TEXTILE TOPICS
INTERNATIONAL TEXTILE CENTER
TEXAS TECH UNIVERSITY • LUBBOCK, TEXAS / USA

Volume 22, No. 4 Summer 1994

IMPACTS OF ENZYME TREATMENTS ON FINISHING OF COTTON AND COTTON/WOOL BLEND FABRICS

It has been shown that application of cellulase enzymes to cotton fabrics and protease enzymes to wool fabrics can improve the surface appearance by removing protruding fibers and naturally accompanying substances.[1] An effective enzymatic treatment results in the degradation of the protruding loose fibers and altered physical and surface properties. However, accompanying impacts on dyeability and related characteristics of enzyme treated fabrics have not been fully evaluated. A recent ITC project focused on an investigation of:

- Effects of enzyme treatments on dyeing and finishing performance of 100% cotton and 80%/20% cotton/wool blended fabrics.
- Effects of the enzyme treatments on selected physical properties of these fabrics.

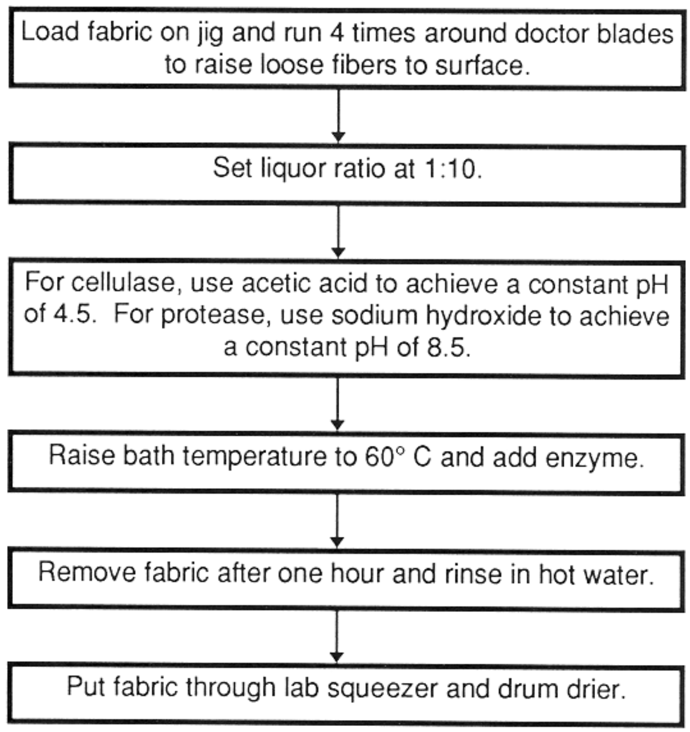
The all-cotton fabric used was a plain weave with 60 ends/inch and 56 picks/inch; yarn sizes were Ne 16/1 in both warp and filling directions. The 80/20 cotton/wool blended fabric used was a special weave with 52 ends/inch and 48 picks/inch; yarn sizes were Ne 14/1 in both warp and filling directions. Both fabrics were desized and bleached before treatment with the enzymes. Cellulase enzymes were applied to both fabrics, while protease enzymes were applied only to the cotton/wool blended fabric. While different concentrations of the enzymes were used, results reported here will be for a concentration of 3% on fabric weight.

Procedures

Steps followed for the enzyme treatments that preceded dyeing are summarized in **Exhibit 1**. The procedure was almost the same for each enzyme, the single major exception being different pH levels--4.5 for cellulase versus 8.5 for protease.

Untreated portions of the experimental fabrics were dyed in the same manner, providing a "control" for measuring the impacts of treatments. For the all-cotton fabrics, two dyes were used: Direct Red 79 and Sumafix Supra Bifunctional Reactive Blue 3RF.

Exhibit 1: Steps in Enzyme Treatment of Fabrics



For the cotton/wool blend fabric, three Sumafix Supra Bifunctional Reactive dyes were used: Red 3BRF, Yellow 3BF and Blue 3RF. Therefore, five distinct dyeings were done on the control fabrics.

Since the cotton/wool blend fabric was treated with both cellulase and protease, three additional dyeings were necessary. Therefore, for the enzyme treated fabrics, there were eight distinct dyeings for evaluating enzymatic action:

1) DRCC - direct red on cotton treated with 3% cellulase.
2) RBCC - reactive blue on cotton treated with 3% cellulase.
3) RRBC - reactive red on cotton/wool blend treated with 3% cellulase.
4) RYBC - reactive yellow on cotton/wool blend treated with 3% cellulase.

5) RBBC - reactive blue on cotton/wool blend treated with 3% cellulase.
6) RRBP - reactive red on cotton/wool blend treated with 3% protease.
7) RYBP - reactive yellow on cotton/wool blend treated with 3% protease.
8) RBBP - reactive blue on cotton/wool blend treated with 3% protease.

Steps used in dyeing with direct dyes (on 100% cotton fabrics) are summarized in **Exhibit 2**. In it, the "liquor ratio" refers to the fabric-to-water ratio, while the acronym OWF means "based on the weight of the fabric". One percent direct dye OWF was used along with 7.5% sodium sulfate OWF.

Exhibit 2: Steps with Direct Dyes

Load fabric on jig and prepare dye bath with a liquor ratio of 1:7.5.

↓

Add 0.5% wetting agent (Tergitol NP9) OWF.

↓

Add 1% dye OWF and raise bath temp. to 90° C.

↓

Add 7.5% Sodium Sulfate OWF and run for 40 min.

↓

Drain bath, refill with cold water and run for 10 min.

↓

Put fabric through lab squeezer and drum drier.

Steps used in dyeing with bifunctional reactive dyes (on both all-cotton and cotton/wool fabrics) are summarized in **Exhibit 3**. Two percent dye OWF was used, along with sequential additions of sodium sulfate and soda ash to the dye baths.

Tests performed and techniques used included the following:

- <u>CIE (Commission International de l'Eclairage) 1976 L*a*b* color difference at equal apparent strength of the dyed fabrics</u> - determined by using Illuminant D 65 and a viewing angle of 10° with a Macbeth Color-Eye 3000 Spectrophotometer.
- <u>Colorfastness to washing</u> - evaluated by AATCC Test Method 61-1993; Wash Test 1A washed for direct dyes and Wash Test 2A used for reactive dyes.

Exhibit 3: Steps with Reactive Dyes

Load fabric on jig and prepare dye bath with a liquor ratio of 1:7.5.

↓

Add 0.5% wetting agent (Tergitol NP9) OWF.

↓

Add 2% dye OWF and raise bath temp. to 90° C.

↓

Add 40 grams/liter of sodium sulfate in 4 equal portions at 5-min. intervals; then run for 20 minutes.

↓

Add 4 grams/liter of soda ash in 4 equal portions at 5-min. intervals; then run for 45 minutes.

↓

Drain bath, refill with cold water and run for 10 min.; boil off; drain, refill and run again for 10 min.

↓

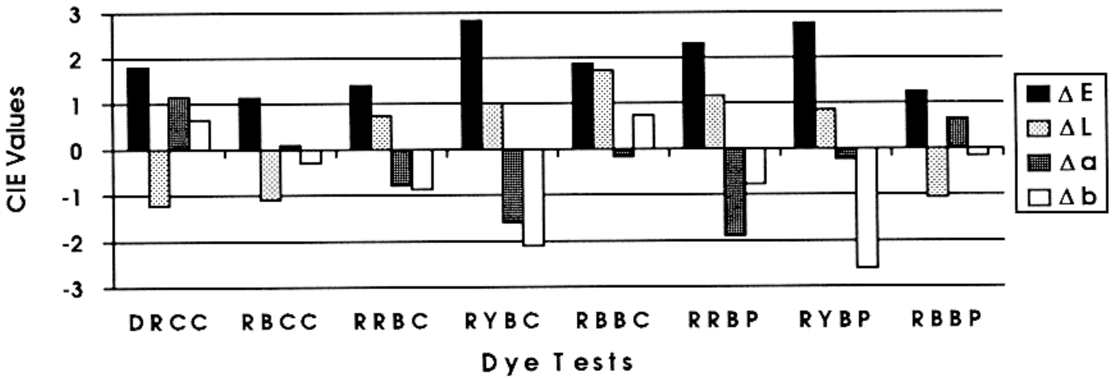
Put fabric through lab squeezer and drum drier.

- <u>Colorfastness to dry and wet crocking</u> - crocking was done with a rotary crockmeter using AATCC 116-1989 test method and evaluated with gray scale for staining.
- <u>Lightfastness</u> - evaluated after 20 and 40 hours of exposure according to AATCC Test Method 16-1993 using an Atlas Sunchex air-cooled Xenon-Arc Lamp; for calibration, used blue wool lightfastness standard L-4 for 20 AATCC fading unit equivalents.
- <u>Breaking strength</u> - measured by ASTM D 1682-75 test method.
- <u>Resiliency</u> - examined by dry wrinkle recovery angle using AATCC 66-1984 test method.
- <u>Pilling Resistance</u> - measured by ASTM D 3512-82 test method.

<u>Results:</u>

Results of the color difference values obtained on the enzyme treated fabrics versus the control are summarized in **Exhibit 4**. The ΔE values give total color difference and indicate that in all cases the enzyme treatment affected the shade of the dyed fabrics.

Exhibit 4: Color Difference Values



The formula for ΔE is as follows:

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

where

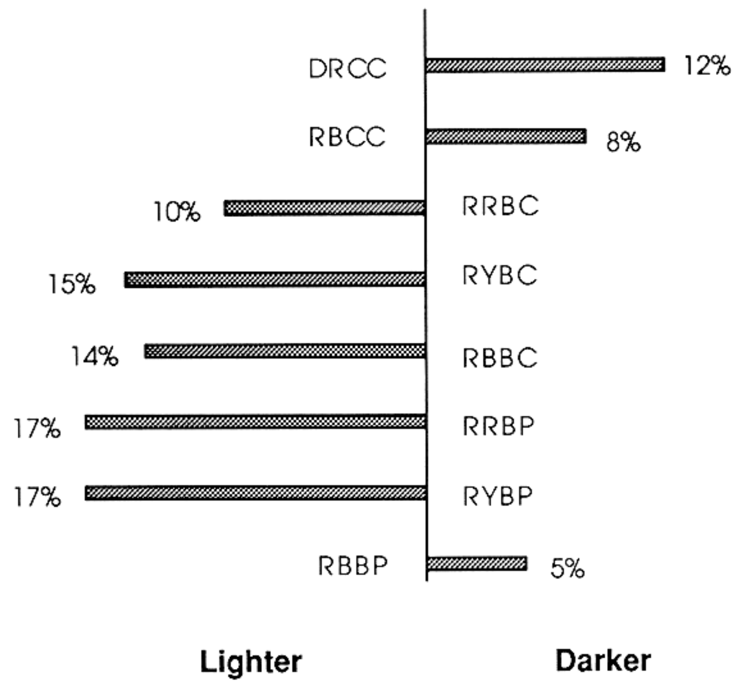
Δ denotes sample value minus standard value,
L denotes lighter (+Δ) versus darker (-Δ) shade,
a denotes redder (+Δ) versus greener (-Δ) shade, and
b denotes yellower (+Δ) versus bluer (-Δ) shade.

The results in **Exhibit 4** indicate that: (1) cotton fabrics treated with cellulase were darker; (2) cotton/wool fabrics treated with either cellulase or protease were lighter except for fabric dyed with reactive blue; (3) cotton fabrics treated with cellulase and dyed with direct dye were redder and yellower than the control; (4) cotton fabrics treated with cellulase and dyed with reactive dyes were redder and bluer than the control; and (5) cotton/wool fabrics treated with either cellulase or protease and dyed with reactive red or yellow dyes were greener and bluer than the control.

Based on the Macbeth Spectrophotometer rating, the percentage lighter or darker the colors were for each dyeing is summarized in **Exhibit 5**. All-cotton fabrics dyed with Direct Red 79 and Sumafix Supra Bifunctional Reactive Blue 3RF exhibited an increase in color strength. The 80/20 cotton/wool blended fabrics, dyed with Sumafix Supra Bifunctional Reactive dyes, generally showed decreased color strengths; the exception was 5% darker with the blue dye on 3% protease treated fabric.

The dyed samples were subjected to colorfastness tests of washing, crocking and light (shown in **Exhibit 6** on the next page). For colorfastness to washing and crocking, the extent of staining was given a numerical gray scale rating in geometric steps of color difference:

Exhibit 5: Color Strength Differences from Macbeth Spectrophotometer



5 - negligibly stained;
4 - slightly stained;
3 - noticeably stained;
2 - considerably stained;
1 - heavily stained.

All dyed fabrics, whether treated with enzymes or not, scored either 3 or 4. Washfastness of the all-cotton fabric appeared to be slightly improved by the 3% cellulase treatment. Results were mixed for the cotton/wool fabric; however, it appears that the 3% protease treatment was slightly more beneficial than the 3% cellulase treatment.

As expected the wet crocking test had lower ratings than the dry; otherwise, results were mixed for the enzyme treated fabrics versus the control.

Exhibit 6: Effects of Enzyme Treatments on Dye Performance of Fabrics

Test	Treatment	Washfastness Rating	Crocking Rating		Lightfastness Rating	
			Dry	Wet	20 Hours	40 Hours
DRCC:	Control	3	4	2.5	4	4
	3% Cellulase	4	3.5	3	4	4
RBCC:	Control	3	4	3.5	4	4
	3% Cellulase	4	3.5	3.5	4	4
RRBC:	Control	3	4.5	2.5	4	3
	3% Cellulase	4	4.5	3	4	3
RYBC:	Control	4	4	3	4	4
	3% Cellulase	3	5	3	5	5
RBBC:	Control	4	4	2	5	5
	3% Cellulase	3	4.5	3	5	5
RRBP:	Control	3	4.5	2.5	4	3
	3% Protease	4	3	2.5	4	4
RYBP:	Control	4	4	3	4	4
	3% Protease	4	3.5	3	5	5
RBBP:	Control	4	4	2	5	5
	3% Protease	4	4.5	2.5	5	4

For Measurements of lightfastness the extent of color transference was ranked as follows:

5 - negligibly changed;
4 - slightly changed;
3 - noticeably changed;
2 - considerably changed;
1 - greatly changed.

Lightfastness of the all-cotton fabric appeared to be unaffected by the cellulase treatment; perhaps there is a slight tendency for the enzyme treatments to improve lightfastness in the cotton/wool blend.

Some key physical properties were affected by the enzyme treatments. These include the following (**Exhibit 7**):

- Breaking strength of treated fabrics was reduced by 8-17% in both warp and filling directions. The 3% protease treatment reduced cotton/wool blend fabric strength more than cellulase; probably because at 3% OWF the protease (affecting wool only) was equivalent to 15% OWF.
- The fabrics treated with cellulase exhibited greatly increased resistance to pilling, whereas those treated with protease showed a moderate increase, likely because the protease had no effect on the cotton fibers.
- The wrinkle recovery angle of all treated fabrics was improved. This may be attributable to the lack of random interference of protruding fibers. The lack of protruding fiber also means that the enzyme treated fabrics will generally have a smoother "hand" than their untreated counterparts.

Conclusion:

It has been demonstrated that, for the dyes used in this study, the cellulase and protease enzymes had

1) significant effects on color properties;
2) marginal effects on durability of the color; and
3) significantly affected physical properties of the dyed fabrics.

On balance, the cotton fabric benefited more from enzyme treatment than did the cotton/wool fabric. Perhaps the 3% threshold used for enzyme concentration was too high to achieve optimum results for the blended fabric. Except for the inevitable weakening of the cotton fabric (7-9% strength

Exhibit 7: Differences in Physical Properties of Fabric with 3% Enzyme Treatment

Dye-Fiber-Enzyme Combination	Decrease in Strength		Increase in Pilling	Increase in Wrinkle Recovery	
	Warp	Filling	Resistance	Warp	Filling
Direct Dye on Cotton (DRCC)	8%	8%	25%	9%	6%
Reactive Dye on Cotton (RBCC)	9%	7%	25%	10%	11%
Reactive Dye On Cotton/Wool Blend					
Using Cellulase (RRBC, RYBC & RBBC)	8%	10%	75%	7%	6%
Using Protease (RRBP, RYBP & RBBP)	12%	17%	12%	2%	10%

strength reduction), a 3% cellulase treatment improved its overall performance.

At this point in time we are not aware of any published research with respect to dyeing related properties of enzyme-treated cotton/wool blended fabrics. However, Koo, et. al.[2], recently investigated the effects of direct and reactive dyes on 100% cotton fabrics. In contrast to the greater color yields we observed with enzyme treatments, they observed a reduction in color yields after treatment. This apparent contradiction may be due to the different colors of dye that were used in each study.

Developing a method which realizes simultaneous improvements in physical properties and dyeability is of increasingly practical significance. Therefore, further research is desirable to better understand and control the effects of enzyme applications on dyeability, physical properties, and related characteristics.

This study was funded by the Texas Food and Fiber Commission and was conducted by Shridhar Chikkodi, Research Associate at the ITC. Requests for further information may be directed to him.

References:

1. Hemp, W. H., "The Surface Modification of Woven and Knitted Cellulose Fiber Fabrics by Enzymatic Degradation," International Textile Bulletin - Dyeing/Printing/Finishing, 37 (3), pp. 5-11 (1991).
2. Coo, J., Ueba, M. And Waked, T., "Cellulase Treatment of Cotton Fabrics," Textile Research Journal, 64, pp. 70-74 (1994).

RESEARCH EFFORT WINS AWARD

Second place in the 1994 International Textiles and Apparel Association's Graduate Student Research Competition has been awarded to Ms. M. Jane VanZandt, a student in the Department of Merchandising, Environmental Design and Consumer Economics of Texas Tech University's College of Human Sciences. Her thesis was among sixteen entries in the competition. The research was conducted at the International Textile Center as a cooperative program between the ITC and the College of Human Sciences.

VanZandt will present her thesis at the 1994 ITAA meeting in Minneapolis, MN on October 22, 1994. An abstract is printed below:

DEVELOPMENT OF FABRICS FROM FOXFIBRE NATURALLY COLORED COTTONS (COYOTE AND BUFFALO) AND EVALUATION OF FLAME RESISTANT CHARACTERISTICS

M. Jane VanZandt

Flammability of textiles, including cotton, continues to be a source of product liability and is a major concern of consumers, legislators, and manufacturers. Naturally colored cotton is an emerging market, welcomed for its environmental aspects. The primary purpose of this research was to test colored cottons for flame-resistance before and after application of U6P flame-retardant finish in a 25% concentration level. A secondary purpose was to assess the effects of the flame-retardant finish on physical properties of the fabrics and the effects of care on the finish and fabrics. The sample for the study included two naturally colored cottons, Coyote brown and Buffalo brown, in weights of 4 oz/yd^2 (plain weave) and 10 oz/yd^2 (twill weave). The procedure included both

flammability and physical testing with the application of a flame-retardant finish. Flammability was tested in accordance with federal standards for children's sleepwear and motor vehicle and airplane interiors. Physical tests included tear and abrasion resistance, dry and wet breaking strength, and oxygen index value. Test results from untreated fabrics were used as a control for comparing test results after application of the flame-retardant finish. The 4 oz/yd^2 fabrics did not meet minimum flammability requirements of the children's sleepwear standard, with the exception of treated Coyote before laundering. In reference to the motor vehicle standard, the 10 oz/yd^2 fabrics met the standard before and after application of the flame retardant and before and after dry-cleaning. For airworthiness, treated fabrics met the standard before and after dry-cleaning.

In conclusion, the flame-retardant finish made the fabrics flame resistant, but physical strength was reduced. Naturally colored cotton fabrics in Coyote brown and Buffalo brown were found to be suitable for use as upholstery in motor vehicles and airplanes.

TEXAS INTERNATIONAL COTTON SCHOOL

The tenth session of the Texas International Cotton School is being held in October 1994. Eighteen students from seven countries are enrolled. Students will obtain an integrated understanding of issues surrounding cotton breeding, production, ginning, processing and marketing. The facilities of the ITC are at the disposal of those who attend, enabling hands-on instruction about the classing, testing, preparation, and processing of cotton. Even the latest developments in handling and processing of naturally colored and organic cottons are covered.

A major focus of the school is on the global cotton/textile markets. It covers every segment of the U.S. cotton and industry and the interactions with the global market. Particular emphasis is given to high volume instrument (HVI) technology and how it is being used by the various market segments.

Outstanding experts from all over the US provide instruction and are available for personal discussions with students. Featured speakers for the October school include experts from: New York Cotton Exchange; International Cotton Advisory Committee; Cotlook, Ltd.; Rieter Corporation; Zellweger Uster and NationsBank.

DR. REIYAO ZHU TO JOIN ITC STAFF

Dr. Reiyao Zhu has accepted the position of Acting Head of Fibers Research at the International Textile Center. It is anticipated that she will arrive at the ITC in early January, 1995. We look forward to having her on the staff.

Currently employed at the University of Bradford, Bradford, U.K., in the Department of Industrial Technology, Dr. Zhu received her Ph.D. from the Department of Textile Industries, The University of Leeds, Leeds, England, in 1992. She earned a Bachelor of Engineering degree at China Textile University, Shanghai, Peoples Republic of China, in 1982 and a Master of Science at the same university in 1985.

SMITH PARTICIPATES IN BRAZILIAN CONFERENCE

Harvin Smith, assistant director of the International Textile Center, attended the 16th National Congress for Textile Technology in Salvador, Bahia, Brazil on September 7-10, 1994. He presented a paper titled "Market Trends in the World," with emphasis on the Brazilian cotton outlook. While in Brazil, he also visited the Textile Research and Education Institute (SENAI/CETIQT) in Rio de Janeiro to discuss possible cooperative research projects relating to Brazilian cotton quality.

NEW EQUIPMENT AT ITC

We are pleased to announce the installation of a Zinser Ringspinner 330 HS free standing ring spinning frame in the short-staple spinning laboratory. The machine has 240 spindles with a spindle gauge of 75 mm and can operate at spindle speeds of 16,000 to 25,000 rpm. Other features include ring diameters of 36 and 38 mm, a positive-drive ring rail, computerized state-of-the-art electronic controls with visual readout and automatic CO-WE-MAT doffing and donning. This versatile equipment will enable evaluation of high-speed ring spinning using the widest possible range of cotton fibers and blends.

VISITORS

Visitors to the International Textile Center since the last issue of *Textile Topics* include the following:

- Gene Wynn, Forche, Levelland, TX;
- Ronaldo Parente, Fontaceza, Brazil;
- Clarence Rogers and Doug Rippy, Clemson University, Clemson, SC;
- Mike Rodriguez, Cotton Incorporated, Raleigh, NC;
- Tim H. Orsak, Seymour, TX;

- James Sweeten, Brownwood, TX;
- Don Bethel, David Harris and Ken McCraw, Lamesa, TX;
- Johnny W. Green, Aprotex, Lubbock, TX;
- Jim Pope and Joe Essick, Zellweger Uster, Knoxville, TN;
- Ian Carmichael, Platt Ginning Equipment Ltd., Manchester, England;
- Mark Stoker, Advanced Manufacturing and Mechatronics Group, Middlesex University, London, England;
- Jim Weatherford, Consolidated HGM Corporation, Columbus, GA;
- Bill Morton, Mission Valley Mills, New Braunfels, TX;
- Zane Willard, Texas Sheep & Goat Raisers Assn., San Angelo, TX;
- Gary K. Fox and Paul C. Anderson, Eagle Aerospace, Inc., Albuquerque, NM;
- Glenn Harvey and Robert A. Holton, Levi Strauss & Co., Richardson, TX;
- Waverly B. Watkins, Levi Strauss & Co., Knoxville, TN;
- Steve Verett, Texas Food & Fibers Commission, Dallas, TX;
- David R. Goldman, Wellman, Inc., Fibers Division, New York, NY;
- Winston P. Moore, Bob Fletcher and Ed Wilburn, Wellman, Inc., Charlotte, NC;
- Peter Fleischhacker, TKM, Gilmanton I.W., NH;
- Robert C. Buffkin, Buffkin Associates, Inc., San Antonio, TX;
- Norma Ritz, Texas Dept. Of Agriculture, Lubbock, TX;
- Neil W. Yeargin and Darlene L. Ball, Burlington Industries, Greensboro, NC;
- Max H. Hance, Burlington Industries, Mooresville, NC;
- Robin Hurrell and Steve Coombes, SDL, Stockport, England;
- Patty Holloway, Levelland Knitting, Levelland, TX;
- C. A. Dishman, Dishman International Co., Inc., Uvalde, TX;
- Harvey Campbell, BC Cotton Inc., Bakersfield, CA;
- LaRhea Pepper, Pepper Farms, O'Donnell, TX;
- Herb Wright, Wright Fibers, Big Spring, TX;
- Randall and Sherry Brooks, Randall Brooks Farm, Lancaster, TX;
- Sayed Basher, Rieter Corporation, Spartanburg, SC;
- Garrett A. Screws, Jr., Novo Nordisk Bioindustrials, Inc., Danbury, CT;
- Michael Schmidt, Novo Allee, Bagsvaerd, Denmark;
- Patrick T. Murphy, Akzo Nobel Fibers Inc., Conyers, GA;
- Jim Blevins, Peyer, Dallas, TX;
- Roger Bolick and Tim Somers, Allied Fibers, Petersburg, VA;
- Danny Gilmore, George A. Goulston Chemical Co., Monroe, NC;
- Larry Harris, Sara Lee Knits, Winston-Salem, NC;
- Duery Menzies, Mohair Council of America, San Angelo, TX;
- Ken Simpson, Dan Boggs and Bill Buchanan, Doran Textiles, Shelby, NC;
- Joe David Ross, Texas Sheep & Goat Raisers' Association, Sonora, TX;
- Christopher J. Lupton, Texas A&M Experiment Station, San Angelo, TX;
- Two cotton officials from Uzbekistan; Nigmatulla Hasanov, Deputy Director General of UZAGROIMPEX and Vallery Evgenyevich Oustugin, Deputy Director General of SIFAT, accompanied by O. A. Cleveland, Mississippi State University, Mississippi State, MS;
- Yuri G. Kabaldin, Gennadly I. Usanov, Leonid P. Razvozzhaev and Lyudmilla F. Serebryannikova, Komsomolsk Na Amure Pedagogical University, Komsomolsk-on-Anur, Russia, accompanied by John Greiner of the Office of International Affairs at Texas Tech University;
- Twenty senior citizens from Pennsylvania, touring the Southwestern states with Perkiomen Tours of Pennsburg, PA;
- Sixteen members of the Lubbock Chamber of Commerce Women's Committee, Lubbock, TX;
- Eighteen participants in the International Dryland Agricultural Institute, accompanied by B. A. Stewart and Leonard Wilson, West Texas A&M University, Canyon, TX. Countries represented by the group included Pakistan, Ethiopia, Senegal, Argentina and Thailand;
- Five students from the Plant Science Department at Texas Tech University accompanied by their instructor, Larry Blanton;
- Twenty-eight young people who were visiting Lubbock, Texas as part of the Japanese Junior Ambassadors program;
- Nine representatives of American Cyanamid Company, accompanied by Gaylan Goddard, Plainview, TX;

- Seven members of the Hale County, TX Women Involved in Farm Economics (WIFE) organization;
- Forty members of the Texas Farm Bureau, Waco, TX;
- Sixteen Texas Tech University Agricultural Economics students, accompanied by their instructor, Dr. James Graves;
- Seven employees of the USDA Cotton Classing Office at Lamesa, TX;
- Twenty 4-H Club members from Floyd County, TX, accompanied by Floyd County Extension Agent Melissa Long.