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Role of coat structure in mechanical properties of yellow and black rape seeds

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A hypothesis of this work was that marked differences in mechanical resistance of black and yellow rape seeds can be explained by structural differences in their seed coats. Mechanical resistance parameters of the seeds were measured by static and dynamic loading and oil-point tests. Structural parameters of the seed coats were estimated from SEM and mercury intrusion porosimetry. Higher porosity, smaller thickness and lower bulk densities of yellow rape seed coats accompanied higher susceptibility of yellow rape seeds against mechanical damage and their lower oil-point.

1. Introduction

Rape (*Brassica napus*) seeds are highly sensitive against static and dynamic damages that cause huge losses of raw material (Calisir et al., 2005; Skriegan, 1992). Dynamic strains occur during harvest transport and silo filling, and the static ones arise mainly during storage. Stepniewski et al. (2003) showed that up to 50% of all seed damage occurs at harvest and that this number increases at further steps of postharvest processing. They reported that the total number of broken seeds at the end of this cycle accounts from 1.6 to 7.5%. Vertical load in silo causes further seeds breaking depending on the bed height (Horabik and Molenda, 2000; Jayas et al., 1989). Accompanying release of oil and enzymes from the seed interior and intensive growth of microorganisms (Tanska and Rotkiewicz, 2003; Yiu et al., 1983) lead to increase in temperature and occurrence of additional horizontal stresses which further spoil the stored material (Ramirez et al., 2009; Tys, 2006). Tys et al. (2006) reported that only 44% of rape seeds delivered to oil factories in Poland were acceptable by technological quality standards (Kachel-Jakubowska and Szpryngiel, 2006) in respect to a number of fractures.

Rape seeds sensitivity to mechanical damage is related to their nonhomogeneous and layered structure, and the protective role against breaking is mainly played by the seed coat (Szpryngiel et al., 1995). The seed coat consolidates rather loosely joined germ cotyledons which easily disintegrate into three parts: outer, inner and germ (Hu et al., 2013). Mechanical properties of rape seed coat are governed mainly by its chemical composition and cell walls structure (Tanska et al., 2008). A particularly important role in seeds mechanical stability is played by a fiber, composed of lignin, cellulose, hemicellulose and pectins (Bell, 1993). High fiber content has, however, negative effect on energetic value and digestibility of rape fodder (Buraczewska et al., 1998). To decrease the fiber content in seeds, new yellow rape varieties have been developed, called “OOO” having also higher protein and saccharose level (Rahman et al., 2001; Slominski and Campbell, 1990). Ochocki and Piotrowska (2002) reported that the total fiber content in black rape seeds (variety Lisek) reached 50.1% (dry mass), whereas yellow

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rape seeds contained 34.7 to 37.3% fiber. They suggested that lower fiber content induces higher sensitivity to damage of yellow rape seeds. We hypothesized that large differences in mechanical resistance of yellow and black rape seeds are caused also by differences in structure of their seed coats. The present paper was designed to compare structural parameters of seed coats and juxtapose them to the resistance of the seeds against dynamic and static loading.

2. Materials and methods

Seeds of yellow rape number 041 selected from 100-member recombinant inbred line and of black rape variety Bojan, both supplied by Plant Breeding and Acclimatization Institute in Poznan (PL), were studied. Mechanically separated seed coats dried at 40 °C and degassed in vacuum at 20 °C were used for microscopic and porosimetric studies. Whole seeds of 7.5% moisture were used for mechanical tests.

Cross section and planar seed coats microphotographs were recorded using SEM TESCAN, VEGA 3 LMU microscope with 10 keV accelerating voltage at high vacuum mode, for 10 seed replicates of each rape variety. Using an image analysis tool of the microscope, the coat thickness, t [m] and radii of individual surface pores, r_{sp} [m], were measured. Also, the pore areas, A_{sp} [m^2], and number of pores present on selected area on the coat surface, N_{sp}, was calculated. Pore size distribution of the studied coats was determined using Autopore IV 9500 mercury porosimeter provided by Micrometrics INC, USA. Stepwise mode of mercury intrusion in the pressure range from 0.036 to 413 MPa was applied, following ISO 2005 (Hajnos et al., 2006). The measurements were repeated thrice for samples containing around 0.4 g of the coats. The intrusion pressure p [Pa] was related to the (cylindrical) pore radius r [m] using Washburn (1921) equation:

$$p = \frac{2\gamma_{Hg}\cos\theta}{r},$$

where: γ_{Hg} is surface tension of mercury (485Jm^{-2}), θ is mercury contact angle assumed to be equal to 130° for all samples studied.

The results were plotted in two graphical forms. In the first form, the cumulative pore volume, v [cm^3g^{-1}], was plotted against a logarithmically scaled pore radius abscissa, and in the second form the differential pore size distribution curve (PSD) based on logarithmic differentiation dv/dlog$r$ was calculated. The apparent solid phase (skeletal) density of the samples, ASD [g cm^{-3}], (that is lower than true skeletal density due to the residence of the finest pores in the solid phase which are not filled by mercury at its highest pressure), total pore area, A [m^2g^{-1}], bulk density, BD [g cm^{-3}], volumetric porosities, P [%], and median pore radius (by volume), r_{med} [m], were calculated by porosimetric data analysis program provided by the equipment manufacturer. Average pore radius, r_{av}, was determined assuming that all pores are straight cylinders of the length L, total pore volume $V_{tot} = \pi r_{av}^2 L$ and total pore area $A = 2\pi r_{av} L$. Thus, r_{av} is equal to $2V_{tot}/A$.

Mechanical resistance of the seeds was determined using two static tests: single seed loading and seed bed loading, and one dynamic impact test. Single seeds placed between two parallel steel plates were quasi-statically pressed using INSTRON 6022 strength testing machine and the stress–strain (force-displacement) dependence was registered. The force F [N] at maximum deformation at the moment of the seed breakdown (Stępniewski, 1998), taken as mechanical resistance of individual seed was measured for 50 replicated seeds of each rape variety. The seed beds were compressed using slightly modified method of Sukumaran and Singh (1989). The seeds poured into a small ($1cm^2 \times 1.2$ cm) cylinder placed on a piece of a filter paper were pressed using a metal piston with 10 mm/min speed, also using INSTRON machine, in 20 replicates for both rapes. The pressure O [Pa] inducing the first appearance of oil on a filter paper ("oil-point") was taken as the measure of the seed beds resistance against loading. The dynamic impact tests were performed according to the procedure of Tys et al. (1997) for individual seeds. The seeds were first selected by sieving to have the same dimensions (2.2–2.3 mm) and next by weighing to have the same masses (±0.0001 g). Twenty seeds placed to expose side surfaces of the cotyledon were one by one struck by a rotating arm. The increasing energy of the arm was applied for the next 20 seeds and so on until at least 90% of the seeds were visibly damaged. The procedure was repeated thrice for 100 seeds applying energies slightly differing to the latter one and the energy E [J] needed to reach the damage performance closest to 90% was taken as the energy of a single seed dynamic damage.

3. Results and discussion

Representative scanning electron microphotographs of the studied seed coats are presented in Figs. 1 and 2. Table 1 summarizes average values of seed coats surface parameters derived from SEM images. As calculated by F-test, all above parameters were statistically different at $\alpha = 0.05$. The black rape seed coat is almost two times thicker and more uniform in thickness than the yellow one. Both seed coats have not flat but rough surface on which the pores form characteristic cavities (Fig. 2), composed from cellulose fibers of tissue cell walls. Approximately twice larger radii (and four times larger areas) of these cavities were observed in yellow rape seed coat. On the same area of the surface of black rape seed coat three times more cavities occur than in the case of the yellow rape. Visually, the black rape coat surface is more regular and homogeneous, and the yellow one is more diversified and heterogeneous.

Exemplary mercury intrusion porosimetry curves showing an amount of intruded mercury (pore volume) versus logarithm of the pore radius (cumulative porosimetry curves) as well as pore size distributions functions for the studied seed coats are illustrated in Fig. 3. Parameters of pore system of rape seed coats derived from mercury porosimetry analysis are collected in Table 2. As calculated by F-test, all above parameters but the apparent skeletal density of the coats were statistically different at $\alpha = 0.05$. The total pore volume of the yellow rape seed coats is higher than of the black rape coats in the whole pore radii range that results in a higher porosity and lower bulk density of yellow rape seed coats. Yellow rape seed coats have markedly more pores in two size ranges: large pores from 20 to 125 μm and small pores from 0.03 to 0.06 μm (majority at around 0.04 μm). Only the latter range corresponds to pores detected by SEM at the applied magnifications. Black rape seed coats have higher number of very small pores below 0.01 μm.

Mechanical properties estimated from static and dynamic tests for the studied rape seeds are summarized in Table 3. As calculated by F-test, all above parameters were statistically different at $\alpha = 0.05$. As evidenced by the analysis of the mechanical resistance of the seeds, black rape seeds are significantly more resistant against damage than yellow rape seeds. This is worth mentioning, that other lines of the yellow rape tribe studied by us exhibited also much lower mechanical resistance that black rape seeds. The extreme resistant yellow rape line was 036 for which values of $F = 11.9$N; $O = 8.9$ MPa; $E = 0.71$J and the least resistant was line 022 ($F = 8.6$N; $O = 7.5$ MPa; $E = 0.56$J). For other varieties of the black rape, our studies showed that the extreme resistant was Margo ($F = 16.2$N; $O = 15.7$ MPa; $E = 1.3$J) and the least resistant was Lirajet ($F = 10.6$N; $O = 9.4$ MPa; $E = 0.87$J).

The oil point of the seed bed can simulate the real conditions