

# Properties of Henequen Cellulosic Fibers

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## SYNOPSIS

The physical properties, thermal stability, and interaction with liquids of cellulosic fibers isolated from henequen fibers (*Agave fourcroydes*) by an alkaline digestion process were determined. Physical dimensions, apparent length, and diameter of henequen cellulosic fibers were determined by optical microscopy in a random sample of 125 fibers. Other properties such as crystallinity, specific gravity, and cellulose type were also determined. The sorption of several liquids in these cellulosic fibers was measured and correlated to the polar and hydrogen bonding components of the solubility parameters of the liquids. The results show that, in general, henequen cellulosic fibers have properties that are in close agreement with those found in other cellulosic materials from natural sources.

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## INTRODUCTION

Natural hard fibers, such as sisal (*Agave sisalana*) and henequen (*Agave fourcroydes*), are valued mainly for their high strength and stiffness. These fibers are used in applications ranging from twisted cords and coarse threads for ropes to various textile products. However, the use of these hard fibers has been steadily displaced by synthetic fibers that offer some advantages in the same applications. Until now, the use of natural hard fibers have centered only on the fibers as a whole, and the possibility of using the components of these fibers in other applications has not been explored.

In the particular case of henequen fibers (*Agave fourcroydes*), earlier reports show that they are composed by approximately 60% cellulose, 28% hemicelluloses, and 8% ligning by weight,<sup>1</sup> which compares favorably with the 40% cellulose and 27% ligning content of wood, making them a potential source for cellulose obtention. Furthermore, it has been reported that cellulose can be isolated from henequen fibers under mild hydrolisis conditions,<sup>2</sup> to afford a cellulose pulp with at least 96% by weight

of  $\alpha$ -cellulose. Potential alternative uses for cellulose isolated from henequen fibers could range from the preparation of cellulose derivatives to their use as a reinforcement in thermoplastic or thermosetting matrices. The latter use seems attractive, and it has been explored for different types of cellulosic materials obtained mainly from wood,<sup>3-16</sup> and the potential use in thermoplastic matrices for cellulose isolated from henequen fibers has been pointed out in a previous publication.<sup>17</sup>

The potential of cellulosic fibers as a reinforcement for polymers is explained by some studies that report cellulose tensile strength as high as 1 GPa, and an elastic modulus between 15 and 30 GPa for fibers extracted from Kraft pulps. The high modulus of cellulose is easily observed in the resistance to rupture of a sheet of paper. Generally, the elastic modulus of different papers ranges between 15 and 30 GPa, and their tensile strength is around 500 MPa.<sup>4-7</sup> Based on these properties, cellulosic fibers could become an excellent reinforcement element for preparation of polymeric composite materials. An additional advantage is that celulosic fibers, given their elastic nature, are easier to handle than some of the stiffer fibers such as glass fibers by the processing equipment. Because the potential uses of cellulosic fibers isolated from henequen fibers would be determined largely by their properties, it is our interest to characterize the physical properties,

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thermal stability, and the interaction that some reactants have with henequen cellulosic fibers.

## METHODS AND MATERIALS

Henequen cellulosic fibers were extracted from henequen whole fibers by an acid prehydrolysis step followed by an alkaline digestion of the whole fibers. In a typical extraction procedure, 100 g of henequen fibers, chopped to a length of 20 cm, are placed on a 4 L beaker with a 0.4% solution of sulfuric acid (H<sub>2</sub>SO<sub>4</sub> reagent grade). The solution is kept at the boiling point (102°C) for 1 h under mechanical stirring (600 rpm); then, it is cooled down to room temperature and the fibers thoroughly washed with distilled water. At this point, the fibers are treated with a sodium hypochlorite, NaClO, solution (35% v/v) at 30°C for 1 h or until the pH reaches between 9–9.2. The NaClO used is a soda bleaching solution with 114 g/L of available chlorine. The fiber is filtered and washed with distilled water. The next step corresponds to cellulose isolation. The fiber is treated with a sodium hydroxide, NaOH, solution (20% w/w) with mechanical stirring at room temperature for 1 h. The cellulosic fibers obtained are filtered and washed with distilled water. A final bleaching step was given with a NaClO solution (5% v/v) and mechanical stirring for 1 h at room temperature and through washing with water. The fibers are extended in large plates and left to dry at room temperature for 24 h. Further drying is attained in a convection oven for 6 h at 105°C. The conditions selected for cellulose extraction from henequen whole fibers are based on a method developed in this laboratory and reported elsewhere.<sup>2</sup>

The physical characteristics of henequen cellulosic fibers such as apparent length and diameter of the fibers were determined by optical microscopy using an American Optical microscope with a calibrated eye piece. Specific gravity measurements were carried out according to the ASTM-D792 standard method, with the fibers suspended in benzene.

Glycerol, 1,2 propylene glycol, 1,4 butanediol, ethylene glycol, diethylene glycol, styrene, and methyl methacrylate, obtained from Aldrich Chemical Co., retention by henequen cellulosic fibers were performed following a method described by Kulrhesthra et al.<sup>18</sup> For these measurements, around 0.5 g of henequen cellulosic fibers dried at 105°C for 1 h were immersed in the test liquid for 24 h. Afterwards, the cellulosic fibers were transferred to centrifugation tubes with a false bottom and were centrifugated at 1000 rpm (200 G) at 4°C for 1 h or

until constant weight was attained. The results are reported as the specific volume of liquid retained by the fibers,  $V_r$ , after centrifugation that was calculated by the following expression:

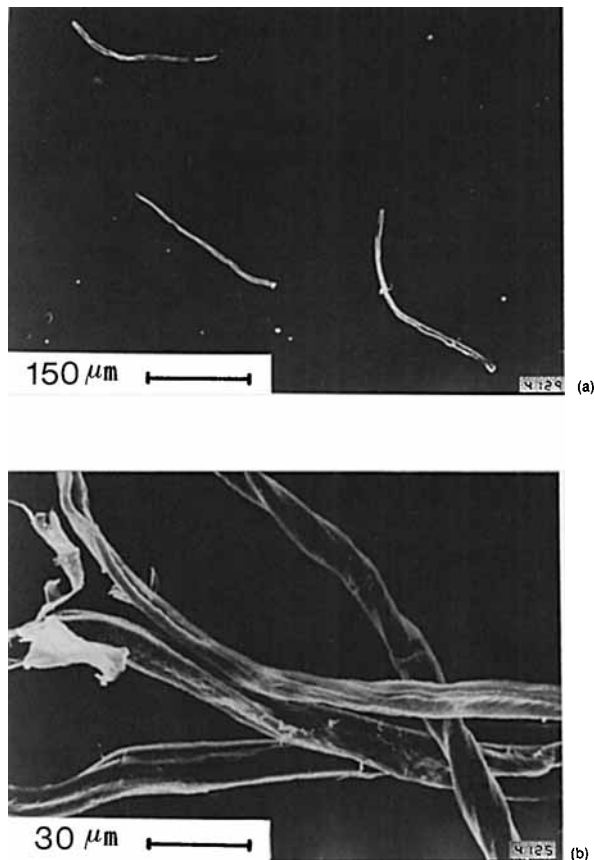
$$V_r = \frac{w_{cl} - w_c}{w_c \rho_l} \quad (1)$$

where  $w_{cl}$  is the weight of the fiber embeded in liquid after centrifugation,  $w_c$  is the weight of the dry fiber, and  $\rho_l$  is the density of the test liquid.

The thermal degradation of henequen cellulosic fibers under air atmosphere was followed by thermal gravimetric analysis in a Perkin-Elmer Inc. TGA-2 instrument, using a scan rate of 10°C/min, between 40 and 700°C. The degradation trace obtained was compared with commercial cotton and microcrystalline cellulose samples, Polycel (Polysciences Inc.). Wide-angle x-ray diffraction measurements (WAXS) of henequen cellulosic fibers were obtained using CuK $\alpha$  radiation in a Philips PW 1140 diffractometer between 4 and 60° in samples compressed manually up to the point where they presented an acceptable surface for x-ray measurements in order to minimize fiber damage. Specimen thickness for the WAXS determination was 0.8 mm.

## PHYSICAL PROPERTIES

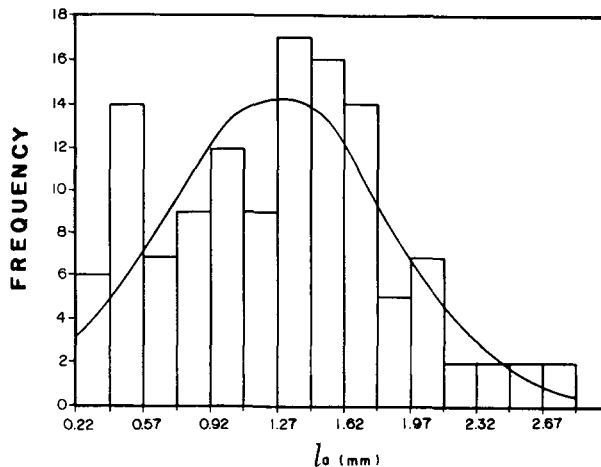
Henequen cellulosic fibers isolated from henequen fibers appear in white fibrous flakes with a slightly grayish color. It has been reported<sup>2</sup> that they consist mainly of  $\alpha$ -cellulose (97–98%) with traces of hemicellulose and lignin (no more than 2–3%). Figure 1(a) and (b) show micrographs at different magnifications of henequen cellulosic fibers after suspension in diethylene glycol. From these micrographs it is apparent that henequen cellulosic fibres are formed by intertwined fibrils twisted in a helical conformation around a central axis of symmetry. This kind of conformation makes it difficult to determine the length and diameter of the fibers because they are not straight and homogeneous, as compared to more rigid synthetic fibers such as glass or carbon; therefore, it was decided to characterize their physical dimensions using apparent length and diameter measurements. The apparent length, as used here, was taken as the end-to-end distance observed in a single henequen cellulosic fiber. Measurements of apparent length were taken by optical microscopy in a random sampling of 125 fibers suspended in diethylene glycol. A distribution of the apparent lengths found is shown in Figure 2. The lengths fol-



**Figure 1** (a) Electron micrograph of henequen cellulosic fibers isolated from henequen whole fibers, suspended in diethylene glycol. (b) Larger magnification of henequen cellulosic fibers where the location of fibrils around a central axis of symmetry can be appreciated in henequen cellulosic fibers.

low a normal distribution the average apparent length,  $l_a$ , is 1.3 mm, and the standard deviation for  $l_a$  is 0.6 mm. The apparent diameters,  $d_a$ , are reported as the observed diameter of the fiber in a random sampling of 45 fibers suspended in diethylene glycol. Figure 3 shows the distribution of apparent diameters observed in henequen cellulosic fibers. The average apparent diameter,  $d_a$ , was 15  $\mu\text{m}$ , with the distribution skewed towards values between 9 and 15  $\mu\text{m}$ . Within the limitations imposed by the nature of the fibers and the difficulty to homogenize them, the average apparent length,  $l_a$ , and diameter,  $d_a$ , found and their distributions are a measure of the physical dimensions of henequen cellulosic fibers.

The specific gravity of henequen cellulosic fibers in benzene was found to be 1.57. Daniel<sup>19</sup> reported a specific gravity value for cellulosic fibers in benzene that is in good agreement with the one reported here.

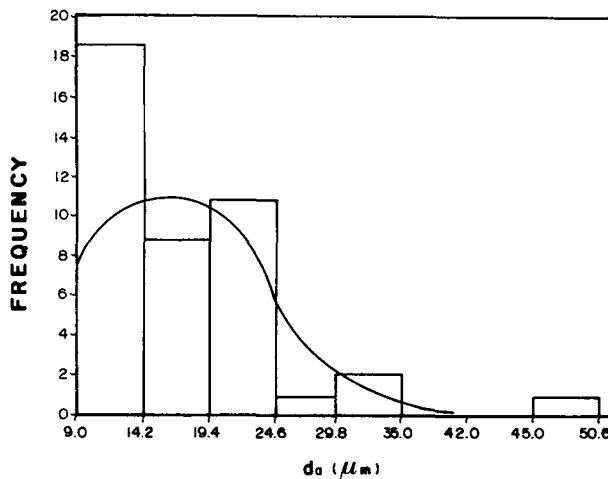


**Figure 2** Apparent length distribution of henequen cellulosic fibers.

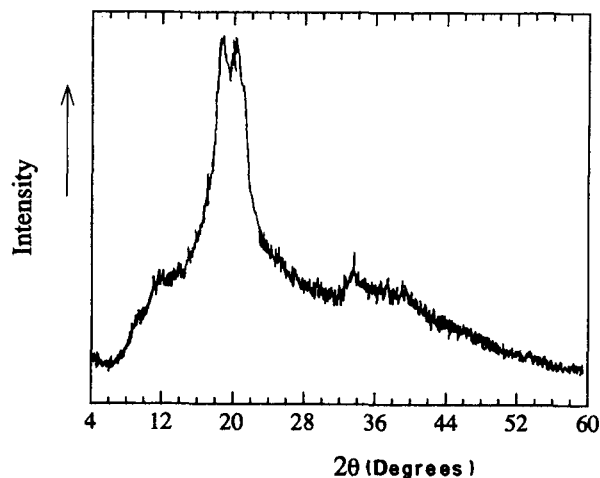
Our value is also similar to the density reported for wood and cotton cellulosic fibers, 1.53 ( $\text{g}/\text{cm}^3$ ).<sup>20</sup>

### X-RAY DIFFRACTION

A typical wide-angle x-ray scattering (WAXS) trace of henequen cellulosic fibers is shown in Figure 4. There is a double peak with maxima at 19 and 21°  $2\theta$  and two small broad peaks at 12 and 35°  $2\theta$ . This x-ray trace is similar to that reported by Patil et al.<sup>21</sup> for a cotton ramie sample hydrolyzed in hydrochloric acid and swollen in sodium hydroxide. A similar pattern is also reported by Moharram et al.<sup>22</sup> for a cellulose sample treated with sodium hydroxide. The similarity in the traces must arise from the



**Figure 3** Apparent diameter distribution of henequen cellulosic fibers.



**Figure 4** Wide-angle x-ray diffraction trace of henequen cellulosic fibers.

method of extraction of the henequen cellulosic fibers, which involves an acid prehydrolysis step followed by an alkaline digestion with sodium hydroxide. These x-ray diffraction traces are assigned to cellulose type (II); therefore, henequen cellulosic fibers can be classified as type (II) cellulose fibers. The crystalline fraction of henequen cellulosic fibers was calculated by the difference in the partial areas corresponding to the amorphous and crystalline portions of the trace obtained. Using this method, the crystalline fraction found is 0.40. This value is the same as the average reported for regenerated cellulose.<sup>19</sup> Table I summarizes all of the above results. In general, henequen cellulosic fibers have physical characteristics that are in close agreement with those reported for other cellulosic fibers obtained from natural sources, such as cotton and wood.

### THERMAL DEGRADATION

A typical thermal degradation pattern under an air atmosphere of henequen cellulosic fibers is shown in Figure 5. In the same figure, the degradation trace for henequen cellulose is compared with those of commercial cotton and microcrystalline cellulose (Polycel). The initial similarity in the degradation traces of the three cellulosic samples is obvious. All of them show a small weight decrease, about 8%, between 40 and 100°C. We attribute this weight decrease to the release of moisture from the samples, because they were not dried prior to the test. The estimated moisture content is slightly larger for henequen cellulosic fibers, but it is in agreement with the moisture reported previously for henequen cel-

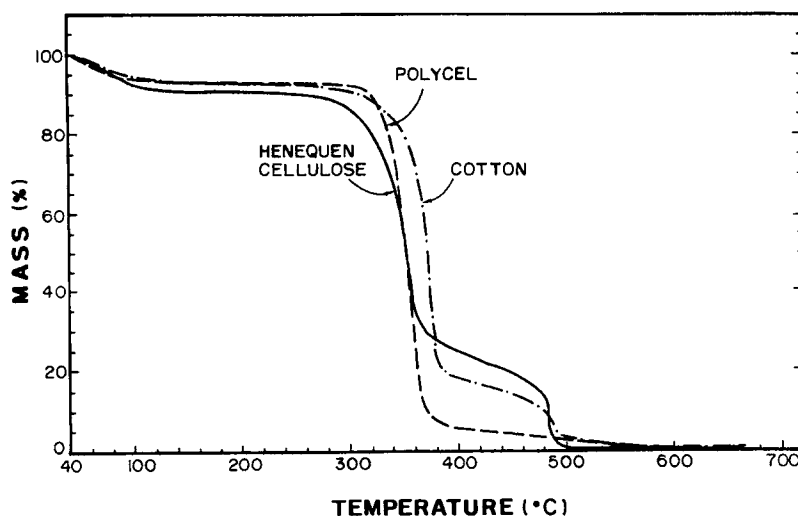
lulosic fibers.<sup>2</sup> A sharp drop in weight at 320°C is apparent for all three cellulosic samples. We believe that this is the onset of cellulose decomposition.<sup>23,24</sup> At this point, microcrystalline cellulose, Polycel, shows a sharp weight loss, approximately 80%, while cotton and henequen cellulosic fibers have a less pronounced weight loss, 70% for cotton and 60% for henequen cellulosic fibers, with a plateau between 380 and 480°C. Thermal degradation studies of cellulose attribute the drop at 320°C to dehydration and depolymerization of cellulose followed by evaporation of the volatile products of combustion.<sup>23,24</sup> The plateau observed between 380 and 480°C, which is more visible in cotton and henequen cellulosic fibers, is attributed to oxidation and burning of the high molecular weight charred residues.<sup>23,24</sup> All three cellulosic samples decompose totally at around 500°C. Therefore, the thermal stability of henequen cellulosic fibers is in close agreement with that observed in other natural cellulosic fibers such as cotton.

### SWELLING AND LIQUID SORPTION

Cellulosic fibers are difficult to dissolve due to their high crystallinity; however, they tend to retain liquids in the interfibrillar space. It is useful to understand liquid retention by cellulosic fibers because of their use as reinforcement in various matrices, such as unsaturated polyesters<sup>25</sup> or polyurethanes,<sup>26</sup> that involves fiber-liquid interactions during the process of incorporation of the components. The retention process can be attributed to the sorption of the liquid in the fibrils. The effect of this sorption is the retention of the liquid by the cellulosic fibers and swelling, possibly increasing the interfibrillar distance. The degree of sorption and swelling obtained with different liquids is determined by the ability of the liquid to interact with the cellulosic fibers. One would expect that liquids with a high degree of hydrogen bonding (H-bonding), such as water or alcohols, tend to swell the fiber more and are retained in larger amounts, than those with a lower degree of H-bonding. Specific volume retention values,  $V_r$ , of a selected number of liquids in henequen cellulosic

**Table I** Physical Characteristics of Henequen Cellulosic Fibers

Apparent length, $l_a$ (mm)	1.3
Apparent diameter, $d_a$ ( $\mu\text{m}$ )	15.0
Specific gravity in benzene	1.57
Crystalline fraction (from x-ray)	0.40



**Figure 5** Thermal degradation of henequen cellulosic fibers under an air atmosphere compared to commercial cotton cellulose and microcrystalline cellulose (Polycel).

fibers are reported in Table II. The liquids were originally selected based on their H-bonding capacity, as defined by the H-bonding component of their solubility parameter,  $\delta_h$ , given in Table II. The  $V_r$  values observed are closely correlated to the H-bonding capacity of the liquid. Polyhydric alcohols, such as glycerol, that have the largest  $\delta_h$ , show a larger specific volume retention value,  $V_r$ , by henequen cellulosic fibers, while styrene and methyl methacrylate, with lower values of  $\delta_h$ , have lower  $V_r$  values under the same conditions.

The sorption of different liquid reactants in paper cellulosic fibers has been reported before by Robertson,<sup>27</sup> who used a methoxylation reaction on the fibers after swelling by different liquids, as a measure of the ability of the liquid to penetrate and swell the

lumen, that is, the empty space of the fibers. Table III shows some values reported by Robertson for several liquids and their respective solubility parameters. An interesting trend is observed by plotting the specific retention volumes obtained in our experiments as a function of the H-bonding component of the solubility parameter of the respective liquids. Figure 6 clearly shows that liquids with little or no H-bonding capacity penetrate the fiber poorly. As the H-bonding capacity of the liquid increases, the retention volume goes up significantly. The largest increase in retention volume occurs between about 20 to 25 MPa<sup>1/2</sup>, which is in the range for the  $\delta_h$  reported for the cellulose used in chromatographic columns,<sup>28</sup> although recently reported  $\delta_h$  values for microcrystalline cellulose are slightly larger than 25

**Table II** Solubility Parameters and Volumetric Uptake of Different Organic Liquids After Centrifugation by Henequen Cellulosic Fibers

Reactant	$V_r$ (cm <sup>3</sup> /g of Fiber)	Solubility Parameter Components <sup>a</sup> (MPa) <sup>1/2</sup>			
		$\delta_d$	$\delta_p$	$\delta_h$	$\delta_t$
1,2 propylene glycol	0.75	11.8	13.3	25.0	30.7
Glycerol	1.32	9.3	15.4	31.4	36.2
Diethylene glycol	0.99	12.4	12.3	23.3	29.1
Ethylene glycol	1.33	10.1	15.1	29.8	34.9
1,4 Butanediol	1.36	15.3	13.6	27.0	33.7
Styrene	0.34	16.8	9.1	0.0	19.1
Methyl methacrylate	0.36	13.5	10.1	8.5	18.9

<sup>a</sup> Values from ref. 28. Meaning of subindices in  $\delta_t$  is as follows:  $d$  = dispersive,  $p$  = polar,  $h$  = hydrogen bonding, and  $t$  = total from  $\delta_t = \sqrt{\delta_d^2 + \delta_p^2 + \delta_h^2}$ .

**Table III Solubility Parameters and Methoxyl Content<sup>a</sup> of Different Liquid Reactants in Paper Cellulose**

Reactant	Methoxyl (%)	Solubility Parameter Components <sup>b</sup> (MPa) <sup>1/2</sup>			
		$\delta_d$	$\delta_p$	$\delta_h$	$\delta_t$
Water	8.15	12.2	22.8	40.4	48.0
Ethylene glycol	8.11	10.1	15.1	29.8	34.9
Diethylene glycol	7.02	12.4	12.3	23.3	29.1
Methanol	7.39	11.6	13.0	24.0	29.7
Ethanol	3.00	12.6	11.2	20.0	26.1
Butanol	0.54	15.0	10.0	15.4	23.7
Chloroform	0.72	11.0	13.0	6.3	18.1
Ethylene dichloride	0.89	14.2	11.2	9.1	20.2
Benzene	0.49	16.1	8.6	4.1	18.7

<sup>a</sup> Values of methoxylation taken from ref. 27.

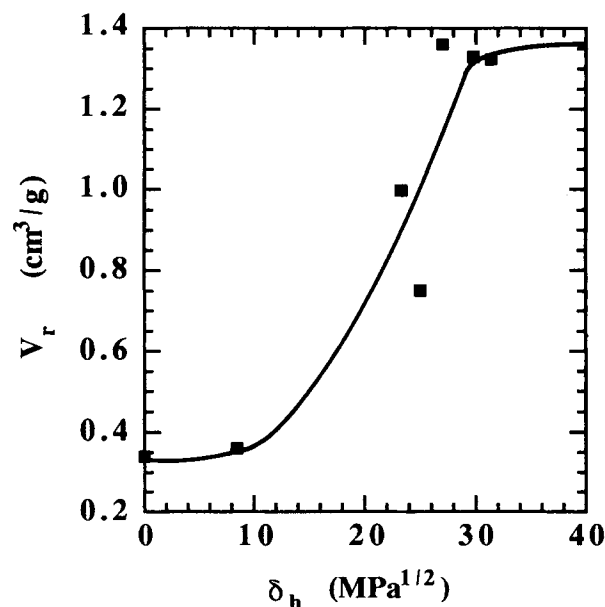
<sup>b</sup> Values from ref. 28.

MPa<sup>1/2</sup>.<sup>29</sup> Past this point, a saturation of the fibers appears to occur with the consequent plateau region on the plot.

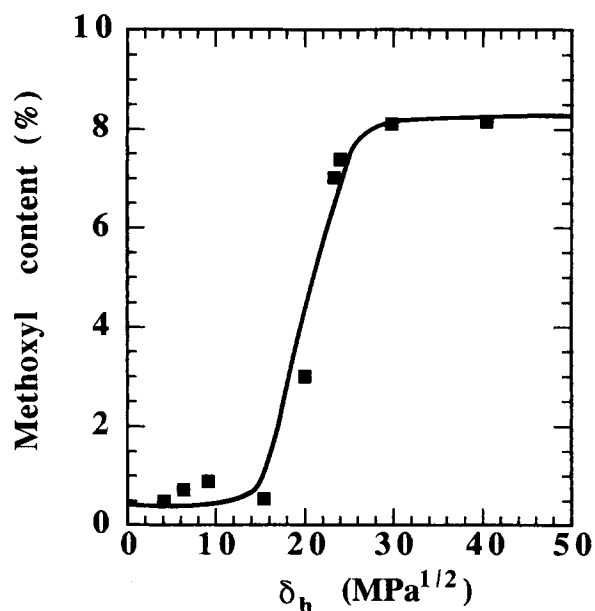
Carrying out a similar exercise as above in Robertson results, where the weight percent methoxyl is a measure of liquid retention, produces the results shown in Figure 7. This plot is strikingly similar to Figure 6. The same sigmoidal behavior is observed with a steep change in liquid retention when  $\delta_h$  for

the liquid becomes about equal to that of cellulose, and a subsequent plateau region at higher  $\delta_h$  values.

It is also fair to state that the polar component of the solubility parameter,  $\delta_p$ , is also useful as a semiquantitative tool to predict liquid retention by cellulosic fibers, as seen in Figures 8 and 9. In both cases,  $V_r$  and the percent of methoxylation follow a similar pattern as a function of  $\delta_p$  as in the previous



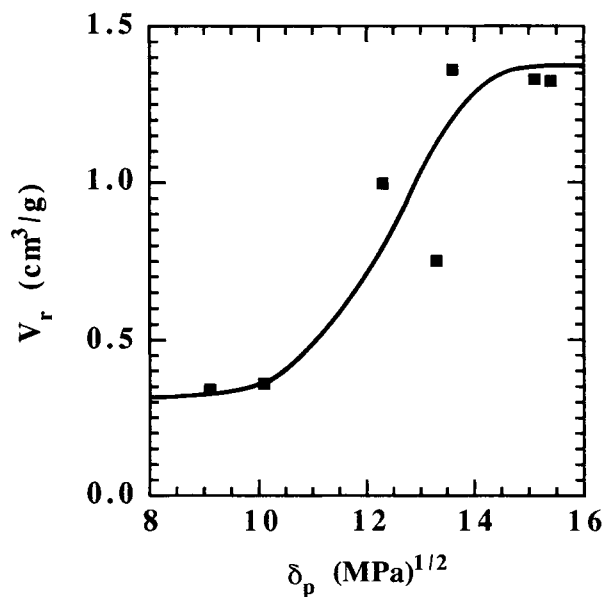
**Figure 6** Relationship between specific volume of liquid retained by henequen cellulosic fibers and H-bonding component of the solubility parameter of the liquid. (Values correspond to the liquids listed in Table II.)



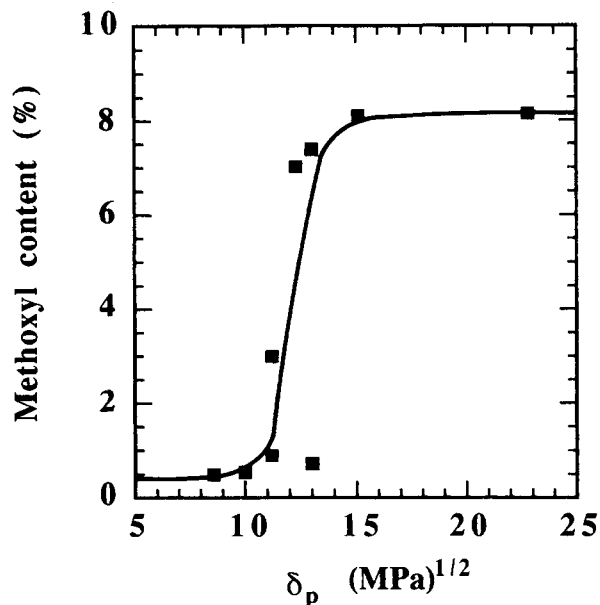
**Figure 7** Methoxyl content in paper cellulose after swelling in different liquids compared to the liquid's H-bonding component of the solubility parameter. Methoxylation values from ref. 27. (Values correspond to the liquids listed in Table III.)

plots where  $\delta_h$  is used as the dependent variable. Apparently, there is a threshold value about 11 to 12 MPa<sup>1/2</sup> for  $\delta_p$  of the liquid to obtain a significant degree of retention by the fiber. This apparent threshold compares with the  $\delta_p$  for cellulose of about 13 MPa<sup>1/2</sup>.<sup>29</sup> The only exception for this trend appears to be chloroform.

We can, therefore, conclude that large polarity and hydrogen bonding character in the liquid favor its sorption by cellulosic fibers. In our previous observation, we speculated that 1,4 butanediol sorption into the fibers was a significant cause for the slower reaction kinetics with an isocyanate, when such a reaction is carried out in the presence of these fibers.<sup>26</sup> The present results confirm that this notion is correct. It is clear, nonetheless, that the ability of the liquid to swell cellulosic fibers shows a direct correlation with the H-bonding solubility parameter component,  $\delta_h$ . A sharp increase in liquid retention is seen when this parameter reaches around 20 MPa<sup>1/2</sup>, with a subsequent constant volume retention or plateau after 25 MPa<sup>1/2</sup>, regardless of the increase in  $\delta_h$ . This result could be used as a predictive tool for the expected amount of swelling or liquid uptake of cellulosic fibers based on  $\delta_h$  values already determined for a large number of liquids or calculated using group contribution methods.<sup>28</sup>



**Figure 8** Relationship between specific volume of liquid retained by henequen cellulosic fibers and polar component of the solubility parameter of the liquid. (Values correspond to the liquids listed in Table II.)



**Figure 9** Methoxyl content in paper cellulose after swelling in different liquids compared to the polar component of the solubility parameter. Methoxylation values from ref. 27. (Values correspond to the liquids listed in Table III.)

### CONCLUSIONS

The physical properties, thermal stability, and the interaction of some liquid reactants with henequen cellulosic fibers obtained from the alkaline digestion of henequen whole fibers were determined. It was found that henequen cellulosic fibers have an average apparent length and apparent diameter of 1.3 mm and 15  $\mu$ m, respectively, with standard deviations of 0.6 mm and 1.0  $\mu$ m. The fiber lengths show an approximately normal distribution, and the diameters show skewness towards small values.

Based on the wide-angle x-ray diffraction trace obtained from henequen cellulosic fibers, their characteristics classify them as cellulose type (II) substrates. Their crystalline fraction was found to be approximately 0.40. The specific gravity of henequen cellulosic fibers in benzene determined to be 1.57, which is the same as for other values reported in the literature for cellulosic fibers.

The onset of thermal degradation for henequen cellulosic fibers in an air atmosphere, under dynamic heating conditions, was found to occur at 320°C. This transition is followed by a plateau between 380 and 430°C, which was assigned to the decomposition of residual material after the initial depolymerization of the cellulose at 320°C, based on similar degradation studies reported in the literature. The

thermal degradation behavior of henequen cellulosic fibers shows only minor differences when compared with that for commercial cotton cellulose.

The interaction between different liquids and henequen cellulosic fibers was also tested. The results of the specific volume of different liquids retained by the fibers were compared to the H-bonding solubility parameter component,  $\delta_h$ , for each liquid. Liquid retention in the fibers increases sharply for  $\delta_h$  values between 20 and 25 MPa<sup>1/2</sup> and levels off after this point. The polarity of the liquid, characterized here by the polar component of the solubility parameter,  $\delta_p$ , also plays an important role in liquid retention by the fibers. This behavior is similar to that observed in other cellulosic materials.

Overall, the present results show that henequen cellulosic fibers exhibit characteristics that are in close agreement with those found in other cellulosic fibers obtained from natural sources.

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