THE EVALUATION OF OIL-FLAX (*LINUM USITATISSIMUM* L.) QUALITY PARAMETERS FOR BIOFUEL PRODUCTION

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Abstract

Oil-flax has a wide range of utilization possibilities, inclusive of bioenergy production. In our research evaluated out two oil-flax varieties 'Scorpion' and 'Flanders' after the calorific value, oil, lignin, and ash content; and also the chemical composition in 2008 -2010. The results show that oil-flax shoves have a high calorific value and high lignin content but a low temperature for ash fusion. It was established that the growth year and some unexplored factors have influence on the ash content. The oil content in the seeds was 336-458 g kg^{-1}, depending on the chosen variety. The flax shoves can be used as a supplement for biofuel combustion, as they have a low fusion temperature, a high calorific value, but a comparatively low yield makes oil-flax unprofitable to grow for biofuel production.

Key words: *Linum usitatissimum* L., shove, lignin, ash, oil, calorific value.

Introduction

Classification of energy crops – dedicated energy crops - can be divided into three subgroups based on the utilization of the plant materials in the conversion process to bioenergy/biofuel: 1) sources of sugar and starches (non-structural carbohydrates); 2) ligno-cellulosic feedstock; and 3) sources of vegetable oils (Biomass...., n.y.). Oil-flax is an annual plant that requires more warmth but less moisture in comparison to fibre flax. From the oil-flax it is possible to obtain: technical fibre, shoves, oil cake, and straw. All parts of oil-flax can be used (Ivanovs and Stramkale, 2001; Stramkale et al., 2008; Груздевене et al., 2009).

The flax shoves are considered as waste products, which are left behind after the cellulose fibre is separated from the flax stems. The shove yield is 2.5 tonnes to one tonne of fibre produced (Cox et al., 1999).

In the world, oil-flax has been studied as one of the energetic plants, and it has been found that other crops are the most promising (Biomass...., n.y.). Studies in Latvia have not been conducted on the possibilities of using linseed biofuel production, although there are studies on oil content and oil composition.

The aim of the research is to evaluate the utilization of oil-flax for bioenergy production.

Materials and Methods

Annual crop – oil-flax (*Linum usitatissimum* L.) from *Linacea* family – was tested in the Latvia and under the conditions described in Table 1. The trial was carried out in Agricultural Science Centre of Latgale in 2008-2010. The nitrogen supplementary fertilizer and agrochemicals were given to the oil-flax in the fir-tree phase (Growth Stages (GS) 4), and insecticide – at GS 1 and GS 4.

Table 1

Oil-flax trial methods

Parameters		Trial year		
		2008	2009	2010
Soil type		Humi-podzolic gley soil		
Soil composition	pH	7.3		7.0
	OM, %	3.8 (Tyrin's method)		6.5 (Tyrin's method)
	P, mg kg^{-1}	36 (DL method)		63 (DL method)
	K, mg kg^{-1}	54 (DL method)		98 (DL method)
Pre-crops		Spring rape		Winter wheat
Complex fertilizers	N:P:K, kg ha^{-1}	6:11.3:24.9, total 300 (N=29.03; P=54.9; K=120.5)		
Sowing time		9th May	4th May	6th May
Sowing rate	kg ha^{-1}	70		
Varieties		'Scorpion'	'Scorpion', 'Flanders'	
N fertilizer rate	kg ha^{-1}	N0, N60, N80, N100		
Harvesting time		23rd September	21st September	10th August
Trial plots	m^2	7.5		
Replication		4		
Agro-chemicals	Insecticide	Fastaks 50 e.c. (alfa- cipermetrin, 50 g L^{-1}) 0.3 L ha^{-1}		
	Herbicide	Glins 75 d.g. (hlorsulfuron, 750 g kg^{-1}) 10 g ha^{-1}		
		Lontrels 300 s.c. (clopiralid, 300 g L^{-1}) 0.3 L ha^{-1}		MCPA Super 500 s.c. (MCPA, 500 g L^{-1}) 1.0 L ha^{-1}

The flax samples were harvested by hand in the growth stage of the early yellow ripeness. The plants were tied up in bundles and left in the field for 5-8 days. When flax was dry, it was crushed with the machine Eddi, and after that the pods were cleaned through a sieve. The seeds were cleaned with a sample cleaner MLN, weighed (accuracy ± 0.001 g) and the seed yield was established taking 100% purity and 9% moisture content.

The sample of 10 g of stems was weighed (accuracy ± 0.0001 g) then scutched with the tool ЛМ-3, broken and shaken until the shoves were withdrawn and weighed again. The result was calculated by the formulae (Freimanis et al., 1980). Five percent may be dust etc., these substances were eliminated from the shove content (formulae 2).

$$C = 100\tfrac{S}{L} \qquad (1)$$

where C – fibre content, %; S – strow mass, g;
L – fibre mass, g.

$$K = 95 - C \qquad (2)$$

where K – shove content, %; C – fibre content, %.

The meteorological conditions for the growth period of oil-flax are shown in Figure 1. The weather conditions during the trial years were different. In 2008, in Latvia the air temperature in the 3rd ten-day period of May was close to the long-term average, but there was no rain. Also in June and July there was no precipitation. In July the average daily temperature corresponded to the long-term average, and the amount of precipitation was abundant. The amount of precipitation in the 1st and 3rd ten-day period of July was only 40% of the long-term average, but in the second ten-day period it was 173% of the long-term average. In the first ten-day period of August, the daily temperature was 15.8 °C, close to the long-term average, but the rainfall was 86% of the long-term average. The weather conditions during trial years were different. In May 2009, the temperature was the same as the long-term average, but the amount of precipitation was only 32% of the long-term average. There was rain in June and July. The amount of precipitation in August was only 22%, and in September – 52% of the long-term average. In July and August 2010, the amount of precipitation was only 30% of the long-term average, but temperature was about 3.8-4.8 °C higher than the long-term average.

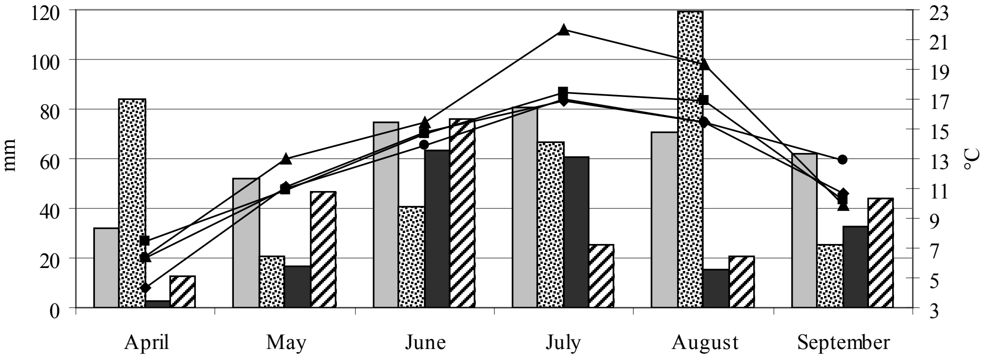

Figure 1. Average air temperature and sum of precipitation:

The following parameters were tested: 1) moisture content in shoves, according to standard ISO 589-81; 2) ash content for dry material, according to standard ISO 1171-81; 3) gross calorific value ($Q_{gr.d}$) with V (volume)=constant for dried fuel at 105 ºC, according to standard LVS CEN/TS 14918; 4) net calorific (Q_{net}) value with V=constant, according to standard LVS CEN/TS 14918; 5) ash behaviour when melting in an oxidising atmosphere, according to standard ISO 540; 6) potassium (K), calcium (Ca), sodium (Na) and silicon (Si) concentrations in mineralized samples in shoves were determined with the inductively coupled plasma optical emission spectrometer Perkin Elmer Optima 2100 DV with concentrated nitric acid; 7) oil content in the seed samples was determined by the grain analyser Infratec 1241tm, which has a specially adapted system, built-in for the analysis of oil content for flax and hemp; 8) determination of lignin of shoves was performed using Clason's method published by Ģ. Zaķis. Clason-lignin is cleaned with 72% sulfuric acid (Zaķis, 2008).

The MS Excel programme was used for data statistical processing. The ANOVA method and correlation and regression analysis were used. The test of statistically significant differences (LSD 0.05) with the Fisher criterion (F-test) and factor density influence was used for the analysis of mean differences.

Results and Discussion

Previous research in Latvia shows that agrometeorological conditions for the 2008 and 2009 growth period did not influence the oil content for the variety 'Scorpion' (Poiša et al., 2010). The air temperature in the 2010 growth period was higher than the long-term average (Fig. 1) the result of which was that the oil content was only 84% of the previous year result (Table 2). The variety's or genotype's, as a factor, influence was essential ($p<0.05$) for the oil content in the flax seeds. The average oil content in the seeds was 415 g kg^{-1}, which corresponds to the data in the literature on the subject (Ivanovs and Stramkale, 2001; Ульянчик and Лукашик, 2008). For the variety 'Scorpion' the oil content was within the range of 33.6-43.8%, and for the variety 'Flanders' the range was 449-458 g kg^{-1}. The standard error for the oil content of variety 'Scorpion' was greater than for 'Flanders', which means that the oil content in the seeds of variety 'Scorpion' has a greater variation than that in variety 'Flanders'.

Table 2

Oil content in oil-flax seed DM, g kg^{-1}

Variety	Year		N fertilizer rate, kg ha^{-1}			
			N0	N60	N80	N100
Scorpion	2008	Average ± standard error	438±7.1	416±7.2	397±10.5	383±8.3
		min	427	413	361	351
		max	454	434	432	416
	2009	Average ± standard error	425±8.8	416±8.4	408±7.4	398±10.1
		min	395	391	384	352
		max	448	447	425	439
	2010	Average ± standard error	358±9.8	351±9.5	342±4.9	336±7.8
		min	339	332	334	325
		max	379	366	350	347
Flanders	2009	Average ± standard error	449±6.5	453±7.2	456±6.6	458±7.9
		min	435	447	451	449
		max	457	465	463	474
	2010	Average ± standard error	453±1.2	453±1.7	452±1.4	451±1.9
		min	451	450	450	448
		max	455	455	455	455

For the variety 'Scorpion', the seed yield increased from 0.09 to 0.59 t ha^{-1} when N fertilizer was used, and the flax straw yield increased from 0.24 to 0.71 t ha^{-1} in 2008 (Fig. 2). As the air temperature was higher in 2010 and there was a precipitation deficit in the second half of the summer (Fig. 1), the flax ripened earlier, which in its turn reduced the yield. Extracted oil varied from 0.36 to 0.88 t ha^{-1}, which is comparatively low, as other oil plants have a higher oil yield. Therefore it is better not to use oil-flax as a biofuel, as it can be better utilized elsewhere.

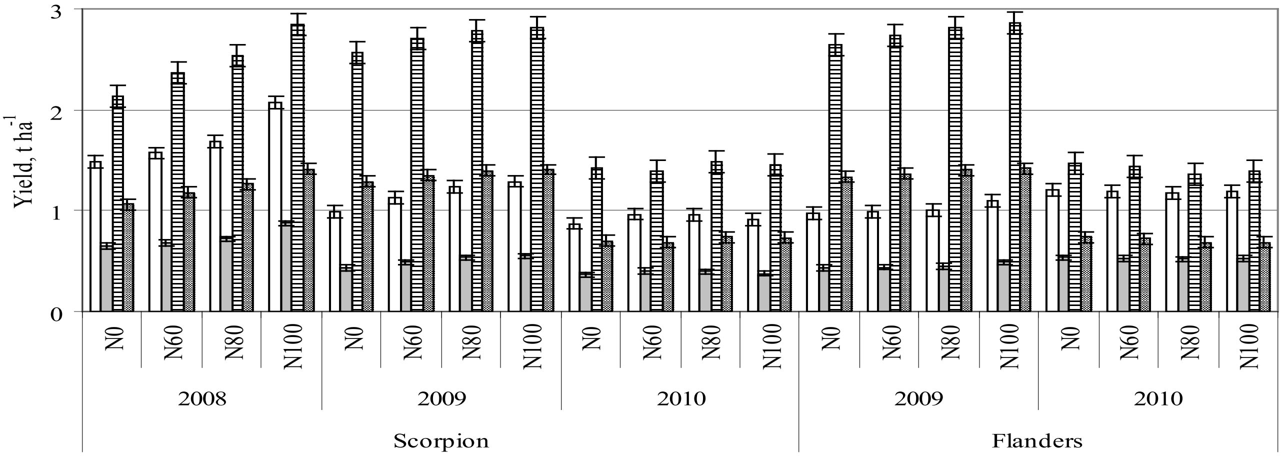

Figure 2. Oil-flax yield depending on the oil-flax variety, growing year, and N fertilizer rate, where
□ Seed yield, ▨ Oil yield, ▤ DM yield, ▩ Shove yield.

Ash represents the mineral content of biomass that depends on the soil and environmental conditions. In general, the ash content of crop materials biomass is significantly higher than that of wood (Volynets and Dahman, 2011).

The ash content in the flax shoves is from 15 g kg^{-1} (Ross and Mazza, 2010; Tamaki and Mazza, 2010). Our research shows that ash content in the shoves varied from 12.7 g kg^{-1} to 36.0 g kg^{-1} (Fig. 3). Which was significantly influenced ($p<0.05$) by the growth year ($\eta=82\%$), and by the interaction between the growth year, the variety, and the small N fertilizer rate ($\eta=1.5\%$).

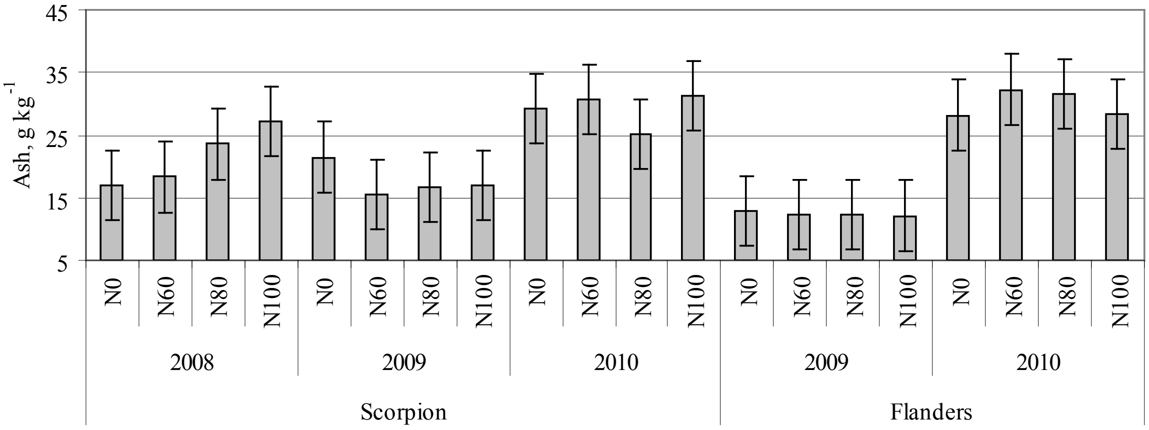

Figure 3. Ash content depending on the oil-flax variety, growing year, and N fertilizer rates.

Biomass ashes have a relatively low fusion temperature the deformation temperature (Dt) is normally in the range from 750 to 1000 °C, in comparison with coal which Dt exceeds 1000 °C; because the ash chemical and mineralogical composition for the coal and biomass is very different (Baxter and Koppejan, 2005). In our research for the oil-flax shoves the deformation temperature was 670 °C, which was not influenced by the growth year, the N supplementary fertilizer, or the chosen variety. The ash sphere temperature formation start was at 780-990 °C. The flax shove ash fusion temperature was much lower than for coal (1400 °C) (Magasiner et al., 2002).

In 2010, the gross calorific value - for the oil-flax shoves was from 18.2 to 19.4 MJ kg^{-1}, and the net calorific value from 15.45 to 16.08 MJ kg^{-1}. Nevertheless, the large amount of alkaline metals in the biomass, which causes corrosion deposit in the boilers, makes this type of biomass unsuitable for combustion (Wright et al., 2000; Volynets and Dahman, 2011).

A significant ($p<0.05$) close linear negative correlation was observed between Ca (x) and K content in the oil-flax shoves (y) ($r=0.771$; $n=10$), and the relationship is reflected in the regression equation $y = -0.60x + 10.92$; $R^2=0.59$.

The variety, the conditions of growth year, and the N supplementary fertilizer rate significantly ($p<0.05$) influenced the amount of chemical elements Ca, K, Na, Si - in the oil-flax shoves (Fig. 4 - 5). The higher level of K, Ca, Mg and Na in the biomass can be explained by the use of pesticides and fertilizers (Wright et al., 2000).

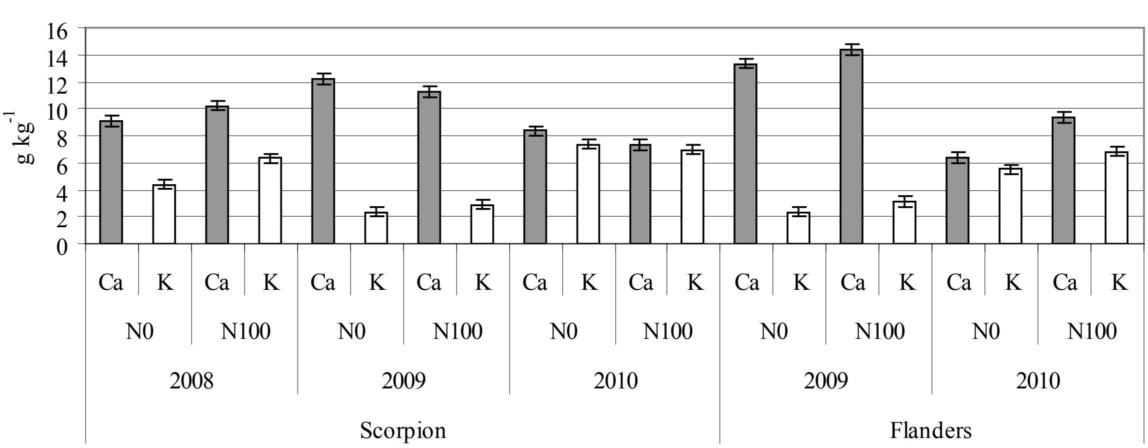

Figure 4. Potassium (K), and calcium (Ca) amount in oil-flax shoves depending on the oil-flax variety, growing year, and N fertilizer rates.

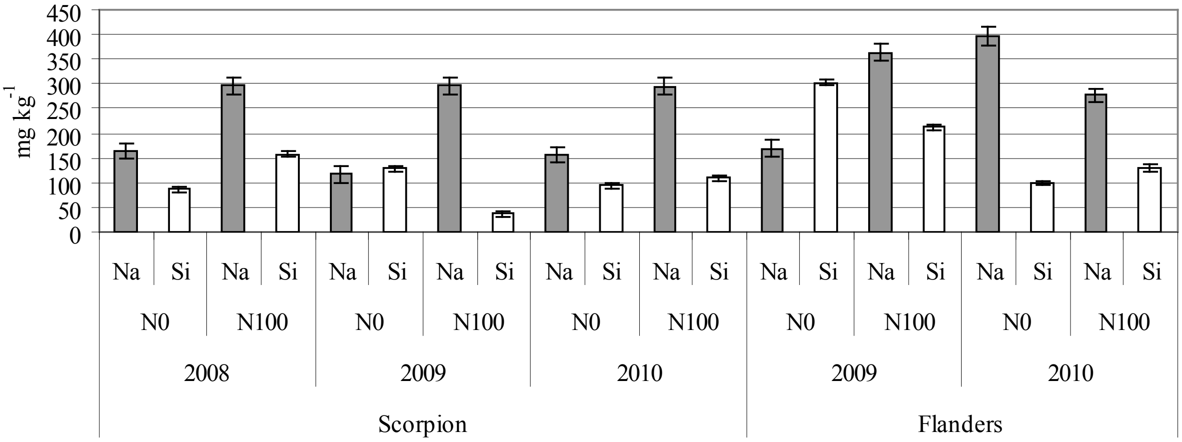

Figure 5. Sodium (Na) and silicon (Si) amount in oil-flax shoves depending on the oil-flax variety, growing year, and N fertilizer rates.

The high lignin content is detrimental for the quality requirements of textiles, cellulose and paper products, as lignin has a dark colouring, and during bleaching it tends to darken as autoxidation and /or photo oxidation tends to age it (Ross and Mazza 2010). The flax shoves have a typically high lignin content (230-310 g kg^{-1}), and high also cellulose (530 g kg^{-1}) and hemicellulose (240 g kg^{-1}) content (Tamaki and Mazza, 2010; Ross and Mazza, 2010.)

In our research, the lignin content in the oil-flax shoves was as follows: for 'Flanders' from 222 g kg^{-1} to 256 g kg^{-1}, and for 'Scorpion' from 245 g kg^{-1} to 271 g kg^{-1}. It was not influenced by the rate of N supplementary fertilizer.

The flax shoves can be used as a supplement for biofuel combustion, as they have a low fusion temperature, and a high calorific value. But a comparatively low yield makes oil-flax unprofitable to grow for biofuel production.

Conclusions

1. The variety's or genotype's, as a factor, influence is significant ($p<0.05$) on the oil content of the oil-flax seeds. The variety 'Scorpion' had an oil content within the range of 336-438 g kg^{-1}, and the variety 'Flanders' - 449 to 458 g kg^{-1}. The oil yield was from 0.36 to 0.88 t ha^{-1}.
2. The ash content in the shoves varied from 12.7% to 360 g kg^{-1}, and was influenced by the conditions of the growth year.
3. For the oil-flax shoves the ash deformation temperature was 670 °C, the start of the sphere formation temperature was at 780-990 °C, which is a lot less than for coal (1400 °C).
4. In 2010, the gross calorific value ranged from 18.2 to19.4 MJ kg^{-1} for the shoves, and the net calorific value - from 15.45 to 16.08 MJ kg^{-1}.
5. The lignin content for the variety 'Flanders' was from 222 g kg^{-1} to 256 g kg^{-1}, and for the variety 'Scorpion' from 245 g kg^{-1} to 271 g kg^{-1}. As the shoves are a waste by-product, they can be a good supplement for the manufacture of biofuel.

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References

1. Baxter L., Koppejan J. (2005) Biomass-coal co-combustion: opportunity for affordable renewable energy. *Fuel*, 84, pp. 1295-1302.
2. Biomass Energy (n.y.) Available at: http://www.seco.cpa.state.tx.us/publications/renewenergy/biomassenergy.ph, 25 January 2011.
3. Freimanis P., Holms I., Jurševskis L., Lauva J., Ruža A. (1980) *Augkopības praktikums* (Practicum of crop production). Rīga, Zvaigzne, 326 lpp. (in Latvian).
4. Ivanovs S., Stramkale V. (2001) *Linu audzēšanas un novākšanas tehnoloģijas* (Technology of flax growing and harvesting). LLU Ulbrokas Zinātnes centrs, 191 lpp. (in Latvian).
5. Magasiner N., van Alphen M., Inkson M., Misplon B. (2002) Characterising Fuels For Biomass – Coal Fired Cogeneration. *International Sugar Journal*, 104, No. 1242, pp. 251-267.
6. Poiša L., Adamovičs A., Stramkale V., Komlajeva L. (2010) Eļļas lini atjaunojamās enerģijas iegūšanai (Oil-flax for renewable energy). *Latgale National Economy research*, *Journal of Social Sciences*. No. 1 (2), 290-300. lpp. (in Latvian).

7. Ross K., Mazza G. (2010) Characteristics of Lignin from Flax Shoves as Affected by Extraction Conditions. *International Journal of Molecular Sciences*, 11, pp. 4035-4050.
8. Stramkale V., Poiša L., Vikmane M., Stramkalis A., Komlajeva Ļ. (2008) Eļļas linu audzēšana un izmantošanas iespējas Latvijā (Oil-flax breeding and application opportunity in Latvia). *Opportunities and challenges of national economic development*: Proceedings of the International Conference, April 17, 2008, Rēzekne, 412-420. lpp. (in Latvian).
9. Tamaki Y., Mazza G. (2010) Measurement of structural carbohydrates, and micro-components of straw and shoves: Effects of extractives, particle size and crop species. *Industrial Crops and Products*, 31, pp. 534-541.
10. Volynets B., Dahman Y. (2011) Assessment of pretreatments and enzymatic hydrolysis of wheat straw as a sugar source for bioprocess industry. *International Journal of Energy and Environment*, 2, pp. 727-446.
11. Zaķis Ģ. (2008) *Koksnes ķīmijas pamati* (Wood Chemistry Basics). LV Koksnes ķīmijas institūts, Rīga, 199 lpp. (in Latvian).
12. Wright I.G., Leyens C., Pint B.A. (2000) *An Analysis of the potential for deposition, erosion, or corrosion in gas turbines fueled by the products of biomass gasification or combustion*, ASME Paper No. 2000-GT-0019.
13. Груздевене Э., Янкаускиене З., Манкевичене А. (2009) Влияние условий окружающей среды и генотипа на урожай и качество семян льна масличного (Influence of environmental conditions and genotype on the oil-flax yield and seed quality). *Enviroment. Technology. Resources*: Proceedings of the 7th International Scientific and Practical Conference, June 25-27, 2009, Vol. 1, Rezekne, c. 154-159. (in Russian).
14. Ульянчик В.И., Лукашик Г.Н. (2008) Влияние ростовых веществ и микроудобрений на продуктивность и качество льна масличного. *Льноводство: реалии и перспективы*: сборник научных материалов международной научно-практической конференции на РУП „Институт льна” 25-27 июня 2008 года. Могилев: Могилевская областная укрепнная типография имени Спиридона Соболя, с. 184-190. (in Russian).