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EFFECT OF DRYING OF RAPESEEDS ON THEIR MECHANICAL PROPERTIES AND TECHNOLOGICAL USABILITY*

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A b s t r a c t. Difficulties in extraction and oil retention by oil-extracted seed meal have been recently observed in several oil plants. An assumption was made that the reason of these problems might be greater susceptibility of seeds to crushing due to differences in the seed microstructure. Changes in the seed microstructure might have resulted from drying in extremal conditions before they were supplied to oil plants. Mechanic properties and microstructure of rapeseeds were determined in the samples with strong diversified extractivity by means of agglomeration test. The results of this test were in agreement with the behaviour of seeds during industrial processing. A score was established for determining bad, medium, good and very good technological quality. Microscope studies of the seed structure (LM, TEM, SEM) revealed significant changes in the cotyledon cells of seeds with poor extractivity. In a model experiment with drying rapeseeds of various moisture at selected temperatures their mechanic properties were determined using agglomeration and modified oil-point tests. A highly significant effect of drying temperature was found on the microstructure changes, mechanic properties and technological usability of rapeseeds.

K e y w o r d s: rapeseeds, mechanical properties, technological usability

INTRODUCTION

Mechanical properties of rapeseed have recently been in the focus of attention of many researchers. Available studies concern often seeds resistance to mechanical damages during combine harvesting and post-harvesting treatment. Results of these investigations show that the main reason of damages is combine harvesting and subsequent operations, namely drying, cleaning and storage of rapeseeds [11,12,14,15]. More and more often it is also pointed to the process of seed drying after harvesting and to the influence of drying temperature on the mechanical resistance of rapeseeds. It has been proved that drying causes considerable lowering of seeds mechanical resistance and that transport after drying can produce up to 50 % of all damages, counted from the moment of harvest to storing in silo. It has substantial significance in the context of the studies by Franzke *et al.* [5] who proved that storage of seeds with considerable number of damages lowers the efficiency of extraction process and evidently deteriorates oil quality measured with acid and peroxide numbers.

Mechanical properties of rapeseed, particularly properties defining their resistance to crushing, play also a fundamental role in processing. The action of compressing and shearing forces are crucial in such mechanical operations as crushing, flaking or hulling [1,2,8]. It is a problem to get moderatly repeatable results of their action, e.g., to obtain constant crushing degree or specified size of flakes. Only low variability of physical properties of material permits selection of stable optimal working parameters of machines [1]. The control of oil extraction process is also considerably impeded by a great variability of mechanical resistance of rapeseed. Excessive seed crushing of new rape varieties widely impedes, in technologists' opinion, the technological process. It is

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often denounced of big losses as a result of poorer oil extraction of press-cake from some portions of seeds, which is caused by a very bad percolation of solvent in extraction layer [4]. Conclusions from published studies referring to changes in rapeseed microstructure observed during various technological operations suggest that they are responsible for the changes of mechanical properties, and above all mechanical seed resistance [2,3,6,8,16]. The results of our preliminary microscope studies confronted with results by Stępniewski *et al.* [11] and Stępniewski and Dobrzański [10] and with information provided by industrial technologists make the ground for an assumption that the reason of poorer extraction resulting from the worse mechanical resistance of seeds could be unproper conditions of drying process after harvesting. Verification of this assumption is the aim of present work.

MATERIALS AND METHODS

Rapeseed of Bolko variety harvested in 1991 and various technological quality rapeseed collected during processing in oil factory in 1992 were investigated. Samples from oil factory were taken after noticing disturbances within the technological process. Process parameters were defined according to plant industry standards. Water and fat content in seeds samples were determined acccording to international standards. Material for model experiments, in which temperature influence on technological usability of seeds was analysed, was obtained by heating seeds with following initial water contents: 10±0.1 %, 14.3±0.2 %, 17.0±0.3 %, 20.1±0.3 % in heating chamber KC-1 at 140 °C, 160 °C, 180 °C, 200 °C for 1 hour. Initial water contents in seeds were obtained by moisturing and conditioning of seeds for 24 h. Analysis of technological usability of seeds was made using quick agglomeration test based on determining the amount of fines after triplicate (without cleaning) sieving of 25 g grinded rapeseeds. The seeds were grinded for 5 s in a rotary grinder WZ-1 and next sieved for 1 min on a sieve with 0.63 mm mesh in SZ-1 screen. The test has provided the information about:

- grinding ability of seeds measured by amount of fraction of fine particles <0.63 mm [%];
- rapeseed meal behaviour during sieving caused by adhesion and cohesion forces.

The difference between the amounts of non-agglomerated fine particles <0.63 mm after first and third sieving referred to as Δ_{I-III} was accepted as a measure of technological quality of rapeseeds. Test results of a number samples with known quality allow to accept the following agglomeration test score:

Δ_{I-III}	Technological quality of seeds
1.0 - 5.0 %	very good
5.1 - 10.0 %	good or medium
10.1 - 20.0 %	bad
over 20.0 %	very bad

Oil-point was determined with a modified method based on Sukumaran and Singh's [12] method. The measurements during compression were made on a uniform 14 mm thick layer of bulk rapeseed in a special measuring unit of INSTRON 1011 at cross-head speed 10 mm/min.

Specimens for electron microscope examinations were prepared according to Stanley *et al.* [9] and analysis was carried out using scanning electron microscope JEOL 5200.

Specimens for light microscopy were prepared according to Yiu *et al.* [16] and were analysed using OLIMPUS BH 2 microscope.

RESULTS AND DISCUSSION

Technological parameters of oil recovery process in industrial conditions were shown in Tables 1-3. Rapeseed with water content from 5.8 % to 7.9 % were conditioned and flaked. Obtained flakes were cooked at temperatures 50-90 °C considered as optimal for the pressing process. Water content in cooked flakes was from 5.2 % to 7.2 % (Table 1). Press cakes were obtained at 14.5-16.0 MPa (Table 2). It is worth noticing that their fat content was diversified and ranged from 17.7 % to 23.3 %. At similar process parameters it can be an effect of different mechanical properties of processed seeds. For samples with the highest fat content, i.e., No. 4, No. 5, No. 6 and No. 9, it can also, even at this stage of process, signalize likely troubles during oil extraction. The extraction of press cake was carried out in Bollmann extractor at solvent temperature 42-50 °C and at elevator speed 26-32 basket/h (Table 3). For rapeseed samples analysed on various days various hexane percolation was observed during extraction. It ranged from very good (samples No. 1, No. 2, No. 7 and No. 8) to very bad, practically without percolation (samples No. 4 and No. 5). Rapeseed meals with water and fat content shown in Table 2 were obtained after final stages (solvent evaporation and toasting). These results demonstrate that

Table 1. Technological parameters of rapeseed pretreatment

Parameter	Number of sample								
	1	2	3	4	5	6	7	8	9
Seed moisture (%)	5.9	6.5	7.9	5.8	7.1	6.0	7.3	6.4	6.0
Flakes moisture (%)	7.4	7.9	8.4	5.7	8.0	6.8	-	6.8	6.6
Moisture after cooking (%)	6.0	6.2	7.2	5.2	6.4	5.4	5.8	6.1	5.8
Cooker temp. (°C) Shelf No.: 2	-	-	-	50	70	60	65	70	60
7	90	90	-	90	85	90	90	80	90
Pressure (MPa)	15.0	16.0	-	15.0	15.0	15.0	14.5	15.0	15.0

Table 2. Moisture and fat content in rapeseed press cake and meal

Parameter	Number of sample								
	1	2	3	4	5	6	7	8	9
	Rapeseed press cake								
Moisture (%)	7.8	7.8	7.5	6.7	7.3	6.6	8.1	8.6	7.2
Fat (% d.m.)	19.3	18.3	20.2	23.3	21.3	23.2	17.7	17.7	22.1
	Rapeseed meal								
Moisture (%)	12.9	14.7	12.3	9.2	10.0	11.1	9.4	11.0	10.9
Fat (% d.m.)	4.4	3.2	5.2	18.8	18.6	12.2	1.5	3.4	10.4

Table 3. Parameters of extraction process

Parameter	Number of sample								
	1	2	3	4	5	6	7	8	9
Hexane temp. (%)	50	45	-	50	46	46	46	42	46
Extraction rate (basket/h)	30	30	-	26	30	26	32	30	30
Percolation	very good	very good	bad	very bad	very bad	bad	very bad	very bad	bad

rapeseeds behaviour during pressing and extraction (especially solvent percolation) was connected with the quality of rapeseed meal. Samples No. 6 and No. 9 were defatted only to 12.2 % and 10.4 % and samples No. 4 and No. 5 merely to 18.8 % and 18.6 % so, respectively, by 4.5 % and 2.7 %.

In order to predict the seeds behaviour during process, a quick agglomeration test was applied in our investigations [4]. The results of agglomeration test for these samples are presented in Table 4. Results of the test proved a high compliance with industrial estimation, which already at the stage of seed purchase can decide about the way of their processing - seeds defatting by pressing and extraction or high pressing and utilization of high fat press cake for fodder.

It has been confirmed that during subsequent sieving of grinded seeds sieve eyes diminished due to cohesion and adhesion forces action, which was the reason of a decreasing amount of sieved material. Such change of physical properties of material could be caused, in our opinion, by strong changes in the seed structure. To confirm this assumption structural analysis of seeds with good and bad extractivity was made using scanning electron microscope. A typical microscope picture of a rapeseed [2,3,9,16] was obtained for sample No. 1 (Fig. 1). Cell walls separating individual cells are clearly visible. Fine-grained protein elements, irregardless of being compact, are separated. Figure 2 illustrates the microstructure of bad quality seeds (sample No. 6) the reason of which could be the formation of expanded structures surrounding the content of cotyledon cells. They can be supposed to be a kind of specific complex of fat and non-fat seed compounds. It is worth pointing out that those structures exist mainly near the cell walls where fat gathers after damaging phospholipide membranes of fat globules. Identical results of earlier microscope studies [2,3] concerning the influence of hydrothermal process on rapeseed microstructure permit to state that seeds with bad extractivity were exposed to a drastic thermal treatment. Very high seed moisture had to be an additional factor causing such strong negative changes in cotyledon cells. This suggests the use of drum dryers for drying of seeds appropriate mainly for green forage. In this kind of dryers, usually coal-fired, it is extremely difficult to keep the temperature of drying medium below 400 °C. In such conditions denaturation changes of protein and weakness of cell walls resistance determine deterioration of seed mechanical properties. The failure of membranes of fat globules leads to their leaking during the action of forces damaging seed structure (grinding, flaking, pressing). Leaking oil can facilitate the formation of agglomerates with powdery protein

Table 4. Results of agglomeration test

Sample No.	Share of fraction <0.63 mm after sieving No. I-III (%)			Δ (I−III) (%)	Seed quality
	I	II	III		
1	62.7	59.0	60.4	1.3	very good
2	59.8	57.6	55.9	3.9	very good
3	69.8	68.0	63.5	6.3	good or medium
4	62.6	58.8	51.7	10.9	bad
5	63.1	60.8	47.8	15.3	bad
6	64.2	47.3	41.4	22.8	very bad
7	55.3	52.9	51.1	4.6	very bad
8	50.8	45.3	44.2	6.6	good or medium
9	59.5	53.0	49.3	10.2	bad

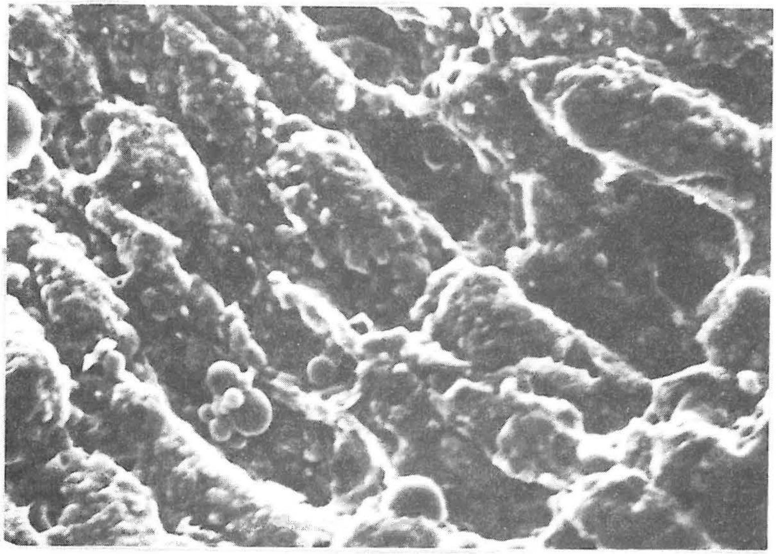

Fig. 1. Microstructure of rapeseed (SEM) of good technological quality - sample No. 1.

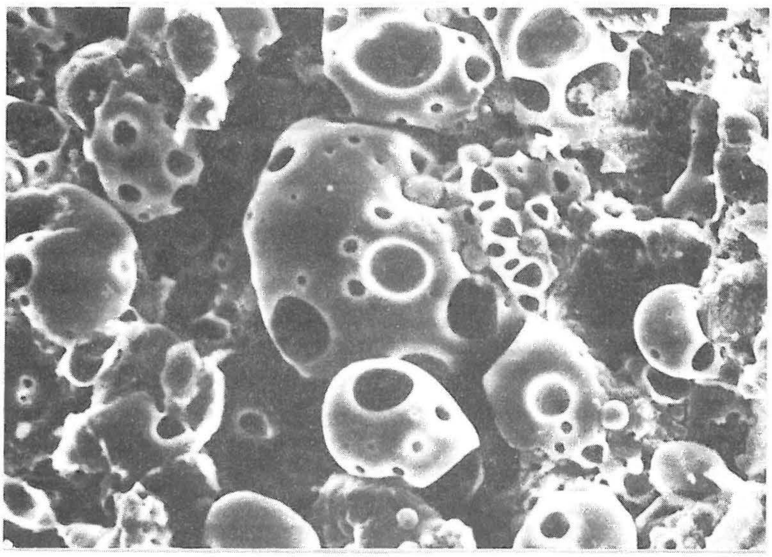

Fig. 2. Microstructure of rapeseed (SEM) of very bad technological quality - sample No. 6.

particles, impeding solvent percolation through the layer of press cake during extraction and sieving throughout the analytical test.

To define the influence of drying temperature and moisture of seeds on their mechanical properties a model experiment was made. The agglomeration test results shown in Table 5 prove significant changes in mechanical properties of seeds and thereby changes in their technological quality with increase of drying temperature and initial seed moisture. Accordingly to accepted quality

Table 5. Results of agglomeration test for rapeseed heated at four different temperatures

Moisture level	Share of fraction <0.63 mm after sieving No. I-III (%)			Δ (I–III) (%)
	I	II	III	
		140 °C		
I	79.2	79.2	77.6	1.6
II	82.4	80.8	78.0	4.4
III	84.8	77.6	74.8	10.0
IV	84.0	79.2	72.0	12.0
		160 °C		
I	78.7	75.7	74.7	4.0
II	80.9	75.7	73.3	7.6
III	74.2	42.2	22.5	51.7
IV	71.6	40.9	32.8	38.8
		180 °C		
I	80.6	66.9	55.5	25.1
II	55.5	16.5	9.9	45.5
III	57.6	18.9	7.5	50.1
IV	52.9	12.9	8.5	44.4
		200 °C		
I	21.0	4.9	3.2	17.8
II	17.6	3.6	2.4	15.2
III	15.9	4.6	3.7	12.2
IV	13.7	2.7	2.2	11.5

classification, seeds with water content 10 % and 14 % dried at 140 °C and 160 °C still had very good and good technological quality. Clear changes in quality were, however, observed at temperature 160 °C in seeds with 17 % and 20 % moisture. The difference between first and third sieving was 51.7 % and 38.8 %, respectively. Similar values were obtained at temperature 180 °C. Heating at 200 °C had a more dramatic effect on mechanical properties of seeds. In spite of lower differences in next sievings (17.8-11.5 % for increased seed moisture), already on the first sieve a distinctly worse sieving ability was observed. For the seeds with 10 % moisture fine share was 21.0 % and for the seeds with 20 % moisture as little as 13.7 %. During third sieving only 3.3 % and 2.2 %, respectively, of material passed through the sieve eyes. Such significant differences should be expected already after observation of grinded seeds. Changes in their colour distinctly suggested non-enzymatic browning as a result of Maillard's reaction. The changes in seed microstructure caused by applied temperatures are clearly visible on the photographs taken with light microscope (Figs 3 and 4). With an increasing temperature of heating more intense yellow colour is observed which proves the failure of fat globule membranes and spilling of fat onto all the cell content. It is worth stressing the disappearance of permeability in vascular boundle which, according to Schneider and Ruette [8], plays an important role in solvent transport.

Electron scanning microscope analysis of seeds heated at temperature 180 °C proved significant structural changes increasing with the water content in the material before drying (Fig. 5). The microstructure of seeds dried at this temperature and of initial moisture 17 % and 20 % was similar to those applied in industrial samples with the worst extractivity (Fig. 2).

The oil-point is the point which occurs immediately prior to the outflow of oil from

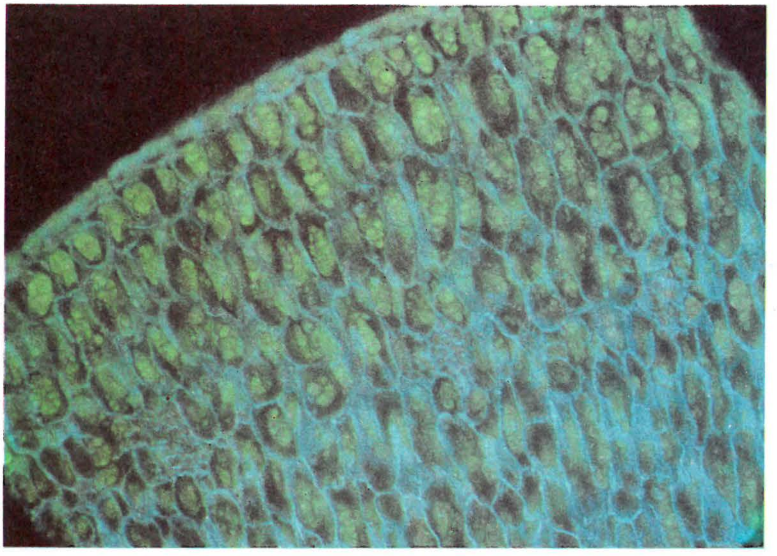

Fig. 3. Structure of rapeseed (LM) - untreated seed.

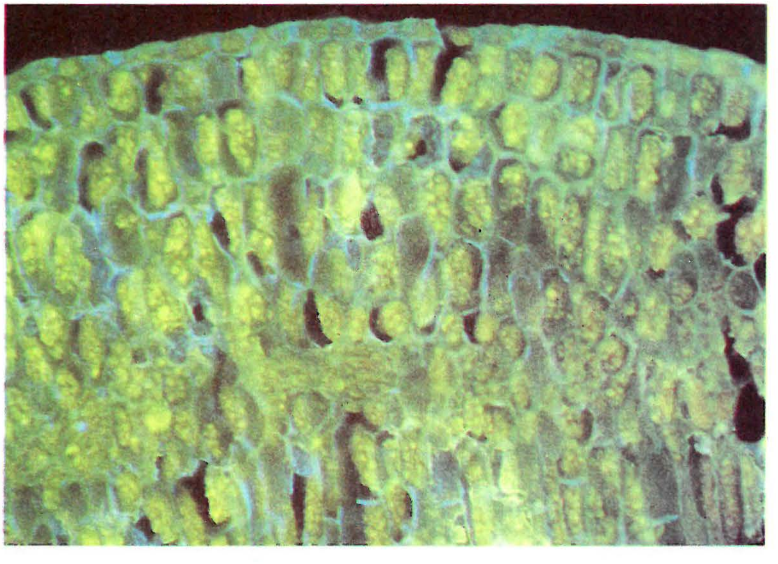

Fig. 4. Structure of rapeseed (LM) heated at 200 ^{o}C.

bulk seeds [12]. The values of pressure, strain and work determined at this point have a considerable practical importance for mechanical expression of oil. The effective pressure for oil equals the applied pressure less than oil-point pressure p_o, thus it could be used as a measure for evaluating the relative effectiveness of alternative pre-treatments of rapeseed [12]. In present paper the results of oil-point test are used for estimation of technological usability of investigated rapeseed. The results of oil-point test are shown on Figs 6 and 7 and in Tables 6 and 7.

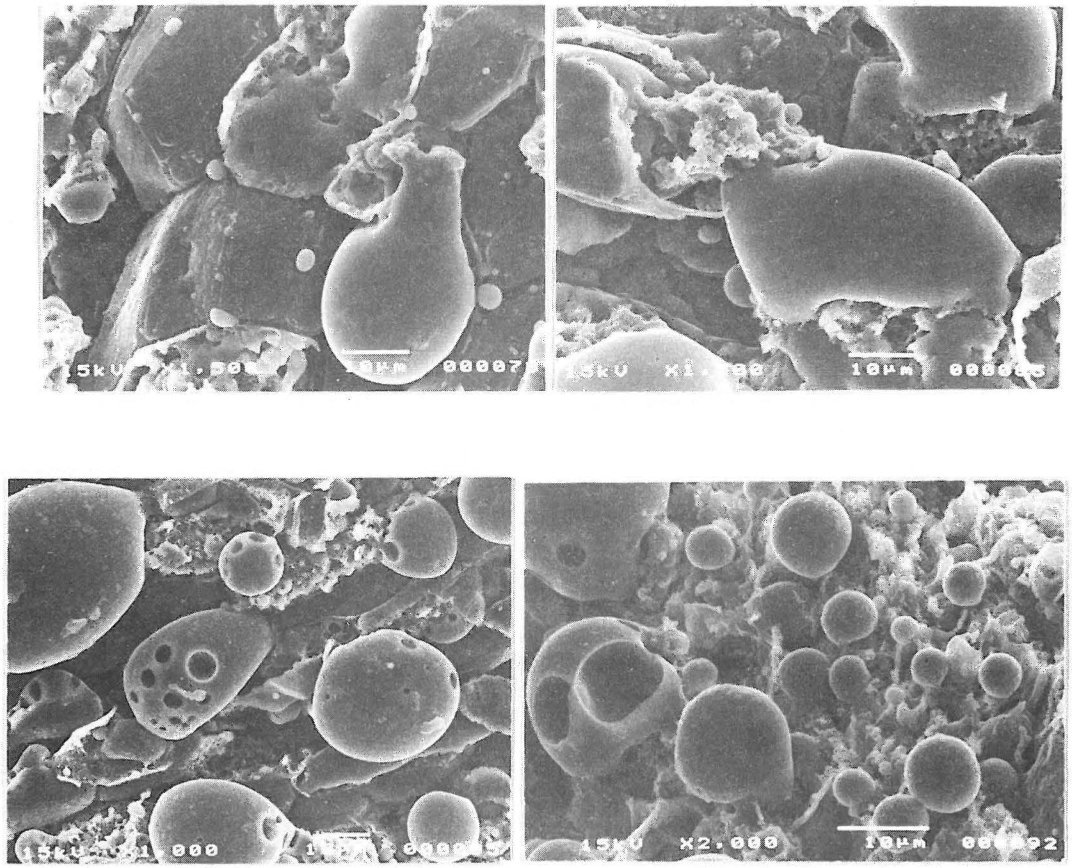

Fig. 5. Microstructure of rapeseed for different water content heated at 180 ^{o}C (SEM): a-10 %, b-14 %, c-18 %, d- 22 %.

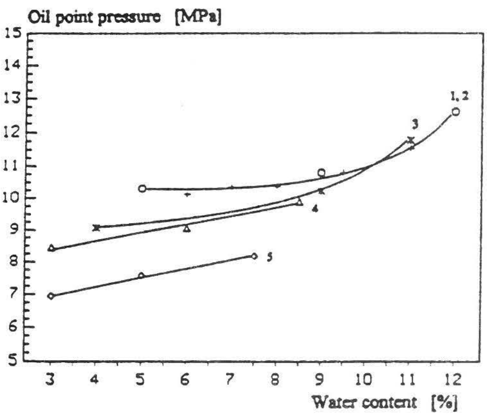

Fig. 6. Relationship between oil-point pressure and water content for rapeseed dried at various temperatures. Seeds: 1-raw, 2-5-dried at 80, 120, 160 and 200^{o}C, respectively.

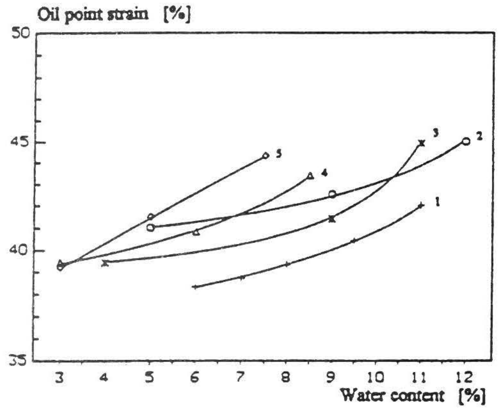

Fig. 7. Relationship between oil-point strain and water content for rapeseed dried at various temperatures. Explanations as in Fig. 6.

Table 6. Drying parameters of rapeseed in industrial dryers

No.	Dryer type	Drying temp. (°C)	Seed moisture (%) before	Seed moisture (%) after
1	M 820	115	11.0	5.0
2	M 820	120	11.0	6.0
3	ŻPŻ 8	130	14.0	6.0
4	M 807	93	13.0	5.5
6	LAW SBC 6	83	11.0	7.0

Table 7. Some mechanical properties of industrially dried rapeseed. Analyses were made with samples of 4.8-5.2 % of moisture

No.	Oil-point Pressure (MPa)	Oil-point Deformation (%)	Oil-point Energy (J)	Agglomeration test (%) $\Delta_{(I-III)}$
1	8.75	36.6	1.76	2.12
2	7.86	37.1	1.57	2.08
Contr.	8.94	38.6	1.81	1.30
3	8.39	36.9	1.65	1.92
4	8.37	37.1	1.68	1.47
Contr.	8.55	36.9	1.72	0.70
6	8.87	36.7	1.75	2.23
Contr.	9.53	37.0	1.99	2.08

Curve No. 1 on Fig. 7 presents the relationship between oil-point pressure and seed water content for raw seeds described as follows:

$$p_o = 9.566 \cdot e^{(0.00142 \cdot W^2)} \qquad (1)$$

where p_o stands for oil-point pressure (MPa) and W for water content (%). High values of correlation and determination coefficients, respectively 0.961 and 0.923, confirm good fitness of function model to experimental data. Presented equation is comparable to equations published by Sukumaran and Singh [12] describing relationship between oil-point pressure or oil-point strain and seed water content [12,13]. Relations expressed as $p_o=f(W)$ for rapeseed dried at various temperatures are shown on Fig. 7. p_o values determined for seeds dried at 80 °C lay on the curve for raw seeds. p_o values for seeds dried at higher temperatures still decrease, irrespective of seed moisture (curves No. 3, No. 4 and No. 5). Such direction of changes results from the microstructure weakness increasing with the growth of drying temperature and facilitating fat movement.

The relations between oil-point strain values and seed moisture for identically dried rapeseeds samples are presented on Fig. 7. The respective strain values for bulk dried seeds regularly increase. Also in this case the course of curve No. 2 (for seeds dried at 80 °C) disturbs a regular course of other curves. It suggests a different character of possible microstructure changes or additional effect of unidentified factors.

The results of oil-point test for randomly collected industrial samples of rapeseed dried in various driers and at various temperatures are another confirmation of usefulness of oil-point determination (Tables 6 and 7). The absence of samples of seeds dried in drum dryer in the analysed set is due to a dry harvest period and, consequently, low water content in seeds (Table 6).

Oil-point pressure values for dried seeds are in all cases lower, and differences between these values for raw and dried seeds are lower for milder conditions of drying. Oil-point energy values for industrial seed samples confirm Sukumaran and Singh's [12] suggestion that the energy expended in reaching the oil-point can be a good index for characterization of bulk rapeseed mechanical properties. The differences in energy values for mildly dried rapeseeds are more distinct than the differences in oil-point pressure values for respective samples. The results of oil-point test showing a good accordance with agglomeration test results (Table 7) confirm proper mechanical properties and very good technological quality of the estimated rapeseeds.

CONCLUSIONS

The results lead to the following conclusions:

1. Bad processing quality of rapeseed resulted from their changes in mechanical properties as a consequence in seeds microstructural changes due to unproper post-harvest treatment.

2. The changes in microstructure of rapeseed are highly affected by heat treatment at post-harvested drying. Initial seed moisture together with high temperature has more pronounced effect than temperature alone.

3. Agglomeration test is a simple and rapid method for predicting of rapeseed processing quality being in a close relation with oil-point method. It enables choosing the right rapeseed processing.

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