RESEARCH NOTE

GUM TRAGACANTH FIBERS INTRODUCING PRIMITIVE GT FIBERS

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Abstract In this research, a method for producing gum tragacanth (GT) fibers is presented. Ribbon type GT of *Astragalus Gummifer* species were obtained from local production lands. The gum was treated with alkali to increase its process ability, and allowed to stand for a period of time to reach the proper viscosity and/or to "ripen". Samples with different ripening times were prepared and their viscosities were measured to determine the effect of ageing time on viscosity. They were injected into a coagulation bath. Calcium chloride with different concentrations was applied as the coagulant agent. The consolidated samples were washed under different pH conditions. Finally, the mechanical properties of these primitive fibers were measured and their scanning electron micrographs were taken. The results show that chemically modified GT can be processed into fibers with different qualities.

Key Words Gum Tragacanth, *Astragalus Gummifer*, Ribbon Type, Alkaline Treatment, Ripening Time, Coagulant Agent, Calcium Chloride

چکیده در این تحقیق برای اولین بار روشی برای تبدیل کتیرا به لیف ارائه شده است. برای انجام آزمایشها از کتیرای نوع مفتولی که در مناطق مختلف ایران به صورت خودرو رویش دارد، استفاده گردید. به منظور افزایش فرآیندپذیری و قابلیت تبدیل این صمغ به الیاف محلولهایی از آن تهیه و تحت عملیات قلیایی قرار گرفتند. برای رسیدن به ویسکوزیته مطلوب یا "زمان رسیدن مناسب"، به نمونه‌ها زمان داده شد. ویسکوزیته نمونه‌ها با زمان‌های رسیدن متفاوت اندازه‌گیری شد و بدین ترتیب تاثیر زمان بر ویسکوزیته مورد بررسی قرار گرفت. نمونه‌هایی با زمان‌های رسیدن متفاوت به داخل حمام انعقاد تزریق شدند. در حمام انعقاد از کلرید کلسیم با غلظتهای مختلف به عنوان ماده منعقد کننده استفاده گردید. نمونه‌های حاصله تحت شرایط پی اچ متفاوت شسته شدند و در نهایت خشک گردیدند. برخی از خواص مکانیکی الیاف تولید شده اندازه‌گیری گردید و میکروگرافهایی از آنها گرفته شد. نتایج نشان می‌دهند که صمغ کتیرا قابلیت تبدیل شدن به الیاف را دارد و با تغییر شرایط عملیات فرآیند پذیر نمودن و یا فرآیند تولید می‌توان الیاف کتیرا را با کیفیت‌های متفاوت بدست آورد.

1. INTRODUCTION

Gum tragacanth (GT) is exudates from several species of shrubs of the genus Astragalus, mostly found in certain areas of Asia and in the semi desert and mountainous regions of Iran, Syria, Turkey and other Near Eastern countries [1,2].

There are almost fourteen species of Astragalus, which produce GT. Among them, Astragalus gummifer, Astragalus kurdicus and Astragalus microcephalus are the most important species for the production of GT [3]. By incision on the trunk of shrub, the gum exudes and after being naturally dried by the air, it is collected as ribbons (the highest quality gum), or flakes [4].

GT has a unique taste. The ribbon type typically occurs as flattened, lamellated, frequently curved fragments or in straight, spirally twisted, linear

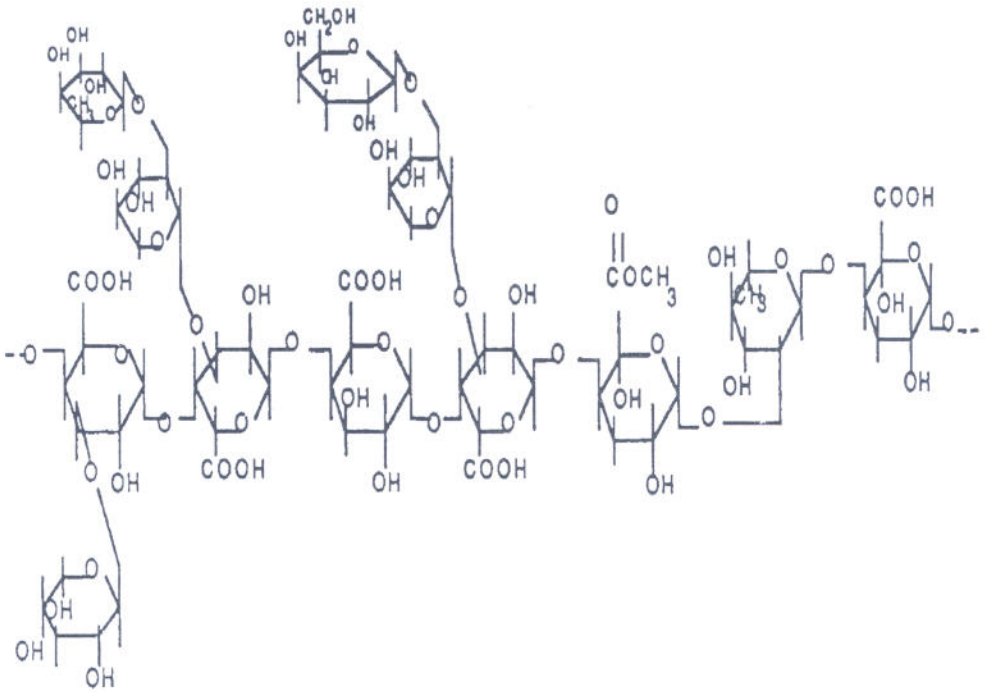


Figure 1. Chemical structure of GT.

pieces. They are hard, odorless, white to off-white in color, 50 to 100 mm in length, and have a diameter of 0.5 to 2.5 mm. On the other hand, the flakes are oval, thick, brittle, and more colored than ribbon type, varying in size from 12 to 50 mm in diameter [4]. Both types exhibit similar pseudoplastic properties, stability to temperature differences, and stability to pH, but there is a marked difference in the viscosity of ribbon and flake types. The ribbon type yields more viscous solutions than the flake type in the same concentration [5,6].

The molecular weight of non-graded GT is about 840,000 and the molecular shape for non-graded high-grade flake type is l=4500 nm and d=19 nm [4].

It is mostly believed that GT consists of at least two major components: a water-swellable (about 60 to 70 %), and a water-soluble portion [1,4]. However, this ratio may be different according to the species of the original shrub, for example the ratio of soluble/insoluble components for Astragalus microcephalus, Astragalus Gummifer, and Astragalus kuridcus are 65/35, 40/60, and 30/70 respectively [3,7].

The water-swellable part is called Bassorin, and the water-soluble part is a colloidal hydrosol, named tragacanthin. There has been some confusion on the nomenclature of these two constituents. Davidson refers to the water soluble, neutral polymer as an arabinogalactan, and the water–swellable polymer as tragacanthic acid (TA) [4].

TA, the major component of GT has a main backbone chain of (1→4)-α-D-galacturonic acid residues similar to pectic acid. The major difference between pectic acid and TA is that the latter has neutral side chains bonded to the acidic backbone. They show different physical properties in solution that may be attributed to the location of galacturonic acid residues, the side chains and the other components present. For example, pectic acid gels easily by cross-linking with calcium ions whereas GT does not. Xanthan gum, as another example, gels when hydrated with locust bean gum whereas GT does not. This might be due to the different location of uronic acid residues, which are parts of side chains in xanthan gum [4].

Kiumarsi reported that the majority of D-galacturonic acid residues in GT carry xylose-containing side chains through C-3 [6]. Three types of side chains have been recognized namely, single β-D-xylopyranose residues, disaccharide units of 2-0 -α-L-fucopyranosyl-D-xylopyranose and 2-0-β-D-galactopyranosyl-D-xylopyranose, and these must account for the majority of the sugar residues in the outer chains [8]. The general chemical structure of GT is shown in figure 1 [9].

GT has been known and used for over five thousand years. It is one of the most widely used natural polymers across the globe. GT can typically be used as the following agents [3,4,6,10,11]:

Stabilizer:
in food industries (e.g. French dressing)

Thickener:
in food (e.g. puddings, dry beverages mixes, quick cooking cereals, sauces), in pharmaceuticals (e.g. water ointments and salves, cosmetic creams and lotions), in textile (e.g. in textile printing pastes).

Emulsifier:
in food (e.g. cheese spread, whipped toppings), in pharmaceuticals (e.g. gynecological jellies, emulsions of mineral oil and fish oil), and in auto and furniture polishes.

Moisture retaining agent:
in food (e.g. ice milk, cheese)

Binding agent:
in food (e.g. gum drops)

Anti-freezing agent:
in food (dairy products, confectionery, dietetic foods).

Adhesive agent:
in cigar wrapper leaf, pills and tablet production.

According to the afore-mentioned literature survey, this natural polymer has not been used for the production of fiber yet, which in the presented article it has been experimented by chemically modifying gum tragacanth.

2. EXPERIMENTAL

2.1 Material Gum tragacanth of Astragalus Gummife (Ribbon type) from local lands near Isfahan, Iran. All chemicals from Merck and Fulka chemical companies.

2.2 Apparatus

1. Grinder: Ball mill type (Damavand Co.).
2. Viscometer: Brookfield model DV-I +
3. Scanning electron microscopy (SEM): Cambrige S360.
4. Strength Tester: Shirley fiber tester Micro 50.

2.3 Methods

Grinding and Sieving GT was cleaned from containments such as wood chips, and then was gradually grinded and sieved several times. Powdered gum with a mesh size 50 to 100 (150 to 250 μm) was used for processing. One consideration accounted for was not to grind GT too much as the mechanical grinding of dry gums could to some extent leads to molecular cleavage [4].

Dissolving of GT (Dope Preparation) 60, 80, and 100 gr (6, 8, and 10 w/w) of GT were gradually added into 1 lit of distilled water to avoid eye-fish affects or agglomeration. During this work, solutions were stirred using a mechanical stirrer. Alkaline solution was prepared by adding 10 gr NaOH to 100 ml distilled water, the solution was added in drops to the GT solutions during stirring. Samples were allowed to stand for different periods of time (12, 72, and 96 hrs) to ripen at 27°C.

Measuring Viscosity For measuring the viscosity, 500 ml of GT solution (6 percent w/w) was prepared in the same way as above. The viscosity was measured for samples with different ripening times (up to 330 hrs) at 1.5 and 3 r.p.m with the spindle number 5. During the different ripening times, GT solution was stirred slowly (10 r.p.m) 27°C.

Spinning, Coagulation Bath, and Measuring Mechanical Properties To spin the dope a 5ml syringe (the diameter of needle was approx. 1 mm)

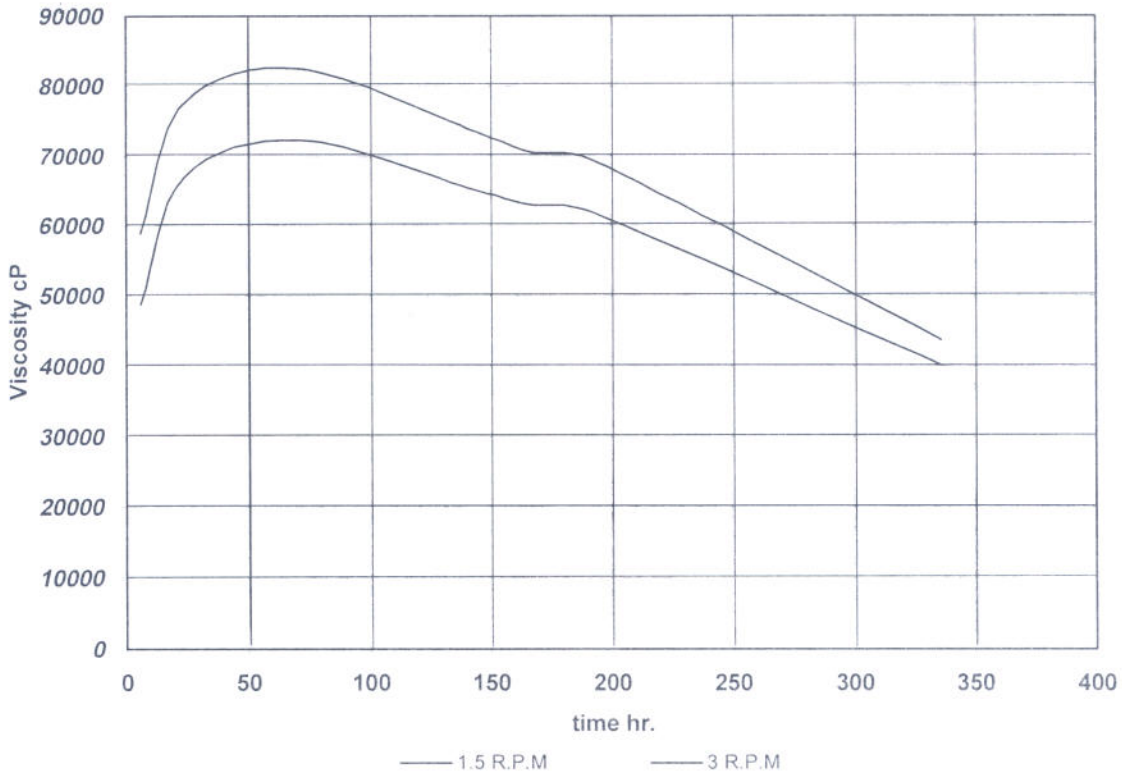


Figure 2. Effect of ripening time on the chemically modified GT solution viscosity.

was used and the contents of it squeezed out into a coagulation bath. $CaCl_2$ solution with different concentrations (2.5, 5, 10, 15, and 25 gr/lit) was used as the coagulant agent. The precipitated fiber was taken out of the coagulation bath immediately and immersed in a washing bath with neutral or acidic pH. The pH of acidic washing bath was set to 5, and acetic acid was used to adjust the pH. Washed samples with the length of 15 cm were dried in a tensionless state at 27°C. After 72 hrs some mechanical properties of them were tested as staple fibers. The produced fibers were 40 tex and the speed of test and the gauge length were 31 mm/min and 10 mm respectively.

3. RESULTS AND DISCUSSION

In this study wet spinning was preferred and chose for the production of GT fibers. The general properties of carbohydrates like GT were one of the major reasons for the selection. The first problem is preparing a viscose and proper dope, but crude GT makes a gel like state at concentration higher than 3% w/w. In this study, to increase solubility and processability, GT was dissolved in an alkaline solution. In the pH range greater than 7 all the galacturonic acid groups are ionized especially on the tragacanthic acid.

According to the repulsion of ionized polymer chains (anionic chains) the tangled or coiled chains, which make the gel like net or state, will separate of each other [4]. Consequently the preparation of more viscose GT solutions with the absence of gel like state will be possible.

According to Figure 2, it is seen that after 72 hrs of ripening time, the viscosity of GT solution is at its maximum value. This ripening time can be considered as the proper time (and/or ripening

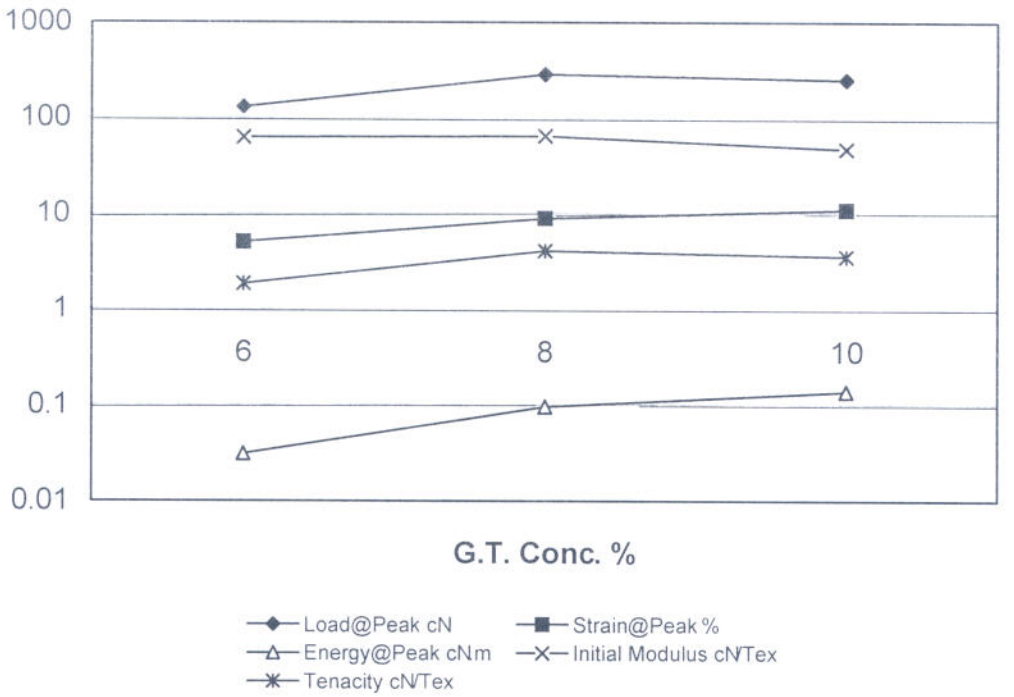


Figure 3. Effect of GT concentration on the mechanical properties of fibers.

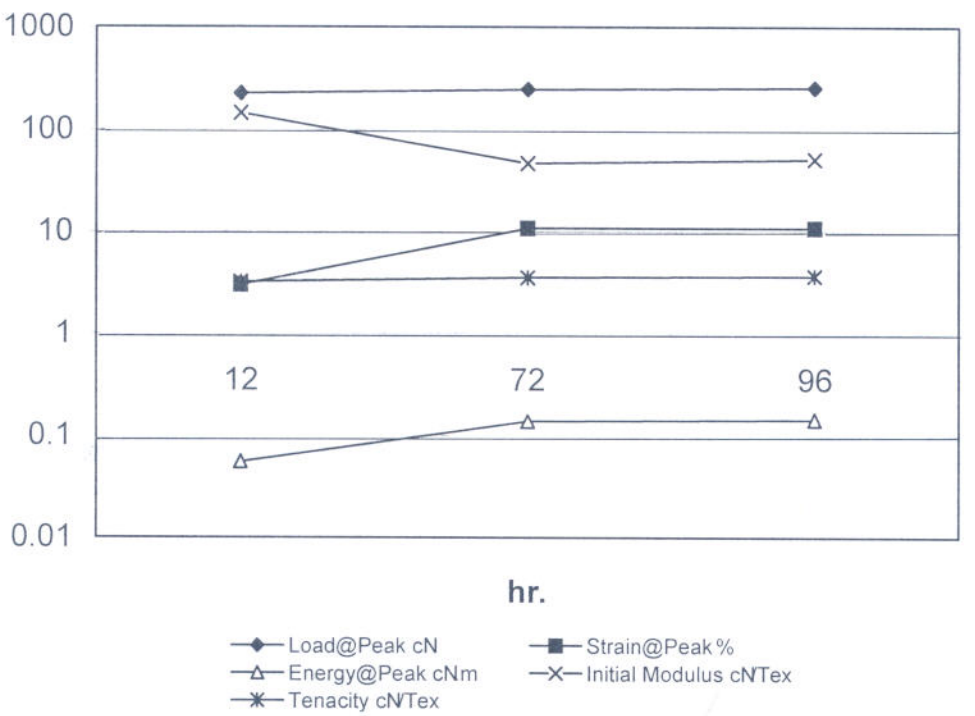


Figure 4. Effect of GT solution ripening time on the mechanical properties.

TABLE 1. Effect of Washing Condition on the Mechanical Properties of GT Fiber.

Washing Conditions	Load cN	Strain %	Energy cN.m	Initial modulus cN/Tex	Tenacity cN/Tex
Unwashed	137.56	16.53	0.1391	46.235	3.8478
Neutral pH	217.98	14.02	0.1772	89.22	6.0973
Acidic pH	248.43	9.58	0.1199	106.19	6.9491

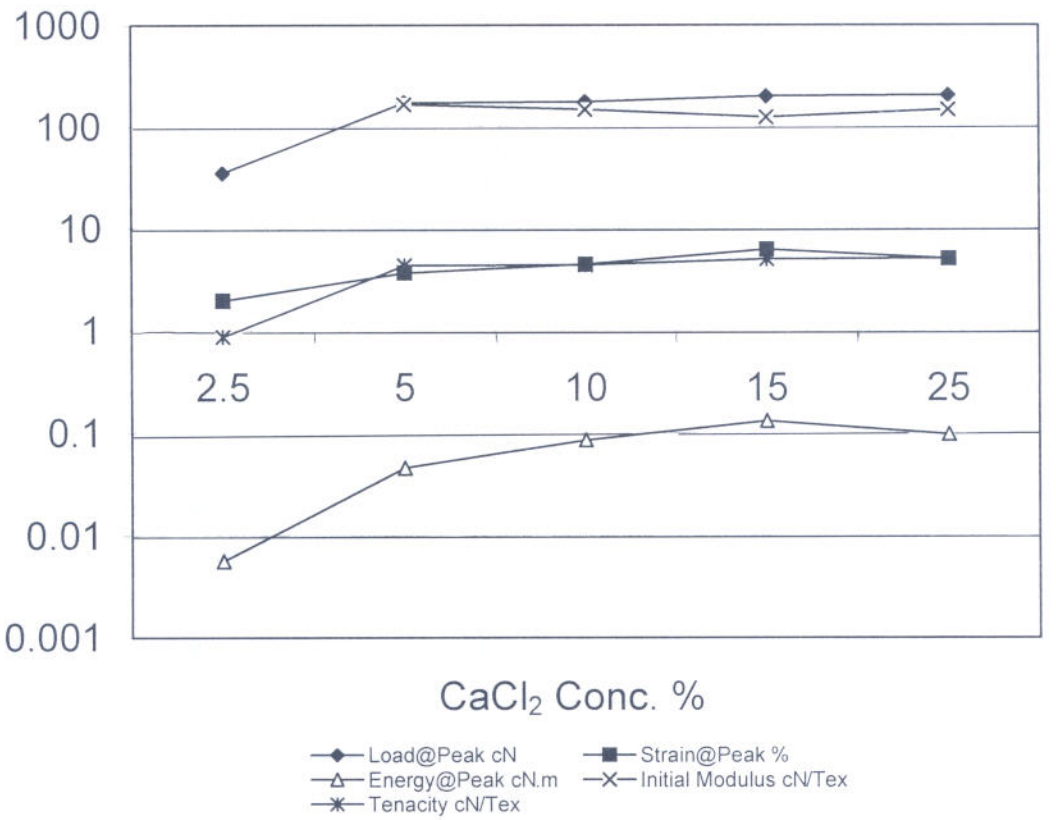
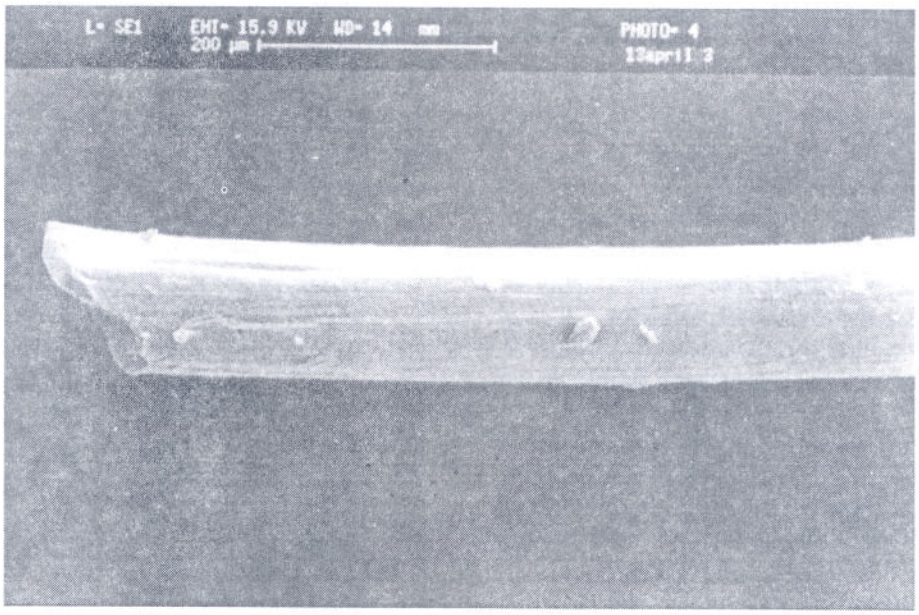


Figure 5. Effect of $CaCl_2$ concentration in the coagulation bath on the mechanical properties of fiber.

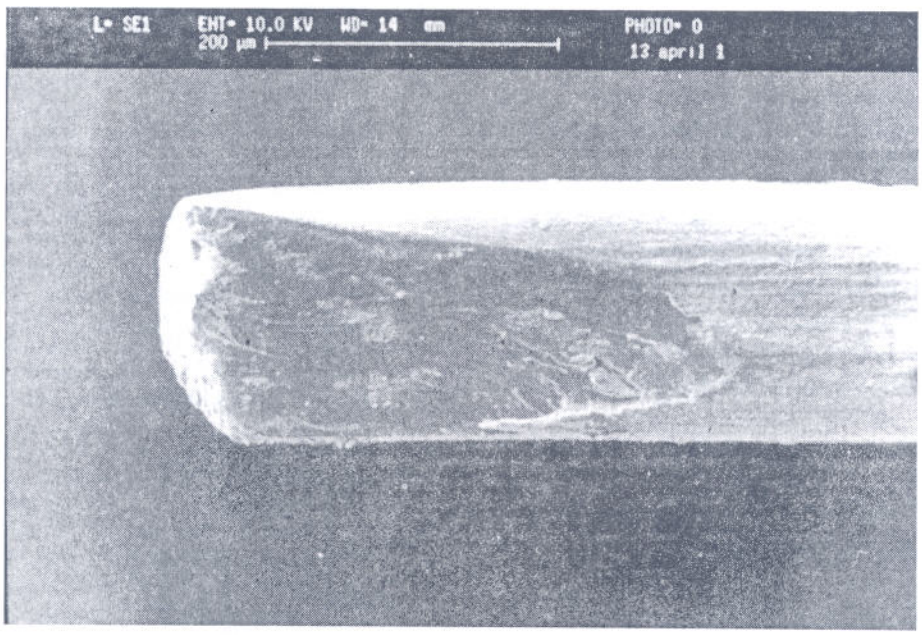
time) for reaching to the maximum effect of alkaline modification. Through the period, the gel like state of the GT solution converted to a viscose state and its color changed from cloudy to bright yellow.

Prolonged ageing time (or high concentration of alkaline) may also cause conformational and/or structural changes on GT. As it is shown in figure 2, with more ageing time, the viscosity of the solution fell down and the color of solution turned opaque gradually.

Some dopes with GT concentration less than 6 %(w/w) were prepared. These solutions were not capable of producing acceptable fibers, due to the



(a)



(b)

Figure 6. SEM micrographs of GT fibers: (a) The surface view, (b) The end view of fiber.

improper viscosities, and the results considered inappropriate to be mentioned here. The 6% GT solution showed relatively acceptable results. With increasing concentration of GT to 8% and 10%, according to Figure 3, some mechanical properties of resultant fibers, such as strain and energy, improved but their processability decreased due to the presence of a gel like behavior in the solutions. The effect of ripening time on resultant fibers is shown in Figure 4. The changes are similar to

Figure 2 in which the mechanical properties of ultimate fibers depended on the initial viscosity of treated GT dope.

Two other important factors in wet spinning are the kind and concentration of coagulant agent, which plays an important role in the quality of produced fibers. Here, only the effect of $CaCl_2$ as coagulant agent was investigated. According to Figure 5, most of the mechanical properties of fibers were improved with increasing $CaCl_2$ concentration solution, but at higher concentrations of $CaCl_2$ (beyond 10%), the GT fibers to some extent were dissolved in the coagulation bath just after they partly consolidated. It can be as a result of increasing the concentration of the salt ($CaCl_2$).

Another determining factor in the quality of produced fibers is the washing bath condition. The effect of washing condition on the mechanical properties of the produced fibers is shown in table I. It is interesting that at acidic pH some of the mechanical properties of final fibers were improved. It should be noted that the produced fibers were brighter in color.

Scanning Electron Microscopy (SEM) was used to obtain micrographs of GT fibers. Fig 6(a) shows the surface of fiber produced by injecting GT into CaCl2 coagulation bath that is smooth and uniform. Fig 6(b) presents the end view of the fiber, which is showing the core of it. Some non-soluble GT also seems to be trapped inside the fiber.

4. CONCLUSION

This work was concentrated on spinability of GT and the evaluation of spinning condition to some extent. Although the primitive fibers produced by injecting the solution into the coagulation bath using a syringe, they present to some extent acceptable quality as expected. The results of mechanical testing and micrographs show the spinability of GT, though further works is being carried out to investigate parameters for improving the quality of these fibers and will be published in due course.

5. REFERENCES

1. Whistler, R. L., and Smart, C. L., "Polysaccharide Chemistry", Academic Press Inc., New York, (1953), 312-314.
2. Anderson, D. M. W. and Grant, D. A. D., "Chemical Composition of the Nitrogen Containing Gum Tragacanth Exudates from Asiatic Astragalus Species Grown in North America", ***Food Hydrocolloids***, Vol. 3, No. 3, (1989), 217-223.
3. Anderson, D. M. W. and Bridgeman, M. M. E., "The Composition of the Proteinaceous Polysaccharides Exuded by Astragalus Microcephalus, A. Gummifer and A. Kurdius- the Source of Turkish Gum", ***Tragacanth Phytochemistry***, Vol. 24, No. 10, (1985), 2301-2304.
4. Davidson, R. L., "Handbook of Water-Soluble Gums and Resins", (1980), Chapter 11.
5. Stauffer, K. L. and Andon, S. A., "Comparison of the Functional Properties of Two Grades of Gum Tragacanth", Food technology, (1975), 46-51.
6. Kiumarsi, A., "The Gum Tragacanth from Iranian Astragalus Microcephalus", PhD Thesis Entitled University of Otago, Newzland, (1998), 2-20.
7. Anderson, D. M. W. and Grant, D. A. D., "The Chemical Characterization of some Astragalus Gum Exudates", Food Hydrocolloids, Vol. 2, No. 5, (1989), 417-423.
8. Aspinal, G. O. and Baillie, J., "Gum Tragacanth, Part I: Fractionation of the Gum and the Structure of Tragacanthic Acid", ***J. Chem. Soc.***, (1963), 1702-1714.
9. Yokoyama, A., Srinivasan K. R. and Fogler, S. H., "Stabilization Mechanism of Colloidal Suspensions by Gum Tragacanth: The Influence of pH on Stability", ***J. of Colloid and Interface Science***, Vol. 126, No. 1, (1988), 141-149.
10. Whistler, R. L., "Industrial Gums", 2nd Ed., Chapter XIII, Academic Press, (1973), 289-299.
11. Aspinal, G. O., "The Carbohydrate Chemistry and Biochemistry", 2nd Ed., Vol. IIB, (1982), 415-421.