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## **EXTRUSION OF RAPESEED INTENDED FOR ANIMAL FEED. PART II. OPTIMALIZATION OF PROCESS PARAMETERS AND THEIR EFFECT ON THE CONTENT OF HARMFUL COMPOUNDS. EXTRUSION WITH AMMONIA**

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Key words: rapeseed, pea, extrusion, glucosinolates, VTO, ITC, sodium chloride.

The extrusion of rapeseed with pea completely eliminates the nutrition-inhibiting agents in pea, and partly eliminates the glucosinolates and VTO of rapeseed. Extrusion with ammonia leads to a further partial reduction of ITC, VTO and glucosinolates. An addition of sodium chloride improves the quality of the extrudate.

In part I of this publication we discussed the technological aspect of the production of the high-energy and protein animal feed component by extruding rapeseed with pea. In subsequent studies aimed at optimizing this process we determined the effect of sodium chloride used as an agent improving extrusion as well as the quality of the obtained extrudate. We also investigated changes in the contents of trypsin inhibitor, ITC, VTO and glucosinolates in the obtained products depending on various temperatures of extrusion, and determined the optimum temperature range for the process. Finally, we studied the effect of ammonia on extrudate quality.

### **MATERIAL AND METHODS**

In addition to the materials and methods described in part I of this publication [6], we made use of Kakade's method as modified by Valdebouze and Gaborit [5] to determine the content of trypsin inhibitor. The contents of glucosinolates, ITC and VTO and macrocomponents such as dry mass, protein, fibre, ash, fats and ammonia nitrogen were determined according to French norms (AFNOR).

## RESULTS AND DISCUSSION

The grain size of ground rapeseed and pea is given in Table 1. As can be seen, uncomminuted rapeseeds ( $> 1.6$  mm fraction) account for only 1.5% of the flour; the  $D_{50}$  diameter of rapeseed particles is 0.52 mm, and of pea particles 0.60 mm. An analysis of the obtained fractions show that the obtained rapeseed-pea mixture is sufficiently homogeneous to ensure uniform and rapid heat flow throughout its mass during extrusion.

Table 1. Grinding conditions of raw materials in Rousselle grinder

Raw material	Diameter $D_{50}$ (mm)	Fraction (%)			
		$< 0.5$ mm	0.5-1.25 mm	1.25-1.6 mm	$> 1.6$ mm
Rapeseed	0.52	54.6	40.3	3.6	1.5
Pea	0.60	48.6	42.8	6.9	1.7

We sought the best parameters of extrusion leading to the best physical properties of the product by modifying the worm structure, the outlet nozzle diameter, worm rotation speed, the amount of water added to the mixture, and the rate of mixture processing [7]. The experiments were performed with a new model of extruder with head length extended to 1000 mm.

An interesting problem was the determination of the optimal amount of sodium chloride to be added to the initial mixture. Sodium chloride vastly improves the physical properties of the extrudate, and this is very welcome given the fact that the fat in the mixture reduced the hardness and plasticity of the extrudate, the effect being further compounded by the fact that these fats are of vegetable origin and hence with a low melting point.

The mean diameter ( $D_{50}$ ) of the applied salt was 0.45 mm; the contents of the various fractions were as follows:  $> 1.000$  mm — 0.6%; 0.800-1.000 — 2.5%; 0.630-0.800 — 19.3%; 0.500-0.630 — 26.5%; 0.420-0.500 — 15.7%; 0.315-0.420 — 11.4%; 0.200-0.315 — 12.4%; 0.104-0.200 — 6.7%;  $< 0.104$  — 4.9%. The size of salt particles and the rate of their dissolution are of crucial importance in extrusion. The smaller the particles, the quicker their complete dissolution in water. The properties of this salt satisfied Harper's requirements [4].

The sodium chloride addition amounted to 1-5% of the rapeseed-pea mixture (Table 2), and extrusion conditions were as follows:

worm structure — 200/50, 100/50, 100/50, 100/35, 100/35, 100/25, 50/25, 50/15, 50/15, 100/15, C: 50/15;

diameter of extruder's outlet nozzle — 3 mm;

extrusion temperature — 200°C;

worm rotation — 200 r.p.m.;

water addition — 11.4 kg/h;

extrudate yield — 104 kg/h;

Table 2. Physical characteristics of extruded mixture rapeseed-pea with NaCl

Physical characteristics	NaCl dose in the extruded mixture (%)				
	1	2	3	4	5
Mixture humidity (%)	11.5	11.5	11.5	11.4	11.4
Extrudate humidity (%)	14.0	13.4	13.1	12.3	10.7
Bulk density (g/l)	362	355	374	360	324
Mass density (g/cm <sup>3</sup> )	0.83	0.85	0.90	0.91	0.92
Expansion rate (Se/So)	2.1	2.4	2.1	1.7	1.6
Hardness (kG)	2.5	2.5	3.6	3.9	4.0
Plasticity (%)	75.8	80.8	80.8	83.8	82.9

energy consumed by extruder — 148.7-159.2 kWh/t at 1-5% salt addition;  
 electric current in extruder engine — 11-13 A (1-5% of salt);  
 time of mixture passage through extruder head — 20 sec;  
 pressure in extruder head — from 30-31 to 36-37 bars (1-5% of salt).

The optimal NaCl additions was determined (Fig. 1) by superimposing curves illustrating the features characterizing extrudate quality (humidity, hardness and plasticity); expansion rate and energy consumed by the extruder are also marked on the diagram. The best results were obtained with a 3% NaCl addition: extrudate hardness in excess of 3.5 kG, plasticity — 81%, and humidity — 13% (Table 2).

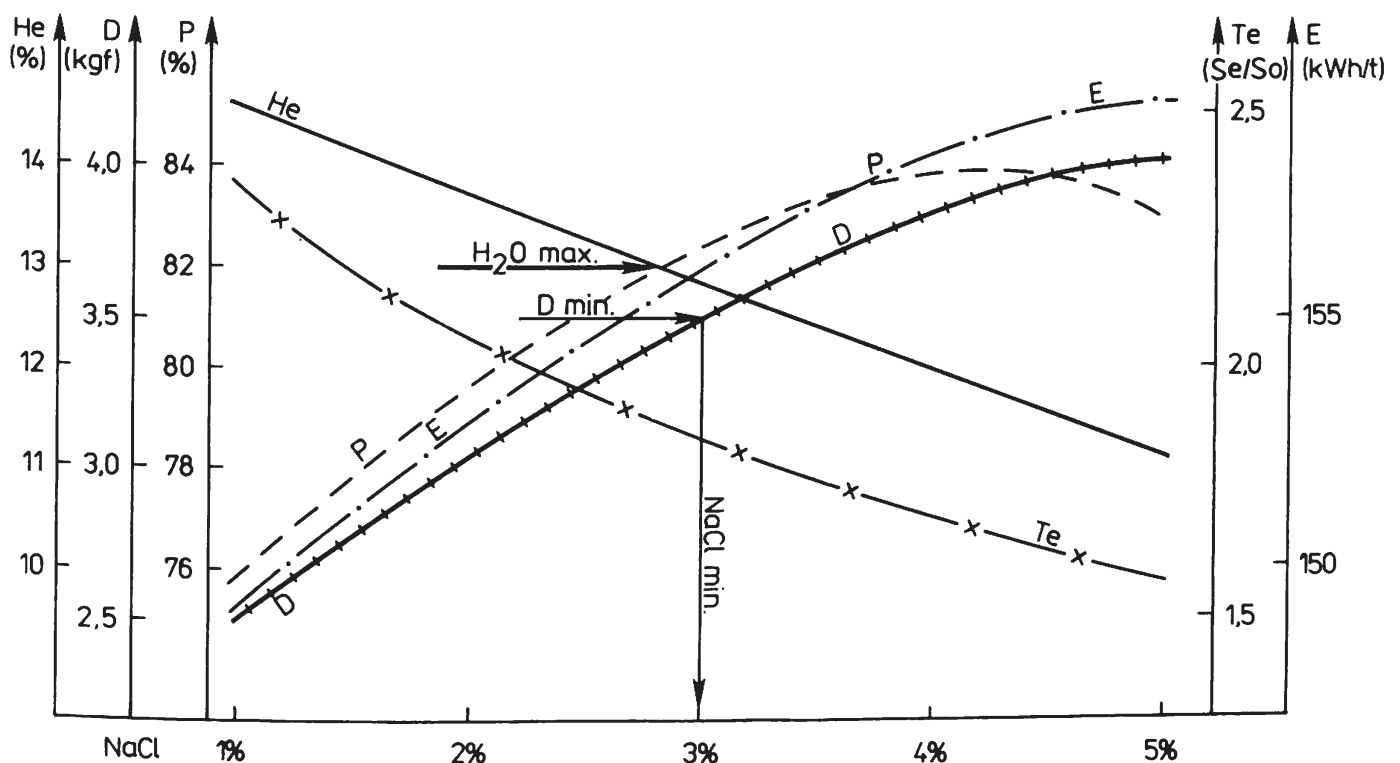


Fig. 1. Determination of optimal NaCl dose

The next parameter to be determined was the optimal temperature of extrusion. Generally speaking, this temperature may range from 110 to 215°C, and usually the process is never performed outside this range. The physical properties of a rapeseed-pea mixture with 3% NaCl, extruded at 120-210°C, are

given in Table 3 (determinations were made every 10°C); the remaining conditions of extrusion were as above. The optimal temperature of extrusion was determined by comparing the humidity, hardness and plasticity of the obtained extrudates, and the trypsin inhibitor content (Fig. 2). The extrudate humidity decreases with the increase of temperature: from 16.5% at 120°C to 11.2% at 210°C (Table 3). The extrudate's hardness stays above the previously determined minimum of 3.5 kG up to 200°C, and then drops to 3.3 kG at 210°C. Plasticity is similarly affected, staying above the minimal value of 70-75%.

Table 3. Physical characteristics of extruded mixture rapeseed-pea with 3% NaCl in the different extrusion temperature

Physical characteristics	Extrusion temperature (°C)									
	120	130	140	150	160	170	180	190	200	210
Extrudate humidity (%)	16.5	15.9	15.1	14.9	14.1	13.3	12.8	11.6	11.4	11.2
Expansion rate (Se/So)	1.2	1.2	1.5	1.6	1.7	1.9	1.9	1.9	2.0	1.7
Hardness (kG)	3.5	3.8	3.6	3.5	3.5	3.8	3.7	3.7	3.5	3.3
Plasticity (%)	80.8	81.0	78.6	80.0	80.7	79.5	82.7	76.9	75.0	74.5

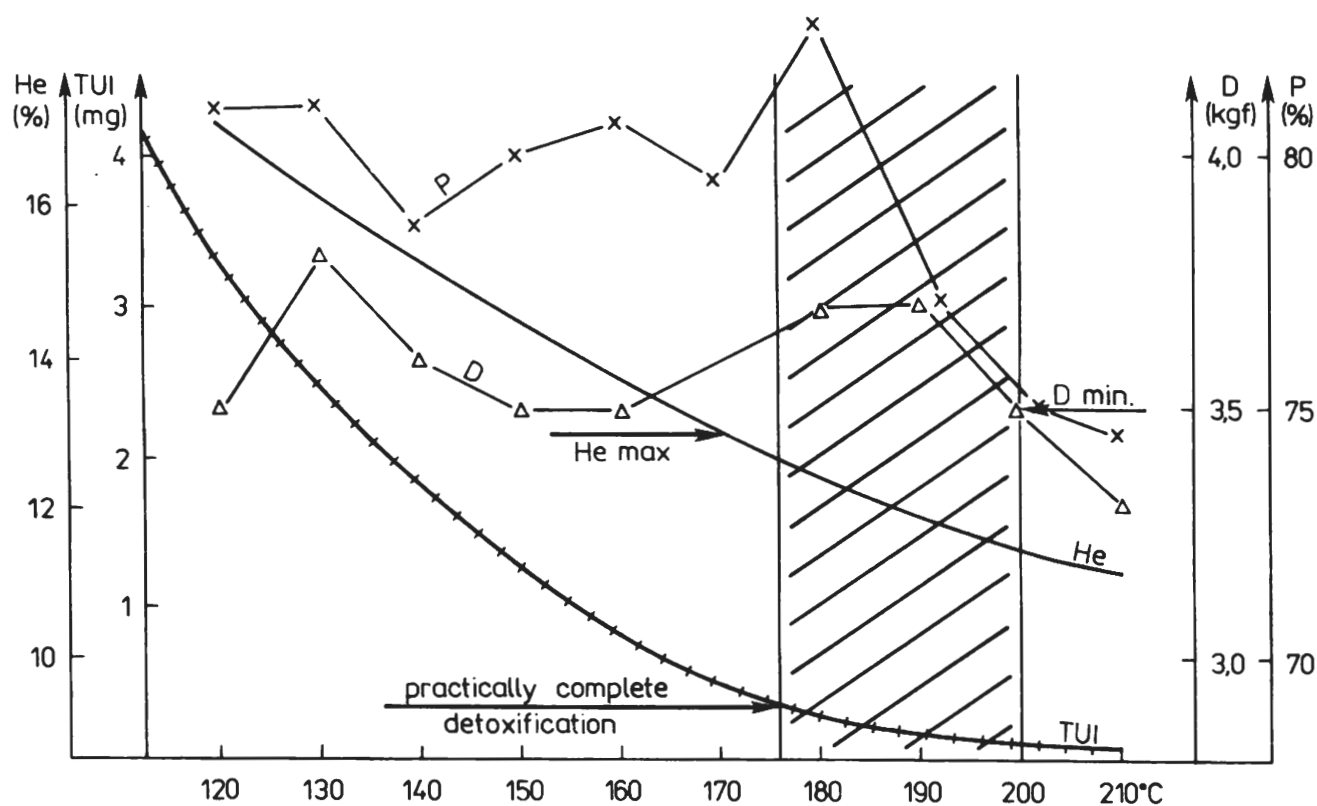


Fig. 2. Determination of optimal extrusion temperature

Determinations of trypsin inhibitor contents depending on extrusion temperature (Table 4) show that detoxification is practically attained at 175°C when the content of the inhibitor, expressed in terms of TUI (trypsin unit inhibitor) in mg of dry mass, drops to 0.35 from 4.2 in the initial mixture. This represents a 12-fold decrease of the inhibitor content; the decrease is still greater (42-fold, or by 98% of the initial content) at 200-210°C. This is an important information, given the presence of protein antinutrients in pea (anti-trypsin agents, haemagglutinins) which limit the amounts of this product (in untreated state) that can be fed to

Table 4. Contents changes of trypsin unit inhibitor (TUI/mg of d. m.) in different extrusion temperature

Extrusion temp. °C	18*	120	130	140	150	160	170	180	190	200	210
Trypsin unit inhibitor	4.2	2.8	2.6	1.8	1.6	0.8	0.4	0.3	0.2	0.1	0.1

\* before extrusion

single-stomach animals to 15% in the case of winter pea, and 30% in the case of spring varieties [2]. The optimal temperature range is limited by pea detoxification at 175°C on the one hand, and extrudate hardness at 200°C on the other. The mean temperature of extrusion is 190°C (approx).

An important problem was the determination of the effect of extrusion on the contents of glucosinolates, ITC and VTO. The findings (for 10°C intervals in the range 120-210°C, and for the initial mixture) are given in Table 5. The ITC content does not change in elevated temperatures and in our experiments it remained unchanged in the initial mixture and in all the extrudates (0.05%). The VTO contents are also unaffected by extrusion temperature changes, amounting to 0.15% dry mass on average. However, the VTO content following extrusion is over 30% lower than in the initial mixture where it was 0.21% dry mass. During determinations of ITC and VTO contents we observed that an additional dose of myrosinase does not increase the obtained amounts of ITC and VTO. We also observed that intracellular myrosinase is destroyed by the time the extrusion temperature reaches 120°C. The glucosinolates content is affected by extrusion temperature changes more visibly. At 210°C it amounted to 15.4 µM/g dry mass as compared to 29.5 µM/g dry mass in the initial mixture (a drop of 48%).

Table 5. Contents changes of ITC and VTO and glucosinolates in different extrusion temperature

Extrusion temp. °C	18*	120	130	140	150	160	170	180	190	200	210
ITC (%)	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
VTO (%)	0.21	0.16	0.14	0.16	0.16	0.15	0.14	0.015	0.14	0.14	0.16
Glucosinolates (µM/g d. m)	29.5	—	20.2	—	20.9	—	18.4	—	15.9	—	15.4

\* before extrusion

The basic chemical composition of the rapeseed-pea (3:7 proportion) extrudate with 3% NaCl obtained in optimal conditions is given in Table 6. The humidity of the product is 11.1%, total protein content amounts to 21%, fat content is 10.1%, and cellulose content is 6.7%. This extrudate is a high-energy protein product, and its use in animal feeding will depend mainly on the nutritional aspect. Theoretically, rapeseed may be rated higher than rape meal,

Table 6. Chemical composition of rapeseed, pea and extrudate (%)

Chemical composition	Humidity	Ash	Fat	Cellulose	Protein
Rapeseed	6.6	4.0	38.6	6.6	20.6
Pea	12.5	3.1	1.3	5.2	24.0
Extrudate (30% rapesead 70% pea)	11.1	6.3	10.1	6.7	21.0

mainly in view of its fat fractions advantageous to poultry and containing unsaturated fatty acids. Also in the case of pigs, a 1 : 2 mixture of rapeseed with a low glucosinolates content (up to 0.3% dry mass) with pea guarantees good weight gains and supplies all the necessary sulphuric amino acids and proteins.

### EXTRUSION WITH AMMONIA

Extrusion of the rapeseed-pea mixture with ammonia was investigated to check the possible destructive effect of ammonia on glucosinolates and products of their hydrolysis. Extrusion was performed at 180°C, with a 1000 mm long extruder head, and outlet nozzle diameter of 3 mm; the remaining conditions of extrusion are given in Table 7. The ammonia doses amounted to 0.5, 0.9, 2.1, and 2.6% of the initial mixture, and ordinary water was replaced with ammonia water containing 10, 15 or 30% NH<sub>3</sub> (supplied to the mixture at the rate of 11,4 kg/h).

Table 7. Extrusion of mixture rapeseed-pea with ammonia

Extruder parameters	Ammonia dose in the mixture (% of NH <sub>3</sub> )			
	0.5	0.9	2.1	2.6
Worm structure	200/50, 100/50, 100/50, 100/35, 100/35, 100/25, 50/25, 50/15 × 2, 100/15 C			
Rotation speed (r. p. m.)	200	200	200	200
Feeding rate (kg/h)	89	77	68	51
Ammonia water addition (kg/h)	11.4	11.4	11.4	11.4
NH <sub>3</sub> concentration in ammonia water (%)	10	15	30	30
Motor amperage (A)	9	10	9	8
Pressure in the head (bar)	22	24	26	24

The humidity of the initial mixture and the obtained extrudates, and the content of ammonia nitrogen in the extrudates are given in Table 8. Remains of ammoniacal nitrogen are not inconsequential for single-stomach animals, and cannot exceed a certain level. According to recommendations of INTR-LTAA in

Table 8. Humidity of mixture and extrudate with ammonia and ammonia nitrogen residue in the extrudate

Ammonia dose (%)	Mixture humidity (%)	Extrudate humidity (%)	Ammonia nitrogen residue (%)
0.5	9.4	11.7	0.3
0.9	9.4	10.5	0.3
2.1	9.4	11.1	0.3
2.6	9.4	11.1	0.3

Nantes, this maximal content of ammonia nitrogen is 0.15% for chicken, 0.3% for laying hens, and 0.1% for fattening pigs. Feed containing more  $N_{NH_3}$  is unsavoury and toxic. In all the obtained extrudates, the ammonia nitrogen content was 0.3%, which suggests that, theoretically, the extrudates may constitute maximally 50% of chicken feed, 100% of laying hens feed, and 33% of fattening pigs feed.

The ITC, VTO and glucosinolates contents depending on the ammonia dose are presented in Table 9. The ITC content was reduced by 20% when the  $NH_3$  dose was 2.1% and more; at lower ammonia doses this content is identical to that in the unextruded mixture (0.05%). The VTO content shows a weak tendency to drop with the increase of ammonia content during extrusion: it fell from 0.19% to 0.17% when ammonia content increased from 0.5% to 2.6%. Nevertheless, the VTO content in the extrudate is 26% lower than in the initial mixture where it stands at 0.23%. This reduction, however, is due to extrusion conditions rather than to the destructive effect of ammonia.

Table 9. Contents changes of ITC and VTO and glucosinolates in different ammonia level

Ammonia dose (%)	ITC (%)	VTO (%)	Glucosinolates ( $\mu M/g$ d. m.)
0.5	0.05	0.19	17.6
0.9	0.05	0.18	16.2
2.1	0.04	0.17	15.5
2.6	0.04	0.17	14.5
before extrusion	0.05	0.23	29.5

The dynamics of glucosinolates content is similar: it drops from 17.6  $\mu M/g$  dry mass for 0.5% of ammonia to 14.5  $\mu M/g$  dry mass for 2.6% of ammonia. Compared to the initial mixture, this reduction amounts to 51%. In all, contrary to reports in the literature [1, 3], it was found that ammonia does not cause significant reductions in the contents of glucosinolates, ITC and VTO.

## CONCLUSIONS

1. Extrusion of a 3:7 mixture of rapeseed and pea gives a high-energy protein concentrate containing 21 % protein, 10.1 % fat, and 6.7 % cellulose.
2. Sodium chloride added to the extruded mixture improves the quality of the product. The optimal NaCl dose is 3 %.
3. Extrusion eliminates completely the anti-nutrient agents in pea, reduces VTO content by 30 %, and glucosinolates content by 48 %.
4. Ammonia did not destroy ITC, VTO and glucosinolates in the performed experiments. The decreases in their contents (by 20, 26, and 51 %, respectively) are due to extrusion conditions rather than to ammonia.

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## EKSTRUADOWANIE NASION RZEPAKU NA CELE PASZOWE. CZ. II. DOBÓR OPTYMALNYCH PARAMETRÓW PROCESU I JEGO WPŁYW NA ZMIANY ZAWARTOŚCI ZWIĄZKÓW SZKODLIWYCH. EKSTRUADOWANIE Z AMONIAKIEM

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### Streszczenie

Technika ekstrudowania rzepaku z grochem daje możliwości otrzymania koncentratu białko-energetycznego dla zwierząt jednożołądkowych, jak i dla przeżuwaczy. Optymalny udział rzepaku w mieszaninie wytłaczanej wynosi 30 %. Ekstrudat zawiera 21 % białka, 10,1 % tłuszczu i 6,7 % włókna (tab. 6). W celu poprawienia właściwości fizycznych ekstrudatu, tj. twardości i plastyczności, zaleca się stosowanie dodatku 3 % chlorku sodowego (rys. 1). Proces wytłaczania eliminuje całkowicie czynniki antyżywniowe grochu (rys. 2, tab. 4) (inhibitor trypsyny) oraz obniża zawartość glukozynolatów o 48 %, VTO o 30 %, podczas gdy zawartość ITC pozostaje niezmienna



(tab. 5). Wpływ amoniaku, jako czynnika niszczącego glukozynolaty i produkty ich hydrolizy został potwierdzony częściowo. W procesie wyłaczania z amoniakiem nastąpiło obniżenie zawartości ITC, VTO i glukozynolatów odpowiednio o 20, 26 i 51 % (tab. 7) i wydaje się, że jest wynikiem głównie warunków wyłaczania. Optymalne warunki wyłaczania przedstawiają się następująco:

układ ślimaków klasyczny — 200/50, 100/50, 100/50, 100/35, 100/35, 100/25, 50/25, 50/15, 50/15, 100/15, C/50/15;

średnica dyszy wyłaczarki — 3 mm;

temperatura wyłaczania — 190°C;

szybkość obrotowa ślimaków — 200 obr./min;

wydajność zasilania mieszanką — 102 kg/h;

wydajność dodawanej wody — 11,4 kg/h;

ilość dodawanej soli do mieszanki wynosi 3%.