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Alterations in flax yield and quality in response to various mineral nutrition

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ABSTRACT

The researchers of Russian State Agrarian University, Moscow Timiryazev Agricultural Academy conducted in 2013-2016 a long-term stationar experiment to study chemical and toxicological properties of fiber flax, Voskhod variety, growing on sod-podzolic soil in the soil and climate of the Moscow region. Test plots were selected with following crop rotation options: (i) without fertilizers, without liming; (ii) without fertilizers, with liming; (iii) $N_{100}P_{150}K_{120}$ (kg ha^{-1}), without liming; (iv) $N_{100}P_{150}K_{120}$, with liming; (v) $N_{100}P_{150}K_{120}$ + manure 20 ton ha^{-1}, without liming; and (vi) $N_{100}P_{150}K_{120}$ + manure 20 ton ha^{-1}, with liming. The agro-climatic conditions of the growing seasons during the research years did not have a negative impact on the growth and development of fiber flax. The hydro-thermal indices were 1.1 in 2013, -1.05 in 2014, 1.5 in 2015, and 1.5 in 2016. The maintained crop rotation and the introduction of a full range of mineral and organic fertilizers were found to contribute to high yields of flax in terms of fiber (18.5-18.9 hwt ha^{-1}) and seeds (7.9-8.3 hwt ha^{-1}). The seeds contained 16.9-19.5% protein and 33.5-39.4% lipids. The yield of flaxseed oil from seeds ranged from 19.5-35.7% on average for different variants of the experiment. The peroxide number index was 1.5-2.5 mg-eq O_2 kg^{-1}, and the acid number index was 1.1-1.9 mg KOH g^{-1}, which correspond to the obtaining high-quality linseed oil in compliance with quality standards for all variants of the experiment. In the fatty acid composition of flaxseed oil, the content of total saturated fatty acids, the amount of unsaturated fatty acids and a high content of essential α-linolenic acid were 9.0-14.1%, 85.9-91.0%, and 46.9-60.9% respectively.

Keywords: Fiber Flax, Fertilizers, Fiber, seeds, Linseed oil, Chemical analysis.
Article type: Research Article.

INTRODUCTION

According to FAO statistics, nowadays a limited number of countries grow fiber flax for commercial purposes: Belarus, Belgium, China, Czech Republic, France, Lithuania, Netherlands, Poland, Russia, Ukraine, and Egypt. France is the leader in crop area, followed by Belarus and Russia. The rise of the flax-growing branch of agriculture in Russia in recent years has been facilitated by the adoption of the State Program for the Development of Agriculture until 2025. This program significantly has deepened and expanded the economic measures of influence on the processes in the agrarian and agri-food spheres of the country, among which a significant place was and is given to subsidies that stimulate the growth of production, including the flax-growing subcomplex (Belopukhov *et al.* 2008; Pozdniakov *et al.* 2015; Belopukhov *et al.* 2015; Mheidi *et al.* 2023). However, in the global production of natural and synthetic fibers, flax occupies a limited market segment (Table 1). This corresponds to the fact that only 0.1 kg of flax fiber is produced per year per inhabitant of our planet, which, of course, is extremely low (Rozhmina 2018). Despite the high productivity of modern fiber flax varieties, their maximum biological capabilities and potential of the variety in production conditions can be 30-50%, which is largely due to the insufficient use of mineral fertilizers in the optimal ratio of nutrients. The use of environmentally friendly, biologically active products can also increase the economic efficiency of flax cultivation through a natural positive effect on productivity. Consequently, by the rational use of soil resources, fertilizers, plant protection products, the results of selection and technological methods of cultivation of flax, it is possible to obtain high yields of fiber and seeds (Guang Hui 2015).

Table 1. Global production volumes of various types of fibers, 1970-2016 (thousand tons).

Year	Chemical fibers	Cotton	Sheep wool	Other animal wool	Flax	Silk	Total
1970	8,397	11,379	1701	-	703	46	22,226
1980	14,182	14,084	1646	-	620	69	30,601
1985	16,336	19,245	1763	-	763	68	38,175
1,990	18,519	17,362	2007	67	688	83	38,726
1995	22,204	18,754	1520	55	716	113	43,373
2000	33,083	19,118	1343	48	528	111	54,232
2005	41,291	26,422	1219	50	1013	152	70,148
2010	51,266	22,480	1121	51	299	165	75,644
2015	67,535	25,916	1156	58	313	169	95,147
2016	68,377	20,912	1141	56	317	169	91,971
2016/1970 (%)	814	184	67	-	45	367	413

The value of fiber flax is due to the variety of its uses. Fabrics for various purposes are produced from flax fiber: clothing (linen fabrics), household, furniture-decorative, industrial, as well as special-purpose fabrics (medical and defense industries). In the EU countries and China, 30-50% of the production of linen fabrics is textile fabrics for the production of clothing. In Russia the production of technical fabrics accounts for about 50% (Belopukhov *et al.* 2015). Flax seeds are a valuable source of various substances: proteins (18-23%), fats (30-40%), phospholipids, macro- and microelements. Flaxseed oil rich in polyunsaturated fatty acids (Omega-3 and Omega-6) is produced from the seeds. Flax seeds and linseed oil are used for food, medical, and technical purposes. Harsh competition in the sales market, including in the flax sector, has set domestic producers the task to obtain high-quality fiber, seeds, and oil, end ensure strict control of their finished product quality and chemical composition (Belopuhov *et al.* 2010; Belopukhov *et al.* 2012). Given the above, the objective of our study was to adapt the fiber flax, Voskhod variety, to the soil and climatic conditions of the Moscow region and to study the chemical composition of flax seeds and flaxseed oil in response to the application of mineral and organic fertilizers and without fertilizers.

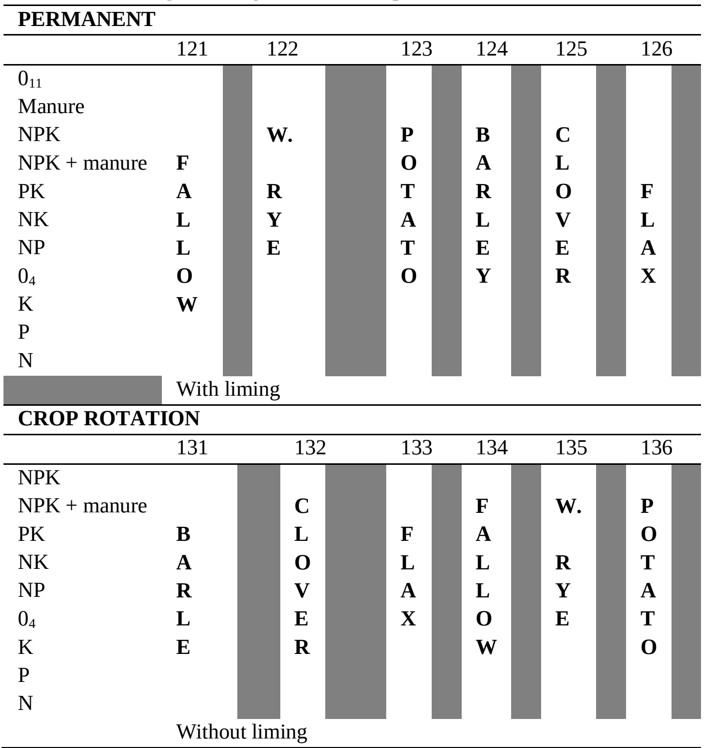
MATERIALS AND METHODS

The studies were carried out in 2013-2016 as part of the long-term field experiment of Russian State Agrarian University, Moscow Timiryazev Agricultural Academy, started by Professor A.G. Doyarenko in 1912. The soil of the experimental plot is sod-medium and slightly podzolic, old-arable (more than 200 years under arable land), naturally acidic and floating (according to the FAO classification - Podsolluvisol). The soil characteristics of the experimental site are presented in Table 2. All fields with permanent crops were divided into 11 plots; fields with crop rotation were divided into 9 plots being fertilized as follows: 1: N; 2: P; 3: K; 4: without fertilizers; 5: NP; 6: NK; 7: PK; 8: manure + NPK; 9: NPK; 10: manure; 11: without fertilizers. The crop rotation plot has no 10th and 11th options. Since 1949 (7th crop rotation), liming has been introduced into the experiment, as one of the essential factors in the cultivation of acidic soddy-podzolic soils. Lime was applied to half of each field, in the form of dolomitized limestone - once per crop rotation (the dose was based on the values of the hydrolytic acidity of the soil). Doses of mineral fertilizers in 1973 were increased and amounted to N_{100}, P_{150}, K_{120} for food elements. Phosphorus and potash fertilizers were applied at the same time for pre-sowing treatment, and nitrogen fertilizers in 2 periods: in the fall (N_{50}) and in the spring as top dressing (N_{50}).

Table 2. Properties of the 0-20 cm soil layer of the experimental plot 60 years after the start of the experiment (Belopukhov *et al.* 2015).

Parameters	**Experimental mean**
Solid phase density (g cm^{-3})	2.65
Soil density (g cm^{-3})	1.53
Maximum hygroscopicity (MH; %)	1.25
pH (pH meter units)	5.2
Humus carbon (C; %)	1.03
Total nitrogen (%)	0.079
C/N	13
P_2O_5 (mobile; mg kg^{-1})	520
K_2O (metabolizable; mg kg^{-1})	160
Total metabolizable alkali (m-eq kg^{-1})	97

Fig. 2. Long-term field experiment scheme.



Since 1973, to study the aftereffect of fertilizers, their plot application was stopped on even fields (132, 134 and 136) of the main crop rotation, and each field were fertilized with a continuous single dose of $N_{100}P_{150}K_{120}$. Manure also was not applied to these fields. For permanent winter rye, 20 tons of manure were applied annually per hectare in 8 and 10 variants Variants of field experiments were selected in the crop rotation: zero level (no fertilizers, no liming): variant 1; zero level (without fertilizers, with liming): variant 2; $N_{100}P_{150}K_{120}$, without liming: vatriant 3; $N_{100}P_{150}K_{120}$, with liming: variant 4; $N_{100}P_{150}K_{120}$ + manure 20 ton ha^{-1}, without liming: variant 5; and $N_{100}P_{150}K_{120}$ + manure 20 ton ha^{-1}, with liming: variant 6. The agricultural flax cultivation techniques were as follows: in the fall: the primary plowing was carried out with MTZ 12+21+UNIA 2+1 vehicle (small reversible plow); in the spring: harrowing (moisture closure) with MTZ-80+BZTS-1.0 vehicle, cultivation with MTZ-80+ZBC-300 vehicle. Seeding rate of seeds was 22 mln.pcs./ha. Sowing was carried out with MTZ-80+AMAZOND9-30 vehicle. The area of the plots and the registration plot area were 50 m^2 and 25 m^2 respectively. The predecessor in all the years of research was the first year clover. Nitrogen fertilizers were applied in the spring before sowing; while phosphorus, potash, and manure in the fall. Liming was carried out once every 4-5 years based on the hydrolytic acidity of the soil. During the years of our research it was not carried out. The soil was soddy-podzolic, medium and light loamy, old arable, according to agrochemical indicators (average values during the years of research): soil density 1.5-1.6 g cm^{-3}; humus content (Tyurin) 2-2.5%; P_2O_5 (Kirsanov) 170-180 mg kg^{-1}; K_2O (Maslova) 90-100 mg kg^{-1}; N, readily hydrolysable (Tyurin) 5-5.5 mg 100 g^{-1}; $pH_{(water)}$ 5.5-6. Agroclimatic conditions of the growing seasons 2013-2016 did not have a negative effect on the growth and development of fiber flax, the yield of which was mainly determined by the studied factors. The HDC (hydrothermal coefficient), which characterizes the degree of moisture in the growing season, were 1.1 in 2013; 1.05 in 2014; 1.5 in 2015; and 1.4 in 2016. Thus, 2013 was a poorly humid year, while 2014 a moderately dry year, and 2015 -2016 a fairly humid year. The fiber and seed yield data were calculated for the variants of the experiment in accordance with the existing guidelines and recommendations. Samples of seeds and oil were obtained from Voskhod fiber flax (Russian selection). Chemical analyses of seeds for the content of the total amount of lipids and proteins, as well as the fatty acid composition of flaxseed oil were performed by near infrared spectroscopy (NIR), SpectraStar XL 2500XL-R, in accordance with GOST 32749. Flaxseed oil was obtained by cold pressing in accordance with GOST 5791. We determined linseed oil yield, acid number according to GOST 50457 and peroxide number according to GOST 51487. Elemental analysis of seeds was determined by atomic absorption spectroscopy (AAS; KVANT-Z ETA instrument model). All tests were performed in triplicate. Confidence intervals with a significance level of 95% were calculated in Excel.

RESULTS AND DISCUSSION

The productivity of agricultural crops is closely dependent on the presence of the basic elements of mineral nutrition in the soil. Therefore, the forms and doses of fertilizers applied under flax directly affect the yield of flax and the quality of the resulting flax products. In our studies, the field experiment was carried out on the territory of the "Long-term field experiment of Russian State Agrarian University, Moscow Timiryazev Agricultural Academy", known abroad as the "Moscow permanent study area". Fiber flax has been grown in this experiment for over 100 years. The multifactorial experience of long-term use of fertilizers, both individually and in various combinations, is a method of understanding the basic patterns of the formation of flax yields and soil fertility conditions in the Non-Black Earth Zone of Russia [10]. Our data show high yields of flax from plots with a full range of mineral fertilizers, together with the introduction of manure and liming, both in fiber (18.5-18.9 hwt ha^{-1}) and in seeds (7.9-8.3 hwt ha^{-1}; Table 3). Plots with $N_{100}P_{150}K_{120}$, without liming (variant 3) produced higher yield, i.e., flax straw by 6.1 hwt/ha, fiber by 0.8 hwt ha^{-1} and seeds by 0.3 ha^{-1} relative to zero level plots (without fertilizers, without liming; variant 1). A similar increase in yield was on plots with liming. The average yield increase over three years of research on plot with $N_{100}P_{150}K_{120}$, with liming (variant 4) was 5.6 hwt ha^{-1} in flax, 1.1 hwt ha^{-1} in fiber, and 0.9 hwt ha^{-1} in seeds relative to the zero-level plot (without fertilizers, with liming; variant 2). The introduction of organic fertilizers (manure) together with mineral fertilizers (variants 5-6) contributed to an average increase in yield by 3.5 - 6.1 hwt ha^{-1} in flax straw, by 0.4 - 1.1 hwt ha^{-1} in fiber and by 0.4 - 0.5 hwt/ha in seeds relative to options with a full complex of fertilizers (variants 3-4) over three years of research. Liming promoted an increase in the yield of flax straw by 1.4 - 5.5 hwt ha^{-1}, in fiber by 0.4 - 1.6 hwt ha^{-1} and in seeds by 0.1 - 1.1 hwt ha^{-1} relative to plots without liming. Thus, the use of mineral fertilizers together with manure and liming (variant 6) has led to the increase in the yield of flax straw by 30%, fiber by 17%, and seeds by 21% compared to the variant without fertilizers and liming (variant 1). The harvested flax seeds were analyzed for the content of the total amount of proteins and lipids (Table 4). Plots treated with a full set of mineral fertilizers together with manure and liming (variant 6) produced seeds with the higher content of protein by 2.7% and lipids by 5.1% relative to plots without fertilizers and liming (variant 1). Liming of plots contributed to the higher content of protein and lipids in flax seeds by 0.3%-0.9% and 0.3-1.8%, respectively, relative to plots without liming on average over three years of the study. An important feature of the quality of the obtained flaxseed oil is its yield (%), acid (AN) and peroxide (PN) numbers. We have determined these indicators when obtaining flaxseed oil from seeds (Table 5). Hydroperoxides are the main primary oxidation products of unsaturated fatty acids. The peroxide number, which characterizes the content of organic hydroperoxides in the oil, is one of the most important indicators of oil quality for its oxidation state. The primary oxidation products of oils and fats are unstable and easily decompose, transforming into secondary oxidation products, which are a complex group of compounds including various aldehydes and ketones, hydrocarbons, epoxy compounds, relatively stable alcohols, acids, hydroxy acids, and others. Aldehydes and ketones impart unpleasant taste, odor, and toxicity to fats. Notably, although usually for edible vegetable oils, maximum admissible level of PN is 10 mg-eq O^2 kg^{-1}, a change in taste (rancidity) and odor of highly unsaturated linseed oil usually begins at PN less than 3-5 mg-eq O_2 kg^{-1} oil. The acid number (AN), which characterizes the content of free fatty acids, should not exceed 2 mg KOH g^{-1} oil.

Table 3. Yield of fiber flax, Voskhod varieties (hwt ha^{-1}).

Yield/variant	**1**	**2**	**3**	**4**	**5**	**6**	**HCP $_{05}$**
2013							
Flax straw	50.5	54.9	56.9	60.4	60.1	65.6	2.6
Fiber	16.4	16.9	17.0	17.6	17.5	18.9	0.8
Seeds	6.9	7.0	7.1	7.9	7.6	8.2	0.4
2014							
Flax straw	48.3	50.2	55.8	57.2	57.2	62.3	2.5
Fiber	15.3	15.7	16.0	17.0	16.6	18.5	0.8
Seeds	6.4	6.6	6.7	7.7	6.8	7.9	0.3
2015							
Flax straw	50.1	52.3	54.5	56.6	60.5	64.6	2.5
Fiber	15.9	16.5	17.1	17.9	16.9	18.5	0.7
Seeds	6.7	6.7	7.1	7.4	7.8	8.3	0.3
2016							
Flax straw	50.0	52.1	54.3	56.5	60.2	64.2	2.4
Fiber	15.5	16.3	17.5	17.4	16.5	18.3	0.6
Seeds	6.4	6.5	7.0	7.3	7.5	8.2	0.3

Table 4. Chemical composition of fiber flax seeds, rate (%) on absolutely dry basis.

Variant	**1**	**2**	**3**	**4**	**5**	**6**	**HCP $_{0,5}$**
2013							
Proteins	17.2	17.5	18.0	18.9	19.1	19.5	0.7
Lipids	34.1	34.7	36.7	37.9	38.4	38.7	1.3
2014							
Proteins	16.9	17.7	18.9	18.9	19.3	20.1	0.8
Lipids	33.5	35.3	36.9	37.5	37.9	38.8	1.3
2015							
Proteins	17.4	17.3	18.3	19.2	19.4	20.0	0.7
Lipids	34.2	34.9	36.5	37.4	38.2	39.4	1.4
2016							
Proteins	17.3	17.1	18.2	19.0	19.3	20.1	0.7
Lipids	34.0	34.5	36.4	37.1	38.1	39.2	1.3

Table 5. Quality indicators of flaxseed oil of fiber flax seeds, Voskhod variety

Indicator / variant	**1**	**2**	**3**	**4**	**5**	**6**	**HCP $_{0.5}$**
2013							
Oil yield (%)	19.5	20.0	25.5	30.4	31.3	35.7	1.1
PN (mg-Eq O_2 kg^{-1})	2.4	2.4	2.0	1.9	1.7	1.6	0.08
AN (mg KOH g^{-1})	1.8	1.8	1.3	1.1	1.1	1.1	0.05
2014							
oil yield (%)	20.0	21.2	25.8	29.6	30.6	34.9	0.9
PN (mg-Eq O_2 kg^{-1})	2.4	2.1	1.9	1.8	1.7	1.5	0.06
AN (mg KOH g^{-1})	1.9	1.7	1.5	1.2	1.1	1.1	0.05
2015							
oil yield (%)	19.8	22.3	24.9	30.0	33.2	34.7	1.02
PN, (mg-Eq O_2 kg^{-1})	2.5	2.3	1.9	1.9	1.6	1.5	0.07
AN (mg KOH g^{-1})	1.9	1.8	1.5	1.2	1.2	1.1	0.05
2016							
Oil yield (%)	19.5	22.0	24.5	30.0	32.9	34.5	1.00
PN (mg-Eq O_2 kg^{-1})	2.3	2.3	1.9	1.9	1.5	1.5	0.07
AN (mg KOH g^{-1})	1.8	1.8	1.5	1.2	1.2	1.1	0.05

The flaxseed oil yield ranged from 19.5-35.7% on average for different variants of the experiment. The yield of oil of flax seeds increased significantly on experimental plots with the use of a full set of mineral fertilizers (variants 3 and 4) was 5 - 10.4% relative to plots without fertilization (variants 1 and 2). Plots treated with a full set of mineral and organic fertilizers (variant 6) exhibited an increase in the yield of flaxseed oil by 14.9-16.2% relative to the zero level plots (variant 1). The PN was 1.5-2.5 mg-eq O_2 kg^{-1} and the AN was 1.1-1.9 mg KOH g^{-1}, which corresponds to the production of high-quality linseed oil in accordance with quality standards (TU U 15.4 - 32448339 - 001: 2005) for all variants of the experiment. Both AN and PN were lower in the fertilized variants relative to the variants without fertilizers. The fatty acid composition of flaxseed oil is represented by the content of the total of saturated fatty acids (9.0-14.1%), the total of unsaturated fatty acids (85.9-91.0%). The composition of unsaturated fatty acids had a high content of diet-essential α-linolenic acid (46.9-60.9%; Table 7). The application of fertilizers, according to the variants of the experiment, contributed to a decrease in the amount of saturated fatty acids and an elevation in the amount of unsaturated fatty acids in linseed oil. Variants treated with the full set of mineral fertilizers (variants 3 and 4) exhibited a drop in saturated fatty acids and an upraise in unsaturated fatty acids (2.1-2.5%) relative to the zero level plots (variants 1 and 2). The same changes were in the composition of fatty acids of flaxseed oil in variant 6 (4.1% - 4.8%) relative to variant 1. Liming slightly influenced the fatty acid composition of the oil according to the variants of the experiment. A wide variety of the content of elements in flax seeds provides a wide range of their medico-biological properties, which allows the seeds to be used as additives for the production of various food products. Table 8 presents the results of elemental analysis of long flax seeds. The content of chemical elements in seeds can be grouped as follows:

- high content, 1500-5620 mg kg^{-1}: Mg, Ca, Fe, K;
- 3.0-25.1 mg kg^{-1}: Zn, Mn, Cr, Si, AI;
- 0.01-0.5 mg kg^{-1}: Cu, Pb, Cd, Hg.

Trace elements with high concentrations in agricultural products are classified as heavy metals of different hazard groups. Our studies have not found the exceeded MPC thresholds in the variants of the experiments.

Table 7. Fatty acid composition of flaxseed oil (%).

Variant	**Year**	**Total saturated fatty acids**	**Total unsaturated fatty acids**	**Total palmitic and stearic acids**	**α-linolenic acid content**
1	2013	13.5	86.5	12.9	47.6
2		13.4	86.6	12.8	47.8
3		11.2	88.8	12.1	50.2
4		11.0	89.0	11.5	55.3
5		9.5	90.5	11.0	55.5
6		9.3	90.7	10.9	60.1
HCP $_{0.5}$		0.4	3.5	0.4	2.0
1	2014	14.1	85.9	13.2	46.9
2		13.6	86.4	12.9	47.1
3		11.6	88.4	11.8	50.5
4		11.2	88.8	11.7	56.2
5		9.3	90.7	10.8	55.9
6		9.1	90.9	10.5	59.8
HCP $_{0.5}$		0.4	3.2	0.4	1.9
1	2015	13.8	86.2	13.1	47.9
2		13.2	86.8	13.0	47.9
3		11.3	88.7	11.6	50.7
4		11.1	88.9	11.3	54.6
5		9.5	90.5	10.9	55.5
6		9.0	91.0	10.6	60.9
HCP $_{0.5}$		0.4	3.4	0.3	2.0
1	2016	13.6	86.4	13.2	48.0
2		13.1	86.9	13.2	47.9
3		11.0	89.0	11.7	50.5
4		11.0	89.0	11.5	54.5
5		9.3	90.7	10.4	55.5
6		9.1	90.9	10.1	60.5
HCP $_{0.5}$		0.4	3.3	0.4	1.9

Table 8. Content of chemical elements in fiber flax seeds, Voskhod variety (mg kg^{-1}) average for 2013-2016.

Element	variant						HCP 0.5
	1	2	3	4	5	6	
Mg	5400	5620	5523	5614	5123	5123	210
Ca	2215	2410	2421	2451	2510	2623	120
Fe	2321	2415	2241	2531	2512	2457	118
K	1652	1655	1685	1701	1670	1589	110
Zn	20.5	20.5	21.1	24.9	24.5	25,1	1.5
Mn	10.5	9.5	9.0	9.1	8.9	9.5	0.9
Cr	11.5	10.0	8.1	10.5	9.3	10.1	0.9
Si	4.3	4.3	4.5	4.5	4.5	4.5	0.5
Al	3.0	3.1	3.5	3.0	3.5	3.0	0.4
Cu	0.5	0.5	0.5	0.5	0.5	0.5	0.1
Pb	0.2	0.2	0.2	0.2	0.2	0.2	0.1
Cd	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Hg	0.03	0.01	0.02	0.01	0.02	0.01	0.01

CONCLUSION

Thus, as a result of our studies conducted in the conditions of the Moscow region on the territory of the Long-term stationary experiment of Russian State Agrarian University, Moscow Timiryazev Agricultural Academy, where fiber flax has been grown for more than 100 years, with the maintained crop rotation and the introduction of a full range of mineral and organic fertilizers, we obtained high yields of flax fiber (18.5-18.9 hwt ha^{-1}) and flax seeds (7.9-8.3 hwt ha^{-1}). The protein and lipid contents in seeds were 16.9-19.5% and 33.5-39.4%, respectively. Acid and peroxide numbers of linseed oil meet quality standards. In the fatty acid composition of flaxseed oil, the total saturated and unsaturated fatty acid contents were 9.0-14.1%, and 85.9-91.0% respectively. There was a high content of essential α-linolenic acid (46.9-60.9%). The chemical elements contents in flax seeds were high for magnesium, calcium, iron and potassium (1500-5620 mg kg^{-1}), while medium for zinc, manganese, chromium, silicon and aluminum (3.0-25.1 mg kg^{-1}), and low for copper, lead, cadmium, and mercury (0.01-0.5 mg kg^{-1}).

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