# Mechanical Properties and a Physical-Chemical Analysis of Acetate Yarns

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Cellulose acetate used in the manufacture of acetate yarns is commonly obtained from cotton-linters or wood-pulp cellulose. Varying in the origin and in the manufacturer, cellulose acetate often differs in its processability. The paper belongs to the investigation the properties of acetate yarns manufactured of the cellulose acetate varied in its origin and manufactured by different suppliers. Mechanical properties (including stress relaxation and frictional behaviour) of acetate multifilament yarns as well as physical-chemical properties of acetate fibre substance are investigated. Regular discrete relaxation time spectrum of acetate yarns is determined using the generalized Maxwell model. Experiments showed that the origin of cellulose acetate does not have great influence on mechanical properties of the acetate yarns, however, the yarn manufactured from wood-pulp-originated cellulose acetate have little advantage against others in strength and extensibility, and also are more uniform in the properties. Physical-chemical analysis revealed that the fibre of wood-pulp-originated cellulose acetate has a higher degree of polymerization and a little higher amount of low-molecular mass fractions. Surface friction of the yarns lubricated with different lubricants is also investigated.

*Keywords*: cellulose acetate, yarn, mechanical properties, stress relaxation, Maxwell model, friction, degree of polymerization.

## INTRODUCTION

Cellulose acetate fibres amounts approximately one tenth of the textile-purpose man-made cellulosic fibres and about 3.8 % of all man-made fibres [1]. The production of man-made cellulosic fibres is on certain decline as several viscose fibre producers have been eliminated during last decades. This is due to the fact that a viscose process is a real burden for the environment and the operating personnel [2]. With the situation a role of acetate fibre in everyday textile usage becomes especially significant. World production of acetate multifilament yarns amounted over 300 thousands tons per year in 1998 [3], and it further continues to increase in $(2 – 3)$ % annually.

Acetate yarns are used in both weaving and knitting: for women's, men's and children's wear, linings and home furnishings. The products made from acetate yarns are comfortable as they breathe and transport moisture away from the body, they present no problems with static electricity, are fully biodegradable, have good drape, pleasant handle, "silky" gloss. They are easily dyed and printed with various colours, do not shrink after washing and do not form pills on the surface. At present time acetate fibre is one of the softest fibres available.

Cellulose acetate used in the manufacture of acetate fibre is obtained from natural cellulose, cotton fibre and cotton linters or wood-pulp being its major source [4, 5]. At present time the choice of these raw materials is market-led. However, the raw materials, varying from one supplier to other, differ in their processability and may have influence to the characteristics of spun thread.

The objective of this research is to identify and compare the mechanical (including time-dependent and frictional behaviour) and physical-chemical properties of acetate yarns made from cellulose acetate of different origin. Special attention is concentrated on impact that the degree of the polymerization and the amount of low-molecular mass fractions in the fibre may have influence to mechanical properties of yarns [6].

## THE INVESTIGATED MATERIALS

A set of multifilament air-jet intermingled acetate yarns, manufactured in Kaunas based joint-stock company "Dirbtinis pluoštas", was chosen for the investigation (Table 1). The yarns were lubricated by lubricants Silastol S1 (Germany), A-1 (Russia) and VIPOIL-AVMP (Italy).

**Table 1.** The investigated acetate yarns

| Yarn code | Nominal linear density, tex | Producer of cellulose acetate | Lubricant |
|---|---|---|---|
| CAA | 16.6 | Celanese (USA) | Silastol S1 |
| CAF | 16.6 | Azot (Uzbekistan) | Silastol S1 |
| CAE | 16.6 | Engels (Russia) | Silastol S1 |
| CAA1 | 16.6 | Celanese (USA) | A-1 |
| CAA2 | 16.6 | Celanese (USA) | VIPOIL-ACMP |
| CAA3 | 8.4 | Celanese (USA) | without lubricant |
| CAA4 | 8.4 | Celanese (USA) | Silastol S1 |
| CAA5 | 8.4 | Celanese (USA) | A-1 |

The origin of cellulose acetate used in the manufacturing of the yarns with codes CAA and CAA1 - CAA5 was wood-pulp cellulose while the cotton cellulose was the origin of cellulose acetate used in the manufacturing of the yarns CAE and CAF.

## EXPERIMENTAL METHODS

**Mechanical properties.** All experiments were carried out in standard atmospheres for testing according to the standard ISO 139.

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Tensile properties of the yarns were determined using universal testing machine "Zwick/Z005". The stress-strain curves of the yarn were obtained following the standard ISO 2062.

Coefficient of kinetic friction of the moving acetate yarn against the steel frictional cylinder with polished chromium surface was measured by two different methods: on "F-Meter" (Rothschild Instruments), and on "Buckle-Pollitt Tester" [7]. In the measurements by "F-Meter" the moving yarn speed was 50 m/min, and in the measurements by "Buckle-Pollitt Tester" – 100 m/min.

The measurement of coefficient of friction ($\mu$) of the moving yarn is based on Euler's formula:

$$\frac{T_2}{T_1} = e^{\mu\theta}, \qquad (1)$$

where $T_1$ is the run-on tension of the yarn; $T_2$ is the run-off tension of the yarn; $\theta$ is the contact angle with the frictional cylinder.

Viscoelastic behaviour of the yarns was revealed by means of stress relaxation tests. The gauge length in the tests was 800 mm. The yarns under pretension of 2.4 mN/tex were subjected to constant rate extension of 1.25 %/s up to the elongation level $\varepsilon_t$ and sustained at this constant level. The elongation levels were different: $\varepsilon_t = 1, 2, 5, 7,$ and 10 %. Stress relaxation was observed and registered from the initial instant as the elongation reached the level $\varepsilon_t$ ($t^* = 0$) up to the time $t^* = 1000$ s.

**Calculation the relaxation time spectrum.** Regular discrete relaxation time spectrum [8] was calculated from the experimental relaxation curves of the yarns using the generalized Maxwell model consisting of a set of Maxwell units connected in parallel. To represent equilibrium stress on the model, a single Hookean spring is also connected in parallel to the set of Maxwell units (Fig. 1).
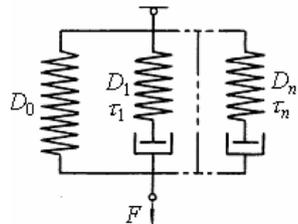


**Fig. 1.** Generalized Maxwell model

Stress relaxation of the constant rate $v_t$ strained model at $\varepsilon_t =$ const is defined by

$$F = D_0\varepsilon_t + v_t\sum_{i=1}^{n} D_i\tau_i G_i\exp\left(-t^*/\tau_i\right), \qquad (2)$$

where $G_i = \exp\left[-\varepsilon_t/(v_i\tau_i)\right]$; $D_0$ and $D_i$ are the elasticity constants of the model springs; $\tau_i$ is relaxation time of the $i$-th Maxwell unit ($i = 1, 2, ..., n$).

As in stress relaxation tests the time $t^*$ approximately covered five orders, i.e., from $t^* = 0.1$ s to $t^* = 1000$ s, we included five Maxwell units in the model ($n = 5$) and a priori determined the following their relaxation times:

$\tau_1 = 0.1$ s, $\tau_2 = 1$ s, $\tau_3 = 10$ s, $\tau_4 = 100$ s, and $\tau_5 = 1000$ s.

Differing the values of relaxation times of the Maxwell units exactly by one order, the model possesses regular discrete relaxation time spectrum. The model coefficients

$D_0$ and $D_i$ were calculated by the same procedure as it has been described in [8].

**Physical-chemical properties.** The investigated physical-chemical-characteristics of the fibre substance of the yarns CAA, CAE and CAF are:

- The degree of the polymerization;
- The amount of low-molecular mass fractions;
- The amount of the conjunct acetic acid;
- The moisture content.

Since the investigated yarns were lubricated in spinning, it was essential to remove the lubricant before the performance of all physical-chemical tests. The following de-lubrication procedure was applied. A test sample of the yarn was dipped in the solution composed of the water, 0.5 g/l of soda and 1.0 g/l of non-ionogenic detergent, and retained for a half of an hour at the temperature $(50 – 60)$ °C. After that, the solution was poured out and the yarn was rinsed in distilled water. The procedure was repeated several times until phenolphthalein test showed the neutral reaction.

After de-lubrication the yarn test sample was dried at the temperature $(105 – 100)$ °C until constant mass, then cut in about 1 mm lengths to prepare test specimens and they are again dried at the same temperature until constant mass. All operations with "clean" fibre specimen were performed following the methodology of the analysis of cellulose acetate [9].

The degree of polymerization of the fibre polymer was established by the method of viscosimetry. Fibre specimen of 0.25 g, weighed at accuracy of 0.0002 g, is dissolved in acetone. The time of the outflow of the polymer solution in acetone ($t_P$) and of the solvent itself ($t_S$) from the viscosimeter is measured separately. Specific viscosity of cellulose acetate is:

$$\eta = (t_P - t_S)/t_T. \qquad (3)$$

The degree of polymerization ($n_P$) is obtained by the Staudinger formula:

$$n_P = \eta/(k \times c_S), \qquad (4)$$

where $k$ is a constant ($k \approx 9.9 \cdot 10^{-4}$); $c_S$ is a concentration of the acetate solution, g/l.

The fibre moisture content in % was determined by masses of the air-dry ($m_a$) and the constant-mass dried ($m_d$) test specimen of 0.5 g (accuracy of weighing 0.0002 g):

$$M = 100(m_a - m_d)/m_a. \qquad (5)$$

To determine the amount of low-molecular mass fractions in the fibre polymer, the method of dissolving the fibre in the acetone-water solution $(55 : 45 \%)$ was applied. The procedure is the following [9]. The air-dry fibre test specimen of 10 g, weighed at accuracy of 0.0002 g, is put into the 250 ml bulb and poured over with the solvent solution. The bulb with the suspension is stirred at the temperature $(20 \pm 2)$ °C for one hour, and then at $(200 – 300)$ mm Hg pressure passed through the Buchner filter, in which the top layer is a cotton cloth and the bottom layer is the filter paper. Both layers and the *petri saucer* of the filter are dried until the constant mass and weighed. The precipitate is twice rinsed in the solution, and later the filters, the percipitate and the *petri saucer* are once again dried until the constant mass and then

weighted. The amount of low-molecular mass fractions is obtained by the formula:

$$F = 100\left(1 - \frac{m_2 - m_1}{m_a(1 - 0.01M)}\right), \tag{6}$$

where $m_1$ is a mass of *petri saucer* with a filter; $m_2$ is a mass of *petri saucer* with a filter and percipate; $m_a$ is a mass of the air-dry test specimen; $M$ is moisture content of the test specimen, %.

The amount of the conjunct acetic acid is determined by the following procedure [9]. The dry fibre test specimen of 0.7 g, weighed at accuracy of 0.0002 g, is put into a glass bulb and poured over with 5 ml of ethyle alcohol. After the specimen moistens, 20 ml of 0.5 N caustic soda solution are instilled into the bulb. The bulb is then stoppled with the Bunzen cork and retained in the thermostat for 2.5 hours at the temperature $(60 \pm 1)\,°C$. At the end of heating the content of the bulb is refreshed to the normal temperature, and 3 – 4 drops of phenolphtalein are instilled into. The inside is titrated by 0.5 N hydrochloric acid solution until the color clears out, and then 4 ml of hydrochloric acid are again added. The inside is stirred during 5 min, then titrated until rose color by 0.1 N caustic soda solution. In parallel the identical procedure is performed with no-specimen inside the bulb.

The amount of the conjunct acetic acid is obtained by the formula [9]:

$$A = \frac{\left[(V - V_1)K_1 - \left(\frac{V_2 - V_3}{5}\right)K_2\right] \cdot 0.03 \cdot 100}{m}, \tag{7}$$

where $V$ and $V_1$ are the amounts of 0.5 N hydrochloric acid solution, consumed in the no-specimen and the test specimen titration correspondingly; $V_2$ and $V_3$ are the amounts of 0.5 N caustic soda solution, consumed in the no-specimen and the test specimen titration correspondingly; $K_1$ and $K_2$ are the corrective coefficients for the titres of hydrochloric acid and caustic soda correspondingly ($K_1 = 1$; $K_2 = 1.02$); $m$ is the mass of the acetate test specimen; 0.03 is the amount of hydrochloric acid (in grams), corresponding to 1 ml of 0.5 N caustic soda solution.

## RESULTS AND DISCUSSION

**Mechanical properties.** Some of characteristic yarn stress-strain curves obtained in the tests are shown in Figure 2, whereas the values of measured tensile parameters are listed in Table 2.

As it can be seen, tensile properties of all types of yarn slightly depend on the origin of cellulose acetate. The yarn CAA manufactured from the cellulose acetate of wood-pulp origin is advantaged against the others both in strength and extensibility. This was confirmed by *t*-test. The yarn CAA is also more uniform – the coefficients of variation of the parameters are lower than of other yarns. The yarn CAF, manufactured from the cellulose acetate obtained from the factory "Azot" is little more rigid, the initial modulus being the highest one.

The experimental stress relaxation curves of CAA yarn are shown in Fig. 3. It is seen from the curves that with higher elongations the intensity of relaxation increases.
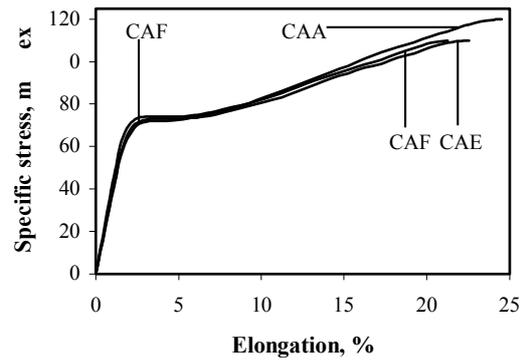


**Fig. 2.** Stress-strain curves of acetate yarns

**Table 2.** Tensile properties of acetate yarns

| Characteristics | Yarn code | | |
|---|---|---|---|
| | CAA | CAE | CAF |
| Linear density, tex | 16.8 | 16.2 | 16.3 |
| Breaking tenacity, mN/tex | 120.0 | 110.0 | 109.0 |
| Elongation at break, % | 24.5 | 22.5 | 21.2 |
| Initial modulus, N/tex | 3.7 | 3.8 | 4.4 |
| Work of rupture, kJ/kg | 22.8 | 20.0 | 18.7 |
| Specific stress at yield point, mN/tex | 70.0 | 71.0 | 71.0 |
| Elongation at yield point, % | 2.4 | 2.5 | 2.2 |
| Coefficients of variation, %: | | | |
| for breaking force | 2.1 | 2.6 | 2.5 |
| for elongation at break | 4.4 | 3.5 | 5.2 |
| for initial modulus | 6.2 | 9.7 | 6.7 |

Within the time range from $t^* = 3$ s to $t^* = 1000$ s yarn stress values at the elongation levels 5 % and 7 %, i.e., above yield point, are even lower than at elongation 2 %. Similar effect for impact-strained acetate yarn has been previously observed by Meredith [10] as well for triacetate yarn – by Vitkauskas [8]. Relaxation curves of CAE and CAF yarns are almost identical to those of CAA yarn. The exception is the behaviour of CAF yarn below the yield point. As the yarn is more rigid, its stress relaxation curve
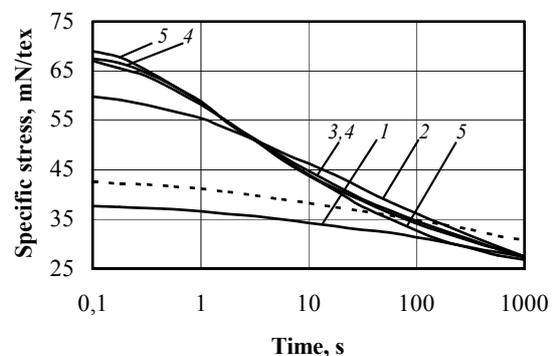


**Fig. 3.** Stress relaxation curves of acetate yarn CAA. Elongation ($\varepsilon_t$): 1 – 1 %; 2 – 2 %; 3 – 5 %; 4 – 7 %; 5 – 10 % (the dotted line is for the yarn CAF at $\varepsilon_t = 1$ %)

at $\varepsilon_t = 1$ % (Fig. 3, dotted line) lies higher than those of other yarns.

In Figure 4 the dependence of the relaxation time spectrum on elongation $\varepsilon_t$ is shown, calculated for the yarn CAA. It is seen that below the yield point with increase of elongation $\varepsilon_t$ the intensity of both the short and long relaxation times in the spectrum slightly increases. Above the yield point with increase of elongation $\varepsilon_t$ the spectrum markedly moves towards the shorter relaxation times with sharp increase of the shortest relaxation times and slight decrease of the longer relaxation times.
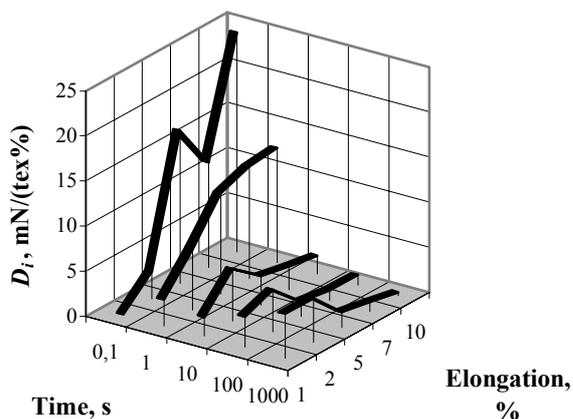


**Fig. 4.** Dependence of the regular discrete relaxation time spectrum on elongation for the yarn CAA

It seems that such characteristic behaviour of acetate yarns is first of all determined by the existence a distinct yield point in the stress-strain curve of the acetate fibre. When the yield point is reached in extension, considerable amount of secondary bonds breaks in a fibre structure and the segments of macromolecules become reasonably released to straighten very fast. The breakage of secondary bonds in the yarn sustained at elongations above yield point continues, therefore stress relaxation is so intensive. It should be also noted that the acetate yarns being tested are air-jet intermingled yarns. This feature of yarn structure also can affect the character of relaxation as the filaments deform and relax in a different way in the intermingled points and in the not intermingled regions.

Low surface friction is commonly desirable in such processing of yarns as winding, warping, etc. Therefore acetate yarns are always lubricated on spinning machine. By frictional tests we intended to acquire more information about the surface peculiarities of the yarns manufactured of cellulose acetate of different origin and also of the yarns lubricated by different lubricants.

The measuring results are given in Table 3. It is seen that coefficients of friction of the CAE yarn are much higher than those of CAF and CAA yarns. It seems, not the origin of cellulose acetate but the other technological peculiarities must be the reason of the difference in frictional properties of the yarns CAE and CAF. The influence of different lubricants on the coefficient of friction is not very distinct, however, the lubricants VIPOIL-ACMP and A-1 are a little superior to Silastol S1 lubricant. It was impossible to measure coefficient of friction of the yarn CAA3 that was not lubricated. In this case the friction formed between the yarn and the friction

cylinder was so high that the yarn always broke down during the test.

**Table 3.** Coefficient of friction of the acetate yarns

| Yarn code | Coefficient of friction | |
|---|---|---|
| | measured by "F-Meter" | measured by "Buckle-Pollitt Tester" |
| CAA | 0.42 | 0.66 |
| CAF | 0.41 | – |
| CAE | 0.53 | – |
| CAA1 | 0.34 | 0.56 |
| CAA2 | 0.30 | 0.52 |
| CAA3 | broken | – |
| CAA4 | 0.42 | 0.66 |
| CAA5 | 0.46 | 0.64 |

As it also is seen in the Table 3, the coefficients of friction measured by the "Buckle-Pollitt Tester" are approximately 1.5 times higher than measured by "F-meter". One of the reasons of this difference may be the different speed of yarn movement in both of testers. Nevertheless, the values of friction coefficient measured by both testers for the set of yarns correlate between themselves: the relationship between the data of both the testers (Fig. 5) is seen evidently to be linear with quite high coefficient of determination.
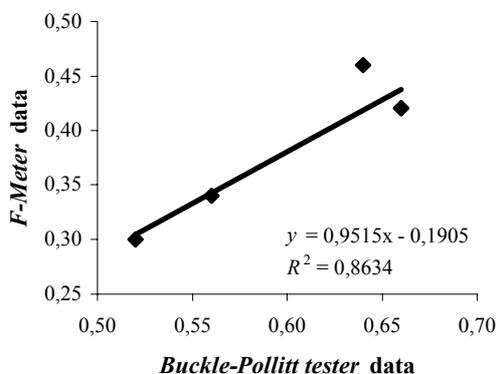


$$y = 0{,}9515x - 0{,}1905$$
$$R^2 = 0{,}8634$$

**Fig. 5.** Relationship between the yarn friction coefficients obtained by different methods

**Physical-chemical properties.** The results of physical-chemical experiments on fibre substance of the acetate yarns are presented in Tables 4 and 5. The fibre codes presented in the Tables are relevant to the codes of the yarns.

**Table 4.** Moisture content and the amount of the conjunct acetic acid in the acetate fibre

| Fibre code | Moisture content, % | Amount of the conjunct acetic acid, % |
|---|---|---|
| CAA | 5.41 | 53.0 |
| CAF | 5.62 | 50.4 |
| CAE | 5.31 | 52.3 |

**Table 5.** The degree of the polymerization and the amount of low-molecular mass fractions in the acetate fibre

| Solution | Time of outflow of polymer, s | Specific viscosity ($\eta$) | Concentration of solution, g/l | Degree of polymerization | Amount of low-molecular mass fractions, % |
|----------|------------------------------|----------------------------|-------------------------------|--------------------------|--------------------------------------------|
| Acetone  | 51.7  | –    | –    | –   | –   |
| CAA      | 105.6 | 1.04 | 4.72 | 224 | 4.6 |
| CAF      | 88.5  | 0.71 | 4.72 | 152 | 3.6 |
| CAE      | 100.9 | 0.95 | 4.71 | 204 | 0.0 |

The experimental results indicate that CAA fibre is composed of longer macromolecules than others as its degree of polymerization is the highest. The fibre also implicates little higher amount of low-molecular mass fractions. Moreover, the fibre CAA contains more acetyle groups in its constitution. This coincides well with the lower moisture content of the fibre

It is interesting to note that CAF and CAE fibres, i.e., the both fibres manufactured of cellulose acetate of cotton origin, markedly differ not only by mechanical but also by physical-chemical properties, the CAE fibre having rather significant advantage. The facts lead to presumption of different peculiarities of cotton cellulose acetylation in Azot and Engels enterprises. It is interesting that the amount of low-molecular mass fractions in the fibre CAE showed to be zero. This points to the fact that the fibre does not contain any fractions that could be leached out in the indicated solution of water and acetone.

## CONCLUSIONS

Stress-strain properties, stress relaxation and frictional behaviour of acetate multifilament yarns as well as physical-chemical properties of acetate fibre substance are investigated. The investigated yarns manufactured of cellulose acetate varied in its origin and produced by different suppliers. The origin of cellulose acetate does not have great influence on mechanical properties of the acetate yarns, however the yarn manufactured from wood-pulp-originated cellulose acetate is little stronger and more extensible, it is also more uniform in the properties.

Stress relaxation experimental data showed nearly identical behaviour of all investigated yarns with the only exception for the behaviour of the more rigid yarn CAF (produced in Uzbekistan) below yield point.

Regular discrete relaxation time spectrum of acetate yarns is determined using the generalized Maxwell model. Below yield point of the yarns both the short and long relaxation times predominate in the spectrum. Above the yield point with increase of yarn elongation the spectrum markedly moves towards the shorter relaxation times with sharp increase of the shortest relaxation times and slight decrease of the longer relaxation times.

The analysis of the yarn friction leads to the conclusion that the most efficient lubricant for acetate yarn should be VIPOIL-ACMP.

Physical-chemical experiments revealed that the fibre of wood-pulp-originated cellulose acetate has a higher degree of polymerization and a little higher amount of low-molecular mass fractions, while the moisture content and the amount of conjunct acetic acid showed no distinct differences between the cellulose acetate of different origin.

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