

Comparative Studies on Cellulose Acetate

By H. STADLINGER, CHARLOTTENBURG, GER.

(Concluded from November issue, page 1113)

The tables appearing in the first part of this article (pp. 1110-1113 Nov. issue) may be subject to the following criticism:

1. Tensile Strength

All determinations were made with an electrically working precision apparatus manufactured by L. Schopper,* Leipzig. Length of the thread 50 cm. Relative room humidity 60%. Temperature 20° C. The given figures pertain to two strands each A and B after the oil was removed and represent the average of 10 single tests in each case.

All tests were checked and also carried out as control tests with the oil not removed.

In the tables, however, are only compiled the tests made with the rayon after the removal of the oil, as the oil contents of the five different brands varied considerably (in the average 1.35%; 3.55%; 2.8%; 4.15%; 2.75%) the contents of oil would influence the actual tensile strength of the acetate thread.

As can be noticed, in the table are given four different values for the tensile strength:

- (a) The tensile strength of the dry material
- (b) " " " " " wet material
- (c) " " " after boiling
- (d) " " " " knotting.

Every one of the test values referred to has its special meaning in the practical application of acetate.

(a) Tensile strength of the dry material:

The tests have revealed the following: I "Setilose" and II "Celanese" have about the same tensile strength (1.23 per den.) both of which are considerably lower than the tensile strength of IV German "Rhodiaseta" and V "Aceta" (in the average 1.34 g. per den.). III French "Rhodiaseta" with 1.25 g. per den. represents about the middle value.

(b) Tensile strength of the wet material:

The reported figures were here obtained in testing the wet cellulose acetate after removal

of the oil, after ½ hour immersion in water. The analytical picture shows a gradual ascending of the values for the tensile strength from I to V, in such a way that brand I "Setilose" and II "Celanese" show the lowest, brand V "Aceta" the highest tensile strength in the wet state. The two "Rhodiaseta" brands III and IV show with equal strength middle values.

(c) Tensile strength after boiling:

We want to emphasize right here the importance of this test for the estimation of acetate cellulose. Before the breaking test is made the rayon is placed in boiling water and subjected for 30 minutes to the boiling temperature so that the strand is nowhere in contact with the walls of the vessel. Then the threads are carefully taken out of the boiling vessel and are left hanging in a room of 60% humidity by volume at 20° C. for a sufficiently long drying period. The dry threads are then being tested for (breaking) tensile strength, gloss and surface changes (see below).

From the boiling test the acetate silks I "Setilose" and II "Celanese" had doubtless suffered the most, because their tensile strength had decreased from 1.22 g. for the dry state to 1.08 g. for the wet material. Contrary to this decrease in strength brand V "Aceta" had in fact slightly increased for it showed 1.36 g. as compared with 1.33 for the dry rayon.

The two "Rhodiaseta" brands III and IV descended from 1.32 g. and 1.22 g. to 1.25 g. relatively 1.12 g.; they again represented the middle values for the wet tensile strength. A high tensile strength after boiling is, of course, of great importance where acetate is used in weavings of mixed or such textile fibers which require a high temperature in the dyeing process, as f.i. mixed fabrics of acetate and wool, which are dyed at high temperatures; furthermore, fabrics of acetate silk and real silk, which have to be degummed by boiling with hot soap solution.

It is, therefore, a matter of great importance to define "fastness to boiling" exactly at the

* American representative: H. Z. Schniewind, 72 Duane Street, New York, N. Y.

coming standardization of rayon, especially as this analytical-physical test does not meet with difficulties.

silks have about the same knotting strength 1.05 to 1.10 Rayon V, however, "Aceta" tops these brands with a knotting strength of 1.15.

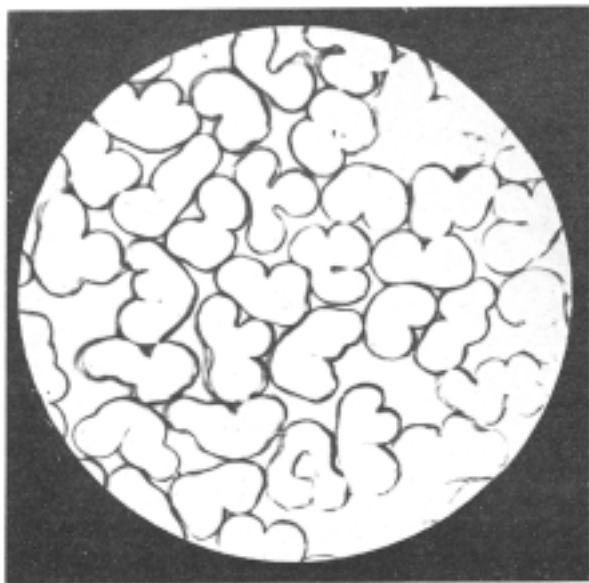


Figure 1
Cellulose Acetate "Setilose" (Belg.)

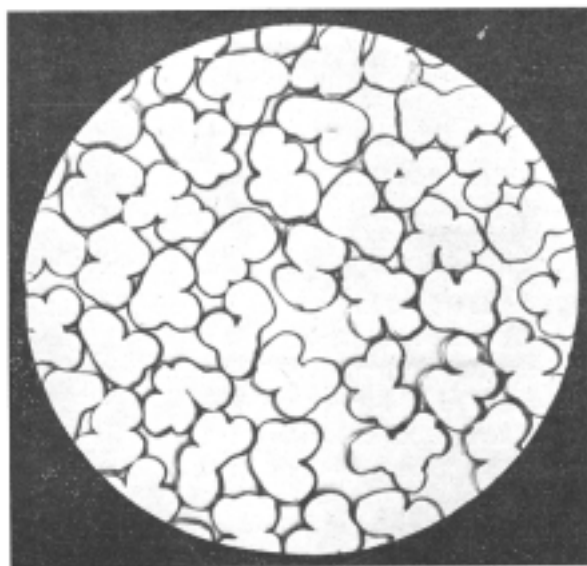


Figure 2
Cellulose Acetate "Celanese" (Eng.)

(d) *Knotting strength:*

This qualification—as far as I know not yet introduced in the rayon literature—shall give an idea about the strength of the threads in the mechanical processes, for instance, especially in the knitting process, where an elastic and pliable thread is of utmost importance. Practical tests have demonstrated that the breaking strength and breaking elasticity furnish a very useful estimation for the tensile strength and elastic properties of acetate silk. These breaking tests are based on the idea that the thread systems as they appear in knitted goods are subjected to highest strain on the knotted point. It is a proven fact indeed that in the pulling test the majority of threads will give way at the knots. A high knotting strength will, therefore, create a favorable opinion on the behavior of acetate silk in the knitting and those processes where rayon is subjected to rough mechanical handling.

The analytical picture shows us that rayon I "Setilose" with a knotting strength of 0.98 is a trifle lower than brand II "Celanese" as well as III and IV Rhodiaseta. These three acetate

2. Breaking Elasticity

Under breaking elasticity is understood the prolongation of a thread in percent of its original length which occurs before breaking. A good acetate silk in dry state should have not less than 22% breaking elasticity, if it is to withstand the mechanical attacks in weaving and more essentially in the knitting process. On the other hand, a further stretching above 28% is not desired, as overstretched threads may give cause to glossy lines.

The five tested brands show with 25% to 27.2% dry breaking elasticities throughout normal values (real silk possesses about the same breaking elasticity).

Far greater are the differences between the figures obtained for wet breaking elasticity. The lowest value of 32.7% was found in the case of I "Setilose" and IV German "Rhodiaseta." Brand V "Aceta" furnished a value of 37.2%. A true picture may be obtained from the percentual increase between the values of dry and wet breaking elasticity.

The figures found:

I	II	III	IV	V
19.5%	23.4%	38.5%	33.0%	39.0%

demonstrate that brand I "Setilose" has gained the least, while brand V "Aceta" has increased to the greatest extent. As I "Setilose" (and similarly also II "Celanese") has in the wet state the lowest breaking strength and the lowest boiling strength, and as "Aceta" V has shown in this regard the highest figure, the conclusion suggests itself, that in acetate silks an increase in the wet tensile strength augments in proportion the breaking elasticity in the wet state.

4. Sorting Results

In general the requirements of acetate silk should be higher than for any other rayon as far as sorting is concerned. The acetate plant is in a much better position to produce only the highest quality threads, because it can return into the process material not entirely satisfactory, a procedure not possible with other type rayon, i.e. Viscose, Cuprammonium or Chardonnet. When tested under the sorting-light IV "Rhodiaseta" and V "Aceta" did not show capillary fractures. The French "Rhodiaseta" III showed a number of burst capillary

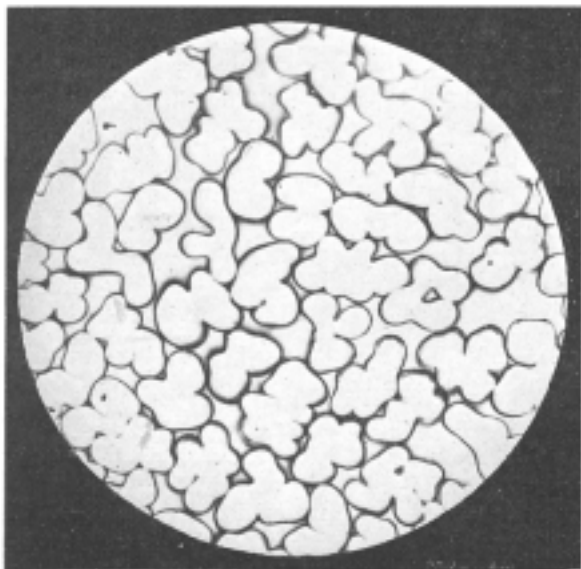


Figure 3
Cellulose Acetate "French Rhodiaseta"

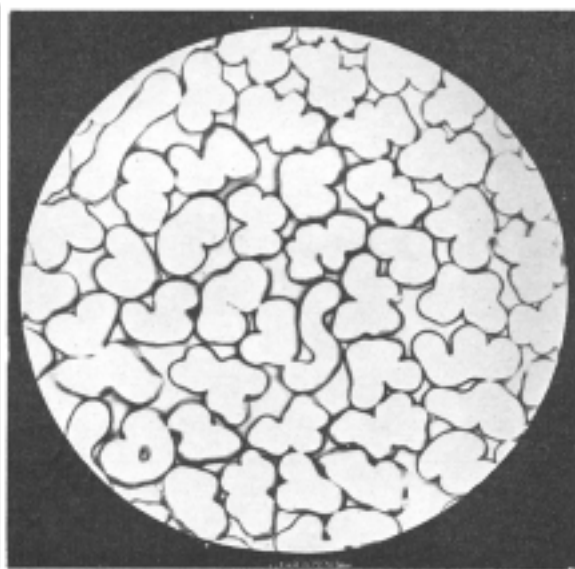


Figure 4
Cellulose Acetate "German Rhodiaseta"

As far as the elasticity of the wet and afterwards dried acetate is concerned no differences could be ascertained in any of the five tested samples.

In general the percentage of prolongation is near 20 and the French "Rhodiaseta" III with an elongation of 21.5% is somewhat out of the range. On the other hand the loss in value for knotting strength of 14.6—15.8% of the German "Rhodiaseta" IV is surprising.

3. The Titer

In all cases the claimed titer is practically attained. The differences observed are all within limit, only "Celanese" II differs to a greater extent in the five tested strands.

threads which increased in number in throwing. In some places the single fibers were interwoven like nests. More favorable were the results in the case of I "Setilose" and II "Celanese"; but also here a number of burst capillary threads could be found.

Of interest is the observation that these latter types show an electrical charge in throwing. During the cutting process for velvets this property is sometimes a most disagreeable interference. This electrical charge could be traced to the lack of oil, as the sample I tested showed the lowest oil content (1.35% average). As the Celanese II with a considerable higher oil content (3.55% average) shows also the electrical phenomenon in throwing other

factors, f.i. the kind of oil also seems to be responsible for that.

5. Feel, Gloss and Color

The ideal product as to gloss, etc., is, of course, the one which comes nearest to the natural silk having a soft feel, and an even quiet and mellow gloss. In this regard the



Figure 5
Cellulose Acetate "Aceta" (Berlin, March, 1930)

samples tested rank as follows: IV German "Rhodiaseta," I "Setilose," V "Aceta," II "Celanese," III French "Rhodiaseta."

As to the color only small differences exist. Almost perfectly white are the brands I, IV, and V, while the types II and III possess a yellowish cast.

6. Ultraviolet Fluorescence

As all fluorescence colors are—as is known—partly hidden in case the material is coated with oils or other preparations, all samples of acetate silk were examined—

- (a) in the original state
- (b) after extraction with ether.

It was found that indeed certain differences were noticeable, which can be seen at a glance at the above table. (See Nov. issue, p. 1112). A practical value can hardly be attributed to these characteristics.

7. The Boil-Test

Already in one of the above paragraphs it was pointed out that the boil-test, that is subjecting acetate yarn to a boil in distilled water for half an hour, furnishes valuable information regarding quality. A good resistance to boiling does not only warrant a high degree of tensile strength but also a good stability as to

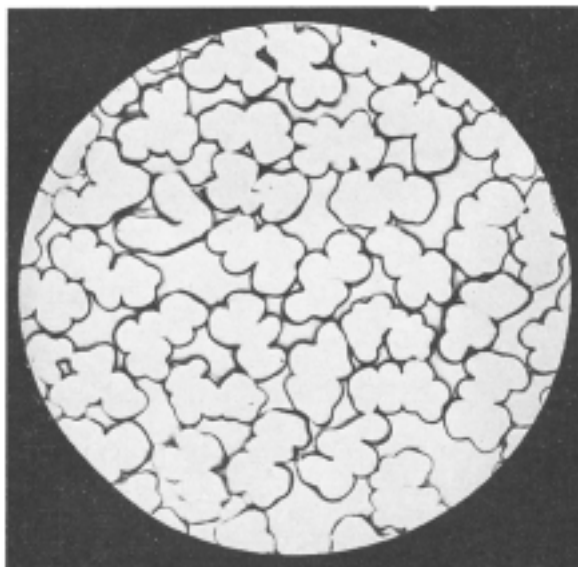


Figure 6
Cellulose Acetate "La Soie de Clairoux S.A." (France)

gloss and smoothness of the thread. No. III French "Rhodiaseta" shows the greatest decrease in gloss. This is less noticeable with I "Setilose" and IV German "Rhodiaseta."

In the dyeing process of mixed fabrics where an extremely hot treatment is often required a high resistance to boiling is of immense value.

8. Dyeing Properties

These experiments were limited to dyeing tests made with a mixture of

- 3 parts Cellit-fast-blue A
- 1 part Cellit-fast-yellow R
- 1 part Cellit-fast-red BB

which mixture furnishes a mauve shade, and it was to be observed whether this mixture goes on the fiber evenly.

The dyeing was preceded by a scouring with a solution of 2 gr. Marseille soap per liter and 1 cc. of ammonia 25%. The goods were en-

tered at 38-40° C. moved for 20 minutes, then rinsed with soft water. The dyeing was carried out with 3% of the above combination calculated on 100 parts of material. The goods were entered lukewarm (38-40° C.) with an addition of 30% (of the weight of the goods) calcined Glauber's Salt, the temperature was slowly raised to 60° C. and the material moved $\frac{1}{2}$ to $\frac{3}{4}$ of an hour at 60-70° C. Then rinsed and dried. During this dyeing process none of the five samples showed an uneven exhaustion of the dyes and the resulting dyeings were level. Only in the shade as well as in the feel and gloss of the dried material certain differences could be ascertained.

also a guarantee that the quality is up to standard.

This is especially true in the case of acetate cellulose. If the pictures are compared with results obtained and compiled in the tables two principal types of cross-sections can be distinguished:

(1) The fuller forms of a clover leaf type having three to six notches being of a more round appearance (see picture 5, brand "Aceta").

(2) The ribbon or flat type having in many cases club-shaped forms of different thickness (see especially pictures 6, 7, 8).

It is obvious that this difference must be of

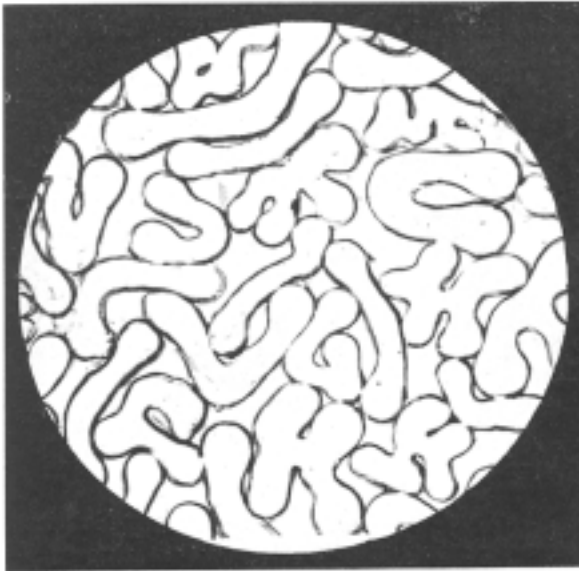


Figure 7

Cellulose Acetate "Brit. Apex Art. Silk" (Eng.)
140 den.; 24 filaments per thread; twist, 190; breaking strength: dry, 1.02 gr., wet, 0.57 gr. p. den.



Figure 8

Cellulose Acetate "Enka-Drya" (Holland)
100 den.; 20 filaments per thread; twist, 214; breaking strength: dry, 1.15 gr., wet, 0.59 gr. p. den.

I "Setilose" showed the highest affinity, followed by II "Celanese"; then came with normal affinity the three other brands.

3: Cross-sections

A modern rayon plant is incomprehensible without a continuous microscopical control of the cross-section of the fiber, because the microscopical picture does not only allow far-reaching conclusions whether the manufacturing process is running smoothly, but furnishes

decisive importance for the light reflection of the single fibers and hence for the gloss of the finished fabrics. A flat ribbon-shaped type of rayon will reflect the light principally in one direction similar to a mirror and will, therefore, furnish a more or less glassy gloss. Contrary to this a fiber of an irregularly curved and notched surface will partly reflect and partly absorb the light, so that a quiet and mellow gloss results.

How greatly the cross-sections of acetate

have changed during the last few years is shown by a comparison of acetate cross-section pictures by A. Herzog from the years 1925 and 1926.

The following picture (Figure 9) shows the cross-sections of "Aceta" as observed by him in 1925. Contrary to these microphotographs the latest photographs (Figures 1-5) demonstrate that the fuller multi-lapped forms and rounder types prevail in today's leading acetate brands, while the flat biscuit-shaped forms are by far in the minority.

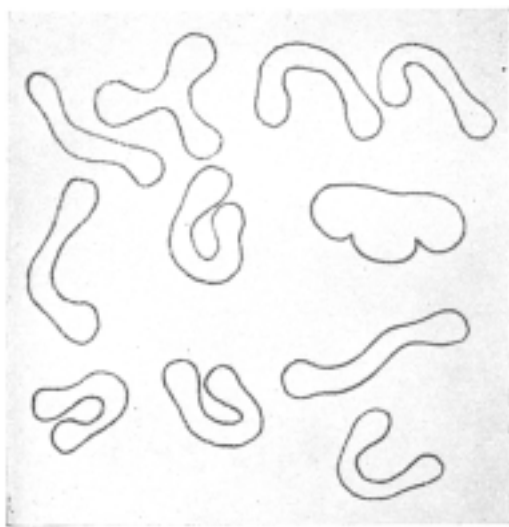


Figure 9. "Aceta" after an illustration by A. Herzog from the year 1925. (Cross-sections are different today.)

This corresponds with the endeavor of the manufacturers to avoid the ribbon-type as much as possible and to obtain filaments of the multi-lapped round shape. The microphotographs shown in recent works, therefore, have to be considered as partly pertaining to history showing pictures of typical ribbon shape. That these types are, however, still being produced demonstrate the pictures 6, 7, and 8.

10. The Acetic Acid Contents

Much has been written regarding the degree of acetylation, especially whether we have before us mixtures of different hydroacetates, for instance, mixtures of di- and tri-acetates. This question has not yet found a final solution. According to newer theories the technical cel-

lulose acetates represent a mixture of celluloses of a different degree of acetylation.

As shown in the table the five analyzed samples contain between 53.2% to 54.9% acetic acid. Brand V "Aceta" has the highest content with 54.9%, brand I "Setilose" the lowest with 53.2% acetic acid. The relation of these acetic acid contents with the better qualities, for instance, the high boiling resistance of the first mentioned brand of acetate silk, shall not be further discussed here. The conclusion that the qualities have some relation to the acetic acid contents is, in any case, obvious.

11. Viscosity

The relative viscosity numbers—determined according to Ost in a 2% acetone solution—are rather low in cases of acetate silks I and II, III and IV (5.70%, 5.80%, 5.55% and 5.75%) while V "Aceta" furnished a very high viscosity value with 8.85%. It may be concluded from these results that the acetylation of the raw material of the last mentioned product must have been carried out with greatest care, in order to prevent as far as possible a decomposition of the cellulose molecule. It is further safe to assume that this fact has also some influence on the high tensile strength in dry and wet condition of the "Aceta" which in this property surpasses all other brands tested.

12. Oil Content

Acetate filaments require a moderate lubrication as a first after-treatment with a non-drying and non-saponifiable oil right after they have left the spindles.

The five tested samples varied in their oil contents quite considerably. The lowest in this respect was "Setilose" with an average oil content of 1.35%, the highest IV German "Rhodiaseta" with an average of 4.15%.

That an insufficient lubrication may cause an unusual strong electrical charge has already been mentioned. The manner in which the oiling process is carried out may also have some influence.

Conclusions

Summing up this analytical and research work the following conclusions may be reached:

(1) The cross-sections of the better known acetate brands have in the course of years undergone such changes that the ribbon-formed shape has disappeared to give room to the fuller multi-lapped shapes.

(2) Ribbon-shaped filaments yield fabrics of a disquiet sparkling gloss, while the fuller formed type furnishes an even, mellow and quiet gloss, similar to real silk.

The disadvantages of the ribbon-formed type appear also in the dyeing of the fibers especially in very dark colors, for instance, in Navy Blue or Dark Black dyeings, as in this case due to reflection the impression of unevenness in dyeing is created.

(3) In the case of the five tested rayons I-V, values for the dry tensile strength were found sufficiently suitable for practical requirements the more if the values of the breaking elasticity are also considered. These values are of great importance especially for the knitting process.

(4) The wet tensile strength and the resistance to boiling show in general a tendency of an improvement from I towards V.

Of interest in this case is the fact, that the last mentioned rayon also possesses the highest degree of acetylation and the highest viscosity. This permits the conclusion that certain physico-chemical connections exist, even admitting that also other factors than the spinning methods apparently play a part in the final quality of the product.

(5) Boiling in water furnishes a valuable criterion. The changes taking place influence

working methods, tensile strength and breaking elasticity as well as gloss and smoothness of the thread. A curling wool-like appearance on the surface of the threads can also be observed here.

(6) Also the knotting strength in connection with the knotting elasticity allows conclusions as to the technical usefulness of the product. This pertains especially to such working methods where rayon is subjected to rough handling and bending, such as is necessary f.i. in knitting.

(7) In the dyeing all five rayons show a good affinity to Cellit-fast-colors; only in feel and gloss certain differences were discernible, in favor of the brands "Rhodiaseta" and "Aceta."

(8) The tests under ultraviolet light did not disclose any pronounced criterion for the quality.

(9) The titer of four acetate silks has proven to be very stable, only in the case of "Celanese" II a marked difference could be stated.

(10) In sorting acetate silk, it is of importance that a minimum of burst capillary threads is found, as these may cause faulty spots and other trouble in the weaving process. Still more objectionable are those capillary thread-ends on account of forming "nests."

(11) The oil content of the five tested samples varied from 1.3% to 4.3%. Too low an oil content or a non-suitable oil may possibly be the cause of an electrical charge.