Effect of chemical pretreatment on dehulling parameters of flaxseed (*cv. Garima*)

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Abstract In the present study, effects of chemical (ethanol, HCl and sulphuric acid) pretreatment on various dehulling parameters of flaxseed (*cv. Garima*) including yield, hull, hullability, extraction rate and embryo (dehulled flaxseed) recovery were studied. Pretreated flaxseed, at 3.1 to 3.6 % moisture range ($p>0.05$) were dehulled for 60 s in a laboratory model rice polisher/dehulling machine at 2,000 rpm followed by aspiration (hull separation) using a laboratory model aspirator. The study revealed that chemical pre-dehulling treatment of flaxseed plays a significant role in the embryo recovery of flaxseed. Both ethanol and HCl pre-dehulling treatment enhanced but sulphuric acid pretreatment reduced the embryo recovery of flaxseed. Moreover, HCL and Sulphuric acid deteriorated the quality of hull obtained during dehulling, hence may not be considered for flaxseed dehulling. The study showed the maximum embryo recovery from ethanol pretreated flaxseed, hence ethanol pre-dehulling treatment with 2 h tempering time may be considered for effective flaxseed dehulling.

Keywords Flaxseed · Chemical pretreatment · Dehulling · Embryo recovery

Flaxseed is emerging as one of the important oilseed in the functional food arena. In addition to being one of the richest sources of α -linolenic acid oil and lignans, flaxseed is an

important source of good quality protein, soluble fiber and phenolic compounds (Oomah 2001). The presence of these compounds makes the grain beneficial and can be used for making food products for human consumption (Mridula et al. 2011; Pohjanheimo et al. 2006). Lignans, present in flaxseed appear to be anti-carcinogenic compounds (Lay and Dybing 1989). Omega-3 fatty acids are responsible for forming a group of hormones-like compound called prostaglandins, which help in fighting many diseases. They make the cell permeable, which helps in the protection of cells against invading bacterial toxins, infections, and viruses (Javed 1999). The α -linolenic acid in flaxseed can reduce the risk of cardiovascular disease (CVD) osteoporosis, rheumatoid arthritis, and cancer (Aldercreutz 2007; Kettler 2001; Spence et al. 2003; Clark et al. 2000; Prasad et al. 1998). Omega-3 fatty acids present in flaxseed help in reducing blood triglycerides, blood pressure, platelet reactivity, neutrophil activity, and increase blood HDL cholesterol thereby helping in lowering CVD risk (Gorder et al. 1986; Cunnane et al. 1993; Li et al. 1999). Though the flaxseed is a unique grain and posses many health benefits but the hard outer surface of whole flaxseed, when eaten whole, the inside kernel and other healthy nutrients may not be easily digested by the human body. The higher level of soluble fiber of whole flaxseed also increases gastric viscosity and leads to reduced nutrient availability in monogastric diets (Bell and Keith 1993). The laxative properties of the whole flaxseed meal arise from its content of pentosans and mucilage (Bhatty and Cherdkiagumchai 1990). As flax mucilage occurs mainly in the epidermis layer of the seed coat (Dev and Quensel 1988), removal of the hull will eliminate most of the mucilage and much of the fiber. The flaxseed hull does not readily detach from the embryo; however, flax mucilage has traditionally been removed by aqueous extraction (Cui et al. 1994; Fedenuik and Biliaderis 1994;

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Garden-Robinson 1994; Mazza et al. 1992; Mazza and Biliaderis 1989; Susheelamma 1987; Wannerberger et al. 1991). Although the mucilage obtained is claimed to be fairly free of contaminants, the wet process may not economical since it involves multiple steps, including drying.

Flax hull removal by mechanical means is an important process for preparing value-added products for human as well as animal consumption. Removal of flax mucilage by dry dehulling i.e. by mechanical means was investigated by Smith et al. (1946) and Dev and Quensel (1988) using a simple process of fractionating ground flaxseed with graded sieves, and also an air-separation method. The moisture content and residence time played an important role in dehulling of flaxseed (Barnwal et al. 2010). Though the lower moisture content facilitates the hull removal but still there are challenges to remove the maximum amount of hull from the flaxseed because of the greater hull thickness, poor seed thickness (0.85 to 1.06 mm) and smaller grain size (geometric mean diameter 3.12–5.6 mm) of flaxseed (Singh et al. 2011) and may also be due to the greater proportion of tougher seed coat i.e. hull in flaxseed. Studies reported the possibility of chemical pre-treatment for dehulling of dent corn (Blessin et al. 1970) and sesame seed (Sastry et al. 1969). Sreedhara et al. (1992) studied the effect of various chemical treatments (viz. hexane, methanol, chloroform, sulphuric acid, hydrochloric acid (HCl), sodium hydroxide and potassium hydroxide, phosphoric acid and acetic acid) and found that treatment with 4 N hydrochloric acid (HCl) was effective in complete removal of the testa, producing a pearl-white palm kernel. With this background, the study of selected chemical (ethanol, HCl and sulphuric acid) pre-treatment of flaxseed was therefore, undertaken to find out their effect on various dehulling parameters of flaxseed.

Materials and methods

The flaxseed (*cv. Garima*) was procured from Chandra Shekhar Azad University of Agriculture and Technology, Kanpur. The seeds were cleaned and graded by sieves to remove broken seeds and other foreign materials. The raw flaxseed was sieved by ASTM Nr 10 sieve and the flaxseed retained on the sieve was used for the experiments.

Pre-treatment of flaxseed The initial moisture content of flaxseed was determined by hot air oven method at 105 °C for the period until the constant weight reached (AOAC 1984). The flaxseed samples at initial moisture content 5.68 % were pre-treated with 20 ml of absolute ethanol, hydrochloric acid (0, 1 N, 1.5 N and 2 N) and sulphuric acid (0, 1 N and 1.5 N) by manual mixing to facilitate the hull removal during dehulling. The time taken in pretreatment i.e. mixing of flaxseed with chemicals was about 40–50 s.

The pre-treated flaxseed samples (sample size 100 g) were packed immediately in self sealable polyethylene bags and kept at room temperature 30 ± 2 °C for three different tempering periods i.e. 2, 3 and 4 h. The pretreated and tempered flaxseed samples then dried in a recirculatory tray dryer at 50 °C to achieve lower moisture contents. The moisture content of all the pre-treated dried samples was in the range of 3.1 to 3.6 % ($p > 0.05$).

De-hulling of flaxseed The pretreated dried flaxseed samples were dehulled in the laboratory model rice polisher (STE-08, Cadence Electronics Systems, Ambala, Haryana, India) at 2,000 rpm of the rotor at 60s residence time. During preliminary trials, flaxseed was dehulled at 40, 50 and 60s residence time, which showed that the lower residence time i.e. 40 and 50s were not sufficient to dehull the flaxseed (*cv. Garima*); therefore, 60s residence times was considered for dehulling of flaxseed. The hull was separated from dehulled mixture by using a batch type laboratory aspirator (6726, M/s Osaw Industrial Products Pvt Ltd, Ambala, Haryana, India). The dehulled flaxseed was sieved using the ASTM Nr 20 sieve to separate the powder from the mixture of the dehulled flaxseed. The powder was weighed and the fraction retained on ASTM Nr 20 sieve was fed in the laboratory aspirator for separation of hull. The hull and flaxseed mixture (unhulled and dehulled flaxseeds) were obtained after aspiration and weighed. The dehulled mixture was manually separated to get the embryo recovery.

Dehulling parameters

Yield of hull (%) It is defined as weight loss of the seed after dehulling of flaxseed (Oomah et al. 1996; Barnwal et al. 2010) and expressed as:

$$Y_{\text{hull}}(\text{fraction}) = \frac{\Delta W}{W_{\text{in}}} \quad (1a)$$

and,

$$Y_{\text{hull}}(\%) = \left(\frac{\Delta W}{W_{\text{in}}} \right) \times 100 \quad (1b)$$

where, ΔW = weight loss of seed = weight of (powder + broken) + machine loss, Machine loss = (feed input–products output) from the machine, and W_{in} = initial sample weight (prior to dehulling).

Hullability (%) It is defined as the ratio of hull removed during dehulling to the total quantity of hull in the seed (Oomah et al. 1996; Barnwal et al. 2010) and written as:

$$H(\text{fraction}) = \frac{\Delta W}{W_{\text{m-hull}}} \quad (2a)$$

and,

$$H(\%) = \left(\frac{\Delta W}{W_{m-hull}} \right) \times 100 \quad (2b)$$

where, W_{m-hull} = total quantity of hull in raw flaxseed (per 100 g seed) obtained by manual hulling of 50 seeds.

Extraction rate (ER)(%) It is an indication of endosperm recovery and calculated as (Oomah et al. 1996; Barnwal et al. 2010):

$$ER(fraction) = \frac{W_{dehulled\ mix}}{W_{in}} = \frac{W_{in} - \Delta W}{W_{in}} = 1 - Y_{hull}(fraction) \quad (3a)$$

and,

$$ER(\%) = 100 - Y_{hull}(\%) \quad (3b)$$

where, $W_{dehulled\ mix}$ is weight of (dehulled + un-hulled) seed mixture after dehulling.

Hull (%) It is defined as the ratio of hull recovered to the total quantity of hull present in the seed and expressed as (Oomah et al. 1996; Barnwal et al. 2010):

$$R_{hull}(fraction) = \frac{W_{r-hull}}{W_{m-hull}} \quad (4a)$$

and,

$$R_{hull}(\%) = \left(\frac{W_{r-hull}}{W_{m-hull}} \right) \times 100 \quad (4b)$$

where, R_{hull} is hull recovered (fraction or %); W_{r-hull} is weight of recovered hull by aspirator (per 100 g seed) and W_{m-hull} is weight of hull (per 100 g seed) obtained by manual hulling of 50 seeds.

Fig. 1 Effect of ethanol pretreatment on dehulling parameters of flaxseed ($n=3$)

Table 1 Analysis of variance for different dehulling parameters of flaxseed pre-treated with ethanol

Dehulling parameters	df	SS	MS	F-value	p-value
Pre-treatment with ethanol					
Extraction rate	3	11.05	3.68	2.17 ^{ns}	0.170
Hullability	3	37.32	12.44	2.17 ^{ns}	0.170
Yield	3	11.05	3.68	2.17 ^{ns}	0.170
Embryo recovery	3	97.03	32.34	38.70*	0.001
Hull	3	68.05	22.68	4.83*	0.033

^{ns} not significant; * significant at $p \leq 0.05$

Table 2 Analysis of variance for different dehulling parameters of flaxseed pretreated with acids

Dehulling parameters		df	SS	MS	F-value	p-value
Pretreatment with HCl						
Extraction rate	H	3	20.61	6.87	3.19 ^{ns}	0.11
	T	2	2.35	1.78	0.55 ^{ns}	0.61
	HT	6	17.07	2.85	1.32 ^{ns}	0.37
Hullability	H	3	69.68	23.23	3.20 ^{ns}	0.11
	T	2	7.93	3.97	0.55 ^{ns}	0.61
	HT	6	57.65	9.61	1.32 ^{ns}	0.37
Yield	H	3	20.61	6.87	3.20 ^{ns}	0.11
	T	2	2.35	1.78	0.55 ^{ns}	0.61
	HT	6	17.07	2.85	1.32 ^{ns}	0.37
Embryo recovery	H	3	34.53	11.51	17.77*	0.002
	T	2	0.16	0.08	0.12 ^{ns}	0.89
	HT	6	4.19	0.70	1.08 ^{ns}	0.47
Hull	H	3	37.76	12.59	4.05 ^{ns}	0.07
	T	2	2.62	1.31	0.42 ^{ns}	0.67
	HT	6	22.53	3.75	1.21 ^{ns}	0.41
Pretreatment with sulphuric acid						
Extraction rate	S	2	11.02	5.51	1.86 ^{ns}	0.269
	T	2	24.28	12.14	4.10 ^{ns}	0.108
	ST	4	29.21	7.30	2.46 ^{ns}	0.202
Hullability	S	2	37.24	18.62	1.86 ^{ns}	0.269
	T	2	81.94	40.97	4.10 ^{ns}	0.108
	ST	4	98.90	24.73	2.46 ^{ns}	0.202
Yield	S	2	11.02	5.51	1.86 ^{ns}	0.269
	T	2	24.28	12.14	4.10 ^{ns}	0.108
	ST	4	29.21	7.30	2.46 ^{ns}	0.202
Embryo recovery	S	2	91.48	45.74	13.09*	0.018
	T	2	0.69	0.35	0.10 ^{ns}	0.908
	ST	4	14.56	3.64	1.04 ^{ns}	0.485
Hull	S	2	198.18	99.09	28.25*	0.004
	T	2	5.77	2.88	0.82 ^{ns}	0.502
	ST	4	59.55	14.89	4.24 ^{ns}	0.095

H Normality of HCl; T tempering time, S Normality of sulphuric acid; ^{ns} not significant; * significant at $p \leq 0.05$

Embryo recovery (%) It is defined as the ratio of dehulled whole flaxseed (embryo), obtained after dehulling to the actually embryo present in the seed. It may be expressed as (Barnwal et al. 2010):

$$R_{\text{embryo}}(\text{fraction}) = \frac{W_{\text{r-embryo}}}{W_{\text{m-embryo}}} \quad (5a)$$

and,

$$R_{\text{embryo}}(\%) = \left(\frac{W_{\text{r-embryo}}}{W_{\text{m-embryo}}} \right) \times 100 \quad (5b)$$

Fig. 2 Effect of hydrochloric acid (with different concentrations) pretreatment on dehulling parameters of flaxseed ($n=2$)

where, R_{embryo} is embryo recovery (fraction or %); $W_{\text{r-embryo}}$ is weight of recovered embryo (per 100 g seed) and $W_{\text{m-embryo}}$ is the weight of embryo in the seed obtained by manual hulling of 50 seeds (per 100 g seed).

Statistical analysis Data pertaining to three replicates of ethanol pretreated samples ($n=3$) and two replicates of hydrochloric acid ($n=2$) and sulphuric acid ($n=2$) pretreated samples were analyzed for analysis of variance using LSD of AgRes software statistical package.

Results and discussion

Results showed that ethanol pre-treatment of flaxseed affected the embryo recovery and hull significantly ($p<0.05$) but other dehulling parameters were not affected by the ethanol pretreatment. The embryo recovery of ethanol pre-treated sample was 70.96 %, 69.88 % and 68.97 %, respectively for 2 h, 3 h and 4 h tempering time as compared to 63.58 % in case of untreated (control) samples (Fig. 1). Though the ethanol pre-treatment enhanced the embryo recovery by 6.3–7.38 % but the tempering time from 2 h to 4 h did not show much difference in the embryo recovery. Mean comparison by

Fig. 3 Effect of sulphuric acid (with different concentrations) pretreatment on dehulling parameters of flaxseed ($n=2$)

LSD showed that embryo recovery of ethanol pre-treated flaxseed tempered for 2 h and 3 h, and 3 h and 4 h were found at par with lowest embryo recovery of control flaxseed sample. As the percent embryo recovery of 2 h tempered ethanol pre-treated sample was found maximum, 2 h tempering time may be considered for dehulling of flaxseed. The recovery of hull was found in the range of 48.48 to 50.26 % in ethanol pre-treated sample as against the 43.88 % of untreated flaxseed. Hull percentage was also found at par in case of 2 h, 3 h and 4 h tempering time for ethanol pretreated sample. Barnwal et al. (2010) reported 54.2 % embryo recovery of flaxseed at 1.9 % moisture content and 45.8 % at 4.5 % moisture content, which was comparatively lower than the finding of the present study for untreated flaxseed. This variation may be due to difference in the flaxseed variety used for dehulling experiments and the residence time for dehulling. Hull percentage was also found higher in the present study than reported by Barnwal et al. (2010) and Oomah et al. (1996), may be because of the better hullability of the seed used in the present study. The other dehulling parameters viz. extraction rate, hullability and yield though varied slightly but not found affected ($p>0.05$) with the pretreatment with ethanol (Table 1).

Table 2 indicated that pretreatment of flaxseed with HCl and tempering time did not affect significantly to various dehulling parameters except on embryo recovery. The embryo recovery of HCl pretreated flaxseed was found in the range of 65.48–67.1 % (Fig. 2) which was higher than untreated flaxseed but the normality of HCl and the tempering time did not indicate any significant effect on embryo recovery after dehulling of flaxseed. The other dehulling parameters viz. extraction rate, hullability, yield and hull percentage though varied but not found affected ($p>0.05$) with the pretreatment with HCl. Though the pretreatment of flaxseed with HCl increased the embryo recovery but it was found that the pretreatment of flaxseed with sulphuric acid lowered the embryo recovery as compared to the untreated flaxseed (Fig. 3). The embryo recovery of sulphuric acid pretreated flaxseed was ranged from 56.49 to 61.47 % as compared to 63.58 % of untreated sample. The recovered hull percentage was also found low i.e. 32.75–42.98 % in sulphuric acid pretreated flaxseed than the HCl pretreated flaxseed (40.19–44.95 %). The extraction rate, hullability and yield percentage of sulphuric acid pretreated flaxseed though varied but statistically similar ($p>0.05$) (Table 2). The embryo recovery and hull percentage in the acid pretreated flaxseed was also found higher than the findings of the Barnwal et al. (2010) and Oomah et al. (1996). This may be due to difference in varietal characteristics of the flaxseed used in this study.

It was observed during the dehulling of flaxseed that there was a slight burning affect of acids at higher normality that made the hull shriveled and lowered the quality of hull, particularly with sulphuric acid. This may be due to the dissolve of outer surface of the seed by the acid. In case of flaxseed

pretreated with sulphuric acid of 1.5 N, it was very difficult to separate the hull from the dehulled mixture because of its shriveled and finer from, which indicated that sulphuric acid of more than 1 N cannot be used for pretreatment of flaxseed.

In the present study, the embryo recovery of ethanol pre-treated flaxseed was higher than the HCl pretreated flaxseed and the quality of the hull obtained from ethanol pretreated sample was also found better on visual observation than the HCl pre-treated flaxseed. Therefore, ethanol pretreatment may be considered for pre-dehulling treatment of flaxseed.

Conclusion

The study was conducted to see the feasibility of using chemical pretreatment namely ethanol, hydrochloric acid and sulphuric acid for dehulling of flaxseed. It was found that chemical pre-dehulling treatment of flaxseed plays an important role in flaxseed dehulling. In contrast to ethanol and HCl, sulphuric acid pretreatment was not found feasible due to lower embryo recovery from flaxseed with this pretreatment. Moreover, the study also revealed that HCL and Sulphuric acid are not suitable because of their adverse effect on quality of hull obtained during dehulling. In view of the maximum embryo recovery from ethanol pretreated flaxseed, ethanol pre-dehulling treatment with 2 h tempering time may be considered for effective flaxseed dehulling.

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