Resistance of Various Textile Fibers to Mildew

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E. I. Du Pont de Nemours & Co.

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Exclusively Contributed

THE rapid deterioration of military fabrics in tropical use, due to growth of mold and rot fungi, has focused attention on the inherent resistance of certain textile fibres to these organisms. From a preliminary search of the literature most investigators claimed cellulose acetate rayon was highly resistant to the common superficial and cellulose destroying molds. As these investigations had all been conducted a number of years ago, and as the identity of the samples and degree of acetylation was not known, it was decided to reinvestigate the problem.

In planning this program, it was further decided to include, for control purposes, fabrics from the various textile fibres in common use for military goods. This list included the following: 1. cellulose acetate rayon, 2. viscose process rayon, 3. cotton, 4. nylon, 5. completely saponified (or deacetylated) acetate rayon, 6. nylon transparent film, 7. du Pont Cellophane (Viscose Process).

This study was conducted by the Pest Control Research Section of the Grasselli Chemicals Department, E. I. du Pont de Nemours & Co., under the direction of Dr. W. H. Tisdale.* Evaluations were made by the following methods:

1. SOIL BURIAL TESTS:—The test specimens were randomized in soil burial beds, containing an active flora of micro-organisms, for twenty-one days.

2. LABORATORY CULTURE TESTS:—The test specimens were placed on glass mats in culture jars and after adding liquid nutrient, spore suspensions of the various test organisms were added. The culture jars were then stored at 80°F and 80% relative humidity for a period of fourteen days. The test organisms used may be classified in two distinct groups:

(a) Active Cellulose Destroyers, and
(b) Superficial Molds.

*Manager Pest Control Research Laboratory, Grasselli Chemicals Department, E. I. du Pont de Nemours & Company.

Courtesy The Du Pont

The new Du Pont Pest Control Laboratory at Wilmington, Del., with its adjacent greenhouse. The new three-story structure offers the most modern facilities available for the development and study of insecticides and fungicides.

3. STORAGE OF CELLULOSE ACETATE AT 80°F. AND 80% R. H.:—Due to recent reports of the rapid tendering and staining of certain rayon fabrics if stored under conditions of high temperatures and high relative humidities, an investigation was made to determine the effect on skeins of cellulose acetate after storage for seven weeks at a temperature of 80°F. and 80% relative humidity. This investigation was made on skeins containing the normal finish applied by the rayon producer and also skeins which had been given a thorough scour at 180°F. to remove the finish.

Skeins were examined weekly for staining, and then tested for physical properties, to determine if any progressive tendering or staining was developing.

Results and Conclusions

1. SOIL BURIAL TESTS (See Table I):—Both the cellulose acetate ("Acele") fabrics and the nylon fabric are very resistant to the attack by micro-organisms, as measured by soil burial tests. The "Acele" fabrics numbered 1, 2, 3 and 4 (See Table I) after exposure retained 81.2, 92.1, 94.7, and 94.2% of their original strength respectively. The 81.2% value for sample No. 1 (scoured and ether-extracted) is not in good agreement with the values for the other three "Acele" fabrics. This is directly traceable to the low values recorded for two of the exposed samples in this series. It is thought, if a larger number of replicate samples had been available, this value would have been more in accord.

The nylon fabric numbered 10 and film No. 11, retained 95% and 96% of their original tensile strength respectively.

The viscose rayon, cellophane, cotton, and completely saponified acetate fabrics were entirely destroyed in the soil burial tests. There was no remaining evidence of any of the viscose rayon or cellophane samples in the soil, and only small shreds remained of the cotton duck and saponified acetate samples. The complete saponification of cellulose acetate, resulted

TABLE I

Comparative Resistance of Viscose Rayon, Cellulose Acetate, Nylon and Cotton Fabric to Deterioration in Soil Burial Tests

Size of samples used 6" x 4". Strength determination made by Grab Test Method as specified by A.S.T.M. Standards. Fabric placed in soil 2/5/43; Fabric removed from soil 2/26/43

Fabric No.	Fabric and Description	Average Tensile strength before burying* (Lbs. required to break)	Average Tensile strength after 3 weeks in soil* (Lbs. required to break)	Percentage of Tensile strength retained after three weeks in the soil
	225 x 90 "Acele" Cellulose Acetate Satin			
1	Washed and ether extracted	37	30	81.2
2	Commercially finished, contains residues of gelatin and sulfonated oils	38	35	92.1
3	Commercially desized and finished to contain 1% to 2% sulfonated peanut oil	38	36	94.7
4	Greige fabric from loom, contains 6% gelatin and sulfonated oils	34	32	94.2
	112 x 66 Viscose Rayon Twill Lining			
5	Commercially finished containing a small amount of sulfonated oil	62	0	0
6	Washed and ether extracted	66	0	0
	115 x 115 Completely Saponified Acetate 1.0 oz. Taffeta			
7	Commercially finished	30	0	0
8	Washed and ether extracted	31	0	0
	Cotton and Nylon Fabric			
9	Cotton Control—8¼ oz. Duck—No fungicide	130	0	0
10	1½ oz. Nylon fabric—"66 Polymer"	78	74	95
	Nylon and Cellophane Films			
11	Nylon film—Type 6 Polymer†	49	47	96
12	Du Pont Cellophane—Type 600 A	42	0	0

* Figures are average for six samples.
† Experimental nylon film. *Differs from textile nylon.*

in a fabric that was just as susceptible to attack as viscose process rayon or cotton fabrics. This definitely establishes that resistance of cellulose acetate is correlated with the ester linkage.

All of the cellulose acetate and nylon samples were relatively bright and clear after being removed from the soil and rinsed in cold water.

It is questionable whether the small reductions in tensile strength recorded for the cellulose acetate and nylon fabrics are of significance, for these losses may be within experimental error; in any event, they are insignificant when compared with the complete decomposition of the cellulose fabrics.

2. LABRATORY JAR TESTS (See Table II):—The cellulose acetate ("Acele") fabrics are very resistant to the attack of *Chaetomium globosum, Metarrhizium, Aspergillus,* and *Penicillium;* but *Stachybotrys* developed abundantly on all four of these fabrics. The growth of *Stachybotrys* did not significantly reduce the strength of the fabric. Neither did the finishing agents greatly alter the amount of growth of this fungus, for the ether extracted sample supported approximately the same amount of growth as did the samples containing the commercial finish. The sample (No. 3) containing one to two per cent of sulfonated peanut oil appeared to stimulate the growth of *Chaetomium,* but did not result in reduced tensile strength.

The viscose rayon and completely saponified acetate samples were alike in that *Chaetomium globosum, Metarrhizium,* and *Stachybotrys* grew luxuriantly on all of these fabrics, and loss in strength varied directly with the amount of growth of the aforementioned organisms.

The nylon fabric like the cellulose acetate sample possesses excellent resistance to the cellulose destroying fungi *Chaetomium Metarrhizium.* Although *Stachybotrys* developed on both the nylon and acetate fabrics, it did not produce a significant reduction in tensile strength of either of these materials. The "Type 6" polymer film was also very resistant; whereas the cellophane film was virtually destroyed by *Chaetomium, Metarrhizium,* and *Stachybotrys.*

The cellulose acetate and nylon samples, which were exposed in these tests, were badly discolored by *Stachybotrys.* It is not clear how this organism was able to grow on the washed and ether-extracted sample of "Acele". It is known that the organism cannot grow on the liquid nutrient, which was used, and consequently, it appears that in some way it must have derived some of the essential nutrients from the acetate yarn. However, this action must be very slow for there was no appreciable loss in strength. Perhaps, if we had been able to use a larger number of replicates we could have detected minor losses in strength, but it was impossible to do this because of the size of the test. *Moreover, it is obvious that the information which has been obtained is adequate to clearly show the relative resistance of these fibers to attack by micro-organisms.*

3. STORAGE OF CELLULOSE ACETATE SKEINS AT 80°F. AND 80% R. H. (See Table III):—The "Acele" skeins were entirely resistant to mildewing through a period of seven weeks storage at a temperature of 80°F. and 80% R. H.

Random skeins were selected weekly and carefully examined for indications of fungus growth. The skeins were then conditioned and physical properties determined and compared with the original physicals before storage.

TABLE II

Comparative Resistance of Viscose Rayon, Cellulose Acetate, Nylon and Cotton Fabric to Mildew and Rot when exposed in laboratory culture jar tests

Fabric samples used 2" x 5"; Test set up 2/12/43: Test completed 2/26/43

Fabric Description	Avg. breaking strength—(2" x 5") Lbs. required to break†	Percentage of Area of Fabric Overgrown by Test Fungi and of Tensile Strength Retained After Exposure											
		% Fabric surface overgrown*	Avg. breaking strength of exposed samples (in lbs.)*	% Tensile strength retained after exposure	% Fabric surface overgrown*	Avg. breaking strength of exposed samples (in lbs.)*	% Tensile strength retained after exposure	% Fabric surface overgrown*	Avg. breaking strength of exposed samples (in lbs.)*	% Tensile strength retained after exposure	% Fabric surface overgrown*	Avg. breaking strength of exposed samples (in lbs.)*	% Tensile strength retained after exposure
		Chaetomium globosum			Metarrhizium			Stachybotrys sp.			Aspergillus Penicillium		
225 x 90 *"Acele" Cell. Acetate Satin*													
1 Washed and ether extracted	28	0	23	82.1	0	27	96.5	71	29	103.5	0	27	96.5
2 Scoured only	34	1	30	88.4	0	27	79.5	73	29	85.5	0	30	88.3
3 Commercially finished	32	19	30	94	0	29	90.7	90	31	97	5	28	87.7
4 Greige fabric	29	2	29	100	3	29	100.	57	29	100	0	29	100
112 x 66 *Viscose Rayon Twill Lining*													
5 Commercially finished	58	82	15	25.9	30	17	29.4	90	12	20.7	0	27‡	46.6
6 Washed and ether extracted	55	42	29	52.8	47	22	40.0	95	15	27.3	0	41‡	74.8
115 x 115 *Completely saponified acetate*													
7 Commercially finished	28	55	17	60.7	28	17	60.7	85	17	60.7	0	25	89.3
8 Washed and ether extracted	25	53	21	84	3	26	104.0	90	12	48	0	25	100.0
9 Cotton Control..No fungicide	62	50	42	67.8	92	45	72.5	57	56	90.5	0	67	107.0
10 1½ oz. Nylon "66 Polymer"	62	20	69	111	9	64	103	87	59	95.4	0	57	92
Films													
11 Nylon Type 6 Polymer§	22	0	17	77.3	0	21	95.7	2	18	81.9	0	17	77.3
12 DuPont Cellophane Type 600A	15	60	0	0	18	0	0	2	60	0			

* Average value for triplicate samples.
† Average for six samples.
‡ Contaminated with Stachybotrys.
§ This is an experimental type of nylon film and differs from textile nylon.

No fungus growth of any type was observed on the skeins after the full seven weeks of storage. The physical properties remained constant throughout the entire storage period.

TABLE III

Physical Properties of 75/24 Bright "Acele" cellulose acetate after storage at 80° F. and 80% R. H. for periods from 2 to 7 weeks

	Period of Storage						
	Control	*2 Wks.*	*3 Wks.*	*4 Wks.*	*5 Wks.*	*6 Wks.*	*7 Wks.*
Unscoured Skeins Containing Standard Finish Applied During Manufacture							
Denier	77.5	77.3	76.6	77.0	77.0	77.6	77.7
Dry Tenacity	96.	95.	96.	96.	94.	97.	96.
Dry Elongation	27.3	26.4	26.4	27.6	26.9	25.3	26.2
Wet Tenacity	61.	59.	58.	58.	58.	57.	58.
Wet Elongation	32.4	31.4	34.2	32.6	33.8	33.0	34.0
Dry Grams/Denier	1.25	1.24	1.25	1.25	1.22	1.25	1.24
Scoured and Ether Extracted Skeins							
Denier	77.3	77.2	77.0	77.5	77.6	78.1	78.0
Dry Tenacity	95.	94.	91.	94	94.	95.	96.
Dry Elongation	26.3	26.9	26.0	26.8	25.9	26.2	26.5
Wet Tenacity	60.	60.	59.	57.	58.	58.	58.
Wet Elongation	32.2	31.6	31.2	30.8	34.2	32.6	34.4
Dry Grams/Denier	1.23	1.22	1.18	1.21	1.21	1.21	1.23

Acetate Rayon and Nylon Have High Resistance to Fungi

The results of this series of tests demonstrate that cellulose acetate rayon and nylon possess a high degree of resistance to attack by fungi and bacteria, and possessing these properties they should withstand very well the severe exposure conditions of the tropics.

These two materials should especially fulfill the requirements for lightweight fabrics, such as nettings. We have established by repeated tests that light weight cotton fabrics i.e., balloon cloth and mosquito nettings, and viscose rayon nettings are difficult to protect with fungicide treatments. Heavy weight cotton fabrics, i.e. 8 oz. duck and heavier, can be more satisfactorily protected by a number of suitable fungicides. Therefore, the inherent resistance of yarns is of particular importance in light weight fabrics which are to be used under severe tropical exposure.

Details and Description of Tests

A standard lingerie fabric was selected as the cellulose acetate test specimen in a 225 x 90 construction using du Pont "Acele" cellulose acetate 55/18 bright yarn in the warp and 75/50 dull yarn in the filling. It was realized that cellulose acetate by itself might be resistant to certain organisms but the presence of finishing agents, sizing materials, etc., would promote the growth of the same organisms. Consequently, it was decided to run four sets of acetate samples. These sets were treated as follows:

1. Thoroughly scoured, rinsed, and ether-extracted to remove all traces of size and finish.

2. Thoroughly scoured and rinsed. This represents a commercial procedure for removing foreign material from the fabric.

3. Thoroughly scoured, rinsed, and treated with a commercial softening oil (sulfonated peanut oil, 1 to 2% on the weight of goods).

4. Greige goods as coming from the loom containing a total of 6% gelatine and sulfonated oils.

The following items were included for control purposes to be compared against the cellulose acetate items:

5. VISCOSE RAYON twill lining 112 x 66 construction from 150 denier du Pont viscose process rayon in both warp and filling. This item was commercially finished in order to include a small amount of sulfonated finishing oil.

6. VISCOSE RAYON twill lining. Same construction as No. 5 which was scoured, rinsed, and ether extracted to remove all finishes and sizing materials.

7. SAPONIFIED ACETATE, 115 x 115 taffeta construction, from a 30 denier completely saponified acetate yarn. This item contained a commercial finish.

8. SAPONIFIED ACETATE—same construction as No. 7. This item was scoured and ether-extracted to remove all traces of finishes and sizing materials.

9. COTTON balloon cloth 3.9 oz./sq. yd. was used for the laboratory culture tests and COTTON duck 8¼ oz./sq. yd. was used for the soil burial test. Both were commercially finished.

10. NYLON parachute fabric woven from 40 denier in the warp and 60 denier in the filling of nylon No. 66 polymer. This fabric contained a commercial finish, free from finishing oils.

11. NYLON film cast from Type 6 polymer and differs from textile nylon.

12. Du Pont Cellophane film, Type 600A.

DESCRIPTION OF SOIL BURIAL TEST: — Twelve samples, 6" x 4", were prepared from each of the fabric samples, which were submitted for testing. The samples were cut slightly larger than the specified dimension, but were reduced to uniform size by raveling. Six of the samples were selected at random from each of the series and kept in the laboratory as reference pieces. The other six were placed in greenhouse soil burial beds. All samples were randomized for position within the bed.

Courtesy Du Pont Co.

Soil burial beds in the greenhouse of the Pest Control Laboratory.

The soil in the test beds contain an active flora of micro-organisms, and it is maintained at a high level of activity by periodically incorporating partially decomposed leaf mold into the soil, and also by spraying the beds with a suspension of spores of active cellulose destroying fungi, molds, and bacteria. The air temperature of the room in which the beds are located varied from 75° to 85°F., while the soil temperature of the bed varied from 66° to 72°F.

The fabric samples were left in the soil beds for twenty-one days after which they were removed and tested together with the exposed reference pieces for tensile strength. These strength measurements were made by the Grab Test Method as specified by the A.S.T.M. Standards on Textile Materials. The results from the soil burial tests are summarized in Table 1.

Description of Laboratory Culture Jar Tests

A—ORGANISMS USED:—Eighteen samples, 2" x 5", were prepared from each fabric. Triplicate samples were exposed to each of the following test fungi:*

1—*Chaetomium globosum*—Kunze

2—*Metarrhizium sp.*

3—*Stachybotrys papyrogena*—Sacc.

4—*Aspergillus niger*—Van Tieg. and *Penicillium sp.* (used as a mixed spore suspension by mixing pure cultures of these organisms at time of inoculation).

The first three fungi are active cellulose destroyers and cause serious damage to the fabrics exposed under humid conditions. *Metarrhizium* has been reported to be causing considerable damage to military fabrics in the tropics, while species of *Stachybotrys* and *Chaetomium* are known to be widely distributed throughout the temperate and tropic regions of the world. *As-*

*Method used by Greathouse et al, Ind. & Eng. Chem. *14*:614-620 Aug. 1942.

pergillus and *Penicillium* are not capable of decomposing cellulose, but are often found growing on certain types of finishes. These organisms are also among the most widely distributed and abundant fungi. *Penicillium, Aspergillus,* and *Stachybotrys* also badly discolor fabric surfaces; the former organism imparting a greenish to yellow color to the surface, whereas the two latter impart a black color to the surface. All of the fungi used in this test, with the exception of *Penicillium,* represent cultures, which were obtained from the U. S. Department of Agriculture and are widely used in fabric testing work. The *Penicillium* culture is one, which we isolated and have used for a number of years in our testing work.

B—TEST CONTAINERS:—Sixteen-ounce glass jars (square in cross-section) equipped with improvised metal caps fitted with a fine mesh glass filter fabric, which permitted exchange of gases but fine enough to prevent contamination of the fabric by other organisms, were used as test containers. In order to keep the fabric to be tested in contact with the liquid medium and prevent it from being submerged, a mat of glass fabric was placed in each bottle to support the fabric sample and serve as a wick. The mats are made from glass and consequently do not support the growth of the test fungi.

C—MEDIA USED AND METHOD OF INOCULATION:—The liquid nutrient used was made up by the following formula:

	Gr/L.
K_2HPO_4	1.394
$MgSO_4$	0.739
NH_4NO_3	1.000
$CaCO_3$	0.005
NaCl	0.005
Fe, Zn, and Mn as SO_4	0.001 (each)

Sufficient of this nutrient was pipetted into each of the test containers until it came nearly up to the upper surface of the glass fabric, when the container was in a horizontal position. The caps were then placed on the jars and they were sterilized in an autoclave at 15 pounds of pressure for twenty-five minutes.

When the jars had cooled, the fabric samples were placed on the surface of the glass mat, and 3 cc. of a spore suspension of the test organism was pipetted onto the surface of the fabric. The jars were stored in a room at 80°F. and 80% relative humidity for the *two weeks* the test was run. At the end of the test the samples were sent to the Physical Testing Laboratory, where tensile strength measurements were made by the same method as described for the soil burial test samples.

Description of Storage Tests of Acetate Skeins

Ninety skeins were made up from du Pont 75/24 Bright "Acele" cellulose acetate yarn. These skeins all contained the standard finish applied during manufacture. Thirty skeins were given a thorough scour and then ether-extracted to completely remove the finish.

The thirty extracted skeins and thirty of the standard skeins were then submitted to the seven-week storage test. The thirty remaining skeins containing the standard finish were retained in the Physical Testing Laboratory for control purposes.

The sixty test skeins were stored in the culture room by being draped over a line running across the room. The area is maintained at a constant temperature of 80° F. and 80% R. H. This area is known to have a high spore population at all times, due to routine tests constantly being run. At the time the test was in progress the paint on the walls and ceiling was badly "mildewed", and consequently, spores were undoubtedly present throughout the room.

After two weeks storage in this area, five skeins were selected at random from each test item. A microscopic examination was made in an effort to determine if any fungal growth was present. The skeins were then tested for physical properties and compared against the control skeins, held for this purpose.

This procedure was repeated each week, through seven weeks of storage. At no time was there any evidence by either the microscopic examination or physical testing, of any fungus growth or degradation of the acetate yarn. Physical tests are listed in Table III.