CLVI. STUDIES OF THE BACTERIAL DECAY OF TEXTILE FIBRES.

II. A PRELIMINARY STUDY OF THE DETERIORATION OF SAMPLES OF ARTIFICIAL SILK THROUGH THE ACTION OF MICRO-ORGANISMS.

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THE investigations described in this report comprise a number of preliminary exposure tests of four different types of artificial silk to the action of micro-organisms, made with a view to determining their rate of decay. The existing literature contains only one reference to the behaviour of artificial silk when exposed to the action of micro-organisms. In this reference Dorée [1920] reports that samples of cellulose acetate silk were exceptionally resistant to the destructive action of the cellulose-decomposing micro-organisms of sea water. In view of the growing importance of the artificial silk industry, the extension of these observations to other types of artificial silk, as well as to other forms of cellulose-decomposing micro-organisms, particularly those of the soil, was thought sufficiently important to justify a renewed enquiry.

The experiments reported in the following pages should not be regarded as an exhaustive answer to this enquiry but rather as of a preliminary nature.

The available material was of a very heterogeneous nature and consisted partly of skeins of artificial silks prepared by the viscose process, the Chardonnet process and the cuprammonium process, and partly of two types of fabric, a lighter and a heavier, woven from cellulose acetate silk, which is known commercially as "Celanese." Since the number of filaments in the threads of the samples varied considerably and the thickness of the various filaments showed considerable divergencies¹ and since, also, some of the samples were

¹ The number of filaments in the threads were:

Viscose	...	...	...	...	21
Nitro	...	...	...	...	19
Cuprammonium; more than	...	...	...	...	40
Celanese (lighter) (warp and weft)	...	...	...	...	17
" (heavier) "	...	...	...	...	39

The relative diameters of the filaments were:

Viscose	...	...	...	...	0.9
Nitro	...	...	...	...	1.0
Cuprammonium	...	...	...	...	0.4
Celanese	...	...	...	...	0.6

in the form of skeins, and others made up into fabrics, it will be realised that great care had to be shown in the interpretation of changes observed in the tensile strength of the samples during exposure. Furthermore, it had to be borne in mind that some of the silks might lose more of their tensile strength than others on prolonged contact with moisture, quite irrespective of the changes caused by micro-organisms.

For the above reasons it was thought preferable when measuring the resistance of the various samples to rely on changes in the macroscopic and microscopic appearance of the filaments and on the number of micro-organisms present on them during and after exposure, rather than on changes in their tensile strength.

It is realised that these criteria are not entirely satisfactory, since they indicate the more pronounced changes only. They are, however, on that account safer to adopt for such heterogeneous material as that used than the determination of changes in the tensile strength of the filaments.

The exposure tests carried out may be divided into three groups: *A*, tests in which the samples were placed anaerobically in a food solution favouring the development of cellulose-decomposing bacteria; *B*, tests in which the samples were fixed in square frames buried vertically in a light garden soil and reaching to a depth of 30 cm., thus ensuring a thorough exposure to the action of aerobic and anaerobic cellulose-decomposing micro-organisms of the soil; *C*, tests in which the samples were fixed in square frames and submerged in sea water, conditions which in previous experiments had been found to ensure exposure to the action of the cellulose-destroying micro-organisms of the sea.

The observations made in these various tests are recorded below in tabular form.

Series A. Rate of destruction of artificial silks exposed to the action of a culture of cellulose-decomposing micro-organisms.

In this series the various samples were placed in 500 cc. flasks filled with a medium consisting of peptone 0.1 %, di-potassium hydrogen phosphate 0.1 %, magnesium sulphate 0.1 %, sodium chloride 0.1 % and ammonium sulphate 0.2 %. Each flask was inoculated with 2 cc. of a culture containing anaerobic cellulose-decomposing bacteria and incubated at 37°.

Under the conditions of this series of experiments the cellulose acetate silk and the nitro silk were probably not attacked by the anaerobic cellulose-decomposing bacteria used. The sample of viscose silk showed less resistance to these forms than the first two mentioned but was still considerably more stable than the cuprammonium sample, which deteriorated completely within a fortnight, an even faster rate than that shown by a sample of raw cotton, of Indo-American origin, which, during that time, had been damaged to 57 %, estimated by the swelling test [Fleming and Thaysen, 1921].

Table I.

Days' exposure at 37°	Cuprammonium silk	Viscose silk	Chardonnét silk (nitro silk)	Celanese silk (cellulose acetate silk)
5	Sample is reduced to a pulp. The filaments show bad fraying. Sulphuretted hydrogen is evolved	Sample is yellowish, discoloured. The filaments show some fraying. Sulphuretted hydrogen is evolved in small quantities	Sample is not discoloured; there is no evidence of damage to the filaments. Sulphuretted hydrogen is evolved	No change
13	Only few filaments remain. Sample discarded	Sample is discoloured and slimy. The edges of the filaments are more jagged than before	Sample still undamaged	No change
19	—	Little change	Sample still undamaged	No change
26	—	The filaments are getting appreciably weaker and their edges more jagged	No change; re-inoculated with a fresh culture of cellulose-destroying micro-organisms	No change; re-inoculated with a fresh culture of cellulose-destroying micro-organisms
33	—	No further change; re-inoculated	No change; re-inoculated	No change; re-inoculated
40	—	The filaments now form a slimy mass	No change; re-inoculated	No change; re-inoculated
47	—	Little remains of the filaments. Sample discarded	No change; re-inoculated	No change; re-inoculated
103	—	—	Some of the filaments feel a little weaker and are discoloured slightly yellow; re-inoculated	No change; re-inoculated
138	—	—	Though weaker, the filaments show no microscopic signs of decay; re-inoculated	No change; re-inoculated
200	—	—	No further change	No change

Series B. Rate of destruction of samples of artificial silk buried in a light garden soil.

The rate of decay of the samples exposed in the soil was of the same order as that observed in the laboratory anaerobic experiments, the cuprammonium sample going first, the viscose sample next. In the soil, however, the nitro sample was also affected and its resistance was only a little greater than that of the viscose silk. The cellulose acetate silk remained unaffected even after 30 weeks' exposure. When examined after 27 weeks, the heavier cellulose acetate fabric showed a greenish patch about 5 cm. from the top. On microscopic examination this was found to be due to a green coenocytic filamentous alga. The fabric was not damaged, however, and remained normal throughout the test. That this resistance was not due to the absence of cellulose-decomposing micro-organisms was shown experimentally by the inoculation of flasks, such as those already described, containing filter paper and food solution with pieces of the exposed fabrics added. The filter paper of these flasks was invariably found to be decomposed after 8-14 days' incubation.

Table II.

Weeks' exposure in the soil	Cuprammonium silk	Viscose silk	Nitro silk	Celanese silk Light and heavy fabric
2	No change	No change	No change	No change
3	Some of the filaments show frayed edges and on gentle pulling the threads break at a spot about 15 cm. from the surface of the soil	Many of the filaments beginning to fray at the edges. The threads are discoloured pink	Some of the filaments beginning to fray at the edges	No change
4	The filaments show much fraying. The threads break fairly easily on gentle pulling	The fraying has not advanced very much. The threads are nevertheless very easily broken. The pink discoloration is intensified	Many of the filaments now show fraying at the edges. The sample is nevertheless still strong and does not break easily	No change
5	The fraying at the edges of the filaments has increased. The threads break at the slightest touch	No appreciable change	No appreciable change	No change
6	In a very poor condition; the bulk of the sample is a slimy mass. Sample discarded	Slightly more pronounced fraying at the edge of the filaments	Perhaps slightly more fraying at the edges of the filaments	No change
7	—	The filaments are more markedly frayed and the threads break easily on gentle pulling. The threads feel slimy	Stronger than the viscose sample; the fraying of the edges of the filaments is more marked than last week	No change
8	—	Markedly weaker. The filaments are now a slimy mass. Sample discarded	The threads are fairly resistant though the fraying of the edges of the filaments has increased	No change

The various samples exposed in the soil were subjected to a bacteriological analysis on each examination in order to determine whether an increase in the secondary micro-flora of these samples occurred during exposure. As is known, it is not possible, or at least is very difficult, to isolate cellulose-decomposing bacteria in pure culture; the enumeration of these organisms on a given sample of material is therefore not feasible. But an increase in the readily cultivable secondary micro-flora, which under natural conditions is associated with the cellulose-decomposing bacteria and lives on their decomposition products, cellobiose and glucose, indicates an activity of the latter. A sudden rise therefore in the numbers of this secondary flora can generally be regarded as a sign of an increased activity of the cellulose-decomposing micro-organisms, particularly when no other cause for an increase is apparent, such as the addition of food materials or seasonal changes in the soil flora.

For the bacteriological analyses 1 g. of the material to be examined was shaken vigorously for 2 hours with 9 cc. of physiological saline solution and

this standard suspension plated out on the following media: ordinary nutrient agar, wort agar, cellulose agar prepared according to the formula of McBeth and Scales [1913], nutrient gelatin, and glucose agar under anaerobic conditions (Burri tubes).

The result of these analyses showed that there was a marked rise in the numbers of the secondary micro-flora in each case, the rise being noticeable first in the cuprammonium silk, a week later in the viscose silk, and three weeks later in the nitro silk. A slight increase in the secondary micro-flora of the cellulose acetate silk took place nine weeks after the increase had occurred in the cuprammonium silk.

The presence of both aerobic and anaerobic cellulose-decomposing bacteria on the various samples of exposed artificial silks was confirmed experimentally by inoculating samples of the exposed silks into a medium containing filter paper and suitable for the growth of cellulose-fermenting bacteria. In every case, even where exposed Celanese silk was used, the filter paper became decomposed.

*Series C. Rate of destruction of samples of artificial silk submerged
in sea water.*

The samples of series *C* were fixed in square aluminium frames and submerged in the sea at the jetty of the Royal Naval Cordite Factory, at a place where previous investigations had shown cellulose to become readily destroyed.

In Table III (p. 1093) the observations made in this series of exposure tests are recorded.

The rate of decay of all samples was markedly faster in the sea than in the soil. This on the whole confirms previous observations made with other types of textiles. More noteworthy is the fact that the difference in the rate of decay between the first three artificial silks named has been almost completely obliterated in the sea exposure tests. The only marked resistance to decay is that observed in the cellulose acetate samples. The enumeration of the secondary micro-flora present on the decaying samples of this series offered little information of interest, and did not show the increase observed in the case of the samples buried in the soil. It was established that both aerobic and anaerobic cellulose-decomposing micro-organisms were present on all the submerged samples which showed definite signs of decay. Before such decay had commenced, cellulose-decomposers were absent from the textiles.

CONCLUSIONS.

From the experimental data collected in the three series of exposure tests it may fairly safely be concluded that the samples of cellulose acetate silk tested showed the greatest resistance to micro-biological destruction. Dorée's observations have thus been confirmed. Evidence has also been obtained of

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a varying degree of resistance among the other types of artificial silk examined, the order of merit of the samples tested being nitro silk, viscose silk and cuprammonium silk.

Table III.

The temperature of the water varied between 11.5°–14.0° during the exposure trials.

Weeks' exposure of samples in the sea	Cuprammonium silk	Viscose silk	Nitro silk	Cellulose acetate silk	
				Light fabric	Heavy fabric
1	Most of the filaments show frayed surface	Same as cuprammonium sample	Same as cuprammonium sample	No change	No change
2	The fraying of the surface of the filaments is much more marked. The threads break fairly easily	The fraying of the surface of the filaments has increased but the sample does not break as readily as the cuprammonium sample	Same as viscose sample	No change	No change
3	Sample almost completely destroyed	The destruction of the filaments has progressed; the sample is however distinctly superior to the cuprammonium sample	Same as the viscose sample	No change	No change
4	Sample reduced to a slimy mass	Sample almost completely decomposed	Same as the viscose sample	No change	No change
9	—	—	—	No change	A few of the filaments show frayed surface
10	—	—	—	Though the fabric tears fairly readily there is no sign of damage to the filaments	The number of filaments showing frayed surface has increased
11	—	—	—	No further change	Considerably worse. The damage to the filaments is more pronounced and the fabric tears readily
12	—	—	—	Both samples are now badly decayed. The thinner fabric is still superior to the heavier and shows a considerable number of normal filaments	

In endeavouring to account for this difference, it strikes the observer that the most resistant form of artificial silk is that in which the cellulose is present as a fully saturated ester. The sample next to this, the nitro silk, is one in

which the cellulose had been at one time a fully saturated ester. In the viscose silk the cellulose may have been esterified but certainly not completely. In the least resistant sample, the cuprammonium silk, the cellulose was present in a hydrated form which had not in any way been chemically combined with acids. It would appear desirable to determine how far a preliminary esterification of cellulose offers a means of protecting it against micro-biological deterioration.

Another point which has undoubtedly to be considered in endeavouring to elucidate the difference in resistance shown by the various artificial silk samples, is their crystalline nature. Of the four types included in the experiments the cellulose acetate silk has been definitely shown to be of an amorphous nature [Herzog and Jancke, 1920], whilst cellulose precipitated from cuprammonium solution and viscose silk are both crystalline, like untreated cellulose. The structure of nitro silk is, it is believed, as yet undetermined.

In this connection it is interesting to recall that a difference in resistance to enzymic decomposition was observed by Pringsheim and Aronowsky [1922] between ordinary inulin and inulin which had been obtained from inulin acetate by hydrolysis. Of these, the former was crystalline and the latter amorphous.

It is intended to continue the study of the resistance of artificial silks from the two above-mentioned points of view.

SUMMARY.

The rates of destruction of four different types of artificial silks on exposure to the action of micro-organisms have been studied. It has been established that the rate varies considerably, being slowest in the case of cellulose acetate silk and quickest in the case of cuprammonium silk.

This difference may possibly be due either to physical or to chemical differences in the various types of artificial silk.

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REFERENCES.

- Dorée (1920). *Biochem. J.* **14**, 709.
Fleming and Thaysen (1921). *Biochem. J.* **15**, 407.
Herzog and Jancke (1920). *Ber. deutsch. chem. Ges.* **53**, 2162.
McBeth and Scales (1913). U.S. Dept. of Agric., Bureau of Plant Industry, Bulletin No. 266.
Pringsheim and Aronowsky (1922). *Ber. deutsch. chem. Ges.* **55**, 1414.