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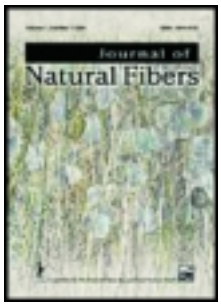

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ABSTRACT. Thermogravimetric analysis (TGA) makes it possible to obtain information on change in physicochemical properties of biopolymeric components of flax fibres when determining their quantitative content by burning microsamples in the range of 25-500°C. The results obtained have shown an individual pattern of TGA and differential scanning calorimetry curves in samples with a different quality of flax fibre. Degradation rate maxima for flax fibre biopolymers were observed over a temperature range of 185-455°C that allowed estimation of the content of water, cellulose, lignin and ash. TGA was shown to serve as a sensitive method for estimating flax fibre composition. *[Article copies available for a fee from The Haworth Document Delivery Service: 1-800-HAWORTH. E-mail address: <docdelivery@haworthpress.com> Website: <http://www.HaworthPress.com> © 2006 by The Haworth Press, Inc. All rights reserved.]*

KEYWORDS. Cellulose, fibre quality, flax (*Linum usitatissimum* L.), lignin, thermogravimetric analysis (TGA)

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INTRODUCTION

The content and quality of bast fibre is a principal criterion for estimating fibre flax (*Linum usitatissimum* L.), a valuable industrial crop. The most commonly used method for analysing the quality of flax products is organoleptic evaluation. Among advanced technological techniques the following should be noted: determination of flax fibre quality by air flow method, estimation of bast fibre breakage characteristics, investigation of fibre cross sections by scanning electron microscopy, application of infra-red spectrometry for characterizing fibre polysaccharide components (Barton, Akin and Morrison, 2002; Baley, 2004). There exists, however, an urgent need for developing a method enabling the carrying out of screening of selectional and industrial plant fibre samples for identification and revealing of structural and functional characteristic of high-quality flax fibre (Oëver, Bas and Soëst, 2003).

The goal of the present research was to develop a fast and reproducible technological procedure for analysing bast fibre based on determination of kinetic parameters of thermal destruction in polysaccharide components.

METHODS

Standard samples of combed flax fibre N 1-13, colour marks A and B, provided for determining the content of incrusting substances in accord with specifications of the Republic of Belarus 00312308.01–99 were taken as a research material. Certified values of the incrusting substance content in the standard samples varied with an increase in the flax fibre quality in the range 4.5% (N 1) to 1.5% (N 13).

A thermogravimetric analysis (TGA) of fibre microsamples (5.0-5.1 mg) was made with thermoanalyser TA-4000 (module TG-50) (Mettler Toledo STAR^e System Switzerland).The TG analysis was carried out over a temperature range of 25-500°C with a heating rate of 5°C/min and air consumption of 200 ml/min. Curves of weight loss were computed by means of STAR^e software. The mean values of the parameters from three analytical replications are given in Table 1.

RESULTS

The experimentally obtained curve of the relationship between weight variation and temperature (TGA) indicates thermostability and sample

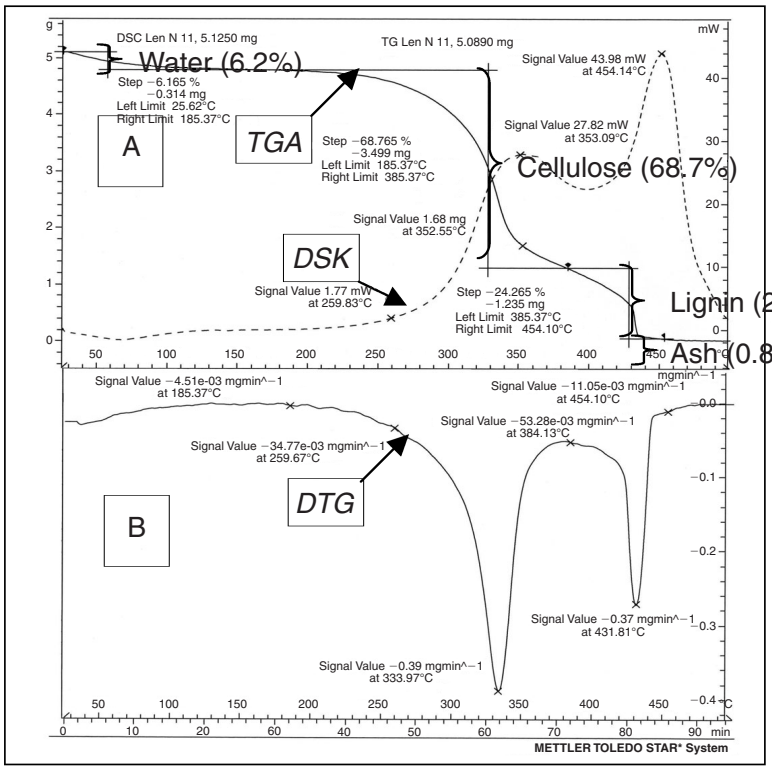
TABLE 1. Component composition (%) and activation energy (E_a) of combed flax fibre samples No. 1-13 of colour marks A and B.

N of Sample	Weight (mg)	Water	Cellulose	Lignin	Ash	E_a (kJ/mole)
1A	5.18	8.89	59.11	21.04	10.96	67
2A	5.12	9.25	59.49	23.71	7.55	78
3A	5.12	8.83	66.10	22.73	2.34	92
4A	5.19	8.23	64.85	20.12	6.80	81
5A	5.12	8.34	64.13	25.49	2.04	84
6A	5.18	8.33	61.60	25.14	4.93	92
7A	5.15	9.26	65.90	22.05	2.79	93
8A	5.16	8.92	61.02	22.95	7.11	87
9A	5.20	7.92	67.53	22.38	2.17	97
10A	5.19	8.19	65.19	24.15	2.47	99
11A	5.24	9.03	64.58	23.23	3.16	101
12A	5.23	8.24	65.07	22.39	4.30	102
13A	5.16	8.55	67.52	22.92	1.01	103
1B	5.15	9.15	54.98	26.13	9.74	61
2B	5.15	9.26	60.63	24.74	5.37	77
3B	5.16	8.77	60.81	22.40	8.02	85
4B	5.20	9.05	62.13	24.58	4.24	82
5B	5.25	9.38	64.51	22.82	3.29	90
6B	5.21	8.71	64.67	21.83	4.79	92
7B	5.19	9.06	64.91	23.12	2.91	92
8B	5.23	8.35	62.55	22.33	6.77	90
9B	5.21	8.48	67.31	23.66	0.55	96
10B	6.19	8.68	67.89	22.51	0.92	101
11B	5.17	8.50	63.63	21.82	6.05	98
12B	5.08	6.87	66.97	25.04	1.12	102
13B	5.14	8.01	69.15	22.43	0.41	100

composition in the initial state, thermostability of intermediate products of degradation and residual ash content (Figure 1A).

The differential thermogravimetric (DTG) curve allows a more complete determination of temperatures of the onset and termination of the reaction and judgement by its peak about temperature of the maximum rate of combustion reaction (Figure 1B). The analysis of the DTG curve has shown that after water removal from the sample, the primary decay

FIGURE 1. Curves of thermogravimetric analysis (*TGA*, loss of weight), differential scanning calorimetry (*DSK*, energy release) [A] and differential thermogravimetry (*DTG*) [B] in the bast fibre sample No. 11A.



peak was in the temperature range of 185-385°C (the maximum at 334°C) that conforms to thermal destruction of cellulose components (Velde van De and Kiekens, 2002). The next peak was located within the range of 385-455°C with the maximum at t = 431°C that conforms to the maximum rate of lignin thermal destruction (Wiedemann, 1993). It should be noted that the analysis of the TGA and DTG curves does not display completely, the thermal destruction processes, of flax fibre samples. For more complete description of thermodestruction processes, it is necessary to evaluate thermal effects of combustion reactions in polysaccharide components by differential scanning calorimetry (DSC) method (Faughey, Sharma and McCall, 2000). The DSC curve

presented in Figure 1A points to exothermal processes of thermal degradation in flax fibre components. Proceeding from the fact that the loss of substance weight at combustion is subject to the kinetics equation of the first order and linear dependence: ln 100/100 $-\Delta$m on T(K°) is satisfied, activation energy was calculated after A. Broido method (Velde van De and Kiekens, 2002), based on mathematical processing of the TG curve:

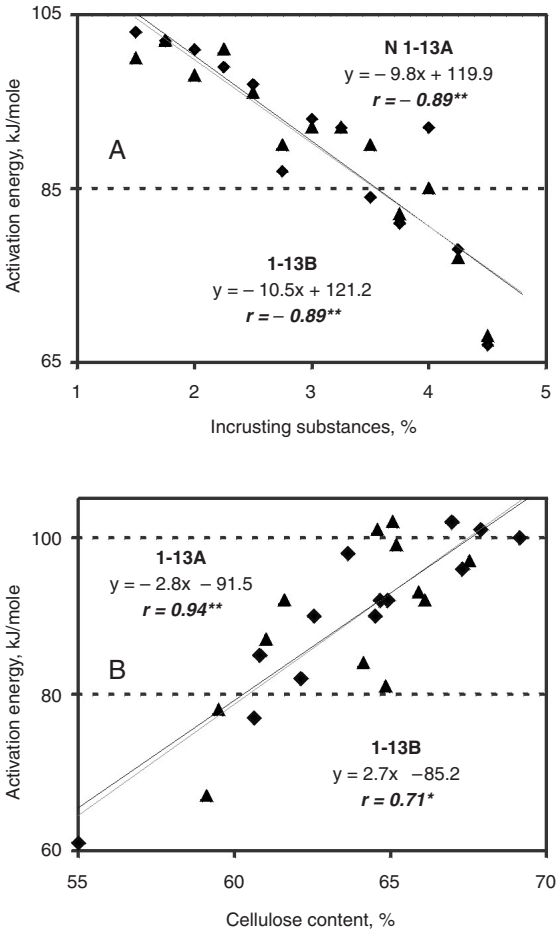
$$\ln (\ln 100/100 - \Delta m) = (E_a/R) + (1/T + C)$$

where Δm is weight loss in %, T temperature in K°, R universal gas constant, E_a-tg_{φ}–8.31 and C const.

The samples under study involved different proportions of cellulose, lignin, hemicellulose and pectin substances by the data of the thermogravimetric analysis depending of the flax fibre quality (Table 1). The results obtained have revealed differences in the component composition and activation energy of cellulose thermodestruction reaction. Flax fibres No. 13A and 13B are distinguished by the relative content of cellulose. They are superior to the rest of the samples studied in this parameter and have the maximum value of activation energy (100-103 kJ/mole). Since the E_a value is equal to the difference in energy between the transitional and initial states of combustion reaction, its values in high-number flax fibre samples indicate pronounced structural homogeneity of cellulose. It should be noted that with a decrease in the incrustation rate and a rise in the cellulose content in flax fibre the ash content tends to decrease at the practically unchanged level of lignin. A significant reverse relationship between incrustation percentage of flax fibre samples N 1-13 A and the activation energy value was observed ($r = -0.89^{**}$) (Figure 2). A similar relationship was found when analysing flax fibre N 1-13 B ($r = -0.89^{**}$). A rise in the specific share of cellulose with an increase in the quality and number of samples is accompanied by an increase in the value of activation energy (Table 1, Figure 2B), the correlation coefficient being equal to 0.71* and 0.94** for samples N 1-13A and 1-13B, respectively, during constructing the regression curve.

Thus, the tool method of the thermogravimetric analysis enables differentiation of the samples under study in conformity with standard technological characteristics of the flax fibre quality. The activation energy value represents the cellulose content in the samples studied, allows judgement on its structural state and indicates the presence of admixtures of other polysaccharides and mineral components. A high

FIGURE 2. Regression relationships between the content of incrusting substances [A] and cellulose [B] in combed flax samples (N 1-13A; N 1-13B) and the activation energy value; * P < 0.05, ** P < 0.01.



correlation between the values of kinetic parameters of flax fibre destruction at the thermogravineteic analysis and the data obtained by scanning electron microscopy should be noted (Zharsky, Titok and Kubrak, 2004). Work is under way to extend databases of the thermogravimetric analysis using flax fibre samples, standard in colour and strength, that makes it possible to improve method potentialities and accuracy of flax product quality estimation. Thus, the thermogravimetric

analysis may be, in our opinion, a reliable and reproducible method for estimating the quality of fibre and yarn and allows quantitative characterization of distinctions in thermal degradation of cellulose. This method may be assumed as a basis in developing common standards for evaluating the flax product quality. In our opinion, combination of such advanced tool methods as thermogravimetric analysis, scanning electron microscopy and infra-red spectroscopy will allow control of a chemical composition and structural characteristics of flax fibre.

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