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Preparation of Flax Roving for Spinning in Electro-Chemical Activated Solutions

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Abstract
A number of environmentally hazardous substances are used in the process of flax roving preparation for spinning. An alternative to them can be the use of electrochemical activated aqueous solutions. However, the metastability of such solutions requires time tracking of their properties' relaxation. This article presents experimental data on the relaxation of relevant solutions that can be used for a well-founded selection of flax roving treatment procedures to ensure a required quality of flax yarn. It has been experimentally proven that the use of ECA solutions for preparing rovings for spinning allows to obtain high quality yarn as well as significantly reduce wastewater pollution and improve the environmental situation in the places of their emissions.

Key words: *electro-chemical activated solutions, relaxation, catholyte, flax roving treatment, flax yarn.*

Introduction

Flax roving, when prepared for spinning, must be chemically treated [1] to remove lignin from the fibers and create conditions for the mutual sliding of fibers in the course of spinning. There are two types of pre-spinning treatment of the roving: bleaching and boiling. The latter implies weakening of the bonds between elementary fibers. However, the natural colour of the flax roving would not change.

The exhausted solutions contain some chemicals of hazard classes 2-4 in amounts that exceed many times the maximum concentration limit for effluents. Before their discharge, alkali and acids must be neutralised (i.e. the pH value will have to reach levels 6.5-8.5) with subsequent desalination. Fermentative treatment [2-5] can be one of the alternative ways to prepare the roving for spinning. However, it has found more extensive use for the modification of flax fiber. We have investigated the preparation of flax roving for spinning in electro-chemical activated aqueous solutions [6-8].

The point of electro-chemical activation [9, 10] is that ordinary water after anode treatment or cathode processing in a flow-type membrane electro-chemical reactor passes into a metastable condition characterised by abnormal physico-chemical activity that gradually decreases in time (relaxes). The solution resulting from the cathode portion is called catholyte, and the one that comes out the anode portion will be anolyte.

ECA-solutions permit, without using any chemical agents, to directionally change between very wide limits the acid base, reductive-oxidative and catalytic properties of ordinary water; and to use such metastable liquids instead of traditional solutions of chemical agents in various production processes.

The production of ECA-solutions is based on the use of redox-processes accelerated many times in special flow-type membrane electro-chemical reactors [10]. To ensure the reactor's correct operation, the water supplied to it must have weak mineralisation under 1 g/l produced by the salt liquor solved in it. The solutions acquired are metastable, and their chemical activity decreases in the course of time, totally disappearing within a given period. In consequence, the production waste to be discharged will be a solution with a general mineralisation of 650 ppm that has no need for additional cleansing. Therefore, the treatment of roving in ECA-solutions is an environmentally-friendly process allowing to substantially reduce the pollution of effluents as well as costs due to the exclusion of cleansing steps from the technological chain.

Experimental justification of flax roving treatment procedures

In order to treat roving without losing its colour (boiling), catholyte is used, which has pronounced reducing properties.

According to the experiments described in [11, 12], the treatment of flax roving in catholyte allows to destroy the incrusts and enhancs the maceration ability of the fiber. A number of methods to prepare flax roving for spinning are based on this effect [6, 7].

It should be noted that in the course of time the relaxation of ECA-solutions, which also contain aqueous catholyte, should be taken into account when selecting treatment conditions [11].

A laboratory-scale plant for boiling roving comprises a stainless steel reactor, which accommodates roving wound on a perforated-shaft bobbin. The plant includes a pump and distributing valve to ensure, as an experimentalist would wish, pumping of the solution from the inside of the bobbin towards its peripheral area and backwards. The heating of the solution and maintenance of the temperature at a required level is ensured by an electric heater.

Preliminary experiments determined that the roving should be treated at about 60 °C. It is common knowledge that with temperature growth the chemical reaction rate increases. For that matter, we can expect the substantial influence of temperature on the relaxation rate of the ECA-solutions with regard to the data

presented in [11]. To investigate this issue, some experiments were conducted to supervise the catholyte pH value in the course of heating the solution prepared. For that end, the reactor of the experimental facility was filled with catholyte prepared at room temperature with pH = 11.43, whereupon the solution was heated by the heater included in the experimental facility with temperature and pH control every five minutes. The results of the experiment are presented in ***Figure 1***.

Comparing ***Figure 1*** (curve 2) with the catholyte relaxation data at room temperature [11], it may be inferred that heating can have a significant influence on catholyte activity. The experimental results show that its activity substantially goes down only in the process of heating, i.e. when the solution is being prepared for the treatment of roving.

Another essential factor that affects ECA-solutions can be the interacting process of the solution with the treated roving. To investigate this phenomenon, the experiment described was repeated until the solution temperature reached 60 °C. That done, a roving bobbin was placed in the reactor, and the treatment started at a constant temperature of 60 °C. In the course of treatment, changes in the solution's pH value were noted every 15 minutes. The results are presented in ***Figure 1*** (curve 3).

By comparing curves 2 and 3 (***Figure 1***), it becomes clear that the reduction in solution activity during the boiling of the roving becomes more intensive. As a consequence of this, the treatment efficiency goes down before the result required is reached. To avoid this phenomenon, the exhausted solution must be periodically replaced with a new, freshly prepared solution.

The analysis of the experimental data made it possible to formulate the following recommendations for the selection of catholyte production procedures most optimal as regards its activity:

- to create mineralisation during catholyte production, it is recommended to
- use sodium salt NaCl [11];
- the mineralisation level can be controlled by the current flowing through the flow-type electro-chemical reactor maintaining it at approximately 5A [11];

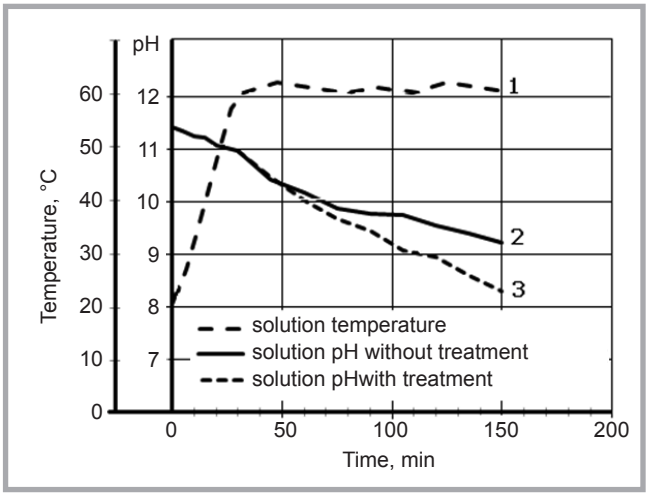

Figure 1. *Influence of temperature and roving treatment time on catholyte activity.*

- the water entering the reactor must be heated up to 60 °C;
- on account of the catholyte pH value relaxation, the treatment should not start later than 0.5 h after the preparation;
- to maintain solution activity during the treatment of roving, the solution in the chemical reactor should be partially replaced every 15 minutes.

Investigation of the flax roving treatment procedure in catholyte

The main factors affecting the degree of roving preparation for spinning are as follows:

- catholyte activity, which can be estimated by the pH level;
- temperature of the solution;
- time of treatment.

The main output parameter of the experiment is the degree of roving preparation for spinning as assessed by the maceration ability, i.e. the strength of 10 cm pieces of roving in a wet condition. Additionally, other output parameters were used to assess the degree of roving preparation, such as the loss of mass during treatment and the content of incrusts in the exhausted solution, determined by the spectrum analysis method.

The preliminary experiments [6] established that the treatment degree of roving rises with an increase in the factors mentioned. The dependencies of the output parameter on the experiment factors do not have extreme values. In this case, one can only talk about optimal treatment procedures with regard to limitations, first and foremost economic aspects expressed by the power consumption for 1 kg of roving.

It is evident that power consumption grows due to an increase in treatment times and solution temperatures; therefore, the optimum procedure will be that which ensures the required maceration ability with the minimum treatment time and temperature.

In order to determine the optimum treatment procedure with regard to the results described in [6] acquired with the experimental facility, the treatment of roving of 760 tex linear density consisting of a mixture of long flax fibers No. 14/16 was carried out. Catholyte of pH = 11.2 obtained from water preliminarily heated up to 60 °C was used for this treatment. The treatment consisted in pumping the solution through a winding layer alternately in two directions, from the bobbin cavity to the peripheral portion of the winding and backwards. The change of flow direction was made manually every 15 minutes of treatment. At the end of 15 minutes, the temperature and pH value were controlled. If a temperature drop exceeded 55 °C, solution heating was activated. If a pH value went down below 10.8, the solution was replaced with newly prepared catholyte of pH = 11.2. The exhausted catholyte was collected for analysis on an IR spectrophotometer with the aim of determining the available/quantity of incrust disintegration products in the exhausted solution.

Temperature and pH control results are presented in ***Table 1***.

The procedure illustrated in ***Figure 1*** was used for the treatment of roving with a total duration of 1, 2 or 3 hours. Besides the treatment procedure shown in ***Figure 1***, treatment in water at a constant

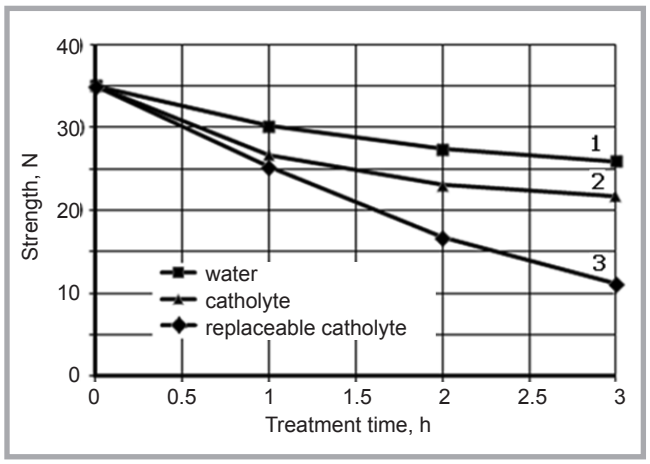

Figure 2. *Dependence of roving strength in a wet condition on treatment procedures.*

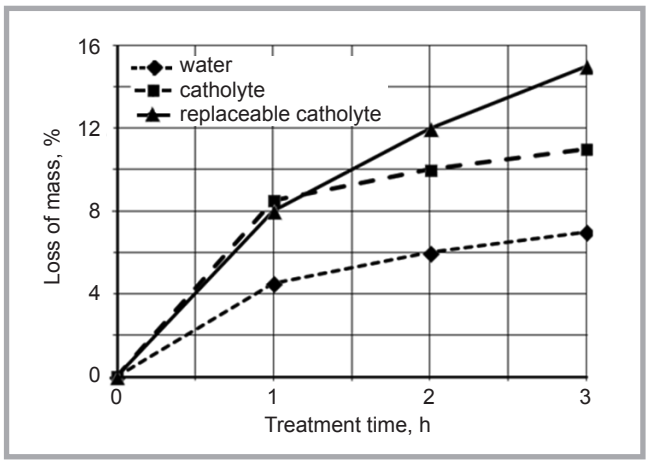

Figure 3. *Dependence of mass loss on roving treatment conditions.*

Table 1. *Changes in pH value and temperature in the course of treatment.*

Treatment time, min.		15	30	45	60	75	105	120	135	150	165	180
pH	beginning of period	11.2	11.1	11.2	11.0	11.2	11.1	11.1	11.2	11.1	11.2	11.0
	end of period	10.8	10.9	10.8	10.8	11.1	11.0	10.8	11.1	10.9	11.0	10.8
Temperature, °C	beginning of period	56	56	58	55	55	58	56	58	55	55	58
	end of period	60	60	63	62	60	62	60	63	62	60	62
Change of solution		yes	yes	yes	yes	yes	no	yes	no	yes	no	

temperature of 60 °C and in catholyte without its replacement were also used. In accordance with the treatment results, the maceration ability, loss of mass and the quantity of incrust decomposition products in the exhausted solution were also controlled. Therefore, in the course of the experiment, nine variants of roving were developed in total. When the final experiments were conducted, the order of the experiments was randomised to exclude any influence of the factors connected with time. The relevant treatment procedures and their enumeration are shown in ***Table 2***.

Figure 2 presents data for the determination of the strength of 10 cm roving pieces in a wet condition that were treated in accordance with the procedures described in ***Table 2***. The strength of grey roving was measured prior to the procedures mentioned as a control variant. As per recommendations [8], some roving samples were put into water for 2-3 minutes with a temperature of 25-36 °C.

Curves 1 & 2 indicate that treatment in catholyte ensures a higher maceration ability of flax fiber in roving. However, this result can be obtained practically within the first hour of treatment. Subsequently, the velocity of changes in roving strength in water and in catholyte do not differ because catholyte activity goes down very fast at 60 °C.

The replacement of catholyte with a freshly-made solution every 15 minutes (curve 3) makes it possible to substantially increase the treatment efficiency to reach a strength of some 11 N after three hours of boiling for the roving that had a linear density of 760 tex in a grey condition.

Experimental results for the determination of mass loss as a result of roving treatment in water and catholyte are shown in ***Figure 3***.

The curves also indicate that by regular replacement of the solution it becomes possible to substantially enhance the efficiency of roving preparation for spinning. Meanwhile, the loss of mass during treatment in replaceable catholyte will conform to the values obtained by lye boiling in production conditions (12-15%).

The process dynamics of roving treatment in catholyte can be traced by analysing the discharge process of incrust disintegration products from the roving into the solution, making use of an infra-red spectrometer – SFK-256 UBI. This device is intended for measuring spectrum factors of the direct transmission of liquid and solid transparent substances in the spectrum region from 1000 to 2500 nm. Two cuvettes made from quartz glass are placed in the spectrophotometer. By comparison, the relation between transmission coefficients is measured through cuvette 2 as opposed to cuvette 1. Distilled water was placed in cuvette 2, and the test solution was put in cuvette 1. ***Figure 4*** shows IR spectra of freshly prepared and exhausted catholyte in relation to the distilled water. Curve 1 represents the reference line made for the sample containing distilled water. curve 2 corresponds to freshly prepared catholyte with pH – 11.2 and curve 4 to the catholyte used in the first 15 minutes of the treatment process, i.e. the one in the reactor for a time range of 0-15 min.

Table 2. *Enumeration of roving treatment variants when prepared for spinning.*

Bath recipe	Time of treatment, h		
	1	2	3
Water	3	6	1
Catholyte pH = 11.2 not changed	2	9	4
Catholyte pH = 11.2 changed every 15 minutes	5	8	7

At the end of that period, the catholyte, as defined in ***Table 1***, had pH = 10.8. Curve 3 corresponds to the catholyte used from the 30th to 45th minute of the treatment process.

The wavelength range 1260 to 1840 nm presumably corresponds to incrust dis integration products. It can be seen from these spectra that the discharge velocity of incrust disintegration products decreases in the course of time. It can also be observed in ***Table 1*** that the alkali content of the catholyte at the beginning of the treatment is reduced by almost 8%, within 15 minutes, and that at the end of the treatment it remains practically unchanged, making it possible to conduct the treatment without replacing the catholyte.

Analysis of yarn quality made from roving prepared for spinning in catholyte

A final conclusion about the applicability of the roving preparation process for spinning in ECA-solutions can only be made from the results of its processing into yarn and analysis of the yarn quality obtained. In the course of preparation for spinning, roving with a linear density of 670 tex was treated at 60 °C in catholyte and water in nine procedures, defined in ***Table 2***.

A PM-88-L8 spinning frame was used to process yarn with two linear densities of 46 and 100 tex from that roving. After drying out to acquire the normal amount of moisture, some analyses were conducted to assess the yarn quality.

By applying a KLA-2M device, spectra of yarn irregularity were constructed, and the number of defects – neps and thick and thin places per 100m of yarn were assessed. The results for the 46 tex yarn are presented in ***Table 3***, and for the 100 tex yarn in ***Table 4***.

Analysis of the given data demonstrates that treatment times significantly affect yarn quality irrespective of the bath recipe. Obviously, with an increase in treatment time, the bonds between elementary fibers in the complexes become weaker. This improves drafting conditions, ultimately reducing irregularity in the linear density.

The reduction in the quantity of yarn defects as well as thick and thin places with an increase in the treatment time is also a result of bond weakening between elementary fibers.

However, when water is used, the process of bond weakening between elementary fibers becomes rather slow. The use of catholyte for the preparation helps accelerate this process, which becomes clear when comparing the data for variants 1 and 2, differing insignificantly in their irregularity and number of defects. However, the duration of water treatment (variant 1) is three hours, while that for catholyte pH = 11.2 is only one hour. As is shown above, catholyte activity goes down rather fast in the course of the roving treatment. Therefore, for variants 5, 8, 7, treatment was conducted with continuous replacement of catholyte by a freshly prepared one. This helped intensify the treatment process and obtain improved values of yarn irregularity. The best values are for variant 7 (three-hour treatment with the replacement of catholyte every 15 minutes.) The best

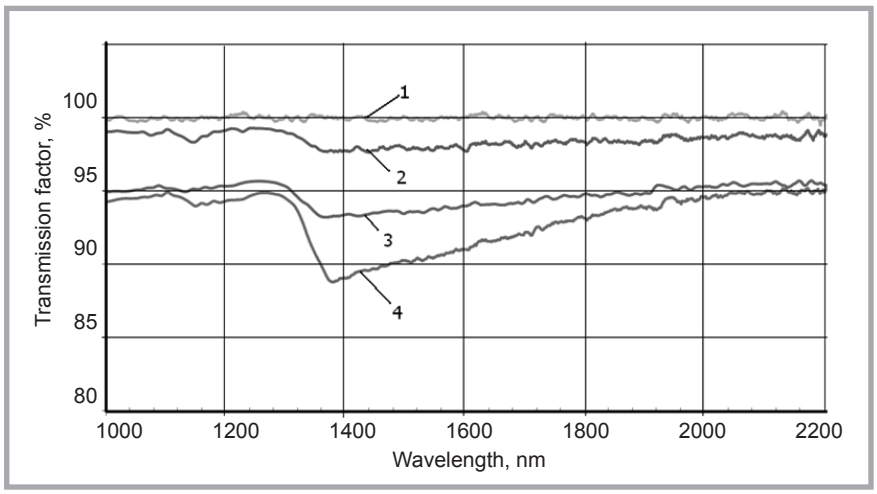

Figure 4. *IR spectra of freshly prepared and exhausted catholyte in relation to distilled water.*

Table 3. *Results of analyses of the irregularity and defects of 46 tex yarn.*

	Number of variant								
	1	2	3	4	5	6	7	8	9
Slub, pcs/50 m	231	258	321	257	239	301	173	198	273
Attenuation, pcs/50 m	228	216	256	179	173	243	136	140	188
Neps, pcs/50 m	18	20	19	17	20	22	14	15	21
Coefficient of variation (Cv), %	49	54	56	56	47	55	41	43	55
Total dispersion (Cv^2)	2401	2916	3136	3136	2209	3025	1681	1849	3025
Dispersion 12-400 mm (Cv^2)	1968	2391	2571	2571	1811	2480	1176	1294	2513

Table 4. *Results of analyses of the irregularity and defects of 100 tex yarn.*

	Number of variant								
	1	2	3	4	5	6	7	8	9
Slub,	91	132	208	114	124	141	91	108	166
Attenuation, pcs/50 m	111	97	135	79	81	103	54	85	100
Neps, pcs/50 m	19	21	25	19	23	24	16	15	22
Coefficient of variation (Cv), %	29	32	39	31	35	34	21	25	32
Total dispersion (Cv^2)	841	1024	1521	961	1225	1156	441	625	1024
Dispersion 12-400 mm (Cv^2)	672	819	1216	7989	1018	960	308	437	850

Table 5. *Physical and mechanical performance of yarn.*

		Number of variant								
		1	2	3	4	5	6	7	8	9
46 tex	Tenacity, cN/tex	13.3	13.7	13.4	17.5	17.1	13.2	21.4	20.0	16.9
	Variation coefficient, %	19	18.7	21.4	19	17.7	24.1	17.6	18.1	18.6
100 tex	Tenacity, cN/tex	16.9	16.7	12.7	18.5	17.1	14.2	22.1	20.2	15.6
	Variation coefficient, %	19	18.7	21.4	19	17.7	24.1	17.6	18.1	18.6

values of yarn defects are for the same variant.

Research results

The main yarn parameter that determines the yarn quality grade is the unit breaking tenacity. The unit breaking tenacity values and variation coefficients are presented in ***Table 5***.

The requirements for the relative breaking tenacity and variation coefficient for 100 m long pieces of all-linen, boiled or bleached yarn are set by GOST 10078-85. Comparing the data obtained with the GOST values, it can be said that the yarn of 100 tex linear density produced for variants 3 and 6 will be ungraded, while that for variants 1 and 2, will correspond to the second grade of the "Ordinary Flax Yarn" group and 4, 5, 9 – to the first grade of the "Medium Flax Yarn" group. The yarn produced for variant 8 corresponds to the first grade of the "High Flax Yarn" group, and for variant 7 – to the first grade of "Special Flax Yarn" group. Similar results were obtained for the 46 tex yarn.

Conclusions

In conclusion, the following has been established :

1. The use of ECA-solutions for the preparing of roving allows to substantially reduce the pollution of effluents and improve the environmental situation at the places of their discharge.
2. The time of roving treatment in catholyte at 60 °C, from the moment of preparation to the beginning of treatment, must be less than 0.5 h.
3. For the preparing of roving, we can recommend treatment in catholyte with an initial value of pH = 11.5 at 60 °C for three hours, with regular replacement of catholyte by a fresh one every 15 minutes.

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