THE NEED FOR STANDARDIZATION OF MECHANICALLY EXTRACTED RAPESEED AS A BY-PRODUCT FROM BIOFUEL PRODUCTION

POTREBA STANDARDIZIRANJA MEHANIČKI EKSTRAHIRANE ULJNE REPICE KAO NUSPROIZVODA U PROIZVODNJI BIOGORIVA

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SUMMARY

A chemical composition and nutritional value of rapeseed expeller available on the domestic market as feed by-product in bio-fuel production was determined. The following mean basal nutrient values were obtained: dry matter 909 g/kg, crude ash 60.8 g/kg, total protein 322 g/kg, crude fat 111 g/kg, crude fiber 114 g/kg, starch 50.4 g/kg, sugar 90.7 g/kg and fiber fractions: NDF 228 g/kg, ADF 162 g/kg and ADL 63.4 g/kg. Metabolizable energy values were 10.2 MJ/kg for poultry and 14.6 MJ/kg for swine. Amino acid contents ofrapeseed expeller appeared to be similar to those in literature. Study results indicate the need for standardization of rapeseed expeller in reference to fat content: low fat (100 g/kg) and high fat (240 g/kg). Concentrations of macroelements: calcium 6.68 g/kg, phosphorus 9.05 g/kg, magnesium 3.84 g/kg,sodium 21.5 mg/kg, potassium 12.0 g/kg and microelements: iron (166 mg/kg), manganese (61.8 mg/kg), zinc (55.8 mg/kg),copper (3.06 mg/kg), cobalt (0.21 mg/kg), molybdenum (0.45 mg/kg), selenium (0.16 mg/kg) and iodine 0.11 mg/kg in most cases were similar to literature data. Undesirable and hazardous substances such as heavy metals (cadmium 0.05 mg/kg, lead 0.13 mg/kg) and other elements (arsenic < 0.1 mg/kg;fluorine < 1.0 mg/kg), mycotoxins (ochratoxin A 7.1 μg/kg , zearalenon 35.2 μg/kg, and deoxynivalenol 108 μg/kg) appeared to be many times lower than permissible limits, which confirmed that studied mechanically extracted rapeseed was valuable and safe feed material.

Key words: rapeseed expeller, nutrient, undesirable substance, standardization

INTRODUCTION

A rational utilization of rapeseed expeller – by-product in bio-fuel extraction – for feed production depends on the knowledge of its chemical composition and nutritional value, namely components for balancing the compound feeds recipes. The safety reasons in applying rapeseed expeller also require the evaluation of a potential risk of

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undesirable and hazardous substances, as well as antinutritive agents (IFSA Standard, 2005).

Different yields of oil extraction make rapeseed expeller, unlike rapeseed extraction meal, non-uniform feed material. Nutritional value of rapeseed expeller depends not only on seed chemical composition (Matyka et al. 1992), but first of all, it varies depending on the oil extraction level (Podkówka, 2004). Therefore, to avoid errors, feed nutritional value and chemical composition tables should be used thoroughly for optimizing the recipes of industrial feeds including rapeseed expeller. Some sources, e.g. Poultry Feeding Standards edited by Smulikowska and Rutkowski (2005) give data on two types of expeller low-fat with about 12% and high-fat with 22% of oil content.

Preliminary studies carried out at National Feed Laboratory revealed that rapeseed expeller may contain varied oil concentrations (Korol et al. 2007). Therefore, the decision was made to continue the study in order to verify not only the oil content in rapeseed expeller, but also other nutrients, including starch and sugars, amino acids, macroelements and microelements, heavy metals and hazardous elements and compounds, mycotoxins, pesticide residues, as well as antinutritive agents.

MATERIAL AND METHODS

Material for the study consisted of 50 rapeseed expeller samples collected throughout Poland in 2006. Samples were taken by official sample-collectors from Veterinary Inspection within feed monitoring or as National Feed Laboratory order.

Basal nutrient analyses, starch, sugar, fiber fractions, and metabolizable energy evaluation on the basis of regression equations were made of all collected samples. Amounts ofbasal nutrients: total protein, crude fat, crude fiber, moisture, and ash were determined by official methods using automatic analyzers in the case of protein, fat, and fiber (Regulation of MARD, 2007). Starch was analyzed using polarimetric method and sugar concentration by Luff-Schoorl's method (Regulation of MARD, 2007). Fiber fractions were assayed according to Ankom methods (Operator's Manual, 1997). Metabolizable energy of rapeseed expeller for poultry was calculated according to Smulikowska and Rutkowski, 2005; for pigs – according to Hoffmann and Schiemann's equation with Müller and Kirchgessner's modifications given in Pig Feeding Standards (1993).

Analyses of amino acids, fatty acids, macroelements, microelements, heavy metals, hazardous elements and compounds, mycotoxins, pesticide remains, and anti-nutritive agents were performed for 20 samples. Calcium, magnesium, sodium, potassium, iron, manganese, zinc and copper contents were determined by means offlame atomic absorption spectrometry FAAS technique in accordance with the ISO 6869:2002 standard after sample dry combustion in muffle furnace at 550 °C. Phosphorus was analyzed spectrophotometrically (Regulation of MARD, 2007). Cobalt and molybdenum by graphite furnace atomic absorption spectrometry GFAAS according to GBC Avanta Ultra Z Methods Manual (2003); selenium by hydrige generation atomic absorption spectrometry HGAAS according to Polish Standard (1998); iodine by spectrophotometric kinetic-catalytic method after sample alkali combustion (Gąsior, 2007). Amino acids (without tryptophan) were determined by ion-exchange chromatography in automatic amino acid analyzer (Commission Directive 98/64/EC, 1998). Tryptophan was assayed by high performance liquid chromatography method (Commission Directive 2000/45/EC, 2000). Mycotoxins – ochratoxin A (OTA), zearalenon (ZEA), and deoxynivalenol (DON) were assayed using high performance liquid chromatography methods (Grajewski et al., 2006). Cadmium and lead were determined by GFAAS after sample wet digestion in microwave digester (EN 15550 : 2007). Mercury was analyzed applying the AAS cold vapors technique (Philips Scientific, 1988); fluorine – spectrometry after sample alkali combusting and fluorine separation in micro-diffusion chamber (Branch Standard, 1978); phytate phosphorus – after precipitating phytic acid, its dissolving, and then determining the phosphorus contained in phytic acid by means of spectrophotometry (Oberleas, 1971). Glucosinolates were determined using a semi-quantitative glucose test according to the Polish Standard (1990). The tannins content was estimated by a spectrophotometric method (Tyczkowska, 1977). Obtained results were statistically processed by calculating mean value, standard deviation and coefficient of variation (Statistica, 8.0) and compared with literature and table data.

RESULTS AND DISCUSSION

Analysis of results from basal nutrients included in rapeseed expeller, namely crude fat, total protein, crude fiber, and fiber fractions (NDF, ADF, and ADL) allowed for distinguishing high-fat samples (Table 1). Those samples made up 16% of tested expeller and contained about 24% of crude fat vs. about 10% in other samples. Rapeseed expeller with 10% of fat was characterized by chemical composition similar to that found in Poultry Feeding Standards (Smulikowska and Rutkowski, 2005). Metabolizable energy appeared to vary – it was similar to table data for expeller with 10% of fat content and by 3-4 MJ/kg higher in the case of fat expeller. Lower coefficient of variation values were achieved within the group of expellers, e.g. the coefficient decreased from 35% to about 13% for fat, while in the case of protein it decreased from 8% to 4%. Thus, rapeseed expeller samples should be standardized depending on fat content. According to Council Directive 96/25/EC and Regulation of Agriculture and Rural Development Ministry on feed labeling (Regulation of MARD, 2007a), a producer who introduces rapeseed expeller onto market, is obliged to declare the crude fat, total protein, and crude fiber contents. No doubt, such requirement is legitimate in the view of here achieved results.

Table 1. Contents of basal nutrients and energetic value of tested rapeseed expellers (n=50)

Tablica 1. Sadržaj osnovnih hranjivih tvari i energetska vrijednost testirane mehanički ekstrahirane uljne repice (n=50)

Nutrient, g/kg	Total rapeseed expellers (n=50) Mean ± SD (CV)	Low fat rapeseed expellers (n=42) Mean ± SD (CV)	High fat rapeseed expellers (n=8) Mean ± SD (CV)
Dry matter	909 ± 22.2 (2.4)	910 ± 22.9 (2.5)	900 ± 5.4 (0.6)
Total protein	322.2 ± 24.8 (7.7)	327.9 ± 14.3 (4.4)	250.0 ± 4.1 (1.6)
Crude fat	111.3 ± 39.0 (35.0)	101.2 ± 13.9 (13.7)	238.4 ± 7.1 (3.0)
Ash	60.8 ± 2.08 (3.4)	61.0 ± 2.09 (3.4)	59.3 ± 2.6 (4.4)
Crude fiber	113.9 ± 7.8 (6.8)	115.0 ± 7.0 (6.1)	99.8 ± 4.3 (4.3)
NDF	227.5 ± 29.5 (13.0)	230.2 ± 29.0 (12.6)	195.0 ± 8.1 (4.2)
ADF	162.5 ± 11.2 (6.9)	164.2 ± 9.6 (5.8)	140.8 ± 5.1 (3.6)
ADL	63.4 ± 4.8 (7.6)	64.0 ± 4.5 (7.0)	56.5 ± 1.4 (2.5)
Starch	50.4 ± 7.8 (15.5)	50.6 ± 8.1 (16.0)	48.1 ± 3.5 (7.3))
Sugars as sucrose	90.7 ± 7.0 (7.7)	92.2 ± 4.3 (4.7)	71.4 ± 2.1 (2.9)
EM_N – poultry, MJ/kg	10.2 ± 1.07 (10.5)	9.7 ± 0.58 (6.0)	13.2 ± 0.38 (2.9)
EM – swine, MJ/kg	14.6 ± 0.72 (4.9)	14.7 ± 0.70 (4.8)	17.2 ± 0.47 (2.7)

SD – standard deviation, g/kg; CV – coefficient of variation, %

Determined concentrations of exogenous amino acids (lysine, methionine, and tryptophan) in rapeseed expeller appeared to be similar to table data (Smulikowska and Rutkowski, 2005), (Table 2). After distinguishing the results for high-fat samples, results of particular amino acids were characterized by slight variability, not higher than 10%, in general.

Results from macroelements determinations (phosphorus, calcium, magnesium, potassium) in rapeseed expeller appeared to be similar to data contained in poultry feeding recommendations and INRA tables (Smulikowska and Rutkowski, 2005, Raw Material Compendium, 1996), (Table 3). The highest differences referred to sodium content: 400 mg/kg according to Smulikowska and Rutkowski (2005), 17 mg/kg according to own study results, and 70 mg/kg according to INRA (Raw Material Compendium, 1996).

Concentrations of iron, manganese, zinc, molybdenum, and selenium in rapeseed expeller determined in present examinations were similar to those of INRA (Raw Material Compendium, 1996) (Table 3). Our own determinations revealed by about 40% lower copper and higher cobalt levels. Rapeseed expeller did not contain excessive iron, manganese, zinc, copper, cobalt, selenium, and molybdenum, therefore there is no risk for maximum levels of these microelements exceeding in compound feeds with the share of this feed material (Commission Regulation 1334/2003/EC, 2003), (Table 3).

Table 2. Amino acid profile of rapeseed expellers (n=20)

Tablica 2. Profil aminokiselina u mehanički ekstrahiranoj uljnoj repici (n=20)

Amino acids, g/kg	Total rapeseed expellers (n=20)	Low fat rapeseed expellers (n=17)	High fat rapeseed expellers (n=3)
	Mean ± SD (CV)	Mean ± SD (CV)	Mean ± SD (CV)
Dry matter	909 ± 22.2 (2.4)	910 ± 22.9 (2.5)	900 ± 5.4 (0.6)
Asparagine	22.4±0.74 (3.3)	22.8±0.64 (2.8)	17.4±0.35 (2.0)
Threonine	13.9±1.70 (12.2)	14.2±1.04 (7.3)	10.8±0.66 (6.1)
Serine	14.1±1.12 (7.9)	14.4 ±0.77 (5.3)	11.0±0.52 (4.7)
Glutamine	46.3±1.87 (4.0)	47.2±1.67 (3.5)	36.0±1.07 (3.0)
Proline	17.7±0.85 (4.8)	18.0±0.75 (4.2)	13.8±0.45 (3.3)
Glycine	15.0±0.96 (6.4)	15.3±0.66 (4.3)	11.7±0.36 (3.1)
Alanine	12.6±1.11 (8.8)	12.9 ±0.94 (7.3)	9.8±0.59 (6.0)
Valine	15.3±0.94 (6.1)	15.6 ±0.74 (4.7)	11.9±0.47 (3.9)
Isoleucine	11.4±0.61 (5.4)	11.6±0.50 (4.3)	8.85±0.37 (4.2)
Leucine	20.2±1.18 (5.9)	20.6±0.88 (4.3)	15.7±0.58 (3.7)
Thyrosine	8.47±0.66 (7.8)	8.6±0.46 (5.3)	6.6±0.36 (5.4)
Phenyloalanine	11.8±0.66 (5.6)	12.0±0.52 (4.3)	9.2±0.38 (4.1)
Histidine	8.8±0.40 (4.5)	9.0±0.38 (4.2)	6.8±0.21 (3.1)
Lysine	16.8±1.62 (9.6)	17.1±1.02 (6.0)	13.1±0.62 (4.7)
Arginine	17.3±0.97 (5.6)	17.6±0.76 (4.3)	13.4±0.47 (3.5)
Cysteine	10.2±1.27 (12.5)	10.4±0.85 (8.2)	7.9±0.57 (7.2)
Methionine	6.50±0.82 (12.6)	6.60±0.52 (7.9)	5.0±0.32 (6.4)
Tryptophan	4.3±0.31 (7.2)	4.4±0.19 (4.3)	3.34±0.15 (4.5)

SD – standard deviation, g/kg; CV – coefficient of variation, %

Table 3. Macro- and microelements in rapeseed expellers (n=20)

Tablica 3. Makro i mikroelementi u mehanički ekstrahiranoj uljnoj repici (n=20)

Mineral components	Range	Mean value	Standard deviation, SD
Dry matter, g/kg	868 - 947	914	23.8
Phosphorus, g/kg	8.06 - 9.70	9.05	0.39
Calcium, g/kg	5.58 - 7.95	6.68	0.73
Magnesium, g/kg	3.21 - 4.24	3.84	0.27
Sodium, mg/kg	7.2 - 50.3	21.5	9.7
Potassium, g/kg	9.3 - 13.2	12.0	1.04
Iron, mg/kg	48 - 476	166	93.4
Manganese, mg/kg	42.1 - 162	61.8	28.3
Copper, mg/kg	2.53 - 4.08	3.06	0.55
Zinc, mg/kg	40.9 - 72.8	55.8	10.1
Cobalt, mg/kg	0.09 - 0.38	0.21	0.09
Molybdenum, mg/kg	0.11 - 0.65	0.45	0.21
Selenium, mg/kg	0.03 - 0.39	0.16	0.13
Iodine, mg/kg	0.10-0.14	0.11	0.015

Concentration of glucosinolates was lower than 25 µmol/g of defatted DM for all analyzed samples indicating that tested rapeseed expellers were obtained from “double-zero” rapeseed cultivars. Level of phytate phosphorus and tannins in studied expeller samples appeared to be similar to that found in rapeseed seeds after recalculation onto defatted sample (Matyka et al., 1992). Phytic phosphorus form dominated in rapeseed seeds, while non-phytic phosphorus made up only about 20% of total phosphorus (Table 4).

Table 4. Contents of undesirable and anti-nutritive substances in rapeseed expellers (n=20)

Tablica 4. Nepoželjne i nehranive tvari u mehanički ekstrahiranoj uljnoj repici (n=20)

Specification	Range	Mean value	Standard deviation, SD
Dry matter, g/kg	868-947	909	22.2
Glucosinolates, µmol/g of defatted DM	<25	<25	nd
Ochratoxin A, µg/kg	1.3-18.1	7.1	6.0
Zearalenon, µg/kg	0-104	35.2	37.4
Deoxynivalenol, µg/kg	0-265	108	90
Phytate phosphorus, g/kg	6.65-8.50	7.45	0.53
Tannins, g/kg	12.3-18.3	16.0	1.72
Fluorine, mg/kg	< 1	< 1	nd
Arsenic, mg/kg	< 0.1	< 0.1	nd
Mercury, mg/kg	<0.01	<0.01	nd
Cadmium, mg/kg	0.02-0.09	0.05	0.02
Lead, mg/kg	0.04-0.24	0.13	0.06

nd – not determined

Mean mycotoxins content (OTA, ZEA, and DON) in tested rapeseed expeller appeared to be safe, and even their highest levels were not the risk for animals and animal-origin products: OTA – 18 μg/kg (ppb), ZEA – 104 μg/kg, DON – 265μg/kg. Therefore, rapeseed expellers available on domestic market are safe for animals as regards mycotoxins concentrations (Table 4), (Grajewski, 2006).

Amounts of arsenic in analyzed expeller samples was lower than 0.1 mg/kg. Mean mercury (0.01 mg/kg), cadmium (0.05 mg/kg), and lead (0.13 mg/kg) content was over 10 times lower than permitted (70-fold lower in the case of lead). Also fluorine amounts were many times lower as compared to permitted ones. Results of performed analyses confirmed that rapeseed expellers produced in Poland was safe.

Performed analyses allowed for evaluating the chemical composition and nutritional value of rapeseed expellers produced in Poland and compare results with literature and table data as well as permitted undesirable and hazardous substances listed inDirective 32/2002/EC (2002). Study results indicate the need to standardize rapeseed expeller in the view of the fat content, because protein, amino acid, and fiber concentrations greatly depend on fat level. Levels of such macroelements as phosphorus, calcium, magnesium, sodium, and potassium as well as microelements like iron, manganese, zinc, copper, cobalt, molybdenum, and selenium in most cases appeared to be similar to table data. Amounts of anti-nutritive substances, tannins, and phytates were typical for studied materials. Recorded amounts of undesirable and hazardous substances – heavy metals and hazardous elements (As, F), mycotoxins (OTA, DON, ZEA) – were much lower than permitted, which confirmed that tested rapeseed expellers were valuable and safe feed material.

CONCLUSION

The obtained results indicated that rapeseed expellers should be standardized depending on the fat content. Lower coefficient of variation of basal nutrient and amino acid values were achieved within the group of expellers low in fat and high in fat, compared to all results irrespective of the fat content. Amounts of basal nutrients, amino acids, macro- and microelements appeared to be similar to those in literature and table data. Low levels of anti-nutritive agents: glucosinolates and tannins were indicated that tested rapeseed expellers were obtained from “double-zero” rapeseed cultivars. No risk of heavy metal (Cd, Hg, Pb)and toxic elements (As, F) as well as mycotoxins (OTA, ZEA and DON) confirmed that tested rapeseed expellers as a by-product of biofuel production are valuable and safe material for animal feeding.

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SAŽETAK

Određen je kemijski sastav i hranidbena vrijednost mehanički ekstrahirane uljne repice na domaćem tržištu kao nusproizvod u proizvodnji biogoriva. Dobivene su sljedeće srednje hranidbene vrijednosti bazalnih hranjivih tvari: suha tvar 909 g/kg, pepeo 60,8 g/kg, ukupne bjelančevine 322 g/kg, sirova mast 111 g/kg, sirova vlaknina 114 g/kg, škrob 50,4 g/kg, šećer 90,7 g/kg i frakcije vlakana: NDF 228 g/kg, ADF 162 g/kg i ADL 63,4 g/kg. Vrijednosti metabolizirane energije bile su 10,2 MJ/kg za perad i 14,6 MJ/kg za svinje. Sadržaj aminokiselina mehanički ekstrahirane uljne repice bio je sličan onima iz literature. Rezultati istraživanja upućuju na potrebu standardiziranja mehanički ekstrahirane uljne repice s obzirom na sadržaj masti:niska masnoća (100 g/kg) i visoka masnoća (240 g/kg). Koncentracije makroelemenata: kalcij 6,68 g/kg, fosfor 9,05 g/kg, magnezij 3,84 g/kg, natrij 21,5 g/kg, kalij 12,0 g/kg i mikroelemenata: željezo (166 mg/kg), mangan (61,8 mg/kg), cink (55,8 mg/kg), bakar (3,06 mg/kg), kobalt (0,21 mg/kg), molibden (0,45 mg/kg), selen (0,16 mg/kg) i jod (0,11 mg/kg) bile su u većini slučajeva slične podacima u literaturi. Nepoželjne i štetne tvari kao što su teški metali (kadmij 0,05 mg/kg, olovo 0,13 mg/kg) i drugi elementi (arsen <0,1 mg/kg, fluor <1,0 mg/kg), mikotoksini (ohratoksin A 7.1 mg/kg, zearalenon 35,2 mg/kg i deoksinivalenol 108 mg/kg) bili su mnogo puta niži od dozvoljenih granica, što je potvrdilo da je istraživana mehanički ekstrahirana repica vrijedan i siguran hranidbeni materijal.

Ključne riječi: mehanički ekstrahirana uljna repica, hrannjive tvari, aninutritivne tvari, standardiziranje