

IMPACT OF CRYOMILLING ON
CHEMICAL COMPOSITION OF
FLAXSEED CAKEObranović M.¹, Škevin D.¹, Balbino S.¹, Čurić D.¹, Kraljić K.¹, Drakula S.¹, Herceg M.¹, Kuraica I.¹¹University of Zagreb, Faculty of Food Technology and Biotechnology, Croatia

INTRODUCTION

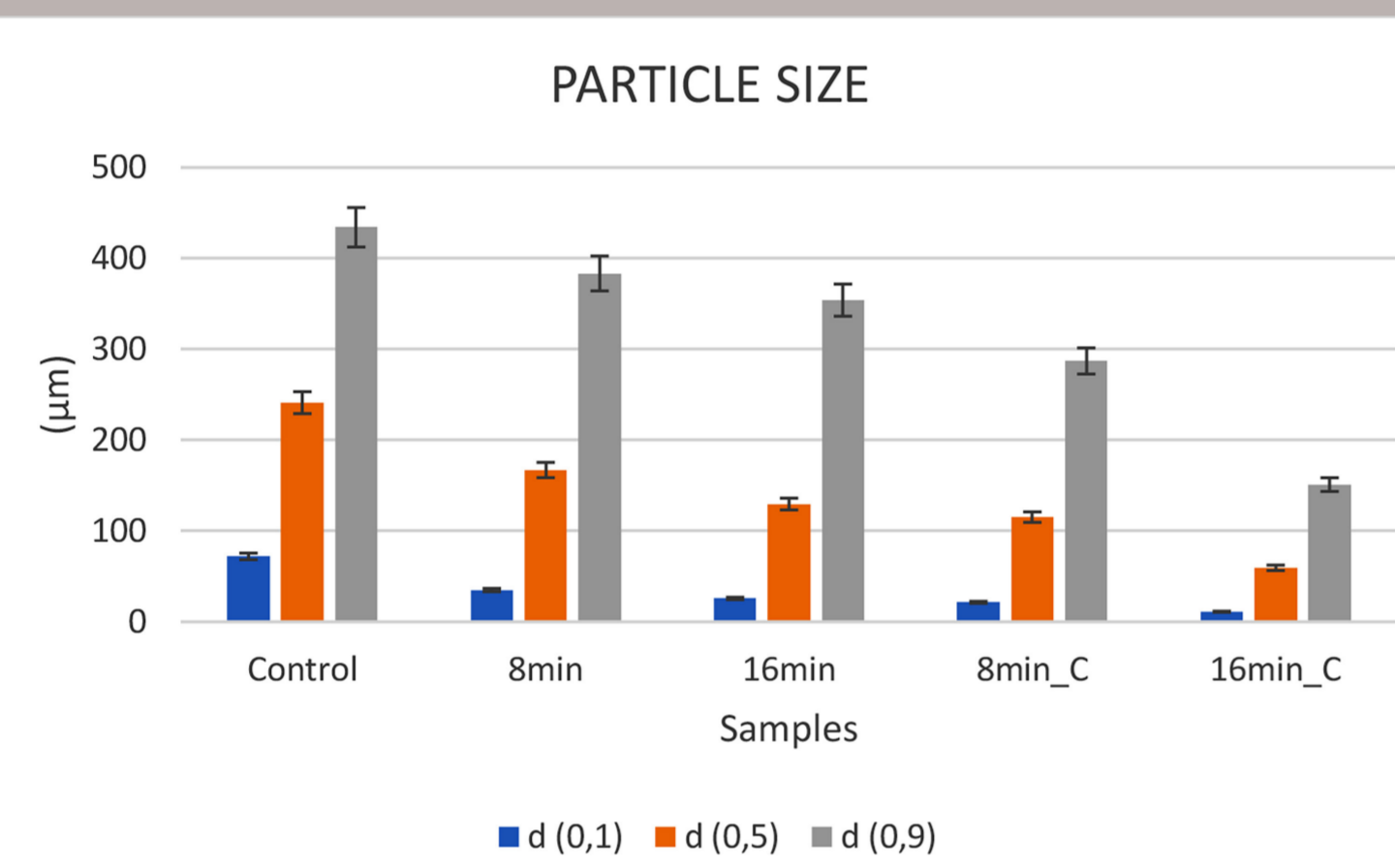
Interest in cold pressed oils in last decade has opened a promising scope for utilisation of their production by-products. One of the most popular oils is the one extracted from flaxseed which is mainly popular because of its high essential α -linoleic fatty acid content (up to 60%). Unfortunately, not all nutritionally valuable compounds are extracted with oil and are left unused in a press cake – solid, defatted by-product of oil production. Apart from that, cold pressing is a low yield process which leaves at least around 10% of oil in press cakes. These press cakes were used as cattle feed but lately they found their way on the shelves of specialised health stores sold as some sort of no gluten flour or used for vegetable protein isolation. Problem of quality of these products and bioavailability of their nutritional compounds is still being investigated.

The aim of this study was to determine impact of milling type (with or without cryocooling) on composition and content of flaxseed cake sterols, phenols, fatty acids, fibers, cyclolipopeptides and antioxidant activity of flaxseed cake.

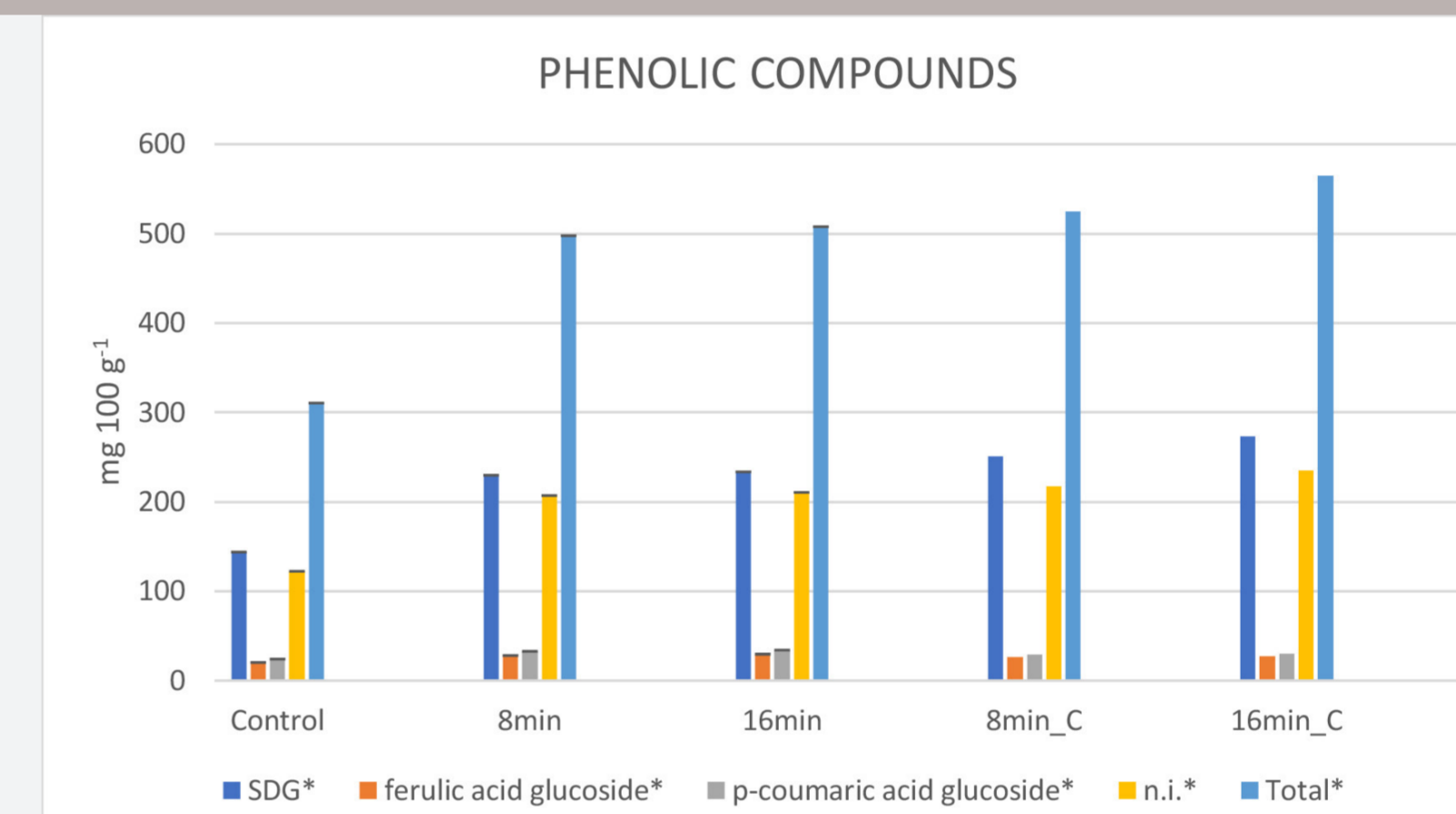
MATERIALS AND METHODS

Flaxseed seed cake was obtained as a by-product of oil cold pressing in a laboratory oil production with a screw press. The samples were milled on "CryoMill" (Retsch, Germany) and preliminary studies were undertaken to select grinding parameters of the cake after which 8 and 16 minutes with and without liquid nitrogen cooling were chosen. Particle size was determined with laser diffraction. Non-polar extracts were prepared with hexane using modified method from Kraljić et al (2013). The fatty acids and sterols were determined by gas chromatography according to HRN EN ISO 12966-2:2017 and HRN EN ISO 12228-1:2014. Polyphenols were extracted using microwaves with modified method from Beejmohun et al (2007) and determined with liquid chromatography. Cyclolipopeptides were extracted with modified method from Aladedunye et al (2013) and determined with liquid chromatography. Fibre content was determined using modified AOAC 2011.25 method (Megazyme, 2017). Antioxidant activity was determined by modified FRAP method from Benzie and Strain (1996).

RESULTS



d(0,1) – 10% of total particles; d(0,5) – 50% of total particles; d(0,9) – 90% of total particles
Figure 1. Average particle size (μm) \pm standard deviation, n=3



*Significant influence of milling time and interaction of milling with and without cooling ($p \leq 0,05$)
SDG – secoisolariciresinol diglucoside
Figure 2. Phenolic compounds ($\text{mg } 100 \text{ g}^{-1}$ \pm standard deviation, n=3)

Table 1. Fatty acid profile (% average \pm standard deviation, n=3) in hexane extracts

Exp.	Control	8min	16min	8min_C	16min_C
C14:0 Y	ND*	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0
C16:0	6,4 \pm 0,1	6,5 \pm 0,2	6,5 \pm 0,1	6,5 \pm 0,0	6,7 \pm 0,1
C16:1	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0
C17:0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0
C17:1	0,1 \pm 0,0	ND*	ND*	ND*	ND*
C18:0 Y	3,6 \pm 0,0	3,5 \pm 0,0	3,7 \pm 0,0	3,7 \pm 0,0	3,5 \pm 0,0
C18:1n9	18,0 \pm 0,2	17,6 \pm 0,2	18,2 \pm 0,2	18,3 \pm 0,0	18,0 \pm 0,2
C18:2t	0,8 \pm 0,0	0,8 \pm 0,0	0,8 \pm 0,0	0,8 \pm 0,0	0,8 \pm 0,0
C18:2c	10,9 \pm 0,1	10,7 \pm 0,2	11,0 \pm 0,1	11,1 \pm 0,0	10,9 \pm 0,2
C18:3n6	0,3 \pm 0,0	0,4 \pm 0,1	0,3 \pm 0,1	0,2 \pm 0,0	0,3 \pm 0,1
C18:3n3	56,7 \pm 0,6	55,5 \pm 0,5	57,2 \pm 0,5	57,5 \pm 0,1	56,2 \pm 0,7
C20:0	0,1 \pm 0,0	0,1 \pm 0,0	0,2 \pm 0,0	0,2 \pm 0,0	0,1 \pm 0,0
C20:1	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0
C22:0 Y	0,3 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0
C24:0 Y	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0	0,1 \pm 0,0
Σ saturated	10,5	10,3	10,8	10,8	10,7
Σ monounsaturated	18,3	17,8	18,4	18,5	18,2
Σ polyunsaturated	68,7	67,4	69,3	69,6	68,2
$\omega 6/\omega 3$	0,2 \pm 0,0	0,2 \pm 0,0	0,2 \pm 0,0	0,2 \pm 0,0	0,2 \pm 0,0
n.i.	2,5 \pm 1,1	4,5 \pm 1,1	1,7 \pm 0,9	1,1 \pm 0,0	2,8 \pm 1,1

ND- note detected; *defined as $\leq 0,05$ %; n.i. - not identified fatty acids*Milling conditions had significant influence on fatty acid content ($p \leq 0,05$)Table 2. Sterol profile (% average \pm standard deviation, n=3) in hexane extracts

	Brassicasterol	Campesterol	Campestanol	Stigmasterol	β -sitosterol [†]	β -sitostanol [†]	Δ -5-avenasterol	Δ -7-stigmasterol	Cycloartenol ^{***}	Δ -7-avenasterol ^{***}	n.i.	Total (mg kg ⁻¹) ^{****}
Control	2,16 \pm 0,01	24,41 \pm 0,00	2,81 \pm 0,00	9,09 \pm 0,00	44,99 \pm 0,01	2,22 \pm 0,00	10,29 \pm 0,00	0,76 \pm 0,01	2,16 \pm 0,00	0,54 \pm 0,01	0,57 \pm 0,01	285,65 \pm 3,58
8min	1,98 \pm 0,00	24,50 \pm 0,01	2,87 \pm 0,00	9,21 \pm 0,00	44,73 \pm 0,00	2,39 \pm 0,00	9,98 \pm 0,00	1,57 \pm 0,00	2,18 \pm 0,00	1,09 \pm 0,00	1,57 \pm 0,01	291,90 \pm 7,84
16min	nd**	22,78 \pm 0,01	2,62 \pm 0,00	8,60 \pm 0,00	39,81 \pm 0,01	3,04 \pm 0,00	9,24 \pm 0,00	nd**	9,62 \pm 0,01	2,36 \pm 0,00	1,94 \pm 0,03	347,73 \pm 11,18
8min_C	1,67 \pm 0,01	25,50 \pm 0,00	2,93 \pm 0,00	9,70 \pm 0,00	46,06 \pm 0,02	2,02 \pm 0,00	10,08 \pm 0,00	nd**	nd**	nd**	2,04 \pm 0,01	280,23 \pm 6,29
16min_C	2,13 \pm 0,01	22,97 \pm 0,02	2,74 \pm 0,00	8,69 \pm 0,01	39,82 \pm 0,02	2,69 \pm 0,00	8,74 \pm 0,00	1,25 \pm 0,00	nd**	nd**	10,98 \pm 0,07	289,10 \pm 16,01

ND- note detected; *defined as $\leq 0,05$ %; n.i. - not identified sterols*Milling conditions had significant influence on fatty acid content ($p \leq 0,05$)

CONCLUSIONS

1. Time and milling type had significant impact on the increase in phenolic concentration ($p \leq 0,05$). Phenolic concentration was increased by 82% in the sample treated for 16 minutes using cryocooling compared to the control sample.
2. Content of secoisolariciresinol-diglucoside (SDG) was higher by 90% in sample treated for 16 minutes using cryocooling which showed statistically significant correlation with antioxidant activity ($r > 0,66$).
3. Cryogenic milling had significant effect on yield of saturated fatty acids ($p \leq 0,05$) - myristic (C14:0), stearic (C18:0), behenic (C22:0) and lignoceric (C24:0).
4. The results showed significant impact ($p \leq 0,05$) of time and milling type on β -sitosterol, β -sitostanol, cycloartenol and Δ -7-avenasterol content.
5. The milling results showed a significant effect of cryogenic grinding on cyclolipopeptides CL_A, CL_B, CL_E and CL_OXID. The cryogenic grinding method did not show any major changes in the proportion of fibers.

LITERATURE

Aladedunye, F., Sosinska, E., Przybylski, R. (2013) Flaxseed Cyclolipopeptides: Analysis and Storage Stability. J. Am. Oil Chem. Soc. 90, 419-428.

Beejmohun, V., Fliniaux, O., Grand, E., Lamblin, F., Bensaddek, L., Christen, P., Kovensky, J., Fliniaux, M. A., Mesnard, F. (2007) Microwave-assisted Extraction of the Main Phenolic Compounds in Flaxseed. Phytochem. Anal. 18, 275-282.

Megazyme (2017) <https://secure.megazyme.com/Total-Dietary-Fiber-Assay-Kit>