Biodegradability Study on Cotton and Polyester Fabrics

Lili Li[1], Margaret Frey[1], Kristie J Browning[2]
[1]Department of Fiber Science and Apparel Design, Cornell University, Ithaca, NY 14853-4401, United States

[2] Cotton Incorporated, Cary, NC 27513, United States.

Correspondence to:
Margaret Frey, Email: mfw24@cornell.edu

ABSTRACT

The purpose of this study was to measure and compare the biodegradability of fabrics in laboratory and large scale composting environments. Cotton jersey fabrics with three levels of finishing treatments (scoured and bleached, softener added and resin added) and a polyester jersey fabric were tested. Under controlled laboratory conditions, the carbon dioxide produced was monitored and integrated to determine the biodegradation rate by ASTM D 5988-03 method in natural soil, and the weight losses were measured after biodegradation in enzyme solutions. The same set of fabrics was buried in the Cornell University Composting Facility for 3 months. The weight losses and the fabric morphology after biodegradation were used to assess and compare the biodegradability with the results obtained under laboratory conditions. The polyester fabric showed a slight initial degradation, but the fabric remained intact under both laboratory conditions and the compost environment. The cotton fabric with softener had an accelerated degradation rate, while the cotton fabric with resin showed a relatively slow degradation rate. All cotton samples were more significantly degraded in the compost environment than under the laboratory conditions and confirmed to be 'compostable'.

Keywords: biodegradation; ASTM D 5988-03; Compost; Enzyme; cotton; polyester; fibers

INTRODUCTION

The disposal of the fabric materials used in textiles [1-3] is a serious challenge to waste management. Conventional methods for fabric waste management include land-filling, recycling and incineration. Increasingly, large scale composting is emerging as a viable disposal method for biodegradable materials. Currently, food, animal and other biodegradable waste streams are being successfully diverted from land-filling to composting waste streams. However, few textile materials are assessed in large scale composting streams although laboratory testing has confirmed their biodegradability [4-7].

ASTM D 5988-03 standard (Standard Test Method for Determination of Aerobic Biodegradation in Soil of Plastic Materials or Residual Plastic Materials after Composting) is designed to

evaluate the extent and rate of aerobic biodegradation of fabric materials in contact with natural soil under controlled laboratory conditions. The CO_2 produced is monitored and measured for each material. The degree of biodegradability is assessed by the amount of CO_2 produced and expressed as a fraction of the measured and calculated carbon content with respect to time [8, 9].

The enzyme biodegradation process is another feasible laboratory method to measure the biodegradability of fabrics. Enzymes have different active components to decompose the corresponding chemical bonds of polymer materials and produce low molecular weight products [10-12].

Composting as an option for waste treatment is a potential way to test biodegradation of the fabrics [13-15]. Compared with these laboratory conditions, the Composting Facility will have more significant microbial and enzymatic active components. The compost is created by: combining organic wastes (e.g., yard trimmings, food wastes, manures) in proper ratios into piles, rows, or vessels; adding bulking agents (e.g., wood chips) as necessary to accelerate the breakdown of organic materials; and allowing the finished material to fully stabilize and mature through a curing process. The composting facility used in this work uses a mature compost which contains approximately 850 tons of pre and post-consumer food scraps and compostables, 3300 tons of animal manure and bedding, and 300 tons of plant materials and soil. The mature compost is a stable material with content called humus that is dark brown or black, and has a soil-like, earthy smell. The production of high temperatures to destroy pathogens and weed seeds is controlled from 50-65 ^{o}C. If the temperature drops below 50 ^{o}C or increase above 65 ^{o}C, the piles are turned by a self powered elevating face turner.

The purpose of this study is to compare the results achieved under laboratory conditions with large scale composting facility conditions in terms of the fabrics biodegradability. All fabrics were laundered 30 times to simulate garments at the end of their useful lives prior to testing. The results of the weight loss obtained in enzyme and composting conditions and the biodegradation rate under ASTM D 5988-03 conditions were used to assess and compare the biodegradability of cotton fabrics and the polyester fabric. The structures of these fabrics before and after degradation by the three testing methods were analyzed by IR spectra. The morphologies of fabric materials after biodegradation by different testing methods were observed by Scanning Electron Microscopy (SEM).

EXPERIMENTAL

Materials

Four knit fabric samples were tested:

1. 100% cotton jersey, scoured and bleached, no finish (Cotton nf).
2. 100% cotton jersey, scoured and bleached, softener only (Cotton softener only).
3. 100% cotton jersey, scoured and bleached, resin plus softener (Cotton resin).
4. 100% polyester shirt purchased at retail (Polyester).

Finish formulations for the cotton samples with softener only and resin plus softener are shown in *Tables I* and *II*.

All fabric samples were washed 30 times prior to testing. The cotton samples were laundered

according to AATCC 135 with a warm wash (105 ^{o}F), using the normal cycle, and tumble-dried (cotton cycle) for 30 minutes. The polyester shirt was laundered according to the manufacture's instructions including a cold wash (80 ^{o}F), using the normal cycle, and tumble-dried low for 30 minutes. AATCC standard liquid detergent was used for all washes and ballast was added to equal a 4-1b load.

TABLE I. Cotton Softener only

Generic Name	% on weight of the bath (owb)
Nonionic wetting agent	0.2
Amino functional silicone softener	1.0
Cationic softener	2.0
Cationic emulsified polyethylene	1.0

Pad-apply finish. Dry only.

TABLE II Cotton Resin plus Softener

Generic Name	% on weight of the bath (owb)
Nonionic wetting agent	0.2
Amino functional silicone softener	1.0
Cationic softener	2.0
Cationic emulsified polyethylene	1.0
Capped, DMDHEU resin	3.0
$MgCl_2$ Catalyst	1.2

Pad-apply finish. Dry. Cure at 340°F for 15 seconds.

For the ASTM D 5988-03 test, natural soil was supplied by the College of Agriculture and Life Sciences at Cornell University. The soil was sieved to less than 2 mm particle size, and stored at 4 ^{o}C for 7 days prior to use. 2 g soil samples were dispersed in 10 mL of distilled water and the pH value of the liquid was measured by a pH meter (Model 215 Denver Instrument). The ash content of the soil was obtained by measuring the remaining weight after incineration at 550 ^{o}C for 3 h. The moisture of the soil was determined from the weight loss after drying in the oven at 105 ^{o}C for 24 h. The water content in the soil was adjusted to 61% by adding water.

The chemicals used were both A.C.S. analysis reagents. The solutions were prepared by adding 4.72 g ammonium phosphate (98%, Sigma-Aldrich) and 28 g potassium hydroxide (98%, Mallinckrodt Baker Inc.) to 1 L distilled water, respectively.

Elemental analyzer

Elemental analyses of the fabrics were performed on a Carlo Erba NC2500 elemental analyzer. Fabrics samples were introduced to the combustion column via a Costech Zero-Blank autosampler.

IR analysis

Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) technique was utilized to collect IR spectra of the samples. The fabric sample was put into the microcup of the diffuse reflectance accessory on a PerkinElmer Nicolet Magana 560 IR spectrometer (Madison, WI), and IR information for the sample was collected and processed with OMNIC software.

Biodegradation methods

ASTM D 5988-03

The evolution of CO_2 from samples was used as a measure of biodegradation according to ASTM D 5988-03. All samples were cut into 25 strips with 2×2 cm dimensions.

The tests were carried out in desiccators at room

temperature. For every sample, a blank, a positive control, and a specimen in positive control were introduced in the desiccators. The blank was only natural soil; the positive control was natural soil in which ammonium phosphate solution was added. The specimen in positive control was the natural soil in which fabric samples and ammonium phosphate solution were added. In each desiccator, 50 mL of 0.5N KOH in a 100 mL beaker and 100 mL of distilled water in a 150 mL beaker were placed on the perforated plate (shown in *Figure 1*) to maintain the moisture in the soil at 61%. The amount of CO_2 absorbed by the KOH solution was measured at pre-determined time intervals. With each sampling, aeration was provided by leaving the desiccator lid open for about 30 min. The CO_2 trapped by KOH was determined by titration with 0.25N HCl.

FIGURE 1. ASTM D 5988-03 method

Composting

Composting experiments were conducted in the Cornell Composting Facility. The Compost Facility consisted of windrows of 270 feet in length, 6 to 8 feet tall and 14 feet wide. The interior of the windrows reached a temperature of 65 ^{o}C (150 ^{o}F). Typically the Compost Facility processed dining hall and animal facility waste from the Cornell University campus.

1×1 foot samples of each fabric were weighed and labeled with a Sharpie marker. Each sample was placed in an onion bag with several shovels full of compost. The onion bags were labeled with a numbered plastic tag on the end of 2 yards of nylon rope. Each bag was buried in the center of a composting windrow with the plastic tag emerging at the top of the windrow (*Figure 2*). The location of each sample was further marked by a flag. Samples were removed before windrows were turned and subsequently re-buried. Samples were examined and SEM images of all fabric samples were taken after 3 months. Weight loss of all fabric samples was calculated after 3 months of composting.

FIGURE 2 Composting method

Enzymatic hydrolysis by cellulase

The fabric sample was added into a small vial containing 30 mL acetate buffer (pH=4.5) with cellulase concentration of 2 g/L. The cellulase was the culture filtrate from Trichoderma viride. The mixture was then incubated at 55 ^{o}C in water bath. After every 48 h, the fabric sample was washed with distilled water, and then dried in a vacuum at 45 ^{o}C for 24 h. The immersion media were refreshed daily to maintain enzymatic activity. The extent of biodegradation was estimated from the weight loss of the fabric sample based on the following equation:

$$W_t(\%) = \frac{W_0 - W_t}{W_0} \times 100\%$$

Where W_t (%) is the percent weight loss after t days of incubation, W_0 is the original weight of the fabric sample before immersion and W_t is the weight of the dry fabric sample after t days of incubation.

RESULTS

Analysis of biodegradation degree by ASTM D 5988-03

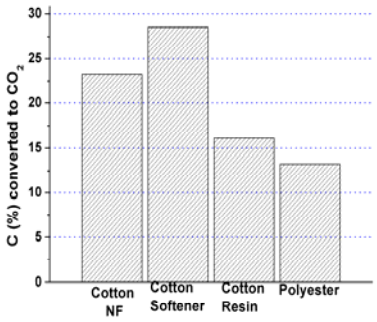

FIGURE 3. Biodegradation of fabrics after 90 days in natural soil

For each fabric, the percent conversions of carbon content to CO_2 were used to determine the biodegradation extent of the specimen in natural soil. The degree of biodegradation after 90 days according to ASTM D 5988-03 is compared in *Figure 3*.

As shown in *Figure 4*, all cotton fabrics (S1#, S2# and S3#) had higher degradation degrees than the polyester fabric. The cotton fabrics had similar degradation degrees in the first 20 days in the ASTM method. After the first 20 days, the cotton fabric with softener showed a significantly faster degradation rate than other cotton fabrics. The cotton fabric with resin degraded more slowly than other cotton fabrics after the 60th day. The degradation rates of all fabrics in ASTM D 5988-03 were compared and are listed below:

Cotton softener > Cotton no finish > Cotton Resin > polyester.

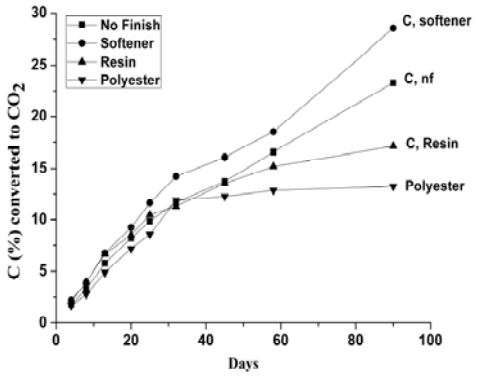

FIGURE 4. Biodegradation of fabrics in natural soil by ASTM D 5988-03

Biodegradation testing by composting

In composting tests, the cotton fabric samples suffered a weight loss of approximately 50-77% after 90 days. Under the same conditions, polyester showed a very slow degradation rate. The weight loss of these fabric samples were compared and shown in *Figure 5*.

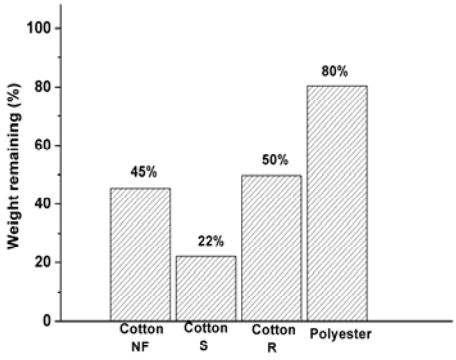

FIGURE 5. Biodegradation of fabrics in composting

The degradation rates of all fabrics in composting

were compared and are listed below:
Cotton softener > Cotton no finish > Cotton Resin > polyester.

Biodegradation by enzymatic hydrolysis

Fabric weight loss was determined by conditioning the samples before and after enzymatic treatment as shown in *Figure 6.* To compare their biodegradability under the same testing conditions, cellulase was used as the only testing enzyme. The polyester fibers had the lowest degradation rate with less than 1% weight loss. Cotton no finish, Cotton softener and Cotton resin had similar biodegradation extents with 8.5%, 8.6% and 8% weight loss, respectively.

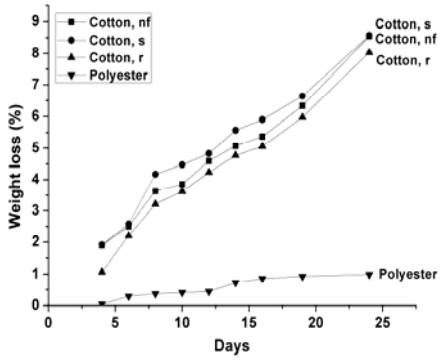

FIGURE 6. Biodegradation of fabrics by enzymatic hydrolysis

Morphological study

The SEM images of the fabric specimens before and after biodegradation testing by ASTM D 5988-03 and composting methods were shown in *Figures 7-8*. The SEM images show the characteristic structure of each fiber type before testing, for example, the twisted ribbon shape of the cotton fibers. The resin-treated cotton fibers showed increased surface roughness. The extruded polyester fibers also showed some evidence of surface finish and did not appear to be round fibers. Evidence of degradation was readily observed on the surface of fibers after 90 days in both the desiccators and the Compost Facility. Degradation of cellulose materials had significantly etched away the fiber structures. The SEM images of polyester by ASTM D 5988-03 after 90 days showed that slight fiber bits peeled off from the surfaces of polyester. When polyester was tested by composting for 90 days, some destroyed fibers were observed on polyester fabric surfaces although most of the fibers were still intact. The SEM images were consistent with the biodegradation results.

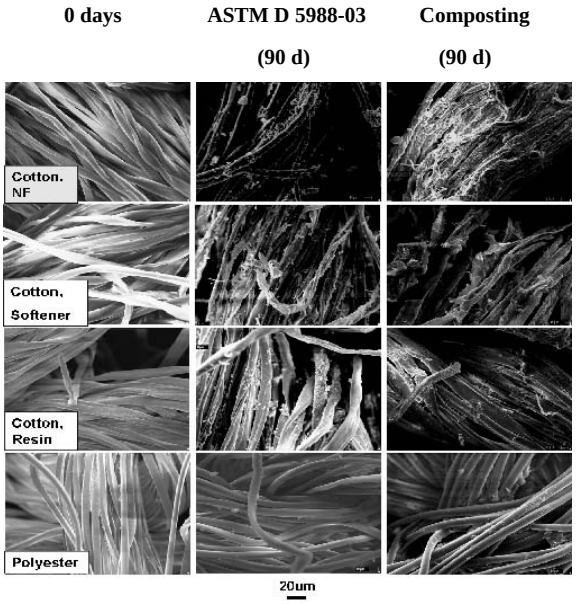

FIGURE 7. SEM images of fabric samples after degradation (1000×)

The SEM images of the ASTM testing and the compost testing were also compared directly. The damage of the cellulose based fibers under compost conditions (elevated temperature, active microbe environment) was significantly faster than the degradation under ASTM conditions (room temperature, soil only). The polyester fabric, however, retained fabric and fiber structure throughout both degradation experiments.

The surface appearances of fabric specimens before and after biodegradation by the cellulase method are shown in *Figure 9*. Cotton nf, Cotton softener only and Cotton resin specimens showed slight degradation morphology as the first several layers were attacked and peeled off from surfaces of fabrics by enzyme after 22 days. There were no discernable changes in polyester fabrics.

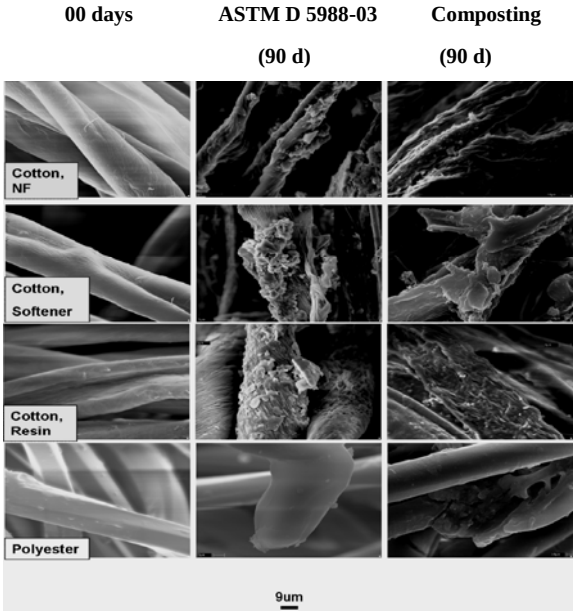

FIGURE 8. SEM images of fabric samples after degradation (5000×)

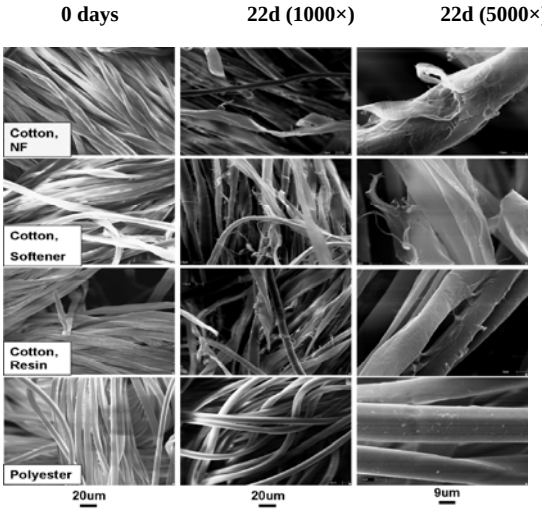

FIGURE 9. SEM images of degradation of fabric samples in cellulase

The differences of the morphology between cellulolytic enzyme biodegradation and other biodegradation method (soil or composting) were caused by their different active components. The cellulases removed the outer layers of the fibers in the cuticle and the primary wall (See *Figure 9*). The natural soil and compost contained multiple types of organisms, cellulose, and other enzymes. The enzymatic hydrolysis firstly removed the outer layers from the fabric surfaces. Subsequently, the organisms converted the interior of the fiber to hydrolysates. These fibers showed completely destroyed structures which could be observed in *Figure 7* and *Figure 8.*

Analysis of polyester fabric

On the 32nd day, polyester showed an unexpectedly high degree of degradation in ASTM D 5988-03 testing, and then the degree of biodegradation leveled off in the following test. Also, the positive samples, including the soil and different quantities of $(NH_4)_2HPO_4$, consumed a different HCl volume. This might be experimental deviation. There were several reasons which might explain the deviation:

1) For every sampling, 10 g out of 100 g KOH was used to titrate, and then the total volume of HCl consumed would be 10 times the titrated HCl volume.

2) Different quantities of $(NH_4)_2HPO_4$ in the soil were used for different positive samples and testing samples.

3) IR data showed that polyester fabric was modified PET (as shown in *Figures 10-14*) which could lead to higher degradation.

Structure analysis by IR

The FTIR spectrums of pure PET, reported from SDBS (*Figure 10*) and the American Institute of Physics (*Figure 11*), had similar peaks. Compared to the FTIR spectra of pure PET from the literature, the polyester sample used in this work (*Figure 12*)

had the same peaks from 1900 to 600 cm^{-1}. However, the polyester sample did not show any peak from 4000-2000 cm^{-1}. The peaks in this range were attributed to aromatic heterocyclic groups, hydrogen bond or hydroxyl groups.

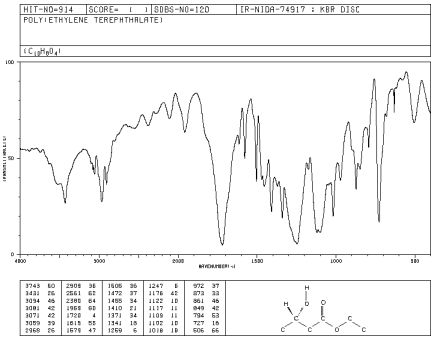

FIGURE 10. IR spectra of PET from SDBS

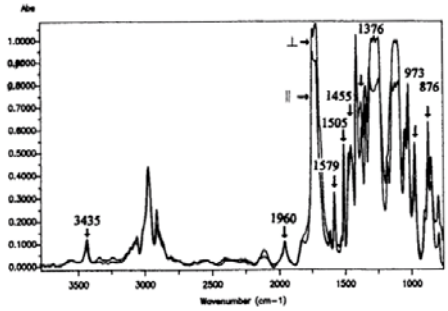

FIGURE 11. IR spectra of PET reported by the American Institute of Physics

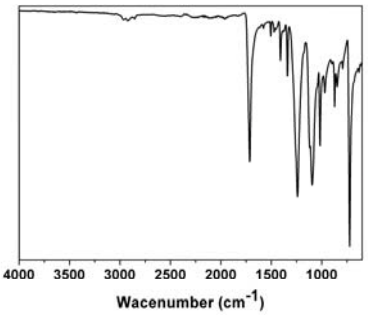

FIGURE 12. IR spectra of PET from the polyester garment

The peaks in the IR spectra of the polyester fabric appeared in the range of 600-2000 cm^{-1} (see *Figure 13*). The waves were assigned as follows:
1717 (C=O), 1407 (aromatic ring), 1339 and 1014 (carboxylic ester or anhydride), 1092 and 1014 (O=C–O–C or secondary alcohol), 971 (C=C), 872 (five substituted H in benzene), 847 (two neighboring H in benzene), 724 cm^{-1} (heterocyclic aromatic ring).

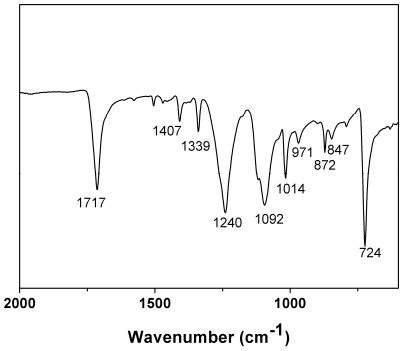

FIGURE 13. IR spectra of Polyester fabric before biodegradation

The main structure of the polyester sample had ester, alcohol, anhydride, aromatic ring and heterocyclic aromatic rings. Alcohol was able to react with anhydride and produce ester groups. That was the reason there was still alcohol and anhydride as residual reactants left in the polyester. The carboxyl, ester, anhydride and alcohol groups showed the polyester fabric was not pure PET. The peak at 1407 cm^{-1} corresponded to the aromatic ring which was a stable group. It was the characteristic absorption peak of PET. The peaks at 1717 and 1092 cm^{-1} were assigned to the ester group which preferred to break under certain conditions. The peak height ratios of the ester group to the aromatic ring group were selected to analyze the biodegradation possibility of polyester fabric as shown in *Table III*. The peak height ratios did not change substantially compared with the starting polyester.

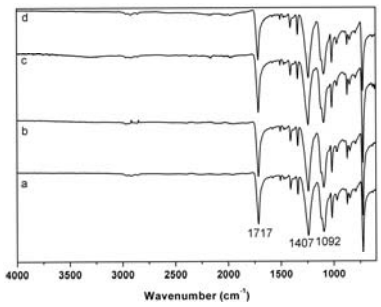

FIGURE 14. IR spectra of polyester fabric before and after degradation: (a) before degradation; (b) composting degradation method; (c) ASTM D 5988-03 degradation method; (d) enzyme degradation method

TABLE III. The biodegradation analysis of polyester by FTIR.

Wavenumber (cm^{-1})	Peak Height		
	Before degradation	soil	composting
1717	10.40	11.89	9.93
1100	9.44	10.54	9.02
1407	11.43	13.29	11.28
1717/1407	0.91	0.89	0.88
1100/1407	0.82	0.79	0.80

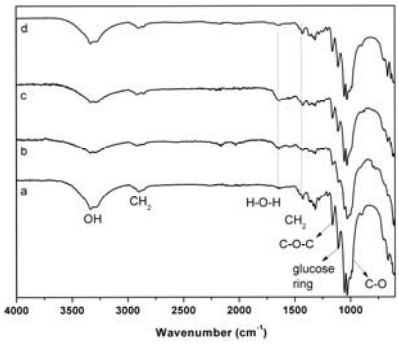

FIGURE 15. IR of Cotton nf before and after degradation: (a) before degradation; (b) composting degradation method; (c) ASTM D 5988-03 degradation method; (d) enzyme degradation method

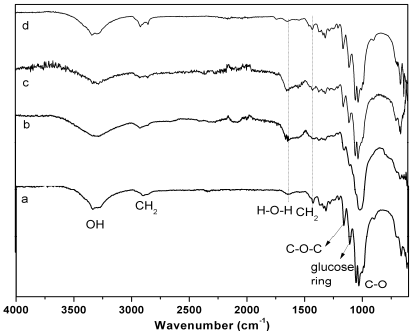

FIGURE 16. IR spectra of Cotton softener before and after degradation: (a) before degradation; (b) composting degradation method; (c) ASTM D 5988-03 degradation method; (d) enzyme degradation method

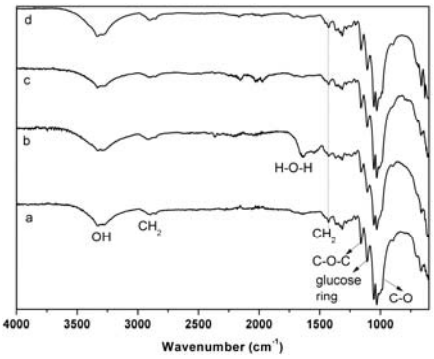

FIGURE 17. IR spectra of Cotton resin before and after degradation: (a) before degradation; (b) composting degradation method; (c) ASTM D 5988-03 degradation method; (d) enzyme degradation method

The intramolecular changes of the cotton fabrics before and after degradation by three methods are illustrated in *Figures 15-17*. The absorption in the region of 3600-3100 cm^{-1} was due to the stretching of –OH groups [16-18] and at 3000-2800 cm^{-1} to the CH stretching, the slight decrease of these contents after degradation indicated that hydrogen bonds and methyl and methylene of cellulose had some rupture, therefore, organisms could attack the cellulose chain easily [18-21]. The band at 1641 cm^{-1} arose from the H–O–H bending of the absorbed water. The symmetric –C–H bending occurred at 1416 cm^{-1}. The band at 1372 cm^{-1} was assigned to –OH bending and at 1317 cm^{-1} to C–C

and C–O skeletal vibrations. While the bands at 1061 and 1032 cm^{-1} indicated C–O stretching at C3, C–O stretching at C6 and C–C stretching [22-25], which decreased significantly by composting and ASTM D 5988-03 methods. These C–O stretching bands gave slight shoulders at 1169 cm^{-1} which represented the antisymmetric bridge stretching of C–O–C groups in cellulose and hemicellulose. The band at 1111 cm^{-1} corresponded to asymmetric glucose ring stretching. After degradation, the bands at 1169 cm^{-1} and 1111 cm^{-1} decreased slightly by enzyme degradation and significantly by composting and ASTM D 5988-03 degradation methods. This indicated that cellulose main chains cleaved between glucose units.

Compared to the slight shoulder bands at 1169 cm^{-1} and 1111 cm^{-1} in cotton nf, cotton softener and cotton resin samples before and after degradation, the possibility of main chain cleavages in cotton nf and cotton softener fabrics were higher than in cotton resin as evidenced by the decrease of corresponding bands.

CONCLUSIONS

This work was conducted to comparatively study the biodegradation of fabrics in laboratory and large scale composting conditions. Cotton jersey fabrics with three levels of finishing treatments (scoured and bleached, softener added and resin added) and a polyester jersey fabric were tested according to ASTM D 5988-03 and enzyme biodegradation methods under controlled laboratory conditions. The same set of fabrics was buried in the Cornell Composting Facility for 3 months. The weight losses after biodegradation were used to assess and compare the biodegradation rate with the results obtained under laboratory conditions. The polyester fabric showed a slight initial degradation, but the fabric was still intact after testing under both laboratory conditions and the compost environment. In soil and compost testing, which included multiple organisms and enzymes, the cotton fabric with softener had an accelerated degradation rate, while the cotton fabric with resin showed a relatively slow degradation rate. All cotton samples were more significantly degraded in the compost environment than under the laboratory conditions and confirmed to be ‘compostable’.

ACKNOWLEDGEMENT

The authors would like to acknowledge Cotton Incorporated for funding this project, the Cornell Center of Materials Research and Mr. Bill Huzinza at the Cornell Composting Facility for supporting this project, and Aric Stocks for helpful discussions.

REFERENCES

[1] Takagi, H., and Asano, A. Effects of processingconditions on flexural properties of cellulose nanofiber reinforced "green" composites, *Composites Part A* 2008, 39A, 685-689.

[2] Duckett, K. E., Bhat, G. S., and Suh, H. Compostable and biodegradable nonwoven fabric of a blend of natural cellulosic and thermoplastic biodegradable fibers and fabric manufacture, *U.S Patent* 1998, 14 pp.

[3] Jahagirdar, C. J., and Tiwari, L. B. Plasma treatment of polyester fabric to impart the water repellency property, *Pramana* 2007, 68, 623-630.

[4] Tian, C. M., Shi, Z. H., Zhang, H. Y., Xu, J. Z., Shi, J. R., and Guo, H. Z. Thermal degradation of cotton cellulose, *J. Therm. Anal. Calorim.* 1999, 55, 93-98.

[5] Zhang, L., Liu, H., Zheng, L., Zhang, J., Du, Y., and Feng, H. Biodegradability of Regenerated Cellulose Films in Soil, *Ind. Eng. Chem. Res.* 1996, 35, 4682-4685.

[6] Hashmi, S. A. R., Dwivedi, U. K., and Chand, N. Graphite modified cotton fiber reinforced polyester composites under sliding wear conditions, *Wear* 2007, 262, 1426-1432.

[7] Tomsic, B., Simoncic, B., Orel, B., Vilcnik, A., and Spreizer, H. Biodegradability of cellulose fabric modified by imidazolidinone, *Carbohydr. Polym.* 2007, 69, 478-488.

[8] Wilson, R. E. Humidity control by means of sulfuric acid solutions with critical compilation of vapor pressure data, *J. Ind. Eng. Chem. (Washington, D. C.)* 1921, 13, 326-331.

[9] Kim, M. Evaluation of degradability of hydroxypropylated potato starch/polyethylene blend films, *Carbohydr. Polym.* 2003, 54, 173-181.

[10] Wang, L.-S., Zhang, Y.-Z., Yang, H., and Gao, P.-J. Quantitative estimate of the effect of cellulose components during degradation of cotton fibers, *Carbohydr. Res.* 2004, *339*, 819-824.

[11] Cortez, J. M., Ellis, J., and Bishop, D. P. Using cellulases to improve the dimensional stability of cellulosic fabrics, *Text. Res. J.*2002, 72, 673-680.

[12] Jang, J., Lee, H.S., and Lyoo, W.S. Effect of UV irradiation on cellulase degradation of cellulose acetate containing TiO2, *Fibers Polym.* 2007, 8, 19-24.

[13] Suman Ghosh, B. P. K., N. B. Singh,. (2000) Composting of cellulosic hospital solid waste: a potentially novel approach, *International Biodeterioration & Biodegradation 45*, 89-92.

[14] Thompson, R. C., Moore, C. J., vom Saal, F. S., and Swan, S. H. Plastics, the environment and human health: current consensus and future trends, *Philos. Trans. R. Soc.* 2009, B 364, 2153-2166.

[15] Song, J. H., Murphy, R. J., Narayan, R., and Davies, G. B. H. Biodegradable and compostable alternatives to conventional plastics, *Philos. Trans. R. Soc*. 2009, B 364, 2127-2139.

[16] Marechal, Y., and Chanzy, H. The hydrogen bond network in Ibeta cellulose as observed by infrared spectrometry, *J. Mol. Struct.* 2000, 523, 183-196.

[17] Blackwell, J., Vasko, P. D., and Koenig, J. L. Infrared and Raman spectra of the cellulose from the cell wall of Valonia ventricosa, *J. Appl. Phys.* 1970, 41, 4375-4379.

[18] Sugiyama, J., Persson, J., and Chanzy, H. Combined infrared and electron diffraction study of the polymorphism of native celluloses, *Macromolecules* 1991, 24, 2461-2466.

[19] Michell, A. J. Second derivative FTIR spectra of native celluloses, *Carbohydr. Res.* 1990, 197, 53-60.

[20] Michell, A. J. Second-derivative FTIR spectra of native celluloses from Valonia and tunicin, *Carbohydr. Res.* 1993, 241, 47-54.

[21] Kokot, S., Czarnik-Matusewicz, B., and Ozaki, Y. Two-dimensional correlation spectroscopy and principal component analysis studies of temperature-dependent IR spectra of cotton-cellulose, *Biopolymers* 2002, 67, 456-469.

[22] Watanabe, A., Morita, S., Kokot, S., Matsubara, M., Fukai, K., and Ozaki, Y. Drying process of microcrystalline cellulose studied by attenuated total reflection IR spectroscopy with two-dimensional correlation spectroscopy and principal component analysis, *J. Mol. Struct.* 2006, 799, 102-110.

[23] Kaczmarek, H., Oldak, D., Malanowski, P., and Chaberska, H. Effect of short wavelength UV-irradiation on ageing of polypropylene/cellulose compositions, *Polym. Degrad. Stab.* 2005, 88, 189-198.

[24] He, Y., Pang, Y., Liu, Y., Li, X., and Wang, K. Physicochemical Characterization of Rice Straw Pretreated with Sodium Hydroxide in the Solid State for Enhancing Biogas Production, *Energy Fuels* 2008, 22, 2775-2781.

[25] Li, J., Zhang, L.-P., Peng, F., Bian, J., Yuan, T.-Q., Xu, F., and Sun, R.-C. Microwave-assisted solvent-free acetylation of cellulose with acetic anhydride in the presence of iodine as a catalyst, *Molecules* 2009, 14, 3551-3566.

AUTHORS' ADDRESS

Margaret Frey

Lili Li

Cornell University

College of Human Ecology

Fiber Science & Apparel Design

299 Martha Van Rensselaer Hall

Ithaca, NY 14853-4401

UNITED STATES

Kristie J Browning

Cotton Incorporated

Senior Textile Chemist

6399 Weston Parkway

Cary, NC 27513

UNITED STATES