained by HFG363 (2.73%) (Table 4b). This value was much lower than that reported by Whistler and Hymowitiz (1979) which was 18%.

- **Carbohydrates by difference:**

There was insignificant difference in carbohydrate content of HFG53 and HFG363 genotypes (P≤0.05). The two genotypes show the lowest values (31.05 - 31.21%), whereas the highest values was given by HFG182 (32.48%) (Table 4b).

4.4.3 Endosperm composition:

- **Moisture:**

The endosperm has a higher moisture content compared to other seed components. There was insignificant difference in moisture content at HFG53 and HFG363 genotypes ($P \leq 0.05$). They gave the least values (6.0-6.05%), while the highest value was shown by HFG182 (6.5%) (Table 4c). Whistler and Hymowitiz (1979) stated that the endosperm moisture content was 10%.

- **Ash content:**

Ash content of the endosperm for HFG182 and HFG363 genotypes show insignificant difference ($P \geq 0.05$). The range of the two genotypes (1.0-1.05%) give the lowest value, but the highest ash content shown by HFG53 (2.0%) (Table 4c). The ash content in this study was greater than that reported by Whistler and Hymowitiz (1979) which was 0.6%.

- **Protein content:**

The protein content of the endosperm show a significant difference in the three genotypes ($P \leq 0.05$). HFG53 has the highest value (4.48%), while the lowest value shown by HFG363 (4.3%) (Table 4c). The value reported by Whistler and Hymowitiz was much lower than that reported in this study (0.6%).

- **Fat content:**

The crude fat of the endosperm for the three genotypes show a significant difference ($P \leq 0.05$). The highest value is shown by HFG53 (0.84%), while the lowest value by HFG182 (0.17%) (Table 4c). Whistler and Hymowitiz (1979) reported that the crude fat of the endosperm was 0.6%.

Table 4c: **Proximate composition of the guar seed endosperm**

Genotype	Moist-ure (%)	Ash (%)	Crude protein (%)	Crude fat (%)	Crude fibre (%)	Carbo-hydrate (%)*
HFG53	6.05[b]	2.00[a]	4.48[a]	0.84[a]	1.25[c]	85.39[b]
HFG182	6.50[a]	1.00[b]	4.44[b]	0.17[c]	1.53[b]	86.37[a]
HFG363	6.00[b]	1.05[b]	4.30[c]	0.19[b]	1.99[a]	86.48[a]
C.V%	0.66	3.02	0.13	1.79	0.57	0.07
SE±	0.03	0.03	0.00	0.00	0.01	0.04
Lsd	0.201	0.142	0.001	0.001	0.001	0.201

- Means values with the same letters in each column are not significantly different (P≥0.05).

* Carbohydrate by difference

- Fibre content:

For the endosperm fibre content there was a significant difference in the three genotypes ($P \leq 0.05$). The highest value was obtained by HFG363 (1.99%), while the least value was given by HFG53 (1.25%) (Table 4c). Whistler and Hymowitiz (1979) reported that the endosperm crude fibre was 1.5%.

- **Carbohydrates by difference:**

The carbohydrates of both HFG182 and HFG363 genotypes showed insignificant difference ($P \geq 0.05$), and the gave the highest value (86.37-86.48%), while the lowest value was given by HFG53 (85.39%) (Table 4c).

4.5 Mineral Content of Guar Seed Components:

4.5.1 Hull Mineral Contents:

(a) Macro-elements:

Macro-elements play an important role in nutrition. For the hull, HFG363 has the highest value in Na, K and Ca. The genotype HFG182 give the highest value in both Ca and Mg, while HFG53 shows the least value in all elements (Table 5a).

(b) Micro-elements:

The micro-elements are very important in determining the level of toxicity. Table 5b shows that the greater values in both Cu and As content are given by HFG53, while HFG182 has the highest value in Zn, Fe, Pb and As with the least value in Cu. But the lowest values of Zn, Fe and Pb were shown by HFG363 genotype.

Table 5a: **Macro-elements of the gaur seed hull**

Genotype	Na (%)	K (%)	Ca (%)	Mg (%)
HFG53	0.25^{b}	0.74^{c}	0.38^{b}	0.11^{b}
HFG182	0.25^{b}	1.13^{b}	0.40^{a}	0.12^{a}
HFG363	0.50^{a}	1.30^{a}	0.40^{a}	0.11^{b}
Mean	0.333	1.057	0.393	0.11
C.V%	0.00	1.98	2.18	5.25
SE±	0.00	0.01	0.41	0.00
Lsd	0.001	0.001	1.836	0.001

Table 5b: **Micro-elements of the gaur seed hull**

Genotype	Zn (mg/kg)	Fe (mg/kg)	CU (mg/kg)	Pb (mg/kg)	As (mg/kg)
HFG53	30.94^{b}	78.84^{b}	3.55^{a}	0.36^{b}	0.26^{a}
HFG182	45.24^{a}	98.69^{a}	3.21^{c}	0.38^{a}	0.26^{a}
HFG363	25.25^{c}	46.05^{c}	3.42^{b}	0.35^{c}	0.25^{b}
Mean	33.81	74.53	3.39	0.36	0.26
C.V%	0.60	0.01	1.233	0.159	0.807
SE±	0.14	0.01	0.04	0.001	0.002
Lsd	0.652	0.001	0.006	1.05×10^{-7}	1.38×10^{-5}

- Means values with the same letters in each column are not significantly different ($P \geq 0.05$).

4.5.2 The Germ Mineral Contents:

(a) Macro-elements:

The high value of K is given by HFG53 with the least value of Ca content. HFG182 has the lowest Na content. The highest values of Na, Ca and Mg are found in HFG363 genotype (Table 6a).

(b) Micro-element:

The germ of HFG53 genotype has the highest value in Fe, Cu, Pb and As, whereas HFG182 give high value of Zn and As. But the least value in all elements is shown by HFG363 genotype (Table 6b).

4.5.3 The Endosperm Mineral Content:

(a) Macro-elements:

The endosperm of HFG53 has the highest Na with lowest Ca and K contents. HFG182 shows the higher Ca but lower K contents, while the highest K and lowest Na contents are given by HFG363. For Mg content there is insignificant difference between the three genotypes ($P \geq 0.05$) (Table 7a).

(b) Micro-elements:

The endosperm of HFG53 genotype has the highest value of Pb and where it has the lowest value of Cu. The greater value of Zn, Fe, Cu and As was shown by HFG182. But the highest As and lowest Zn, Fe and Pb was given by HFG363 genotype (Table 7b).

Table 6a: Macro-elements of the guar seed germ

Genotype	Na (%)	K (%)	Ca (%)	Mg (%)
HFG53	0.40^{b}	3.45^{a}	0.40^{c}	0.22^{b}
HFG182	0.35^{c}	3.03^{ab}	0.47^{b}	0.22^{b}
HFG363	1.00^{a}	2.98^{b}	0.48^{a}	0.23^{a}
Mean	0.58	3.15	0.45	0.22
C.V%	0.70	7.35	1.59	2.62
SE±	0.00	0.16	0.01	0.00
Lsd	0.001	0.719	0.001	0.001

Table 6b: Macro-elements of the guar seed germ

Genotype	Zn (mg/kg)	Fe (mg/kg)	CU (mg/kg)	Pb (mg/kg)	As (mg/kg)
HFG53	28.57^{b}	190.79^{a}	2.60^{a}	0.36^{a}	0.25^{a}
HFG182	40.47^{a}	151.32^{b}	2.41^{b}	0.35^{a}	0.25^{a}
HFG363	23.75^{c}	078.94^{c}	2.20^{c}	0.34^{c}	0.24^{b}
Mean	30.93	140.09	2.40	0.35	0.25
C.V%	0.03	0.01	0.16	0.004	0.00
SE±	0.01	0.01	0.26	0.001	0.00
Lsd	0.001	0.001	$5x10^{-6}$	$2.7x10^{-6}$	0.00

- Means values with the same letters in each column are not significantly different (P≥0.05).

Table 7a: Macro-elements of the guar seed endosperm

Genotype	Na (%)	K (%)	Ca (%)	Mg (%)
HG 53	0.10[a]	0.70[b]	0.30[c]	0.11[a]
HG 182	0.05[b]	0.75[b]	0.37[a]	0.11[a]
HG 363	0.01[c]	0.95[a]	0.35[b]	0.11[a]
Mean	0.05	0.80	0.34	0.11
CV %	0.00	5.10	2.11	0.00
SE	0.00	0.03	0.01	0.00
LSD	0.001	0.142	0.001	0.001

Table 7b: Micro-elements of the guar seed endosperm

Genotype	Zn (mg/kg)	Fe (mg/kg)	CU (mg/kg)	Pb (mg/kg)	As (mg/kg)
HG 53	40.88[b]	105.26[b]	2.61[c]	0.38[a]	0.24[a]
HG 182	44.05[a]	111.84[a]	3.81[a]	0.35[b]	0.24[a]
HG 363	29.55[c]	52.634[c]	2.81[b]	0.34[c]	0.24[a]
Mean	38.16	089.91	3.08	0.36	0.24
CV %	0.02	0.01	0.188	0.00	0.00
SE	0.00	0.00	0.005	0.00	0.00
LSD	0.001	0.001	1.04×10^{-4}	0.00	0.00

- Means values with the same letters in each column are not significantly different (P≥0.05).

4.6 Proximate Composition of Mechanically Purified Guar Gum:

The mechanically purified endosperm of the three guar seed genotypes were analyzed to determine the degree of purity of the two grades, the overs 0.5 and throughs 0.5mm sieves.

- **Moisture content:**

The moisture content of the overs endosperm was greater than that of the throughs of 0.5mm sieve in the three genotypes. This was because the overs was mostly gum while the throughs was contaminated with hull and germ. Therefore, the water retention was greater in the gummy materials.

There is a significant difference between moisture of overs 0.5mm of the three genotypes ($P \leq 0.05$). The highest value is shown by HFG53 (13.25%) while the lowest value is given by HFG182 (9.97%) (Table 8).

For the throughs 0.5mm, there was a significant difference in moisture between the three genotypes ($P \leq 0.05$). HFG53 showed the highest value (8.25%), whereas the least value was given by HFG182 (4.65%).

- **Ash content:**

The ash content of the throughs is greater than that of the overs 0.5 mm of the three genotypes. This was due to the presence of hull and germ in the former.

The ash content of the overs 0.5mm is insignificantly different in HFG53 and HFG363 genotypes ($P \geq 0.05$). The lowest ash content is shown by HFG182 (0.6%).

Table 8: Proximate composition of mechanically purified guar gum (%dry matter basis)

Genotype	Particle size (mm)	Moisture (%)	Ash (%)	Protein (%)	Fat (%)	Fiber (%)	Carbohyd-rate (%)
HFG53	Overs 0.5	13.45^{Aa}	0.70^{Ba}	3.06^{Bc}	0.09^{Bc}	0.99^{Bc}	81.32^{Ac}
	Throughs 0.05	8.25^{Ba}	2.50^{Aa}	7.29^{Ac}	0.30^{Ac}	5.68^{Ac}	75.96^{Bb}
HFG182	Overs 0.5	9.97^{Ac}	0.60^{Bb}	4.35^{Ba}	0.15^{Bb}	1.48^{Bb}	83.47^{Aa}
	Throughs 0.05	4.65^{Bc}	2.25^{Ac}	6.67^{Ac}	0.36^{Ab}	9.63^{Aa}	76.46^{Ba}
HFG363	Overs 0.5	11.28^{Ab}	0.70^{Ba}	4.15^{Bb}	0.26^{Ba}	1.72^{Ba}	81.91^{Ab}
	Throughs 0.05	5.18^{Bb}	2.35^{Ab}	7.44^{Aa}	0.57^{Aa}	9.15^{Ab}	75.33^{Bc}
C.V%		0.615	2.692	0.530	2.438	0.121	0.104
SE±		0.050	0.041	0.029	0.007	0.006	0.080
Lsd		0.130	0.100	0.070	0.020	0.010	0.200

* Comparison in the same column.

\- Capital letters for comparison within the genotype for overs and throughs; while small letters is between genotypes for overs and throughs separately.

() Means with the same letters are not significantly different ($P \geq 0.05$).

For the through 0.5mm, the ash content is significantly different in the three genotypes ($P \leq 0.05$). The highest value is shown by HFG53 (2.5%), while the least value by HFG182 (2.25%) (Table 8).

- **Crude protein:**

The throughs show higher crude protein compared to the overs (0.5mm), this was due to the presence of large quantities of friable germ in the former. The highest value is shown by HFG363 (7.44%), while the lowest value by HFG182 (6.67%).

The overs (0.5mm) show significant difference in protein content in the three genotypes ($P<0.05$). The highest value is shown by HFG182 (4.35%), while the least value of protein is shown by HFG53 (Table 8).

- **Crude fat:**

The highest value of fat is shown by the throughs (0.5mm) in all genotypes. The overs 0.5mm endosperm of HFG363 showed the highest value of fat content (0.26%), while the lowest value of fat was recorded by HFG53 (0.09%).

For the throughs 0.5mm, HFG363 showed the highest value (0.57%) whereas the least value given by HFG53 (0.34%) (Table 8).

- **Crude fiber:**

The throughs 0.5mm endosperm have the highest crude fiber in all the three genotypes. In HFG363 genotype the overs (0.5mm) has the highest value of crude fiber (1.7%), while HFG53 shows the lowest value (0.99%).

In case of the throughs (0.5mm), genotype HFG182 has the ghest value of crude fiber (9.63%) whereas HFG53 shows the ast value (5.68%) (Table 8).

Carbohydrates by difference:

The overs (0.5mm) showed the highest value of carbohy- ate in all genotypes. The highest value of carbohydrate is own by HFG182 (83.47%), while the least value is given by G53 (81.32%).

For the throughs (0.5mm) endosperm, the highest value of rbohydrate was shown by HFG182 (76.46%), while the least lue was given by HFG363 (75.33%) (Table 8).

7 Guar Gum Rheological Properties-Viscosity:

Viscosity of guar gum of the three genotypes HFG53, G182 and HFG363 was studied with regards to concentration, mperature and time. The influence of sodium chloride, sugar d combined salt and sugar was determined on two commercial od-grade guar gum (200 mesh and 80 mesh). Three types of ar gum grades prepared at laboratory level are: (a) pure dosperm (b) over 0.5 mm and (c) through 0.5 mm sieve.

7.1 Viscosity of Pure Endosperm Gums:

The endosperm separated from the hull and germ manually hand was ground into fine particle sizes (1 mm sieve) and ed in all the measurements.

- Ostwald Method:

The viscosity of gum solutions from three genotypes behave linearly at varying concentrations 0.1-0.5 mg/ml at 25°C. The guar gum solutions at this dilution behave Newtonian (Fig. 12). Figure 12 shows insignificant difference at low concentration up to 0.5 mg/ml at $P>0.05$ for the three genotypes. The order of the variation indicated in Fig. 12 was HFG182, HFG53 and HFG363.

- **Redwood Method:**

The effect of temperature increase from 40 to 80°C by Redwood method, on endosperm gum solutions (0.5%) decreased the viscosity for all three genotypes (Fig. 13). The order of temperature stability of the genotypes was as follows HFG53, HFG182 and HFG363. The variation in temperature effect on gum solutions can be attributed to genetic difference since the gum content of these genotypes have insignificant difference ($P>0.05$).

- **Brookfield Method:**

This method measures the rate of gum dispersibility and hydration in relation to viscosity (Fig. 14). The viscosity versus time from 30 up to 180 seconds showed non-linear increase. There was a significant variation between the three genotypes ($P\leq0.05$).

The order of the dispersibility and hydration was HFG363, HFG182, and HFG53 This ability was usually influenced by the particle size distribution, hardness and quality of the gum.

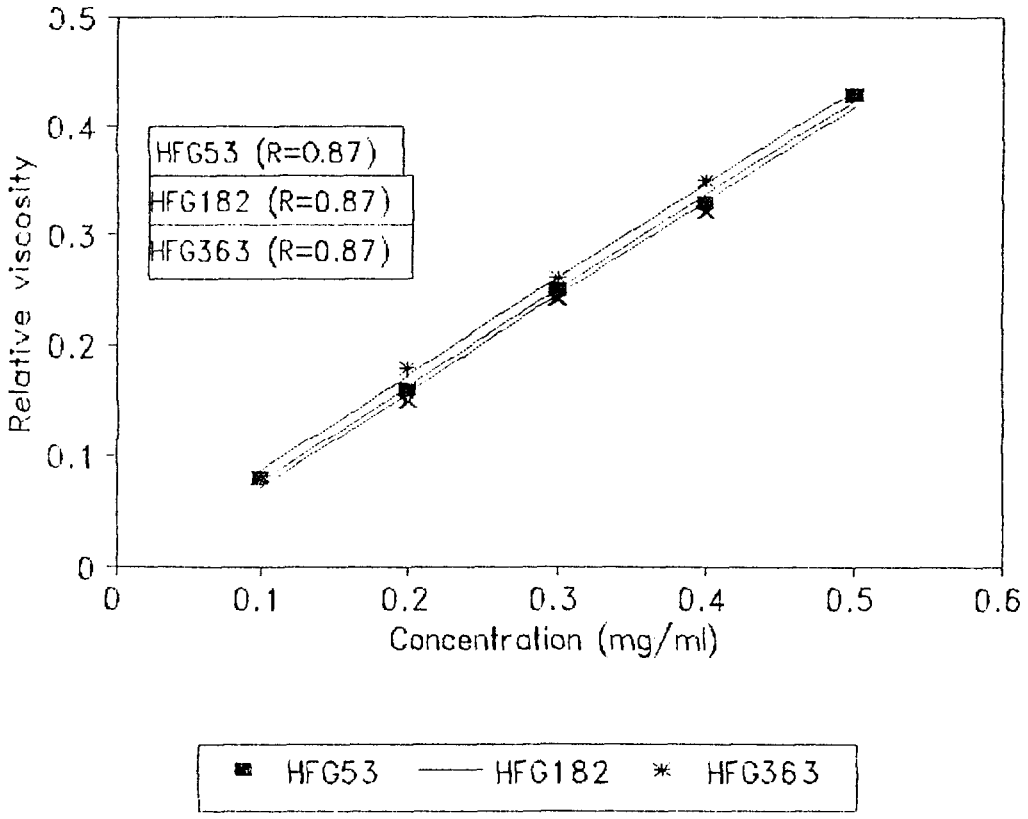

Fig. 12: Viscosities of pure manually purified guar gum as measured by Ostwald method

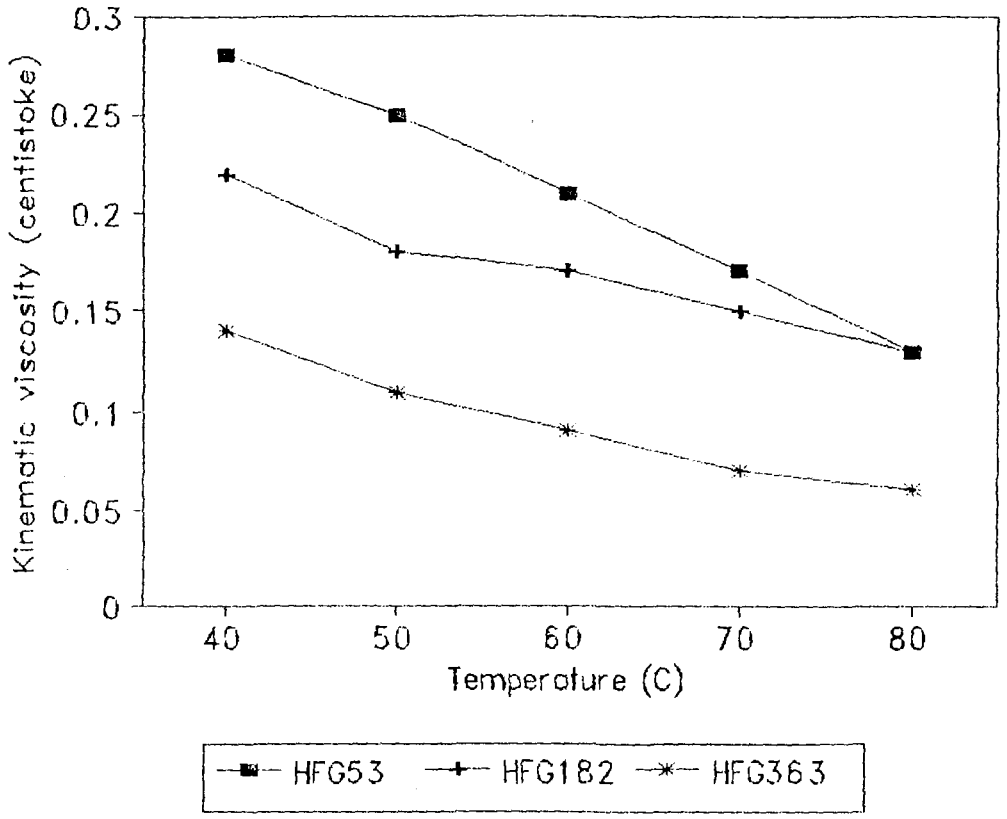

Fig. 13: Viscosities of of manually purified guar gum as measured by Redwood method

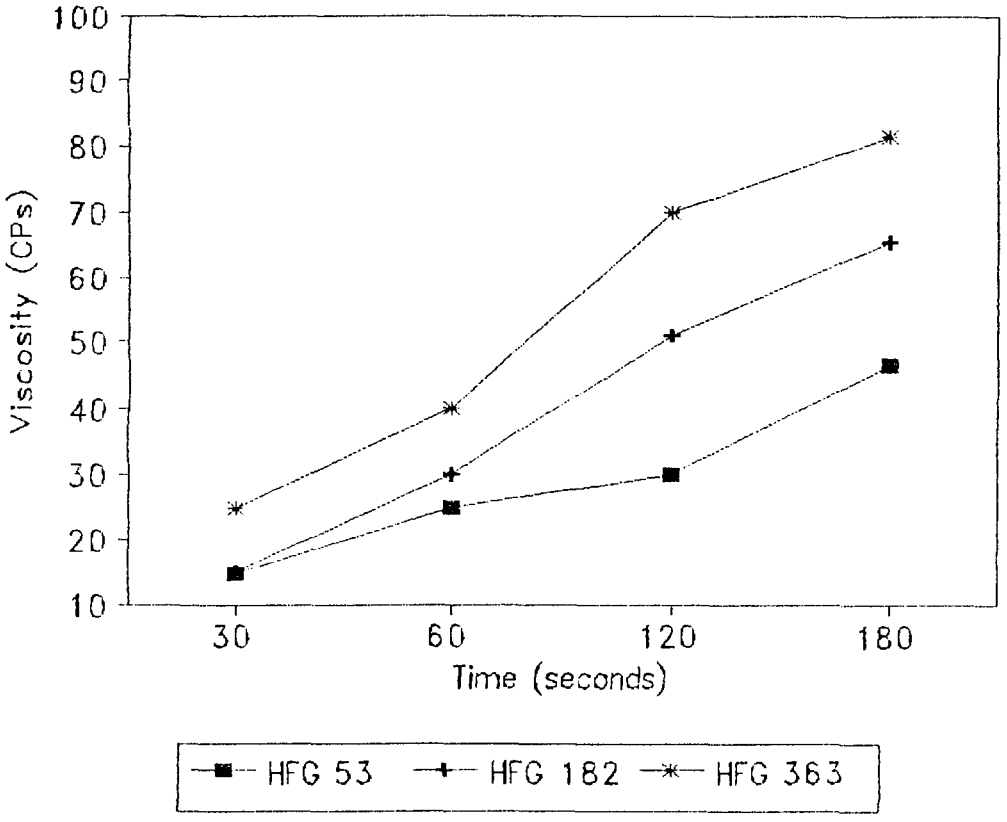

Fig. 14: Viscosities of of manually purified guar gum as measured by Brookfield method

n this case the main factor is uniformity of the particle ize and time allowed for hydration.

.7.2 Viscosities of Mechanically Purified Guar Gum:

Guar gum produced by mechanical method of purification as used in viscosity studies of the three genotypes HFG53, FG182 and HFG363. The purified splits was ground in laboratory hammer mill (1.0mm) to powder.

The powder was separated into two samples in the hammer ill (0.5mm sieve), the overs and throughs. Guar gum produced y mechanical purification was contaminated by the hull and erm as shown in the proximate analysis of the two samples Table 8).

.7.2.1 Viscosity of Guar Gum (overs 0.5mm):

The particle size distribution was less than 1mm and eater than 0.5 mm.

Ostwald Method:

Figure 15 shows linear behaviour when viscosity of guar um (overs 0.5mm) were plotted against the concentration anging from 0.1 mg/ml to 0.5 mg/ml. There is a significant ariation between genotypes gum at low concentration up to 0.3 g/ml. At 0.5 mg/ml there was no significant difference. Since ie overs 0.5 mm granulation powder are made up from harder ortion of endosperms. The purity is shown in the linearity in ie three genotypes (Fig. 15). HFG182 gave the best line of inearity indicating better degree of purification. But the

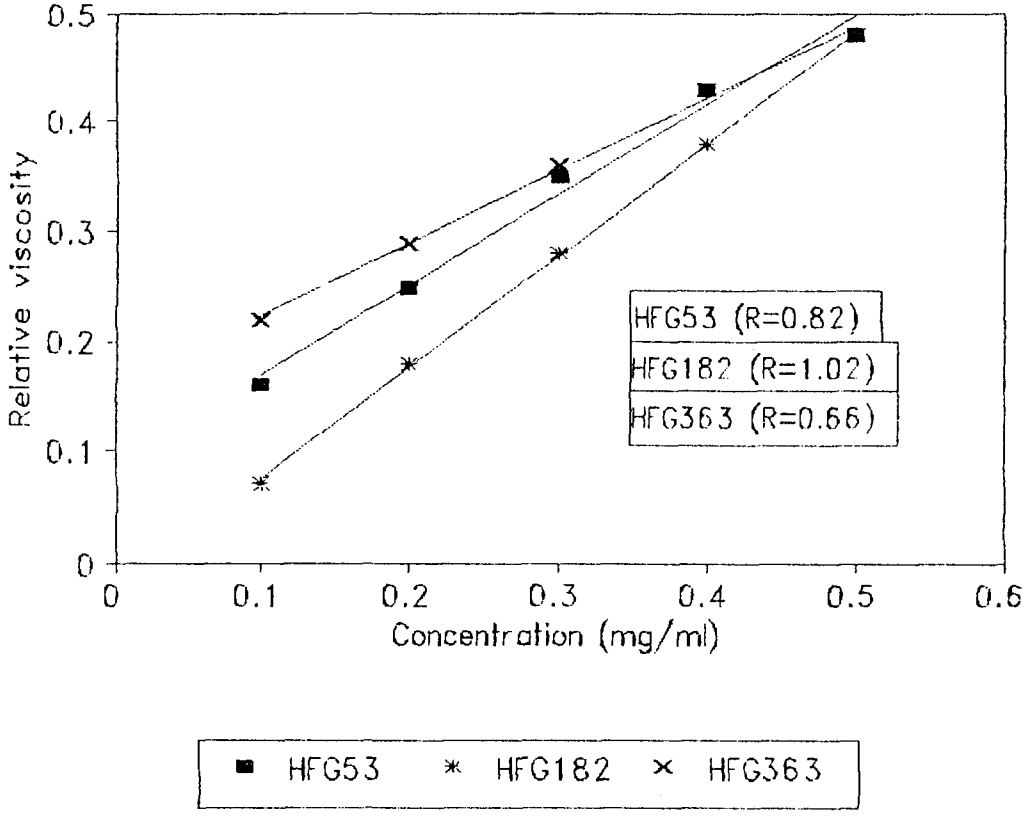

Fig. 15: Viscosities of mechanically purified guar gum fractions (overs ≥0.5 mm) as measured by Ostwald method

viscosity was the lowest compared to HFG363 and HFG53. The order of genotypes gum viscosities levels between 0.1-0.4 mg/ml is as follows in decreasing order HFG363, HFG53 and HFG182.

- **Redwood Method:**

The Redwood viscosities expressed in CPs (CPs = mg/cm/sec) decreased with increasing temperature from 40 to 80°C for the three genotypes (Figure 16). There was great variation in temperature stability of guar solutions (0.5%) between genotypes as determined by Redwood method. The gum of HFG53 was much stable than the two genotypes HFG182 and HFG363. There is a significant difference ($P \leq 0.05$) between HFG53 and the other two genotypes. There seems to be an effect of hard seeds percentage on gum stability of HFG53 (6.15%) and for HFG363 (13.6%) (Table 1).

When comparing the protein and fibre contents in the overs 0.5mm, HFG182 has high results than HFG53 (Table 8). This indicates that HFG53 gum is much pure than HFG182 and HFG363 genotypes.This results in better stability for HFG53.

- **Brookfield Method:**

The result of mechanically purified over 0.5mm solutions (1.0%) guar gum of the three genotypes showed a wide variation in the viscosities of increasing time (Fig. 17). The variation in viscosities increased with the time of dispersibility. The genotypes viscosities in decreasing order is HFG182, HFG53 and HFG363.

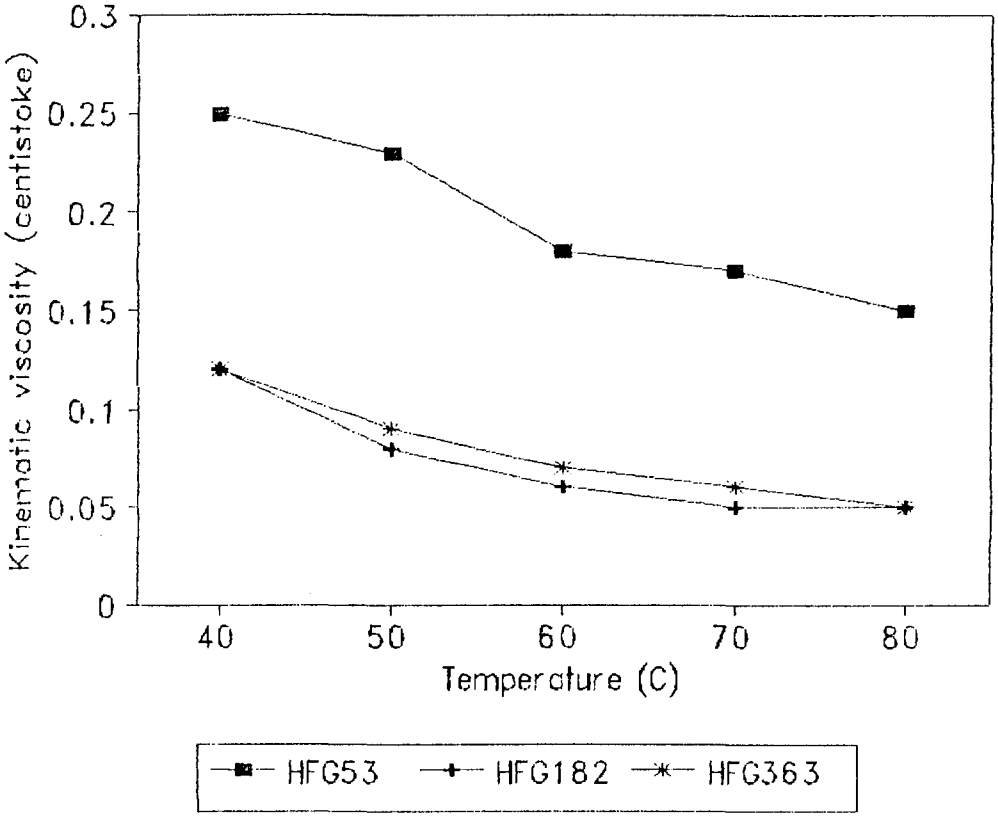

Fig. 16: Viscosities of mechanically purified guar gum fractions (overs ≥0.5 mm) as measured Redwood Method

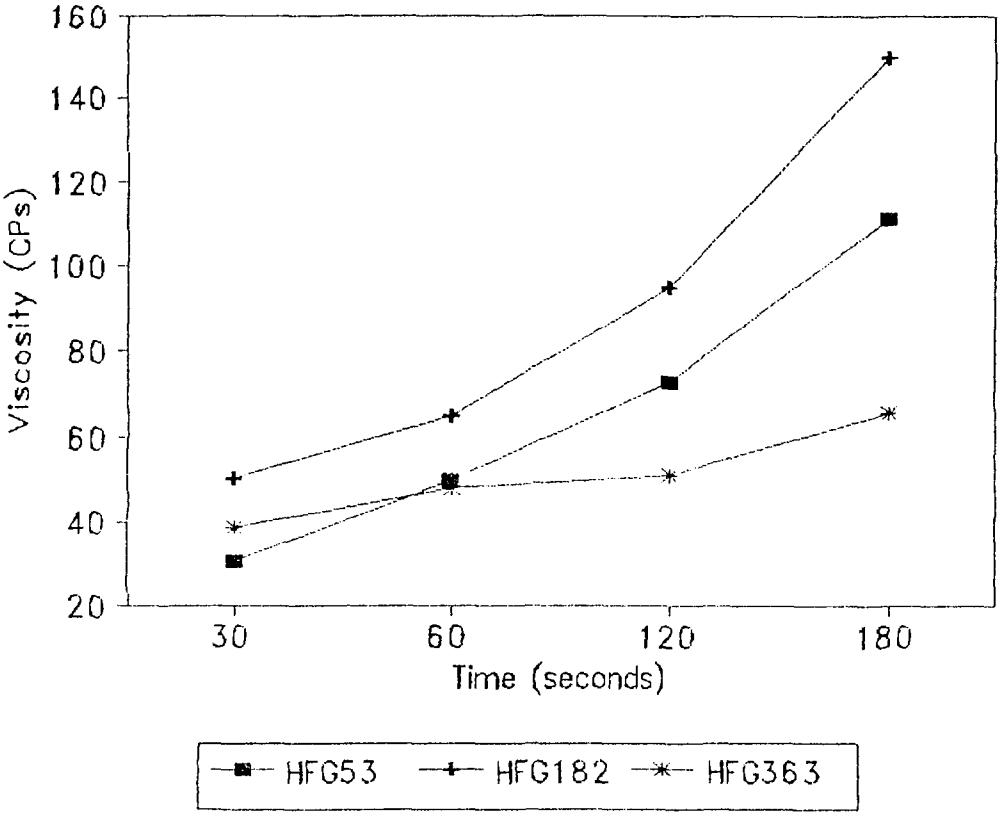

Fig. 17: Viscosities of mechanically purified guar gum fractions (overs ≥0.5 mm sieve) as measured by Brookfield Method

The factors that effect dispersibility and water hydration are particle size distribution, endosperm hardness and contamination. These are hull, protein and fat which were present in the whole guar seed.

4.7.2.2 Viscosity of Guar Gum Throughs 0.5 mm:

The particle size distribution was less than 0.5 mm.That is through seive .05mm.

- **Ostwald Method:**

The viscosities of the three genotypes gum solutions at varying concentrations ranging from 0.1 to 0.5 mg/ml behave linearly i.e. Newtonian (Fig. 18). There was a significant difference ($P \leq 0.05$) between the genotypes. Genotypes HFG53 and HFG363 have no significant difference at 0.4 and 0.5 mg/ml. For HFG182 there is no significant difference at 0.1 mg/ml and 0.2 mg/ml with HFG53 and HFG363 respectively. The variation in the three genotypes is due to the degree of purification. The non-guar gum component in each genotype mechanically purified is shown in Table 8.

- **Redwood Method:**

The heat effects on guar gum solutions (0.5%) show low stability for HFG53. Generally these viscosities are lower than those measured for the overs 0.5 mm particle size. This is due to the level of contamination with the hull and germ of the seed as they are fragile and easily pulverizes into fine powder compared to the endosperm. The contamination level is clearly shown in the quantities of fibre, protein and fat in proximate analysis composition of the throughs 0.5 (Table 8).

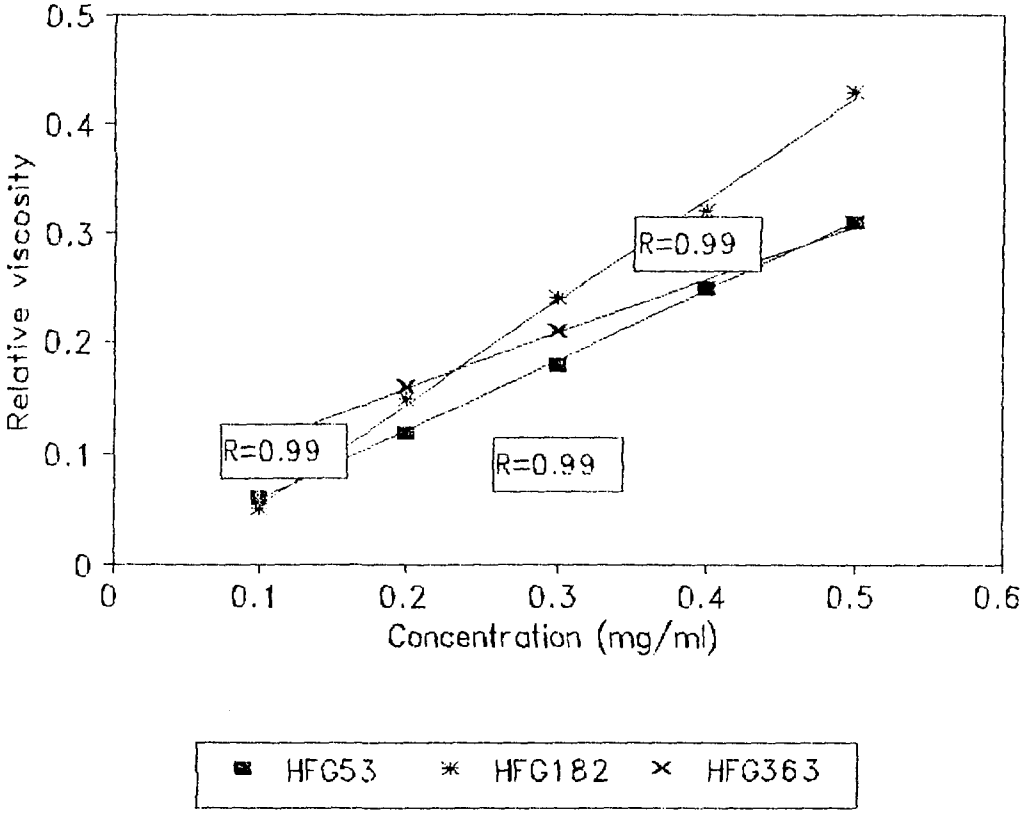

Fig. 18: Viscosities of mechanically purified guar gum fractions (throughs 0.5 mm sieve) as measured by Ostwald Method

The main factor affecting the viscosity level is the gum quantity rather than quality in these results (Fig. 19). The order of genotype gums stability is HFG363, HFG182 and HFG53.

- **Brookfield Method:**

The rate of dispersibility of guar gums of the three genotypes in 1% solution shows great variation. The hydration rate was faster for HFG363 and HFG182, while HFG53 was gradual almost showing some linearity (Fig. 20). This variation is due to the presence of contaminants or low degree of purification. The order of variation between genotypes is HFG363, HFG53 and HFG182. The gum in HFG363 throughs indicates higher gum quantity gum than the other two genotypes.

4.8 The Effects of Purification on Viscosity Characteristics of Genotype HFG53 Guar Gum :

Three types of gums were prepared from genotype HFG53 using manual and mechanical methods. These gums were classified as pure endosperm gum (manually separated) and mechanically purified gums overs 0.5mm sieve and throughs of 0.5 mm sieve.

- **Ostwald Method:**

The purity of the three guar gums plays an important role in the viscosity behaviour as determined by Ostwald method (Fig, 21). The relative viscosity of gums of overs 0.5 mm is the highest followed by that of endosperm and throughs 0.5 mm is the lowest. The proximate composition of these gums (Tables 4C and 8) shows that the order of purity from highest to

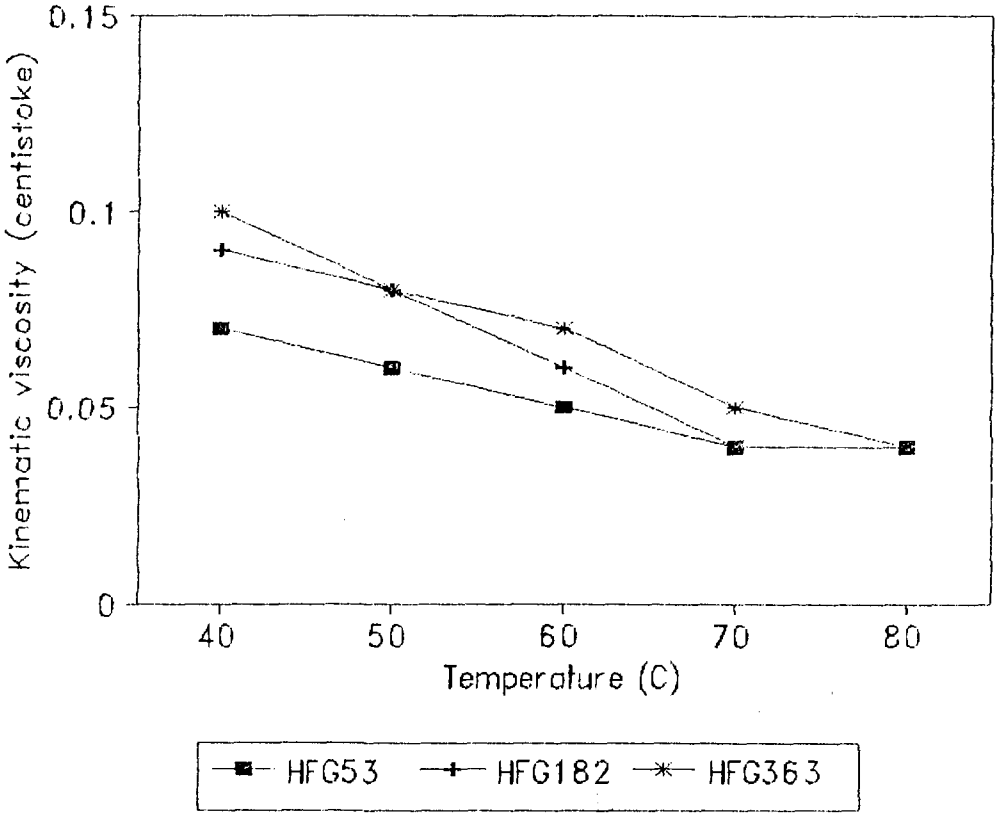

Fig. 19: Viscosities of mechanically purified guar gum fractions (throughs <0.5 mm sieve) as measured by Redwood Method

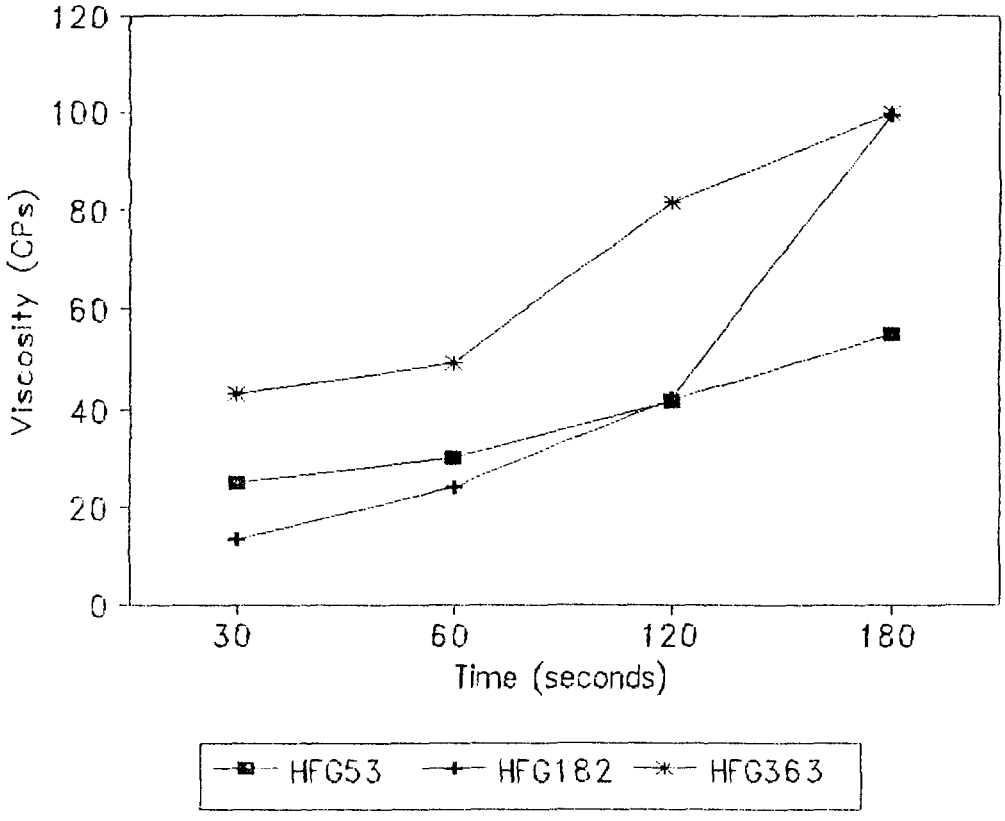

Fig. 20: Viscosities of mechanically purified guar gum fractions (throughs <0.5 mm sieve) as measured by Brookfield Method

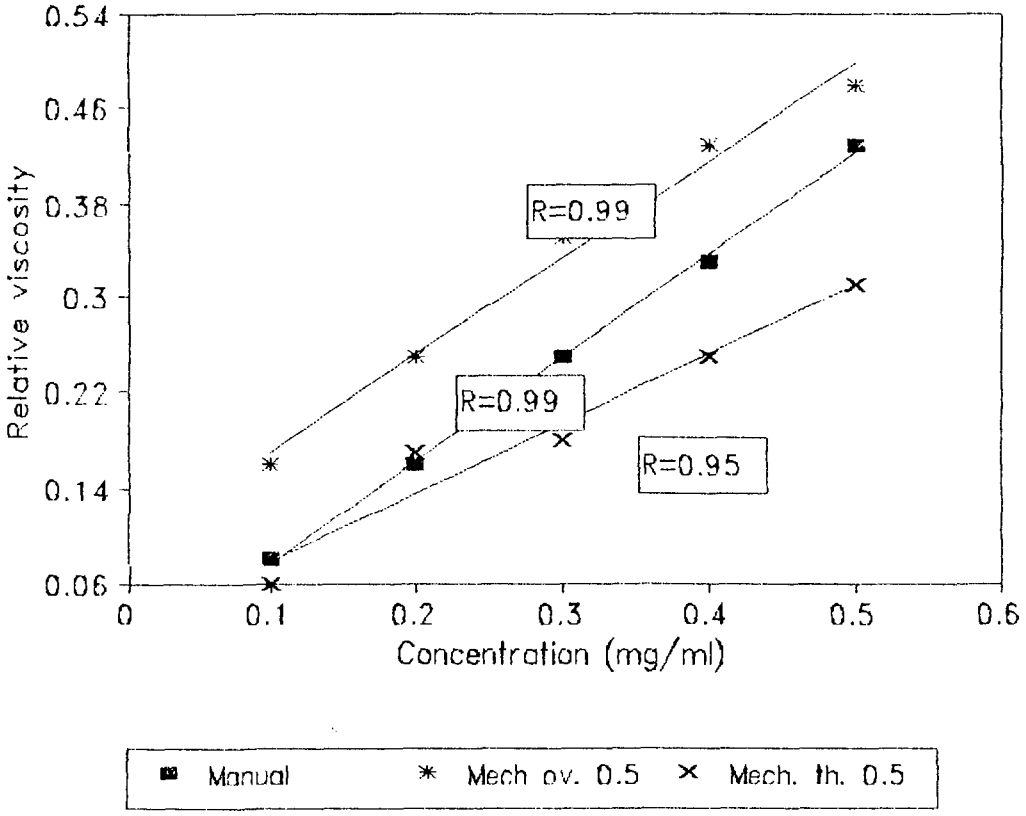

Fig. 21: Effects of purification methods on the viscosity of guar gum as measured by Ostwald Method in HFG53

lowest for the three types of gums is overs 0.5 mm, endosperm and throughs 0.5 mm. Therefore, the quantity of contaminants in the gums reduces the viscosity when determined by this method.

- **Redwood Method:**

The endosperm gum has higher contaminants than overs 0.5 mm, but has higher carbohydrates content (85%) compared to 81% in the overs 0.5. Therefore the purity of gums plays an important role if the contaminants level was within a small range (Fig. 22).

- **Brookfield Method:**

For genotype HFG53 the viscosity curves show distinct wide variation as shown in Figure 23. The characteristics of the curves are not directly correlated to contaminants nor gum quantity. The throughs which have high impurities than pure endosperm give better viscosity contents.This attributed to particle size distribution which is finest for throughs.

4.9 The Effects of Purification on Viscosity Characteristics of Genotype HFG182 Guar Gum :

Three types of gums are used to study the effect of purity within the genotype and they are pure endosperm gums overs 0.5 mm and throughs 0.5 mm gums.

- **Ostwald Method:**

The Ostwald method produced relative viscosity versus concentration with best linearity for overs 0.5 mm, pure endosperm and throughs 0.5mm as shown in Fig. 24. The viscosi-

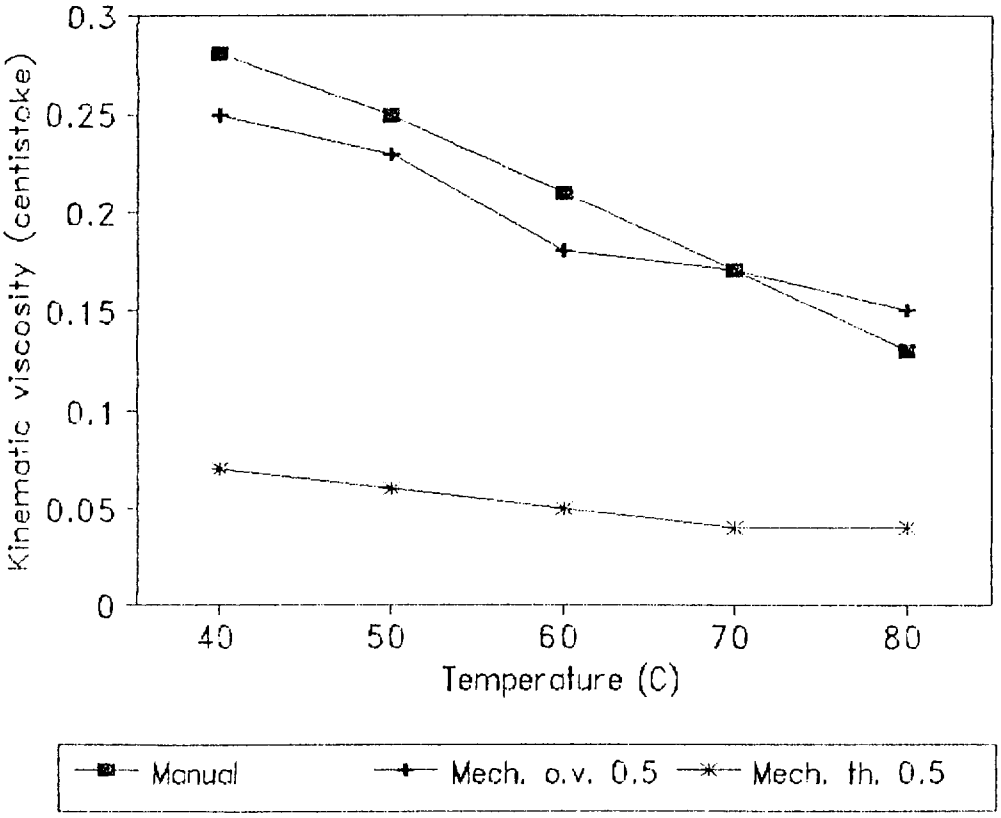

Fig. 22: Effects of purification methods on the viscosity of guar gum by Redwood Method in HFG53

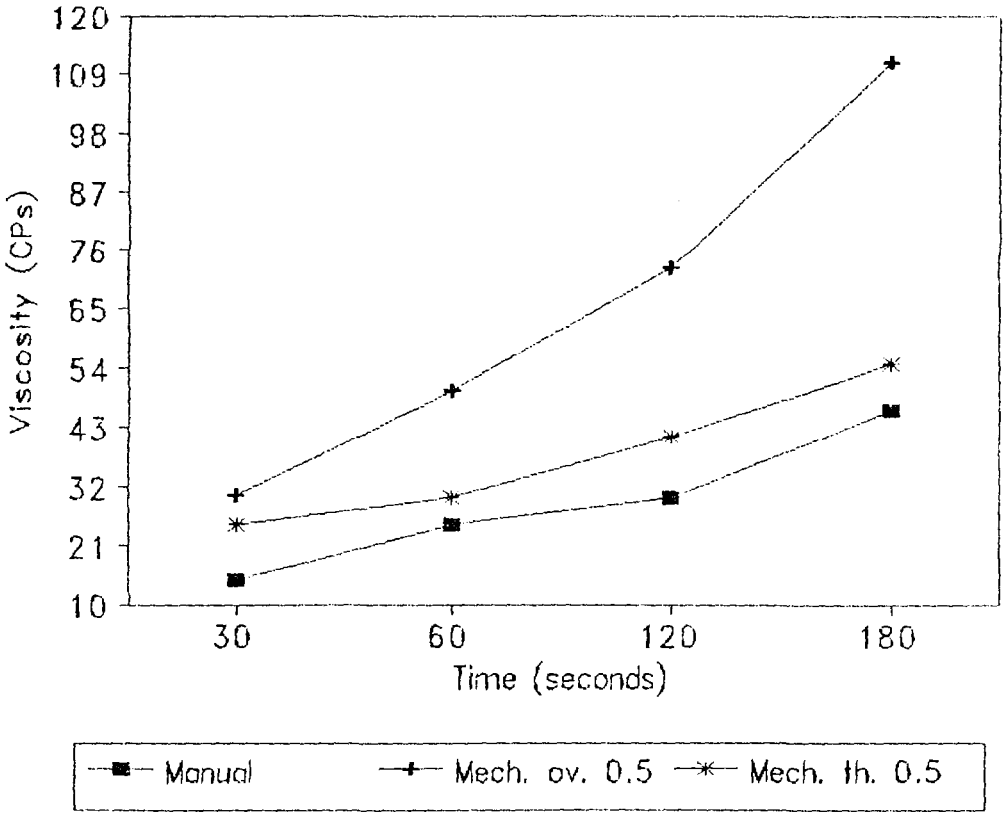

Fig. 23: Effects of purification methods on the viscosity of guar gum as measured by Brookfield Method in HFG53

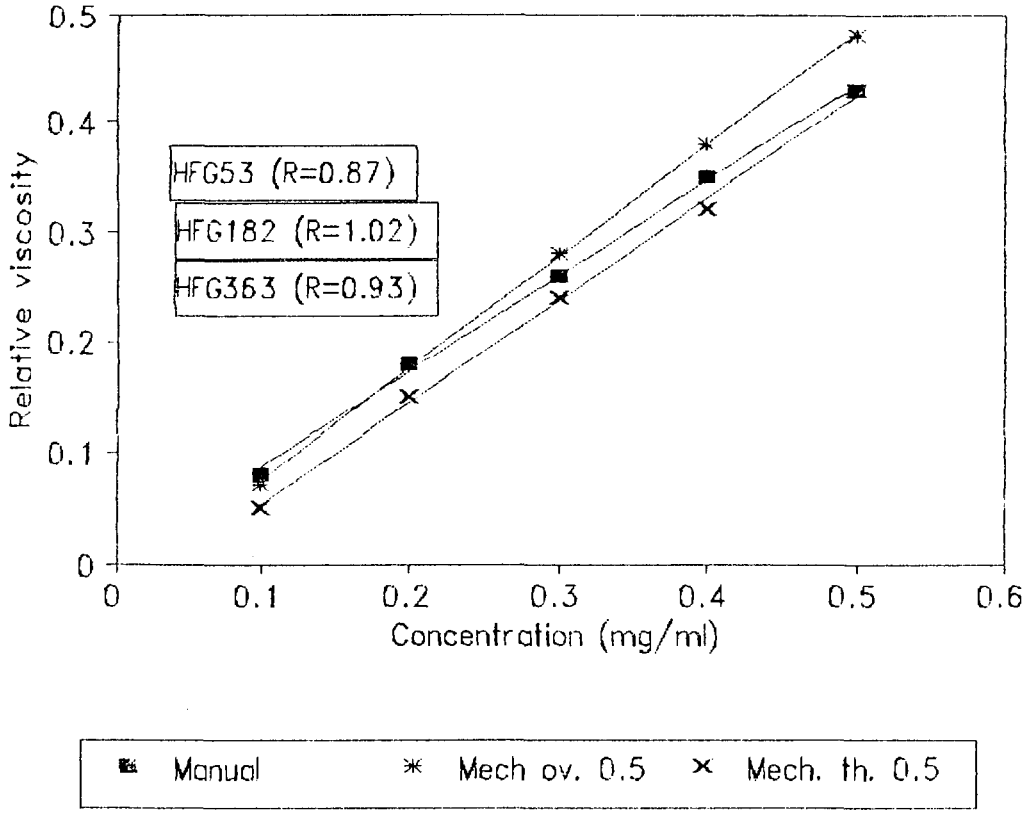

Fig. 24: Effects of purification methods on the viscosity of guar gum as measured by Ostwald Method in HFG182

ty level and linearity correlated well with the degree of contamination in the three gums (Tables 4c and 8).

- **Redwood Method:**

The measurement of viscosities of gums at increasing temperatures show a drop (Fig. 25). The highest curve is that of gums from pure endosperm although the presence of contaminants were not significant ($P \geq 0.0\ 5$) in pure endosperm and overs 0.5 mm (Tables 4c and 8). Therefore the influence in this genotype HFG182 is due to carbohydrates quantities in gums as shown to be 86.4% and 83.5% for endosperm and overs 0.5 mm, respectively.

For gums of overs and throughs, the curves variation was insignificant at various temperatures. This method is less sensitive to contaminants level if the range was small (Table 8). The main contaminants intended in this case were fiber, protein and fat.

- **Brookfield Method:**

For genotype HFG182 the viscosity curves, time of dispersibility and rate of hydration was influenced by the degree of contamination as shown in Fig. 26 for overs 0.5, pure endosperm and throughs 0.5 mm as the lowest curve. There is greater variation in the curves of pure endosperm and throughs 0.5 mm gums at 180 seconds.

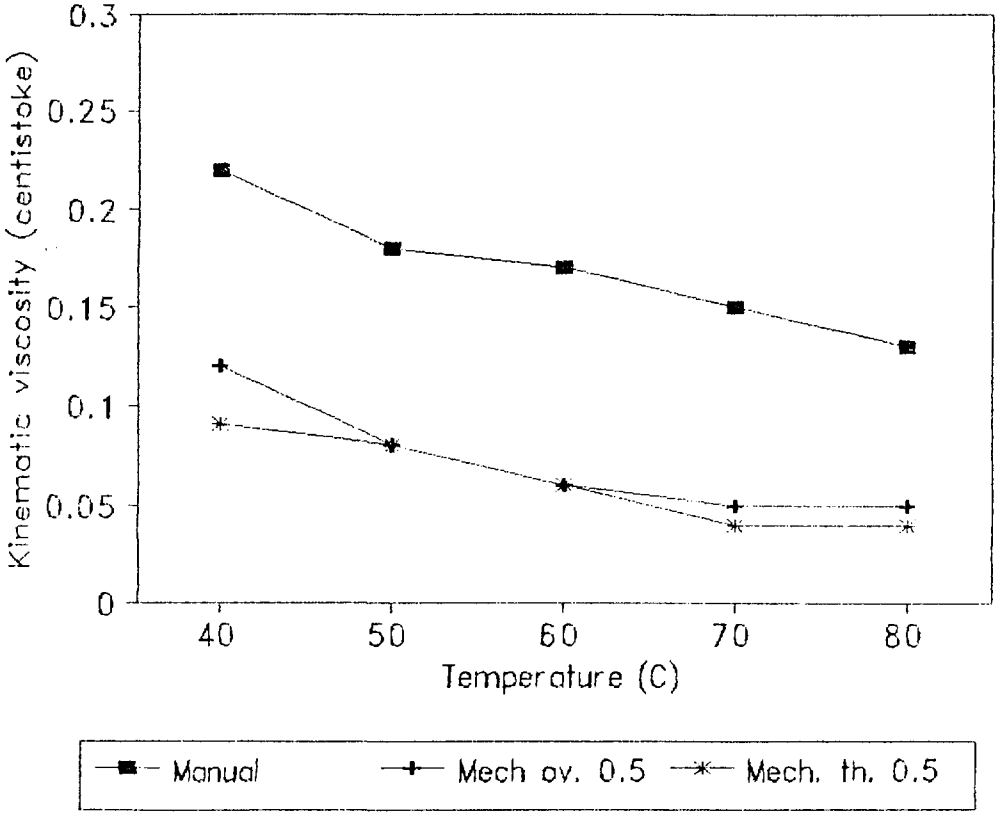

Fig. 25: Effects of purification methods on the viscosity of guar gum as measured by Redwood Method in HFG182

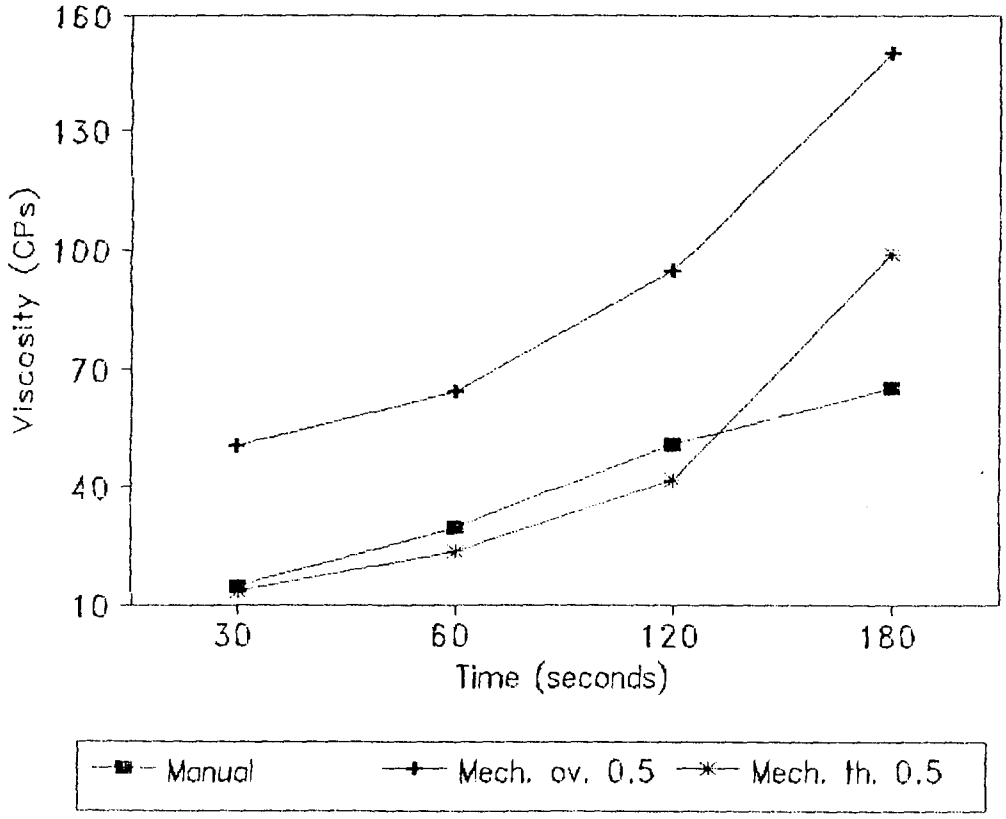

Fig. 26: Effects of purification methods on the viscosity of guar gum as measured by Brookfield Method in HFG182

4.10 The Effects of Purification on Viscosity Characteristics of Genotype HFG363 Guar Gum:

Three types of gums used in this study for purity effects within the genotype are pure endosperm, overs 0.5mm and throughs 0.5 mm.

- **Ostwald Method:**

The viscosity curves in Fig. 27 shows the linearity of the three gums solutions at concentrations ranging from 0.1 to 0.5 mg/ml. There is a wide variation between overs 0.5 mm on one hand and pure endosperm and throughs 0.5 mm on the other hand. This is due to the variation in the contaminants level shown in Tables 4c and 8.

- **Redwood Method:**

The viscosity characteristic curves for genotype HFG363 are similar for the three gums (Fig. 28). The contaminants and gum quantity have no greater influence on the viscosity. The viscosity levels are lower than all viscosity of other genotypes HFG53 and HFG182 and the temperature has greater influence on gum stability, this is not experienced in other genotypes.

- **Brookfield Method:**

The viscosity curve for genotype HFG363 gum show two stages. The first stages at 30 and 60 seconds of measurement. In this stage dispersibility and hydration rates were better for throughs 0.5 mm than the overs 0.5 and endosperm gum that could be due to the particle size and hardness (Fig 29). In the second stage of the curves the trend was similar to the

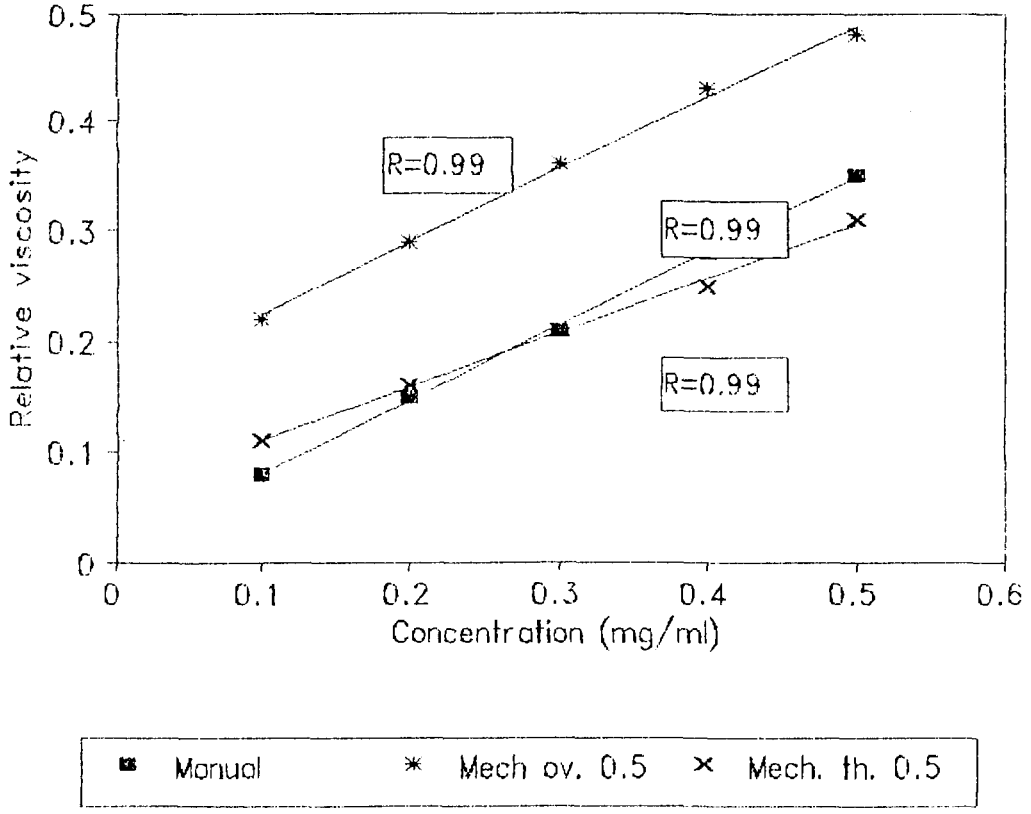

Fig. 27: Effects of purification methods on the viscosity of guar gum as measured by Ostwald Method in HFG363

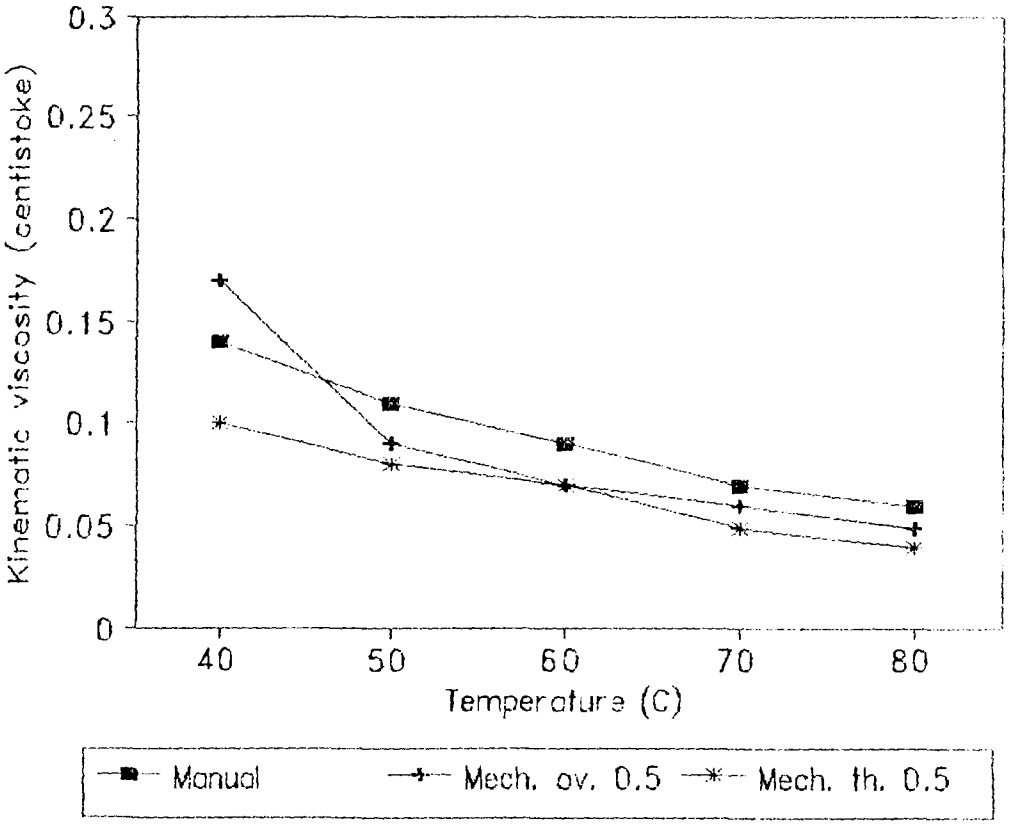

Fig. 28: Effects of purification methods on the viscosity of guar gum as measured by Redwood Method in HFG363

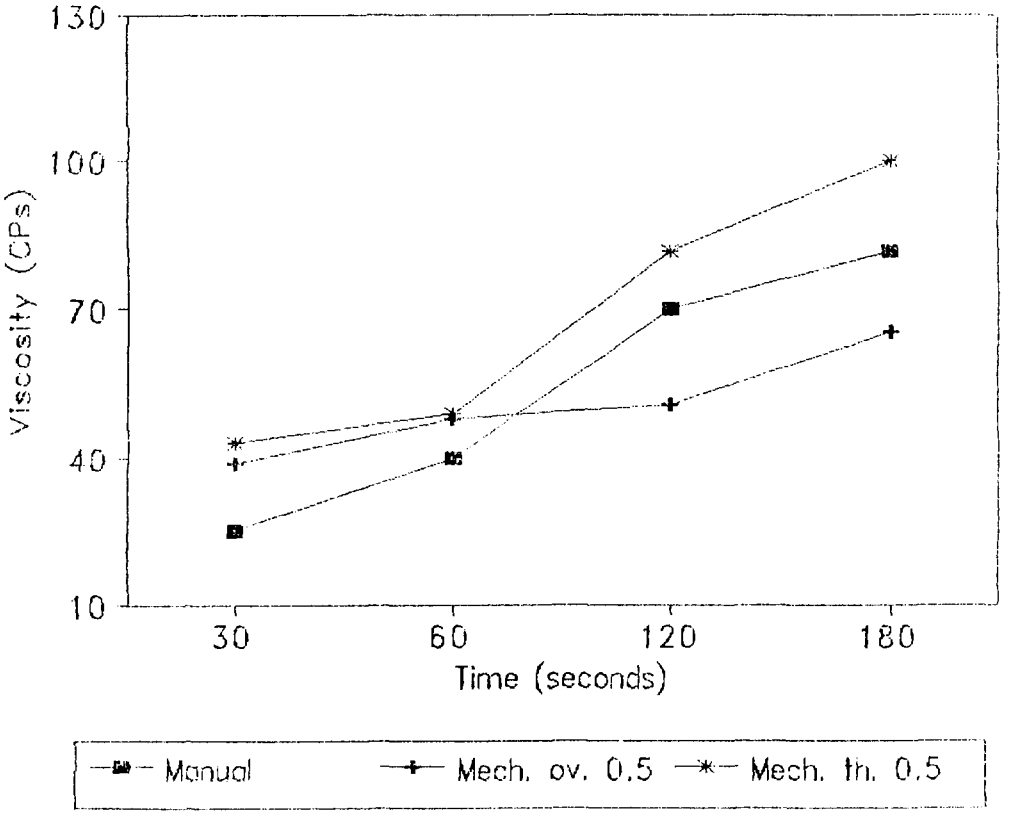

Fig. 29: Effects of purification methods on the viscosity of guar gum as measured by Brookfield Method in HFG363

first stage but the variation become wider and endosperm gum hydration rate started to increase faster than overs 0.5mm (Fig. 29). This bevhaiour could have been much clear if the time of the dispersibility was increased beyond 180 second.

The effect of the concentration on the viscosity of guar gum reported by Glicksman (1969) was stated that in general, viscosity increased linearly (Newtotnian) with the increase in the concentration up to about 0.5%.

At higher concentration guar solution behave as non-Newtonian, thixotropic systems mainly as a result of the molecular interaction at higher concentrations.

Redwood method behave similar to the finding of Whistler and Hymowitiz (1979) who reported that the viscosity of fully hydrated 1.0% guar gum solution varies almost directly with the changes in temperatures over the range of 20-80°C. The decrease in viscosity caused by the degradative effect of prolonged heat.

4.11 The Influence of Salt (sodium chloride) and Sugar on Guar Gum Solution:

The Redwood Viscosity Method was modified to measure the effect of salt, sugar and combined salt and sugar concentrations on solutions (0.5%) of commercial guar gums. These gums are specified with the powder fineness 80 mesh and 200 mesh. The salt concentration studied are 1,0%, 1.5% and 2.0%. The sugar concentrations used are 5%, 10% and 15%. The third study investigated the combination of salt and sugar on the guar gum solutions.

The results of the effect of salt and sugar are shown in the Figures (30, 31, 32 and 33) for viscosities versus the temperature range from 40°C to 80°C.

4.11.1 Effect of Salt Concentration on Viscosities of Guar Gum (200 mesh):

The addition of salt concentrations to guar gum solution (0.5%) resulted in a synergistic effect on viscosities at all temperatures (Fig.30). At 40°C, 1.0% salt gave the highest viscosities followed by 1.5% and 2.0%. The control solution viscosity was by far the lowest with decreasing rate proportional to temperature increase (Fig. 30). The temperature increase has a reducing effect on viscosity of all treated solutions.

4.11.2 Effect of Salt Concentration on Viscosities of Guar Gum (80 mesh):

The effect of salt concentrations on guar gum (80 mesh) solution is different from that experienced in guar gum (200 mesh). The effect of 1.5% salt on viscosity is the highest at all temperatures 40-80°C. The control solution viscosity is higher than those for 1.0% and 2.0% at 40°C. There is no significant difference ($P \geq 0.05$) for viscosities for control, 1.0% and 2.0% salts at 60-80°C as shown in Fig (31). The effect of salt on guar gum 80 mesh is less than in the guar gum 200 mesh. There is a positive effect of particle size and impurities which decreases with fineness of the gum grades.

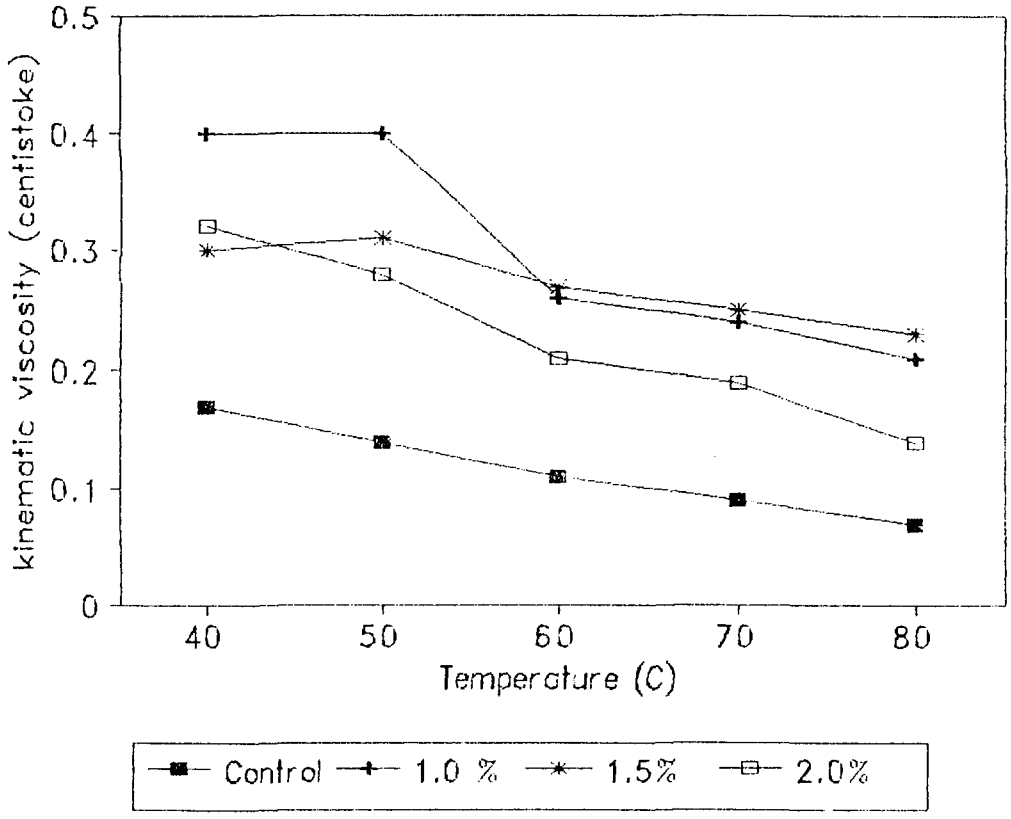

Fig. 30: Effect of salt concentration on the viscosity of commercial guar gum (200 mesh) solution 0.5%

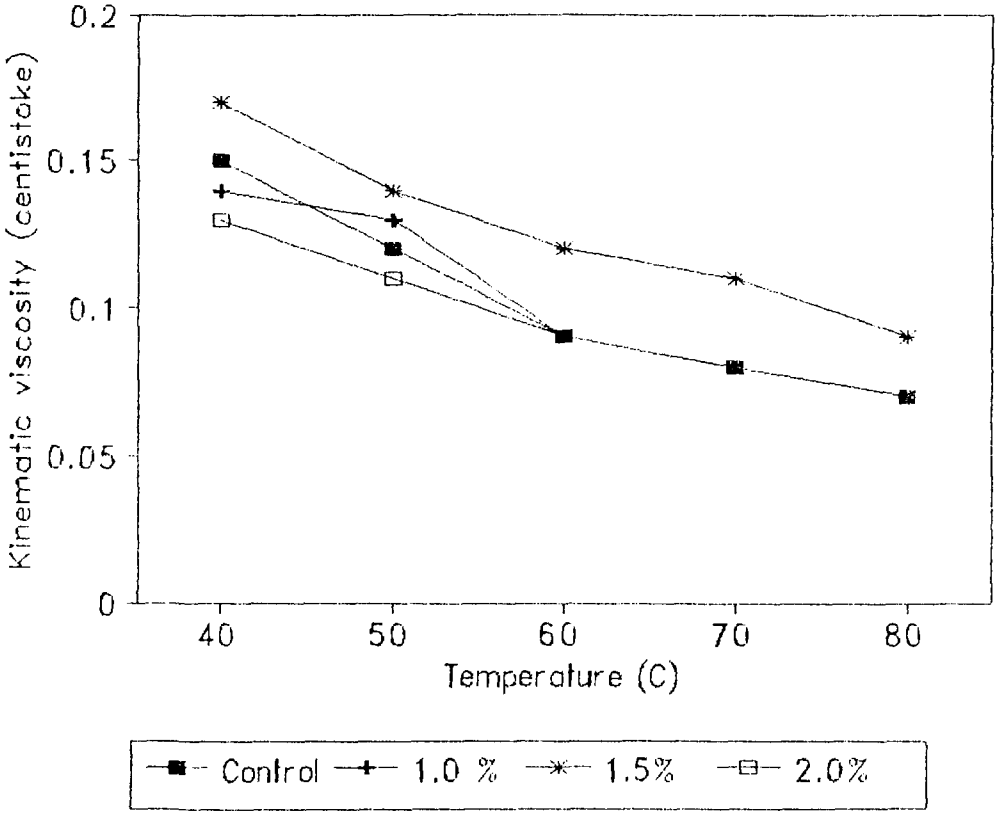

Fig. 31: Effect of salt concentration on the viscosity of commercial guar gum (80 mesh) solution 0.5%

4.11.3 Effect of Sugar Concentrations on Viscosities of Guar Gum (200 mesh):

The results of adding sugar concentrations 5%, 10% and 15% to 0.5% gum solution increased the viscosities by a wider margin (Fig. 32). The highest seudo-plastic effect was experienced with 5% sugar followed by 15% and 10% as the lowest. The effect of temperatures was similar to all solutions curve shown in Figure 32 . The control solution (0% salt) is shown to have the lowest viscosities at all temperatures when compared to these containing sugar. There is significant variation ($P \leq 0.05$)in all the solutions viscosities added 5%, 10% and 15%. According to Allen 1964, the viscosity of guaran-sugar solutions decrease gradually proportional to the sugar concentration.

4.11.4 Effect of Sugar Concentration on Viscosities of Guar Gum (80 mesh):

The addition of sugar to coarse guar gum (80 mesh) gave lower viscosities than those found in fine guar gum (200 mesh) as shown in Figures (32 and 33). The highest viscosities are shown by adding 10% followed by 15%, 0% (control) and 5% the lowest (Fig. 33). The effects of sugar in coarse guar gum (80 mesh) is not similar to those experienced in fine guar gum (200 mesh). The variation between the guar-sugar solution with coarse particle size is narrower than that shown by fine size (200 mesh). The degree of purity might have also played a role as 80 mesh is less purer than 200 mesh.

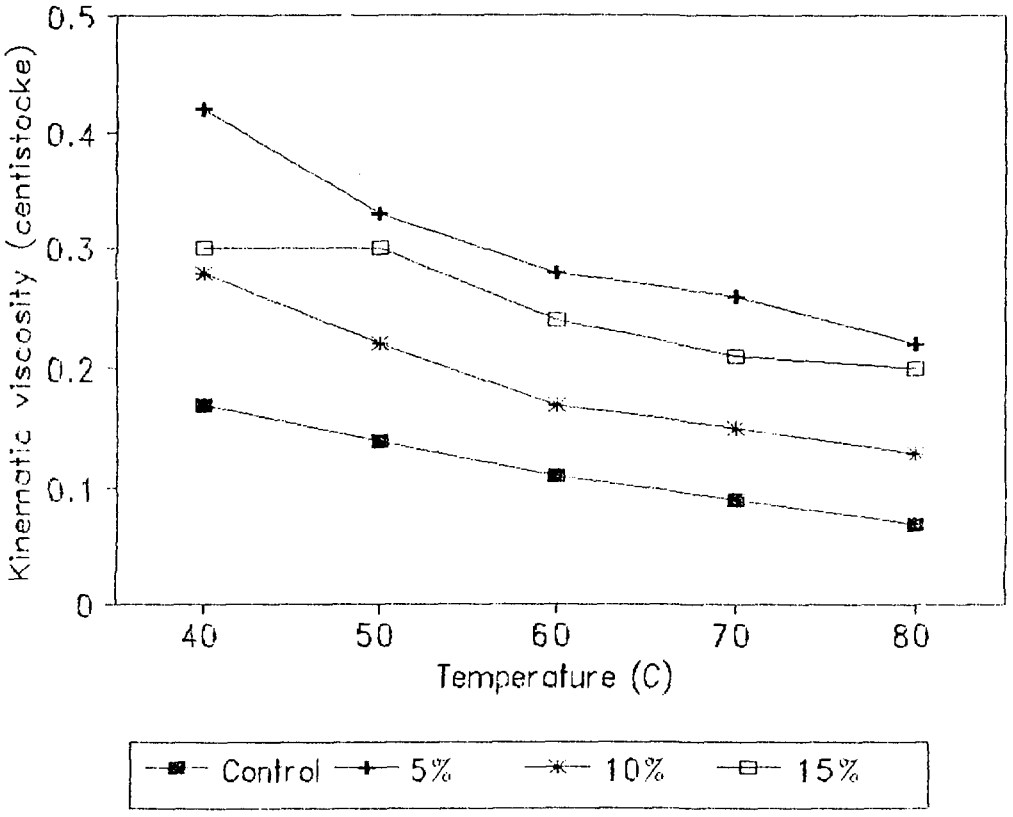

Fig. 32: Effect of sugar concentration on the viscosity of commercial guar gum (200 mesh) solution 0.5%

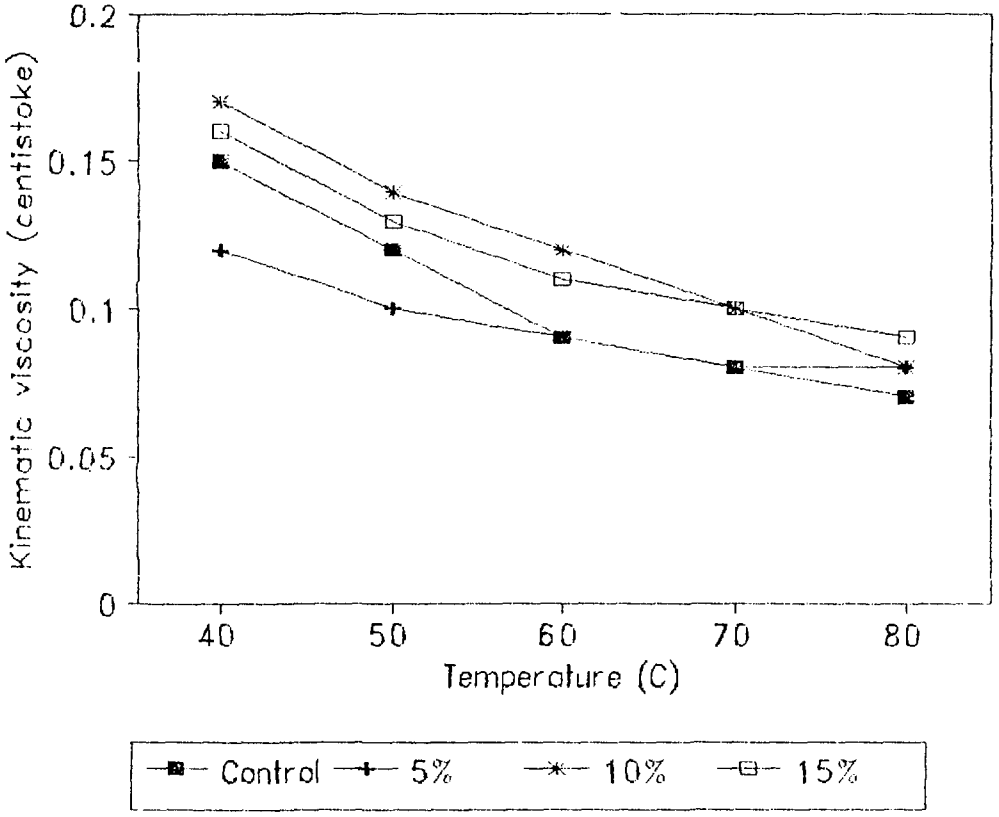

Fig. 33: Effect of sugar concentration on the viscosity of commercial guar gum (80 mesh) solution 0.5%

4.11.5 Effect of Combined Salt-Sugar on Viscosities of Guar Gum (200 mesh):

The effect of combining salt (1.0%) and sugar (5, 10 and 15%) in guar gum (0.5%) solution resulted in the decrease in viscosities of all solutions (Fig. 34), as compared to individual effects of salt and sugar. The order of the effect remain similar for the combined salt-sugar except that the variation of salt-sugar (1/5, 1/10 and 1/15) has narrowed greatly (Fig. 34).

The effect of heat on reducing viscosities rate in combined salt-sugar curves is not increased. The effects of combined salt (1.5%) and sugar (5%, 10% and 15%) on guar gum solution (0.5%) show a significant ($P \leq 0.05$) increase in viscosity for salt-sugar, but there is no significant increase compared to individual actions of salt and sugar (Figures 30 and 32). The only marked effect is shown in the combination of salt-sugar (1.5/10 and 1.5/15 where the 1.5/10% effect decreased to the level of 1.5/15% that show no significant difference (Fig. 35). This effect is not shown in Figures (30 and 32) for salt (1.5 and sugar 10% and 15%).

For the combination of salt-sugar (2.0/5, 2.0/10 and 2.0/15) (Fig. 36), the viscosities of guar gum (200 mesh) are greatly reduced if compared to individual enhancement of gum solution by salt and sugar (Figures 30 and 32). The effect of salt-sugar is still higher than the control (0%). There is a narrowing down of the variation in the effects of slat-sugar solution if these are compared to the curves in Figures (30, 32, 34 and 35).

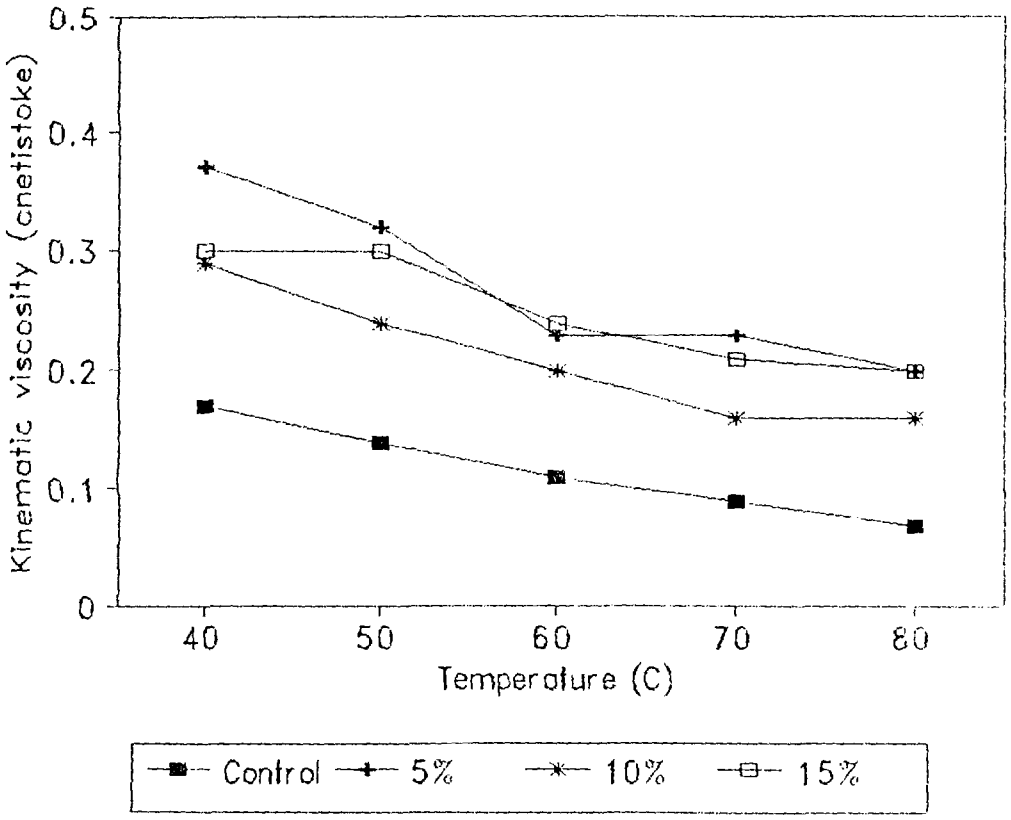

Fig. 34: Commercial guar gum (200 mesh) viscosity at 1.0% salt with varying sugar concentrations

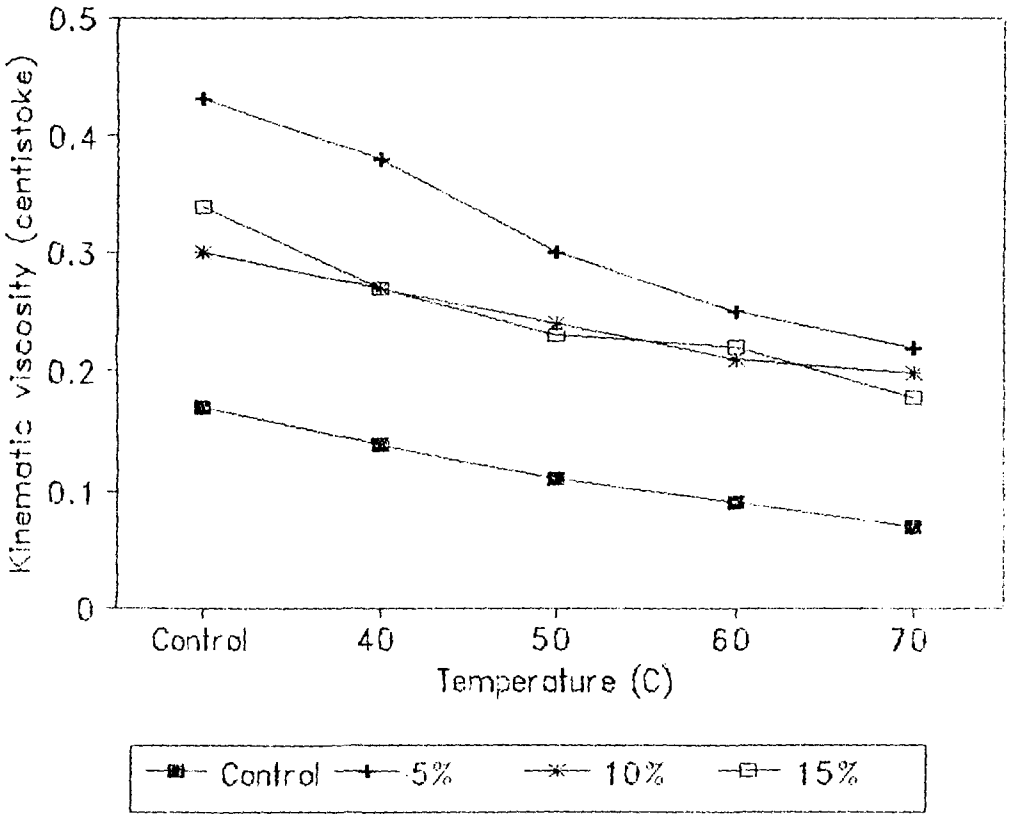

Fig. 35: Commercial guar gum (200 mesh) viscosity at 1.5% salt with varying sugar concentrations

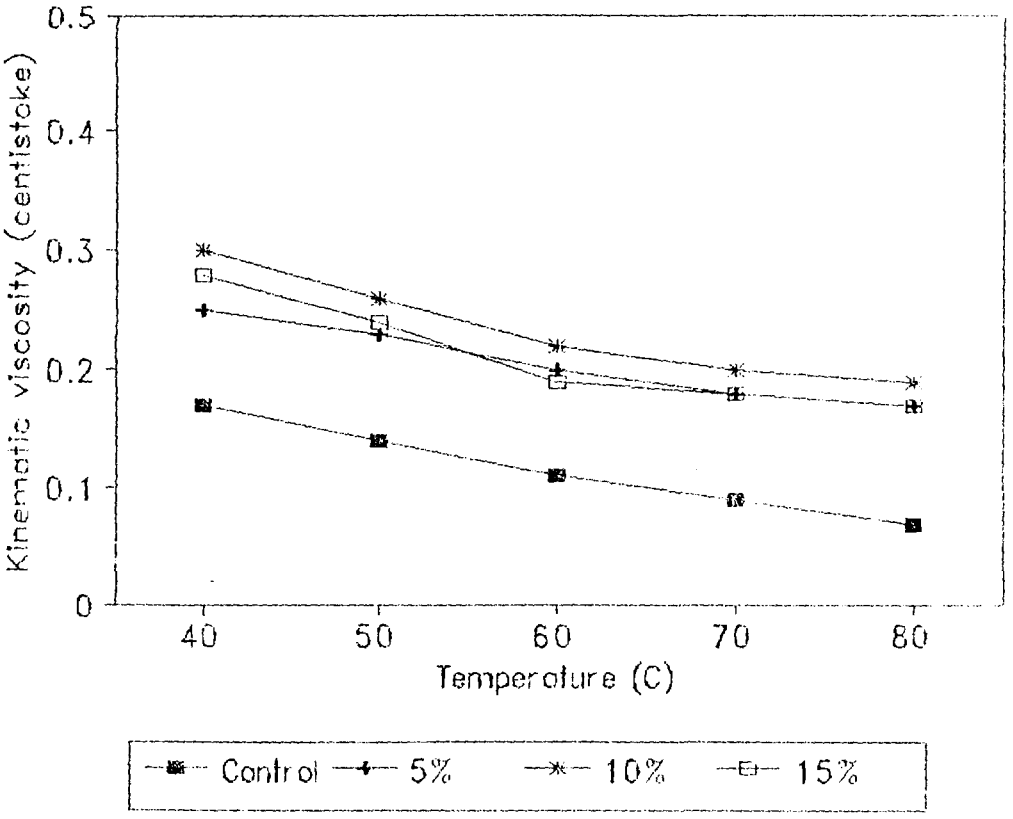

Fig. 36: Commercial guar gum (200 mesh) viscosity at 2.0% salt with varying sugar concentrations

Generally, an increase in salt concentration on sugar has a negative effect on the viscosities on guar gum (200 mesh).

4.11.6 Effect of Combined Salt-Sugar Action on Viscosities of Guar Gum (80 mesh):

The effect of adding 1.0% salt and sugar concentrations (5, 10 and 15%) to guar gum solution (0.5%) produced a decrease in viscosity at 40°C (Fig. 37). The variation of the curves at 50-80°C is similar to the individual effect of salt and sugar. The decreasing rate due to high temperatures seems to be less than those in Figures (31 and 33). The effect of 1.0% salt on 10% and 5% sugar is the same as control at 40°C.

There is no significant difference for the effect of salt (1.0%) and 60°C and 70°C for 10% and 15% sugar. The temperature effect was less on 5% sugar combined with 1.0% salt. Addition of 1.5% salt and sugar concentrations to guar gum solutions (00.5%) gave same results for 5% and 15% sugar at 40°C. There is a significant variation at all temperatures between 5 and 15% against 10% sugar as shown in Fig (38). At 80°C there is no variation for 5% and 10% sugar. There is negative effect by combined action of 1.% salt and 15% sugar that produced lower viscosities than 5% and 15% sugar. When salt was increased to 2% on sugar concentrations, the variation of the curves becomes very close as shown in Fig. (39).

The results for 5% and 10% sugar and salt 2% are the same at 60, 70 and 80°C shown in Fig. (39). There are some variation at 40 and 50°C for 5 and 10% sugar. The effect of 2% salt and 15% sugar give lower viscosities than the 5% and 10%.

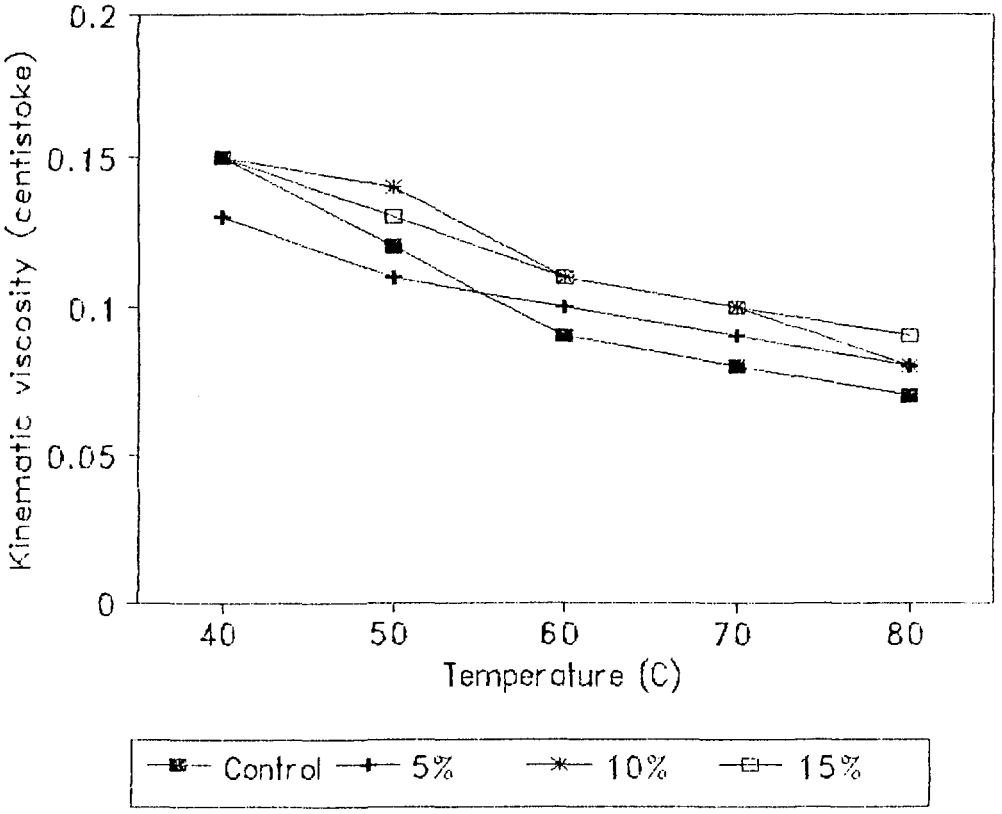

Fig. 37: Commercial guar gum (90 mesh) viscosity at 1.0% salt with varying sugar concentrations

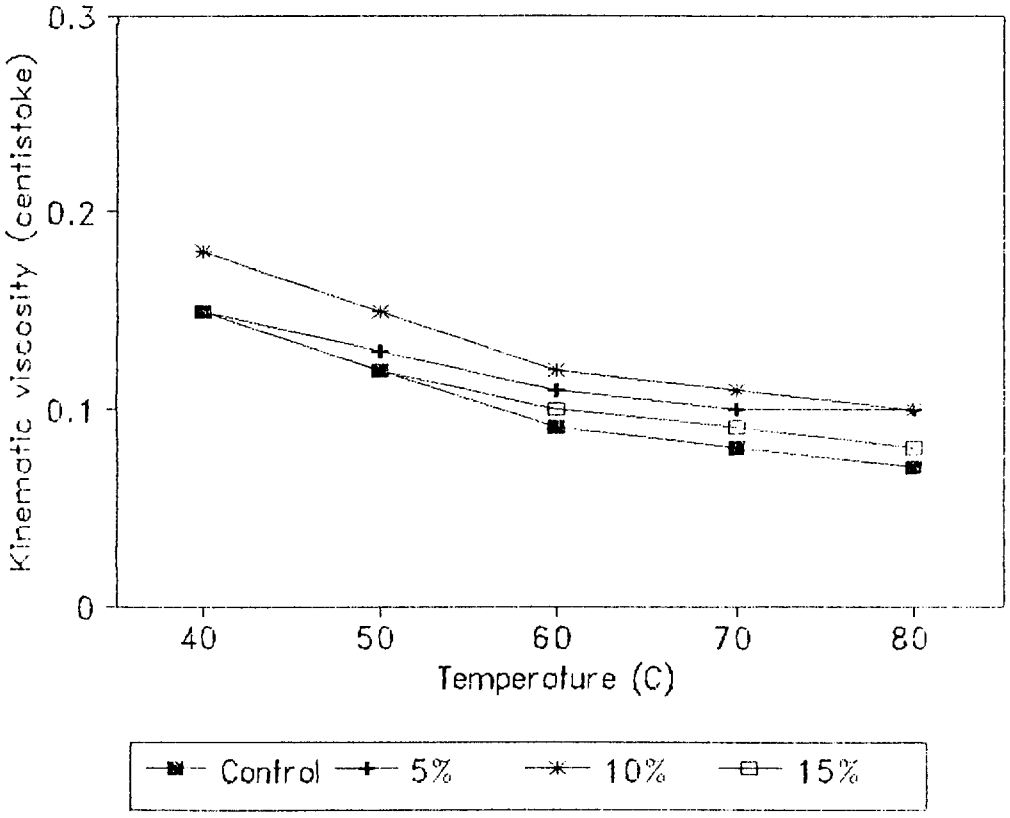

Fig. 38: Commercial guar gum (80 mesh) viscosity at 1.5% salt with varying sugar concentrations

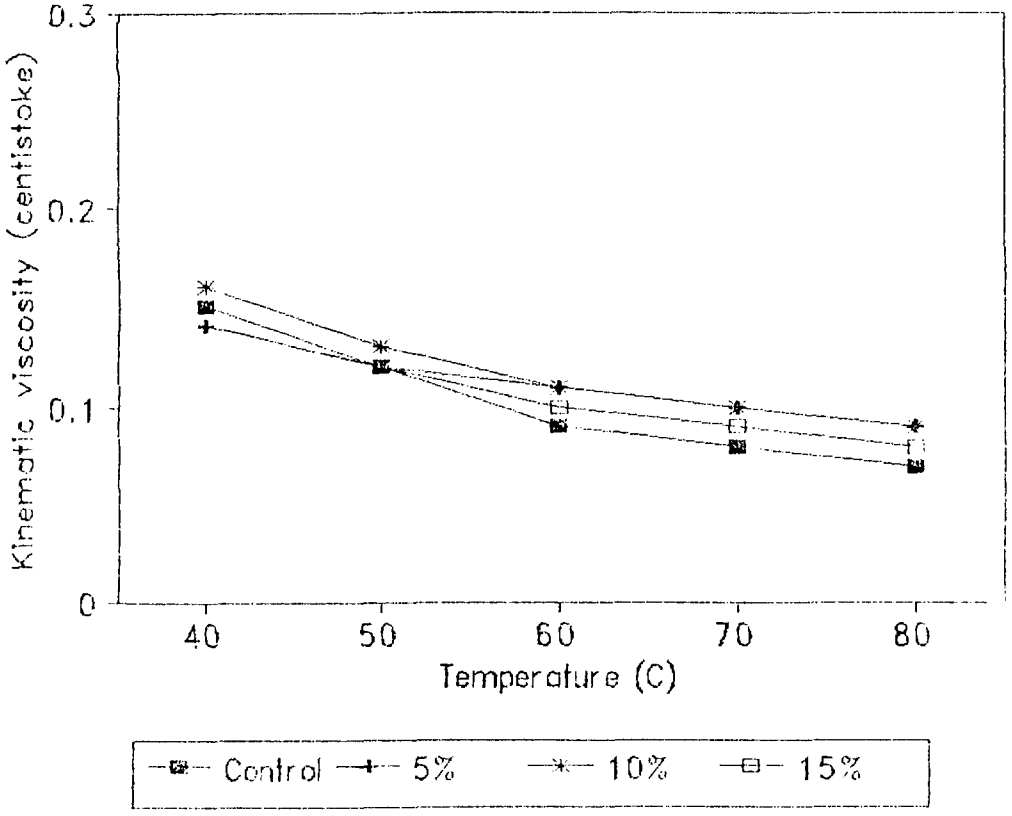

Fig. 39: Commercial guar gum (80 mesh) viscosity at 2.0% salt with varying sugar concentrations

In conclusion, 2% salt and 15% sugar have pronounced reduction on viscosity of guar gum solution (0.5%). The heat compatibility of gum (80 mesh) solution treated with salt-sugar concentrations was better than individual action of salt and sugar.

Comparative study on viscosity of two commercial guar gums grades of 200 mesh and 80 mesh shows that the former gives high viscosities (centistoke) by Redwood than the latter. Addition of salt, sugar, and salt-sugar combination to guar gum increases the viscosities of fine grades (200 mesh), but in coarse grade (80 mesh) the increase was not similar as the untreated guar gum was high or the same at 40°C and 50°C. The rate of viscosity decrease was much reduced in the combined action of salt-sugar on guar gum. The addition of salt (sodium chloride) and sugar gave high viscosities for the two guar gums (200 and 80 mesh) than control solution (untreated guar gum). In combining salt-sugar action there was a general viscosity reduction at all temperatures.

Addition of sugar should decrease the viscosity, surprisingly the result in Fig. (32) showed that 15% showed reincrease in viscosity, a phenomena which should be explained.

SUMMARY:

The endosperm of locally grown guar seeds was found to represent 35-40% of the whole seed. Guar gum, is a polysaccharide, is estimated as carbohydrates of the endosperm at an average 86.38±0.04%.

The mechanical purification methods produced better guar gum than pure endosperm prepared manually by hand. This depends on the mechanical purification efficiency in removing the germ and hull plus the degree of polishing the splits. When the endosperm splits are ground in a hummer (0.5mm sieve) the overs are more purer than throughs. Throughs are highly contaminated with germ and hull as these two components are friable.

Guar gum in solution at concentration up to 0.5 mg ml behaves linearly according to Newtonian laws. The modified Redwood method was found to be the most suitable technique of studying guar gum heat stability between the temperatures 40-80°C. The best heat stable guar gum was given by genotype HFG53.

Hydration rate or dispersibility of guar gum from genotype HFG 363 gave better results using Brookfield method. The influence of sodium chloride(1, 1.5 and 2%) and sugar (5, 10 and 15%) on commercial guar gum (200 mesh) increased viscosity level and heat stability, but the effect was negligible with guar gum 80 mesh. The effect of combined salt-sugar concentrations on commercial guar gum 200 mesh was better than for guar gum 80 mesh.

RECOMMENDATIONS:

For further studies the following recommendations are suggested:-

1. Guar gum applications in food products eg. bread, biscuits,- pasta, ice cream, jellies, squashes, cheese, sausages and pharmaceutical products.
2. Detection techniques for guar gum in food items.
3. Effect of combination between guar gum and gum arabic or others.
4. Rheological studies of guar gum under different conditions.

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