

## OIL POINT DETERMINATION OF SELECTED BULK OILSEEDS UNDER COMPRESSION LOADING

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**Abstract.** The oil point in oilseeds refers to the minimum pressure, which is required to cause oil to emerge from the oil bearing material. Mechanical parameters relevant to expression of oil from three emerging oilseed crops were determined at their oil points and peak compression under the applied force. Oil contents of the batch of oilseeds used were determined using the soxhlet method. The efficiency of the compressive bulk expression scheme was determined as a function of oil yield determined during mechanical expression and the oil contents of the oilseeds. The oil point, oil yield and pressing performance varied significantly between the three crops. The oil points and volume deformation energy at oil points of camelina, pumpkin and sesame seeds were 8.33, 7.43 and 3.36 MPa and 1.44, 0.96 and 0.56 MJ·m<sup>-3</sup>, respectively. Oil expression efficiency for these crops at 35.4 MPa, were 56.0, 53.6 and 28.3 %, respectively.

**Keywords:** oilseeds, recovery, pressure, performance, oil point.

### Introduction

The oil point in oilseeds is normally referenced with respect to compressive expression of oil from oilseeds, being the threshold pressure at which oil emerges from the oil bearing material [1-2]. This in essence is the minimum compressive stress that must be induced in the oil bearing material to occasion both rupture and noticeable show and flow of oil. Available theoretical and empirical treatments of the expression of oil from oilseeds have attempted to relate the oil point to porosity and densities of bulk seeds [2-4]. Effective pressure for oil expression has been suggested to be the net applied pressure beyond the threshold of the oil point with compressibility being a limiting factor [2]. Different studies aimed at improving compressibility through size reduction, moisture and thermal conditioning are reported [1;5;6]. Deformation characteristics, requisite compressive stress and energy requirement for the expression of oil vary with oilseeds and their varieties [7]. *Camelina sativa* L. (Crantz), *Cucurbita pepo* L. and *Sesamum indicum* L. are emerging oilseeds of great economic importance in food, industry and biofuel applications [8-9]. Very little is reported on cold mechanical expression of oil from these oilseeds under bulk compression schemes. In this study, the behaviours of bulk seeds of camelina, pumpkin and sesame under uni-axial compressive load were investigated with a view to determining the threshold values of requisite mechanical parameters, which are essential to effective cold expression of oil from the oilseeds.

### Materials and methods

#### Samples

Whole seeds of camelina (*Camelina sativa* (L.) Crantz), pumpkin (*Cucurbita pepo* L.) and sesame (*Sesamum indicum* L.) purchased in the Czech Republic were used for this study.

#### Experimental setup and test procedures

The oil expression apparatus used for this study was a cylindrical steel pressing vessel having an internal bore diameter of 60 mm, provided with a 20 mm thick circular base plate stepped inwards diametrically at 10 mm depth, 8 mm from its circumference. Lateral orifices (ϕ3 mm) spaced 36° apart along the circumference of the pressing vessel, 15 mm from its base. The vessel had a close fitting solid piston, stepped 1mm, 30 mm from its base. The vessel was mounted on the bed of a Tempos (ZDM 50) model universal test rig and loaded compressively through a spherical base, flat topped disc. The cleaned seeds of camelina, sesame and pumpkin at ≈7.04 % moisture content (d.b.) were bulk fed into the pressing vessel to the depths of 80 mm. Compressive force of 100 kN was gradually applied to the constrained seeds at a crosshead speed of 10 mm·min<sup>-1</sup> until the achievable peak deformation under the applied force was attained. The tests were conducted under laboratory conditions of 20 °C. Each test was repeated three times. A completely randomised design was adopted for the conduct of the experiment. All mechanical test data were electronically logged. Moisture contents of the oilseeds were determined using the oven drying technique as described in the ASAE

standards S352.2 for unground grains and seeds. The masses of the samples used were determined using the Kern 440–35N (Kern & Sohn GmbH, Stuttgart, Germany) weighing balance.

#### ***Evaluation of properties of oilseeds and compression parameters***

Strain in the compressed material  $\epsilon_l$  (–) was determined using Eq. 1

$$\epsilon_l = \frac{\delta_c}{\delta_o}, \quad (1)$$

where  $\delta_c$  – represents the highest value of deformation, which was obtained at peak load, mm;  
 $\delta_o$  – the initial height of the pressed bulk seeds, mm.

The initial volume of the compressed material,  $V$  (mm<sup>3</sup>) is given by Eq. 2

$$V = \frac{\pi D^2}{4} \times \delta_o, \quad (2)$$

where  $D$  – the internal diameter of the cylindrical pressing vessel, mm.

Bulk density of an oilseed prior to oil expression was determined as the ratio of the mass of a sample of the oilseed to its known free-fill volume. The bulk density of the compressed oilseed material,  $\rho_{bc}$  (kg·m<sup>-3</sup>) after oil expression was determined as a function of the mass ( $m_c$ ) and volume ( $V_c$ ) of the oilseeds after compression (Eq. 3)

$$\rho_{bc} = \frac{m_c}{V_c}, \quad (3)$$

where  $V_c$  – the final volume of the compressed material in mm<sup>3</sup>, obtained using Eq. 4

$$V_c = \frac{\pi D^2}{4} \times \delta_f, \quad (4)$$

where  $\delta_f$  – the final height of the compressed oilseeds in the constraining vessel, mm.

The oil point is indicated by the show of oil, observed through lateral orifices located 15 mm from the base of the pressing vessel and with the aid of an auxiliary digital indicator mounted by the pressing rig enabling real-time visual observation of both the force and deformation.

The bulk density of the oilseeds at the oil point was determined as  $\rho_{bo}$  (kg·m<sup>-3</sup>) a function of the deformed volume, at the oil point. The deformed volume  $V_o$  (mm<sup>3</sup>) at the oil point was obtained using Eq. 5.

$$V_o = \frac{\pi D^2}{4} \times (\delta_o - \delta_{op}), \quad (5)$$

where  $\delta_{op}$  – is deformation at the oil point, mm.

Strain in the material at the oil point  $\epsilon_o$  (dimensionless) was calculated as (Eq. 6):

$$\epsilon_o = \frac{\delta_{op}}{\delta_o}, \quad (6)$$

Deformation energy  $E$  (J) is the energy required to achieve a given deformation of the compressed product mass at the specified force and conditions (Eq. 7). This is the area beneath the force–deformation curve and is numerically computable as

$$E = \sum_{n=0}^{n=i-1} \left[ \left( \frac{F_{n+1} + F_n}{2} \right) \times (\delta_{n+1} - \delta_n) \right], \quad (7)$$

where  $i$  – the number of subdivisions of the deformation axis;

$F_n$  – the compressive force (N) for a known deformation,  $\delta_n$ , mm;

$E$  – the deformation energy, J.

Volume deformation energy,  $e$  ( $\text{N}\cdot\text{mm}^{-3}$ ) was calculated as the ratio of the deformation energy to the volume of the deformed oilseeds (Eq. 8).

$$e = \frac{E}{V}, \quad (8)$$

At the oil point,  $\delta_n = \delta_{op}$  and  $F_n = F_{op}$ ,  $F_{op}$  being the applied compressive force at the oil point. Deformation energy  $E_o$  (J) at the oil point was obtained using Eq. 11 up to the oil point. The volume deformation energy at the oil point,  $e_o$  ( $\text{N}\cdot\text{mm}^{-3}$ ) was therefore obtained as the ratio of the deformation energy at the oil point to the volume of the deformed oilseeds (Eq. 9).

$$e_o = \frac{E_o}{V}, \quad (9)$$

The Modulus of Elasticity of the compressed bulk material,  $M_n$  (MPa) was numerically computed using Eq. 10.

$$M_n = \left[ \frac{4 \times \delta_o}{\pi \times D^2} \left( \frac{F_{n+1} - F_n}{\delta_{n+1} - \delta_n} \right) \right]_{n=0}^{n=i-1}, \quad (10)$$

Oil yield,  $OY$  (%) during mechanical expression was determined as a function of the ratio of the expressed oil to the total seed mass (Eq. 11)

$$OY = \frac{m_o}{m_{ss}} \times 100, \quad (11)$$

where  $m_o$  – the mass of the expressed oil, g;  
 $m_{ss}$  – the mass of the seeds pressed, g.

#### ***Determination of seed oil content using Soxhlet extraction technique***

The maximum quantities of oil present in each batch of the oilseeds used for this study were determined using the Soxhlet extraction technique. This was done according to the ISO 659: 2009 reference method for oilseeds. The seeds were milled sufficiently to pass through a size 10 sieve. The samples were defatted in a soxhlet unit using petroleum ether. Standard evaporation techniques were used for solvent recovery as stipulated in the standards. Each experiment was repeated three (3) times. Oil content was determined as the ratio of the extracted oil to the mass of the seed sample (Eq. 12)

$$OC = \frac{m_{OC}}{m_s} \times 100, \quad (12)$$

where  $OC$  – the percentage of oil contained in the sample, %;  
 $m_{OC}$  – the mass of the oil extracted, g;  
 $m_s$  – the mass of the sample, g.

#### ***Determination of oil expression efficiency***

Mechanical oil expression efficiency  $\eta_{OE}$  (%) was determined as a ratio of the expressed oil to the total quantity of oil contained in the oilseed (Eq. 13)

$$\eta_{OE} = \frac{OY}{OC} \times 100, \quad (13)$$

#### ***Data analysis***

All test data were subjected to the analysis of variance using the generalised linear model in Minitab®, release 17. Numerical computations and graphical plots were done using MS Excel. The main treatment effects were compared using the Duncan's multiple range test.

## Results and discussion

The mechanical response parameters of the 3 oilseeds at the oil point are presented in Table 1. Highly significant effects ( $p < 0.0001$ ) were observed in all parameters, except volumetric deformation at the oil point. Although significant differences were observed among linear deformations at the oil point ( $p < 0.0001$ ), volume deformations of the three oilseeds at the oil point were statistically at par ( $p = 0.107$ ). When these effects were compared using the Duncan's multiple range test, higher linear deformation (46.3 mm) was observed in pumpkin than in sesame and camelina, in which linear deformations at the oil point were 34.1 and 30.9 mm, respectively (Table 1). Induced strain at the oil point ranged between 0.37-0.39, 0.41-0.43 and 0.57-0.58 in bulk seeds of camelina, sesame and pumpkin, respectively.

Table 1

**Oil point parameters of the three oilseeds (Mean  $\pm$  SD\*)**

Parameter	Oilseed		
	Camelina	Pumpkin	Sesame
Oil point deformation, mm	30.9 $\pm$ 1.1	46.3 $\pm$ 0.3	34.1 $\pm$ 1.1
Oil point pressure, MPa	8.33 $\pm$ 0.34	7.43 $\pm$ 0.59	3.36 $\pm$ 0.05
Volume deformation at oil point, 10 <sup>4</sup> mm <sup>3</sup>	8.75 $\pm$ 0.31	13.10 $\pm$ 0.08	9.63 $\pm$ 0.31
Bulk density at oil point, kg·m <sup>-3</sup>	1135.30 $\pm$ 25.21	1014.90 $\pm$ 9.38	1157.80 $\pm$ 27.80
Strain at oil point	0.387 $\pm$ 0.014	0.579 $\pm$ 0.004	0.426 $\pm$ 0.014
Deformation energy at oil point, J	57.04 $\pm$ 23.52	57.11 $\pm$ 18.45	20.75 $\pm$ 9.16
Volume deformation energy at oil point, MJ·m <sup>-3</sup>	1.44 $\pm$ 0.08	0.96 $\pm$ 0.09	0.56 $\pm$ 0.01
Compression ratio at oil point	0.236 $\pm$ 0.010	0.210 $\pm$ 0.017	0.095 $\pm$ 0.001
Gain in bulk density at oil point, kg·m <sup>-3</sup>	88.0 $\pm$ 25.2	400.1 $\pm$ 9.4	154.1 $\pm$ 27.8
Percentage gain in bulk density, %	8.40 $\pm$ 2.41	65.08 $\pm$ 1.53	15.3 $\pm$ 2.80

\*SD = Standard deviation

Deformation energy at the oil point was significantly higher in camelina than in the other oilseeds (Table 1). Whereas 128 J was required to cause the show of oil in sesame, 216 and 325 J were required for attainment of the oil points in similar bulk volumes of pumpkin and camelina seeds. The volume deformation energy at the oil point was 0.56, 0.96 and 1.44 MJ·m<sup>-3</sup> for sesame, pumpkin and camelina, respectively.

The oil point is marked by significant loss of porosity in oilseeds [2-4] and this is indicated by relative gains in bulk densities of the compressed material. By basing computations on the instantaneous mass and indicated deformation at the first show of oil, it was possible to determine bulk densities of the compressed oilseeds at their oil points. Gain in bulk density at the oil point was significantly higher in the seeds of pumpkin ( $p < 0.0001$ ) than in the other oilseeds. This is explained in part by its apparent loss of a larger void volume during compression, compared to the other oilseeds. Gain in the bulk density by compressed seeds of camelina, sesame and pumpkin at the oil point ranged between 60.2-109.3, 122.5-174.9 and 389.2-405.6 kg·m<sup>-3</sup>, respectively. These represented 5.75-10.4, 12.2-17.4 and 63.3-66.0 % gains in the bulk densities, respectively, at the oil point (Table 1).

Oil point pressures of bulk seeds of camelina, pumpkin and sesame were 8.33, 7.43 and 3.36 MPa, respectively (Table 1). This implies that at a reference pressure of 35.4 MPa, pressure ratios of 0.236, 0.210 and 0.095 were required to occasion the show of oil in the 80 mm deep beds of camelina, pumpkin and sesame seeds.

The mechanical characteristics of the compressed oilseeds at peak load are presented in Table 2. Highly significant effects ( $p < 0.001$ ) were observed in all mechanical response parameters of the three oilseeds. Volumetric deformations in the three crops at peak load were significantly different ( $p < 0.001$ ). When the treatment means were compared, deformation at peak load was higher in pumpkin than in the other seeds, followed by deformation in sesame; the least deformation at peak load occurred in camelina (Table 1). Deformation at peak load ranged between 44-45, 48-49 and 53-54 mm in camelina, sesame and pumpkin seeds. Volumetric deformation was significantly higher in pumpkin than in the other seeds (Table 2).

Table 2

**Mechanical parameters of the bulk oilseeds loaded compressively to 100 kN at 10 mm·min<sup>-1</sup> and 9 % moisture content (Mean ± SD\*)**

Mechanical parameter	Oilseed		
	Camelina	Pumpkin	Sesame
Deformation, mm	46.00±0.67	53.87±0.37	49.12±0.90
Volume deformation, 10 <sup>4</sup> mm <sup>3</sup>	92.73±0.19	15.23±0.10	13.89±0.25
Strain	0.563±0.008	0.673±0.005	0.614±0.011
Deformation energy, J	963.92±16.39	558.68±46.33	574.37±8.46
Volume deformation energy, MJ·m <sup>-3</sup>	4.261±0.072	2.470±0.205	2.539±0.037

\*SD = Standard deviation

Strain in bulk seeds of camelina, sesame and pumpkin at a peak compressive force of 100 kN was 0.563, 0.614 and 0.673, respectively. Mean deformation energy at peak compression was 964, 574 and 559 J, for 80 mm deep beds of camelina, sesame and pumpkin, respectively. These values represented the volume energy demands of 4.26, 2.54 and 2.47 MJ·m<sup>-3</sup> (Table 2). The energy demands for the deformation of sesame and pumpkin at peak load were at par ( $p = 0.529$ ) and significantly less than the energy requirement for the deformation of camelina seeds at the same load (Table 2).

The pressing performance parameters of the bulk compression scheme at peak application of compressive force are presented in Table 3. The maximum amounts of oil present in the seeds of camelina, pumpkin and sesame, determined using the soxhlet method are also presented in Table 3. Oil contents of camelina, pumpkin and sesame seeds ranged between 35.1-37.2, 35.0-38.9 and 43.9-45.0 %, respectively. Similar amounts of oil ( $p = 0.330$ ) appear to be present in the batches of camelina and pumpkin seeds used for this study; the oil content of sesame was significantly higher being 44.6 %. Mean oil contents of camelina and pumpkin seeds were 36.0 and 37.0 %.

The oil yield during mechanical expression using a compressive force of 100 kN applied at a cross-head speed of 10 mm·min<sup>-1</sup> for similar bulk volumes of pumpkin, sesame and camelina seeds ranged between 9.03-12.3, 23.6-24.2 and 19.5-20.8 %, respectively. These were measured as ratios of the quantities of the oil expressed to the total seed mass. Average values of the oil yield were 10.4, 20.2 and 23.9 %, respectively (Table 3).

Table 3

**Mechanical performance of bulk compression schemes of the oilseeds (Mean±SD\*)**

Parameter	Oilseed		
	Camelina	Pumpkin	Sesame
Oil Content OC, %	36.00±1.07	37.00±1.93	44.57±0.56
Oil Yield OY, %	20.16±0.65	10.42±1.66	23.87±0.31
Compressive Oil Expression Efficiency, %	56.03±1.83	28.33±5.83	53.58±0.98
Bulk density of compressed seeds, kg·m <sup>-3</sup>	1270.8±30.9	1171.5±26.2	1311.9±37.1
Modulus of elasticity of compressed seeds, N·mm <sup>-2</sup>	429.0±46.7	644.3±100.3	611.1±60.9
Gain in bulk density after compression, kg·m <sup>-3</sup>	223.6±30.9	556.8±26.2	308.1±37.1
Percentage gain in bulk density after compression, %	21.35±2.95	90.56±4.25	30.70±3.69

\*SD = Standard deviation

Mechanical oil expression efficiency was determined as the ratio of the quantity of oil mechanically expressed to the maximum quantity of the oil present in the batch of oilseeds used for the study. The oil expression efficiency at the applied force of 100 kN was 56.0, 53.6 and 28.3 %, respectively for bulk seeds of camelina, pumpkin and sesame. The least pressing performance was obtained with pumpkin seeds. Pressing performance with camelina and sesame seeds were statistically similar ( $p = 0.477$ ). Gain in the bulk density at peak compressive force was 21.4, 30.7 and 90.6 % in seeds of camelina, sesame and pumpkin, respectively. The higher the gain in the bulk density by the material, the less the quantity of oil that was expressed from it (Table 3). Whereas high values of strain were observed, these correlated strongly and positively with the loss of the void volume and may not necessarily result in productive stress required for substantial deformation and capable of expelling oil

from their storage media. This appears also to be supported by the corresponding values of the moduli of elasticity of the compressed oilseeds. The modulus of elasticity is a measure of a material's strength and indicates its resistance to permanent or plastic deformation. Higher values of the modulus of elasticity, at the applied force, were observed in the seeds of pumpkin and sesame, compared to camelina (Table 3).

### Conclusions

In this study, the mechanical parameters for cold expression of oil from bulk seeds of camelina, pumpkin and sesame were determined at an applied force of 100 kN and compression speed of 10 mm·min<sup>-1</sup>.

1. The oil points of camelina, pumpkin and sesame seeds were found to be 8.33, 7.43 and 3.36 MPa, respectively.
2. Volumetric deformations of bulk constrained camelina, pumpkin and sesame seeds were similar at the oil point, being 8.75, 13.1 and 9.63 x 10<sup>4</sup> mm<sup>3</sup>, respectively.
3. At an applied pressure of 35.4 MPa, the compressive mechanical oil expression efficiencies for camelina, sesame and pumpkin seeds were 56.0, 53.6 and 28.3 %, respectively.
4. At peak compression, gain in bulk density correlated negatively with the oil expression efficiency and with the volume energy demand.
5. High strain pumpkin seeds appeared to correlate more with loss of void volume than actual induction of productive stress capable of causing the release of oil from the oil bearing material.

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