# TEXTILE TOPICS

## INTERNATIONAL TEXTILE CENTER

TEXAS TECH UNIVERSITY • LUBBOCK, TEXAS / USA

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## SPRING SESSION OF TEXAS INTERNATIONAL COTTON SCHOOL HELD

The eleventh session of the Texas International Cotton School was conducted at the International Textile Center from April 3 through April 14, 1995. Attending were thirteen students from five countries (shown in Exhibit 1).

Outstanding experts from all over the US gave lectures and had personal discussions with the students. Featured speakers for the April school included experts from: New York Cotton Exchange, Lubbock Cotton Exchange, International Cotton Advisory Committee, Schlafhorst Inc., Zellweger Uster, NationsBank, Texas Agricultural Experiment Station, and USDA's Agricultural Marketing Service and Agricultural Research Station.

The next session of the Texas International Cotton School is scheduled for October 2 through October 13, 1995. Information on the school may be obtained by contacting Ms. Mandy Howell at the Lubbock Cotton Exchange, 1517 Texas Avenue, Lubbock, Texas 79401; telephone 806/763-4646; FAX 806/763-8647.

Exhibit 1: Class XI of Texas International Cotton School

Front Row (left to right): **Shoji Naruse**, Kondo Cotton Spinning Co., LTD, Japan, **Mohammad Sayeed Rahim Ullah**, Padma Textile Mills, LTD., Bangladesh, **Brenda Wynn**, TICS Assistant Coordinator, **Mandy Howell**, TICS Coordinator, **Henry Spikes,** Texas Dept. of Corrections, Texas and **George Edens,** Edens Farms, North Carolina;
Middle Row (left to right): **Eric Nelson**, Weil Brothers Cotton, Inc., Texas, **Richard June**, Schlafhorst Inc., North Carolina, **Oscar Guerrero Garibay**, Industrial Lajat, Mexico, **Philip Bogel III**, Bogel Cotton Company, Texas, **Gary Chestnutt**, Chestnutt Cotton Company, Texas and **Seiji Tsuzuke**, TNS Mills, Inc., South Carolina;
Back Row (left to right): **Tony D'Chuna**, Texas Dept. of Corrections, Texas, **William Upchurch, Jr.**, North Carolina Dept. of Agriculture, North Carolina, and **Ralph Knecht**, W. Schlafhorst AG & Co, Germany.

# A SIMPLE METHOD FOR PREPARATION OF HIGHLY ABSORBENT COTTON FIBERS

## Introduction

Highly absorbent cellulose products have been made by grafting acrylonitrile on wood pulp, followed by alkaline hydrolysis of pendant nitrile group (1) or grafting acrylic acid (5). Carboxymethylation of cellulose by treating alkali cellulose with monochloroacetic acid is probably the most important method for preparation of superabsorbent products (6). These processes mostly involve nontextile fibrous products.

In the previous two issues of Textile Topics, we examined the water retention of cotton fibers with different micronaire values (Vol. 23:1), then measured the impact on water retention by these fibers from graft polymerization of acrylamide in the presence of ceric ammonium nitrate as a free radical initiator (Vol. 23:2). It was shown that after alkaline hydrolysis, cotton fibers with low micronaire exhibited a higher rate of water retention than did high micronaire cotton.

The previous study of enhanced water retention for cotton (Vol. 23:2) used a **two-step method**, with acrylamide being used to form a graft polymer within the growth layers of the fibers, then using sodium hydroxide to convert the amido groups of the polyacrylamide into carboxyl groups. This paper gives results on water retention and water absorption of cotton fibers using a **one-step method** of carboxyethylation using acrylamide and sodium hydroxide. Furthermore, the study reported here utilized both **staple cotton fibers** and **cotton from gin waste** (gin motes).

The staple cotton fibers used in this study were a subset of those used in the previous reports; micronaire values were 2.7, 4.4 and 7.0 (as measured on the Shirley FMT3 Fineness Maturity Tester). The two cottons with the lower values were American Upland varieties, while the 7.0 micronaire was an Asiatic cotton variety. Methodology and results for these staple fibers are given in the next section, followed by a brief report on the gin motes.

## Staple Fibers

Fibers were scoured with 4% sodium hydroxide on the weight of the fibers; they were boiled for 90 minutes, in the absence of air, and with a 1:20 material-to-liquor ratio. Then they were washed repeatedly with deionized water until a neutral pH was obtained. After hydroextracting, the fibers were dried under ambient conditions.

Besides an unmodifited sample to provide an experimental control (call it "Treatment A"), three distinct treatments were applied to the staple fibers:

| Treatment | Acrylamide | Sodium Hydroxide |
|---|---|---|
| A | None | None |
| B | 10% | 10% |
| C | 10% | 15% |
| D | 20% | 20% |

It is noteworthy that the 20% sodium hydroxide solution used in Treatment D is actually a mercerizing strength solution.

### *Preparation of Samples*

- 10 – 20 grams of acrylamide were dissolved in 20 – 30 ml of water and volume was increased to 50 ml by adding water.
- 10 – 20 grams of sodium hydroxide were dissolved in water, cooled at room temperature, then adjusted to a final volume of 50 ml by adding water.
- 10 grams of cotton fibers were immersed in an aqueous solution of acrylamide and kept for 10 minutes.
- The sodium hydroxide solution was added to the beaker containing cotton fibers in acrylamide solution and mixed thoroughly by stirring. The material-to-liquor ratio was 1:20.
- The beaker was covered with a polyethylene sheet and aluminum foil to prevent the escape of ammonia that evolved during the reaction, then kept overnight at room temperature.
- The fiber mass was then filtered under suction and washed with hot and cold water until the filtrate pH was approximately neutral.
- The resulting fiber mass was then washed with alcohol, filtered, and dried in air or in a flash evaporator.

### *Carboxyethylation*

In the above procedure, the carboxyl groups were introduced into cotton fibers by wet treatment with acrylamide and sodium hydroxide in an

aqueous medium, as developed by Reinhardt and Bruno (3). In this procedure the relationships among reactants, time, temperature, and reaction medium control the amount and ratio of carboxyethyl and carbamoylethyl ether substituents. Carboxyl groups were estimated by a back-titration technique used by Reinhardt et al. (4) and nitrogen was determined by the Kjehdahl technique of nitrogen analysis.

Initially, acrylamide reacts with the hydroxyl group of cellulose via conjugate addition, which introduces carbamoylethyl groups. Then hydrolysis of amido groups proceeds in the presence of sodium hydroxide with liberation of ammonia to produce the sodium salt of carboxyethylated cotton.

Chemical analysis of the unmodified fibers (Treatment A) indicated that carboxyl content of the scoured cotton varied form 0.06% to 0.14% (Exhibit 2). There was not a significant difference in carboxyl contents of the 2.7 and 4.4 micronaire cottons, but the level was about twice as high for the 7.0 micronaire cotton. With Treatment B, the carboxyl contents increased to over 3% in the two lower micronaire cottons and to over 4% in the 7.0 micronaire cotton. With Treatment C, the carboxyl levels rose to between 5% and 6% for all the micronaire groups, then to between 7% and 8% with Treatment D. Therefore, significant differences among carboxyl contents of the alternate micronaire levels disappeared with higher concentrations of treating solutions.

Exhibit 2: Carboxyl Content of Cotton Fibers

| | Micronaire Readings | | |
|---|---|---|---|
| Treatment | 2.7 | 4.4 | 7.0 |
| A | 0.07% | 0.06% | 0.14% |
| B | 3.36% | 3.19% | 4.27% |
| C | 5.95% | 5.39% | 5.72% |
| D | 7.30% | 7.87% | 7.57% |

*Centrifuge Method of Water Retention*

For Treatments A, B and C, water retention of the cotton fibers was determined by the centrifuge method as per ASTM Standard Test Method D 2402-78. For Treatment D, the mass of fibers became so swollen that the following modified procedure was used: The mass of fibers was centrifuged for one hour without the perforated disk in the centrifuge tube. The supernatant liquid was decanted, and the fiber mass which settled at the bottom of the centrifuge tube was placed over a perforated buchner funnel to remove any remaining liquid. The wet fiber mass was placed in a weighing bottle, weighed, dried in the oven at 110° C, then re-weighed. The weight of the water retained by the fiber mass was expressed as a percentage of the oven-dried weight of the fibers.

As expected, water retention of the unmodified staple fibers decreased with an increase in micronaire, going from a high of 65% to a low of 50% (Exhibit 3). After modification with 10% acrylamide and 10% NaOH (Treatment B) the water retention of the fibers increased to a 300% – 400% level. Those treated with 10% acrylamide and 15% sodium hydroxide (Treatment C) gave water retention readings in excess of 600%. Finally, the treatment of cotton fibers with 20% acrylamide and 20% sodium hydroxide (Treatment D) resulted in the dramatic increase in the water retention levels exceeded 5000%.

Exhibit 3: Water Retention by Cotton Fibers

| | Micronaire Readings | | |
|---|---|---|---|
| Treatment | 2.7 | 4.4 | 7.0 |
| A | 65 | 55 | 50 |
| B | 414 | 323 | 321 |
| C | 668 | 618 | 610 |
| D | 5157 | 5336 | 5711 |

Along with the huge increase in water retention with Treatment D came a reversal in the direction of the relationship between micronaire and water retention; the higher micronaire cottons now retained more water (Exhibit 3). This is attributable to the high level of carboxyl groups substituted in the presence of mercerizing strength sodium hydroxide. Since the fibers were first immersed in the acrylamide solution, the latter would have penetrated the existing amorphous regions of the cotton cellulose. Then in the presence of 20% sodium hydroxide, new amorphous regions would occur and more acrylamide would be reacted. Ammonia, evolved in situ from the hydrolysis of amido groups, would also help in opening and swelling the structure of cotton fiber. The very high water retention of the cotton fibers receiving Treatment D could be attributed to increased substitution of carboxyl groups in the swollen state of the cotton fiber.

*Absorption (Filtration Method)*

The cotton fibers were ground to 20 mesh in a Wiley mill. A dry sample (0.1g) was immersed in 50 ml of distilled water for 30 minutes and poured into a 40 mesh sieve of known weight. Water was allowed to drain for one hour, then the wet sample and the screen were weighed. Absorbency was determined as follows:

$$\text{Absorbency (g-water/g-sample)} = \frac{(c-b)-a}{a}$$

where a = weight of the dry sample, b = weight of the sieve, and c = weight of the wet sample plus the weight of the sieve.

Actually, this technique measures water that has been adsorbed (adhered to the surface), absorbed (held in the void spaces), and that retained by hydrogen bonding with hydroxyl groups and carboxyl groups on and within the cotton fibers.

Exhibit 4: Absorbency of Cotton Fibers

| | Micronaire Readings | | |
|---|---|---|---|
| Treatment | 2.7 | 4.4 | 7.0 |
| A | 13.6 | 17.6 | 18.8 |
| B | 25.6 | 19.6 | 24.1 |
| C | 28.8 | 27.1 | 27.5 |
| D | 65.5 | 62.4 | 62.7 |

* Expressed as g water/g sample

Absorbency as measured by the filtration method is directly (i.e., positively) related to micronaire in the unmodified cottons (Treatment A in Exhibit 4). This is due largely to the greater surface area. This direct relationship with micronaire disappears, however, with any of the three treatments used to modify the fibers. Both Treatment B and Treatment C result in substantial increases in water absorbency, from the vicinity of 13 to 19 grams to the vicinity of 20 to 30 grams. Again, the impact of Treatment D was remarkable, with absorbency exceeding 60 grams of water in all three samples.

## Gin Motes

During the ginning process substantial amounts of cotton waste (motes) are generated. Utilization of these motes in the manufacture of absorbent cotton could be cost-effective. For this study, the motes were mechanically separated from the trash using the Shirley Analyzer. Since raw cotton is not wetted by water, and in our process cotton fibers must first be saturated with the acrylamide, a one-percent nonionic wetting agent on the weight of the fiber was incorporated in the aqueous solution of acrylamide. The motes were treated with 20% acrylamide with nonionic wetting agent and 20% sodium hydroxide (Treatment D). Exhibit 5 shows that results obtained compared favorably with those obtained for the staple fibers.

Exhibit 5: Results of Treatment D on Gin Motes*

| | |
|---|---|
| Carboxyl Contents (%) | 6.76 |
| Water Retention (%) | 5973 |
| Absorbency (g water/g sample) | 67.2 |

* With use of wetting agent

## Conclusion

It has been shown on a laboratory scale that cotton fibers, including waste cotton (motes), can be chemically modified using acrylamide and sodium hydroxide in aqueous solution at room temperature to obtain highly absorbent materials. This process is simple in comparison with a two-step process wherein in the first step acrylamide was graft polymerized on cotton fibers using ceric ammonium nitrate as a free radical initiator and in the second step amido groups of the grafted polymer were saponified at high temperature (2).

The chemicals used (acrylamide and sodium hydroxide) are commercially available and economical to use. Based on a per-pound cost of 74 cents for acrylamide and 17 cents for sodium hydroxide, the chemical costs for manufacturing one pound of absorbent material, using 20% acrylamide and 20% sodium hydroxide, are 14.8 cents and 3.4 cents, respectively. It is likely that the chemical cost for manufacturing could be reduced after optimization of the process. Since the wetting agent worked very well on the raw cotton motes, it could also be used on raw staple fibers to avoid the expense of scouring. Furthermore, the ammonia evolved during the process could be recovered for use as a fertilizer.

This research was funded by the Texas Food and Fibers Commission and was conducted by Dr. R. D. Mehta, Head of Finishes/Chemical Research at the ITC.

**References:**

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2. Mehta, R. D., "Effect of micronaire on water retention of cotton fibers," Book of Papers, AATCC International Conference & Exhibition, pp. 324-329, 1994.
3. Reinhardt, R. M. And J. S. Bruno, "Carboxyethylation of cotton by treatment with acrylamide," Journal of Applied Polymer Science, 10, pp. 387-397, 1966.
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6. Young, R. A., "Crosslinked cellulose and cellulose derivatives," Absorbency, (P. K. Chartterjee Ed.), pp. 217-255, Elsevier, NY, 1985.

**NOTICE:** The recycled demin fibers reported in the previous issue of Textile Topics (Vol. 23:2) were processed by Wright Fibers using a process for which a patent is pending.

## BOONE RETIRES . . .

Mrs. Harriet Boone, Information Specialist at the ITC, retired April 30, 1995, ending nineteen years of employment. She was involved in all aspects of communication/public relations; including production of all types of written reports, answering written and telephone inquires, scheduling and conducting tours, and coordinating meetings and seminars. Harriet also managed the ITC library, which she recently helped to remodel. We wish Harriet the best of everything, and we look forward to her continued friendship in the years ahead.

## . . . ALSPAUGH JOINS ITC

Pam Creel Alspaugh has filled the Information Specialist position, effective May 15, 1995. She comes to the ITC after 7 1/2 years in Video Services of Texas Tech University. In 1995 she was awarded a national honor for promising new professionals by Agricultural Communicators in Education (ACE). Pam comes to meet the challenge of expanding the communication/public relations outreach of the ITC, including the execution of written, audio and video projects under contractual arrangements. We are excited by the new initiatives she will bring to our center.

## TEXAS COTTON QUALITY EVALUATED

The fifteen annual fiber and spinning test report on selected commercial varieties of Texas cotton is now available for distribution. Results are provided on representative bales of cotton from all major production areas throughout Texas. For the second consecutive year, combed yarn data are provided for selected Upland cotton varieties with longer staple lengths. For the first time, results on roller-ginned Pima cotton are reported.

To request a complimentary copy of this report, contact Pam Alspaugh, Information Specialist at the ITC.

## TEXTILE SEMINAR SCHEDULED

A two-day seminar of the principals on Fibers and Textiles has been scheduled for June 20–21, 1995. This seminar was designed for Cotton Breeders, Seedsmen and Agricultural Consultants. It will cover spinning test measurements, terminology, test variation, and relationships among fiber properties and processing performance. On completion of the seminar, the participants will be able to interpret all the data contained in a fiber and spinning report.

Further information may be obtained by contacting Charlotte Anderson, Administrative Assistant at the ITC.

## THE SDL MICROMAT PLACED IN SERVICE

A new "micromat" instrument for measuring fiber fineness and maturity was installed in April. It was provided by SDL International Ltd. of Stockport, England. The International Textile Center has been involved in the evaluation of four models of Shirley FMT instruments since the 1970's. Early test results from the "Micromat" indicate that the repeatability is superior to our FMT III instruments, while the test levels are consistent among them.

## ETHRIDGE VISITS MAINLAND CHINA

Dr. Dean Ethridge, Director of the ITC, toured through China for two weeks during March and April. He traveled with a group organized by the Texas Department of Agriculture. Major areas visited were located near Beijing, Xian, Guilin, and Guangzhou. Throughout the tour, he visited with various people in research and in industry regarding cotton, wool, mohair, and cashmere fibers. He also took the advantage of the opportunity to visit the Hong Kong office of Cotton Council International.

## CHIKKODI RECEIVES ACADEMIC HONORS

Mr. Shridhar Chikkodi, Research Associate at the ITC, is also in the Ph.D. program of the College of Human Sciences, Texas Tech University. His outstanding performances has been recognized by:

- the Educational Foundation of Phi Upsilon Omicron, which awarded him the Diamond Anniversary Fellowship for 1995, and
- Texas Tech University, which awarded him the Carl Cox Scholarship for 1995.

Mr. Chikkodi's research focus in his Ph.D. program is on enzyme technology in textile processing. His duties at the ITC fall into two major categories: assisting in all aspects of the Chemical Finishing Laboratory and managing the ITC computer system.

## SPEED VISITS THE TEXAS HILL COUNTRY

Rainey Speed, Manager of Long Staple Processing at the ITC attended the "Seventh Annual Texas Mohair Tour" on April 27 through April 30, 1995. The tour is sponsored annually by the Mohair Council of America. Mr. Speed toured the Texas Hill Country with a diverse group of people connected with the Mohair industry–textile manufacturers, designers, reporters, buyers and mohair producers. In addition to exchanging information and ideas, participants observed ranching, shearing, warehousing, scouring and topmaking.

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## VISITORS

Visitors to the International Textile Center during the past three months include:

- Robin Hurrell, KSR Instruments, Leigh, ENGLAND;
- Carson County 4-H, Panhandle, TX;
- Benny Lanssens, Picanol, Greenville, SC;
- David Goldman, Wellman Industries, New York City, NY;
- Johnie Nehring, G & P Seed Co. Inc., Aquilla, TX;
- Dan Pustejovsky, P&H Seeds, Inc., Hillsboro, TX;
- Dr. G.D. Hefer, Tongaat Cotton, Warmsbath, SOUTH AFRICA;
- Mel Ueckermann, Tongaat Cotton, Warmsbath, SOUTH AFRICA;
- Joe Essick, Zellweger Uster, Atlanta, GA;
- Joe O'Neill, President, New York Cotton Exchange, New York City, NY;
- Ed White, Zellweger Uster, Memphis, TN;
- Keth Henley, Cotton Outlook, Memphis, TN;
- Onnie Sumangil, NationsBank, Dallas, TX;
- Terry Townsend, International Cotton Advisory Committee, Washington, D.C.;
- Ed Hughes, USDA-ARS, Mesilla Park, NM;
- Sahail Barlas, Vital, LTD., Sialkat, Pakistan;
- Frank Jones, Lubbock, TX;
- Shelley Harp, Associate Professor, MEDCE, College of Human Sciences, Texas Tech University, Lubbock,TX;
- Vicki Evans, Evans Associates, Huntsville, AL;
- Tom Hunt, Tom Hunt Textiles, Inc., Dallas, TX;
- Joe Amason, Lorenzo Textile Mills, Lorenzo, TX;
- Cal and Susan Brints, Oak Creek Products, Lubbock, TX;
- Sheri Dragoo, Texas Women's University, Denton, TX;

- Tommy Fondren, Lorenzo Cotton Producer, Lorenzo, TX;
- Pat Helton, Texas Tech University Small Business Development, Lubbock, TX;
- Carol Jean Bartlett, Sen. John T. Montford's Office, Austin, TX;
- Norma Ritz, Texas Dept. of Agriculture, Lubbock, TX;
- Barbara Upsal, Research Analysis & Maintenance Inc., El Paso, TX;
- Elizabeth G. Haley, Dean, College of Human Sciences, Texas Tech University, Lubbock, TX;
- Jane Craig, The University of Texas, Austin, TX;
- Janie VanZandt, CT&M Doctoral Candidate, Pampa, TX;
- Brian Murray, Texas Dept. of Agriculture, Austin, TX;
- Jinger Eberspacher, Associate Professor, MEDCE, College of Human Sciences, Texas Tech University, Lubbock, TX;
- Bobby Champion, Texas Dept. of Agriculture, Austin, TX;
- Mark Ellison, Texas Dept. of Agriculture, Austin, TX;
- Charles Johnson, Johnson Filtration, Amarillo, TX;
- Betsy Henderson, Assistant Professor, MEDCE, College of Human Sciences, Texas Tech University, Lubbock, TX;
- Jane Ann Stinnett, Stinnett Enterprises Inc., Lubbock, TX;
- Michael A. Mendoza, Dallas Market Center, Dallas, TX;
- Zane Willard, Sheep & Goat Raisers Association, San Angelo, TX;
- JoAnn Shroyer, College of Human Sciences, Texas Tech University, Lubbock, TX;
- Barbara Williams, Tarleton State University, Stephenville, TX;
- Jeri Pool-Marcus, Texas Food & Fibers Commission, Dallas, TX;
- Mary Curl, Director of External Relations, College of Human Sciences, Texas Tech University, Lubbock, TX;
- Mohamed Fouad Kabbaj, B.B.K., Berrechid, MOROCCO;
- Abdelhamid Seddiki, COTEF, Fes, MOROCCO;
- Abdou Belhkayat, FILCOP, Fes, MOROCCO;
- Mohammed Ghazlani Jamai, Ghazafil, Casablanca, MOROCCO;
- Dimitris Petratos, ICOZ, Casablanca, MOROCCO;
- Aziz El Idrissi, Le Petit Poussion S. A., Casablanca, MOROCCO;
- Omar Chraibi, SATEX, Casablanca, MOROCCO;
- Salah Eddine Mezouar, SETTAVEX, S.A., Settat, MOROCCO;
- Aziz Abdelali, Agricultural Specialist, American Embassy, Rabat, MOROCCO;
- Will Bettendorf, Cotton Council International, London, ENGLAND;
- Vaughn Jordan, Cotton Council International, Washington, D.C.;
- Kim, Lee and Katie McDonald, Pampa, TX;
- Wayman Jackson, Levelland Knitting Mill, Levelland, TX:
- Don Slease, Levelland Knitting Mill, Levelland, TX;
- Jack Bruce, Levelland Knitting Mill, Levelland, TX;
- David Butterworth, Johnson & Johnson Medical, Arlington, TX;
- Hans Odoo, Ekhamn, SWEDEN;
- Matilde Vretblad, SIS Environmental Labelling, Stockholm, SWEDEN;
- Urban Zonolor, SIS Environmental Labelling, Stockholm, SWEDEN;
- Jaclyn Harman and Helen Tierney, No. Montpelier, VT;
- Norma Tierney, Wolfforth, TX;
- Roger T. Walker, Federal Aviation Association, Lubbock, TX;
- Keith Harwood, SDL International Ltd., Manchester, ENGLAND;
- Elaine Warner, Edmond, OK;
- Michael Reeves, Lubbock Tourism and Visitors Bureau, Lubbock, TX;
- Six students from Hale Center High School, Hale Center, TX;
- Six students from Roosevelt High School, Acuff, TX.