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## Physico-chemical and functional properties of flour prepared from native and roasted whole linseeds

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**Abstract**

The present research work was conducted to analyze the physico-chemical, functional, morphological properties and fatty acid profile of the flour, obtained from the raw and roasted whole linseeds (Padmini variety). The moisture, carbohydrates and crude fibre were found lower whereas, protein, fat, ash and energy value were found significantly higher, after roasting. Water absorption capacity of raw linseed flour was found significantly higher than roasted linseed flour. Whereas, water solubility index and oil absorption capacity of roasted linseed flour was found significantly higher than raw linseed flour. Fatty acid profile of roasted linseed flour showed slight increase in fatty acid values, than raw linseed flour. The morphological analysis revealed that irregular shaped particles of raw linseed flour changed to cluster like structure in the roasted linseed flour. XRD profile revealed diffused and large peaks of raw and roasted linseed flour indicating the presence of amorphous materials within its matrix.

**Keywords:** Linseed, roasting, fatty acid profile, morphology, XRD profile

**1. Introduction**

Linseed (*Linum usitatissimum*) belongs to family *Linaceae* is categorized as an important oilseed crop, globally i.e. grown under the cool climatic conditions. Canada, China and India are the largest producers of linseed in the world and are ranked in terms of production in their respective order. In the world, India ranks first and third for 23.8% of the total land under linseed cultivation and 10.2% of the total world's production, respectively among the leading producers of the linseed<sup>[1]</sup>. With increasing demand of nutritional products, the consumption of linseed (*Linum usitatissimum*) is increasing day by day. Linseed is used as a functional food which has unique nutritional profile and contains 40-45% of oil which has high portion of polyunsaturated fatty acid (70-72%), moderate quantity of unsaturated fatty acid (18-20%) and low quantity of saturated fatty acid (7-9%)<sup>[2]</sup>. Linseed is also a good source of bioactive components i.e. oil, phenolic compounds, protein, soluble polysaccharides, dietary fibre, lignans, mineral (P, Mg, K, Na, Fe, Cu, Mn and Zn) and ALA (alfa linolenic acid) and vitamins (A, C and E) which are stable under normal processing conditions, like development of flaxseed fortified macaroni and spaghetti<sup>[3, 4, 5]</sup>. The nutritional value of processed food can be increased by fortification with linseed that will enable decreasing the risk of diabetes, breast cancer and cardiovascular diseases<sup>[6]</sup>.

Roasting is a cooking method using dry heat and a commonly used technique, it has been reported that roasting improves the edibility, texture and digestibility of grains, reduces their anti-nutrient factors and also prevents the loss of nutrients<sup>[7]</sup>. The roasting of linseed is very important process for enhancing its color, texture and flavour due to various reactions taking place and thereby revealing the organoleptic characteristics of linseed. In roasting process, the color change in product occurs by browning mainly by non-enzymatic browning reaction (Maillard reaction) where free carbonyl group of reducing sugar reacts with amino group of amino acid (protein) producing high molecular brown color edmelanoids<sup>[8]</sup>. Autoxidation of oilseeds primarily produces odor less hydro peroxides which are responsible for stale flavour and decomposition of food forming hydrocarbons, carbonyl compounds, furan and other compound involved in biological oxidation<sup>[9]</sup>.

Systematic and scientific investigations are needed for the efficient utilization of linseed flour for food uses. An increasing demand for easily available functional food has been created the need for new sources of linseed flour. The investigations on characterization of linseed flour are scanty and it is imperative to be exploring as a new source of functional foods for different food formulations. However, keeping the above facts in mind, this study evaluated the effect of roasting on the physicochemical, functional, morphological properties, crystalline and fatty acid profile of linseed flour (Padmini variety).

## 2. Material and Methods

### 2.1 Material

Linseed kernels (Padmini variety) grown in India, were procured from Dr. Hari Singh University, Sagar, Madhya Pradesh, India, subjected to air classifier to remove the dirt, dust, chaffs, lighter particles as well as damaged kernels. Linseed kernels were kept in air tight HDPE containers and stored at dry and cool environment for further treatments.

### 2.2 Roasting condition

Roasting time and temperature combination for roasting of linseed kernels was optimized by the authors based on preliminary trails. The roasting condition (temperature and time combination) was optimized by response surface method (RSM) on the basis of hardness, color analysis and sensory evaluation of roasted linseed kernels with two variables as temperature (150-290°C) and time (34-46 sec). According to RSM, temperature 150°C for 40 sec was found the lowest time temperature combination whereas, 290°C for 40 sec was the highest time-temperature combination, for roasting of the linseed kernels. Hardness of the kernels was found maximum i.e. 65.68 N at 150°C for 40 sec and minimum (31.39 N) at 290°C for 40 sec whereas, at 220°C for 40 sec its hardness value was found within the range i.e. 53.69 N. Color of the roasted linseed (L, a and b value) was also found within range after roasting at 220°C for 40 sec whereas, L, a and b values of roasted linseed were found lowest at 290°C for 40 sec and highest at 150°C for 40 sec. The sensory evaluation of the roasted linseed show highest score (7.84) for the sample roasted at 220°C for 40 sec whereas, lowest sensory score was noted when sample was roasted at 150°C for 40 sec and 290°C for 40 sec. Roasting treatment of the linseeds at temperature 220°C for 40 sec was found best with the references of hardness, color score and sensory evaluation of the roasted linseed kernels.

### 2.3 Sample preparation

Linseed kernels were roasted at 220°C for 40 sec in electric

$$\text{WAC } \left(\frac{g}{g}\right) = \frac{(\text{wt. of centrifuge tube with sample after drying} - \text{wt. of centrifuge tube}) - \text{Sample wt}}{\text{Sample Weight}} \quad (1)$$

$$\text{WSI } (\%) = \frac{\text{Weight of dissolved solids in supernatant}}{\text{Weight of flour sample}} \times 100 \quad (2)$$

#### 2.4.2.2 Oil absorption capacity (OAC)

The oil absorption capacity of the linseed flour samples was analyzed using the method also described by Sosulski, Humbert, Bui and Jones [11]. Linseed flour (1 g) in a pre-weighed centrifugal tube was thoroughly mixed with 10 ml of

$$\text{OAC } \left(\frac{g}{g}\right) = \frac{\text{wt. of centrifuge tube with sample after removing oil} - (\text{centrifuge tube wt.} + \text{Sample Wt.})}{\text{Sample Weight}} \quad (3)$$

### 2.4.3 Fatty acid profile

GC-MS analysis of the fatty acid of linseed flour was carried out after methylation. The fatty acids composition of linseed flour was analyzed using agilent gas chromatograph (GC) attached to a DB-225 polar column (30m, 0.322mm, 0.25µ) and flame ion detector (FID), used for the detection of fatty acid composition of the samples. The oil sample (20-25 mg) in a glass stopper flask was treated with 5 ml of 0.55 sodium methoxide solution. The content was heated for 10-15 min at around 50°C and then 0.1 ml glacial acetic acid was added followed by 5-10 ml of water. The organic phase of the

rotary roaster (Make: S & SB Associated Engineers, India) having a temperature controller at a constant speed. After roasting, roasted linseed kernels were brought to cool and stored at room temperature (20±2°C) in a cool and dry place. The roasted sample was milled using mixer grinder (Make: Sujata, India; Model: Dynamix, 810Watts) and sieved through a 250 µm mesh screen to obtain roasted whole linseed flour. The roasted whole linseed flour was packed in a high density polyethylene (HDPE) bag and stored for further analysis. The native whole linseed flour was also prepared using same methodology. Native linseed flour was treated as control for further study.

## 2.4 Methods

### 2.4.1 Proximate composition

The moisture, ash, protein, fat and crude fibre of linseed kernels was analyzed according to AOAC [10]. Protein content and fat content of the linseed flour was determined by Kjeldhal method and Soxhlet method, respectively. Carbohydrate content of the linseed flour was calculated by differential method whereas, total calories values were theoretically derived from the content in fats (9 kcal/g), carbohydrates (4 kcal/g) and proteins (4 kcal/g). The results of the compositional analysis are the mean of three experimental measurements.

### 2.4.2 Functional properties

#### 2.4.2.1 Water absorption capacity (WAC) and water solubility index (WSI)

WAC and WSI were determined using the method described by Sosulski, Humbert, Bui and Jones [11]. Linseed flour (1g) was dispersed in 10 ml water for 30 min at room temperature and then centrifuged at 3000g for 25min. WAC (Water absorption capacities) of the flours were expressed as grams of water absorbed by 1 g force of linseed flour (Eq.1). WSI (Water solubility index) expressed as the weight of dry solids in the supernatant, as a percentage of the original weight of sample (Eq. 2).

refined oil at room temperature for 30 min. The tubes were centrifuged at 3000g force for 25 min. OAC (Oil absorption capacity) was calculated as grams of oil absorbed by 1 g of linseed flour (Eq. 3).

sample was extracted with hexane (15 -20 ml) and washed with water till neutral pH. The extract of the hexane was dried over anhydrous sodium sulphate and concentrated under reduced pressure to get the methyl ester. The temperature of oven, injectors and detector blocks has been maintained at 210, 230 and 250°C respectively. Nitrogen was used as the carrier gas and the peaks were identified by comparison with relative retention times of the standard fatty acid methyl esters (FAMES). By normalization of peak areas using GC post run analysis software, the concentration of each fatty acid in percentage and manual integration was recorded.

### 2.4.4 Scanning electron microscopy (SEM)

The morphology of linseed flours was analyzed by scanning electronic microscopy (Hitachi, S-3400N, Tokyo, Japan). The flours were placed on an adhesive tape attached to a circular aluminum specimen stub. The samples were coated with a thin layer of gold and fixed for photographed at an accelerator potential of 5 kV.

### 2.4.5 X-ray diffraction analysis

X-ray diffraction analysis of the linseed flour samples were carried by using PAN analytical apparatus (Model No. X-Pert PRO, Phillips, Holland) with these preset conditions: target Cu-anode X-ray, 30 kV, 40 mA with scanning speed of 0.5°/min. The Origin Pro software package was used to determine the total area under the curve and the area under each prominent peak. The percentage crystallinity of the linseed flour was estimated by using the following formula (Eq. 4):

$$\% \text{ Crystallinity} = (\text{Area under peaks}) / \text{Total area} \times 100 \quad (4)$$

### 2.8 Statistical analysis

All the data of linseed flour were determined in triplicates and results were represented as mean $\pm$ SD. Statistical analysis was done using Statistical-log software package version 7 (M/s. Stat Soft Inc., OK, USA) by using one-way analysis of variance (ANOVA) followed by Duncan's multiple range test at significance level of  $p < 0.05$ .

## 3. Result and Discussion

### 3.1 Physico-chemical characterization of raw and roasted linseed flour

Physico-chemical composition of raw and roasted linseed

flour is presented in Table 1. Whole linseed kernels were roasted at 220°C for 40 sec and milled into flour whereas raw whole linseed kernels were milled into flour without roasting. Roasted linseed flour was found to have significantly ( $p < 0.05$ ) higher protein content (24.19%), fat content (46.02 %), ash content (3.57%) and energy value (550.63 kcal) than raw linseed flour protein content (20.79%), fat content (41.78%), ash content (3.39%) and energy value (503.88 kcal) (Table 1). The increase in protein content of roasted linseed flour may be due to the fact that roasting treatment increases the activity of proteolytic enzymes which hydrolyzed inherent proteins to their constituent amino acids and peptides [12]. Fat content of roasted linseed flour increased due to the increase in a surface area resulting by the demolition of cells resulting in enhancing the extraction of lipid content [13]. Ash content increases in a minor amount may be due to increase in total solid content in roasted linseed flour [13]. Increase in the fat and protein value during roasting may be the probable reason to increase in the energy value of the roasted linseed flour.

Roasted linseed flour has significantly ( $p < 0.05$ ) less moisture content (3.65 %), carbohydrate content (16.29%) and fibre content (9.93%) in comparison to raw linseed flour moisture content (5.71 %), carbohydrate content (22.86%) and fibre content (11.18%) (Table 1). This decrease in moisture content of roasted linseed flour might be due to the effect of heat produced during roasting of whole raw linseed kernels. The decreasing trend in carbohydrate content in roasted linseed flour might be due to increase in the fat and protein content of the flour after roasting. The crude fibre content reduces after roasting because of the available polysaccharides in linseed are mainly insoluble fibres which are very stable in nature but some fibre insignificantly participate in Maillard reactions [13].

**Table 1:** Physico-chemical characterization, Functional properties and Fatty acid profile of raw and roasted whole linseed flour

Physico-chemical Properties							
Linseed flour	Moisture (%)	Ash (%)	Fat (%)	Protein (%)	Fibre (%)	Carbohydrate (%)	Energy (Kcal)
Raw	5.71 <sup>a</sup> $\pm$ 0.05	3.39 <sup>b</sup> $\pm$ 0.06	41.78 <sup>b</sup> $\pm$ 0.21	20.79 <sup>b</sup> $\pm$ 0.97	11.18 <sup>a</sup> $\pm$ 0.42	22.86 <sup>a</sup> $\pm$ 0.63	503.88 <sup>b</sup> $\pm$ 3.21
Roasted	3.65 <sup>b</sup> $\pm$ 0.08	3.57 <sup>a</sup> $\pm$ 0.07	46.02 <sup>a</sup> $\pm$ 0.75	24.19 <sup>a</sup> $\pm$ 0.038	9.93 <sup>b</sup> $\pm$ 0.12	16.29 <sup>b</sup> $\pm$ 0.68	550.63 <sup>a</sup> $\pm$ 6.38
Functional Properties							
Linseed flour	WAC (g/g)		WSI (%)		OAC (g/g)		
Raw	1.99 <sup>a</sup> $\pm$ 0.40		8.95 <sup>b</sup> $\pm$ 0.62		1.66 <sup>a</sup> $\pm$ 0.18		
Roasted	1.79 <sup>a</sup> $\pm$ 0.20		11.51 <sup>a</sup> $\pm$ 1.07		1.99 <sup>a</sup> $\pm$ 0.61		
Fatty acid profile							
Linseed flour	Palmitic acid (C16:0) (%)	Stearic acid (C18:0) (%)	Oleic acid (C18:1) (%)	Linoleic acid (C18:2) (%)	Linolenic acid (C18:3) (%)		
Raw	3.05	1.72	7.83	5.81	23.97		
Roasted	3.17	2.00	8.97	5.86	25.58		

Results are expressed as mean values  $\pm$  standard deviation of three determinations. Mean values in the same row which is not followed by the same letter are significantly different ( $p < 0.05$ ). WAC=Water absorption capacity, WSI= Water solubility index, OAC= Oil absorption capacity

### 3.2 Functional properties of raw and roasted linseed flour

Water absorption capacity (WAC) of raw linseed flour (1.99 g/g) was higher than roasted linseed flour (Table 1). The higher WAC of raw linseed flour may be due to the presence of hull mucilage polysaccharides which adsorb water and swell [14] or due to the gelation of carbohydrates and swelling of crude fibres [15]. Water solubility index (WSI) of roasted linseed flour (11.51%) was found significantly ( $p < 0.05$ ) higher as compared to raw linseed flour (8.95%) (Table 1). WSI of roasted linseed flour may have increased due to

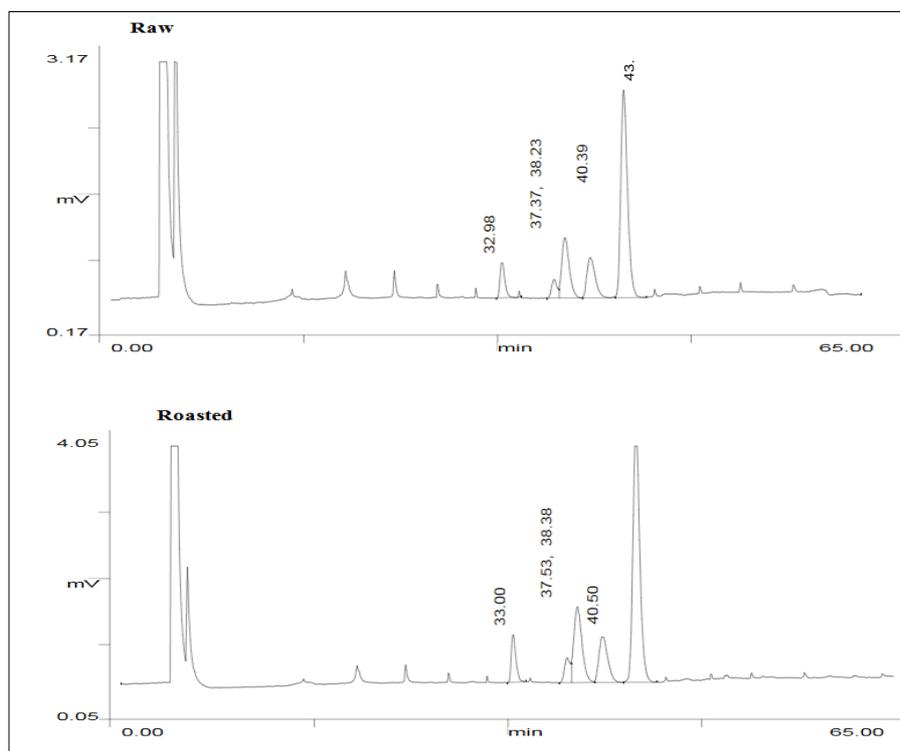
presence of higher solubilized native protein than raw linseed flour [16]. The oil absorption capacity (OAC) is a prominent factor in food formulations because it improves flavour and increase mouth feel of most of the foods. The roasting treatment of linseed kernel significantly ( $p < 0.05$ ) affected oil absorption capacity of the roasted linseed flour i.e. 1.99 g/g, found higher than OAC of raw linseed flour (1.66 g/g).

### 3.3 Fatty acid profile

The comparisons between fatty acid profile of raw and roasted linseed flours are displayed in Table 1. Fatty acids i.e. Palmitic acid (C16:0), Stearic acid (C18:0), Oleic acid (C18:1), Linoleic acid (C18:2), and Linolenic acid (C18:3) are increased slightly in roasted linseed flour than raw linseed flour. Comparatively, higher amount of palmitic acid (3.05 %), stearic acid (1.72 %) and oleic acid (7.83) was found in

roasted linseed flour than raw linseed flour having palmitic acid (3.17 %), stearic acid (2.0 %) and oleic acid (8.97 %) (Fig.1). Fatty acids, like omega 3 (linolenic acid) and omega 6 (linoleic acid) of roasted linseed flour are increased than raw linseed flour in the range of 23.97-25.58 % and 5.81-5.86 %,

respectively. The 3 and 6 omega fatty acids having higher market demand due to their meditational profile and are beneficial for infant brain development, reducing blood lipids and also useful in the prevention of cardiovascular diseases and may also have anticancer properties.

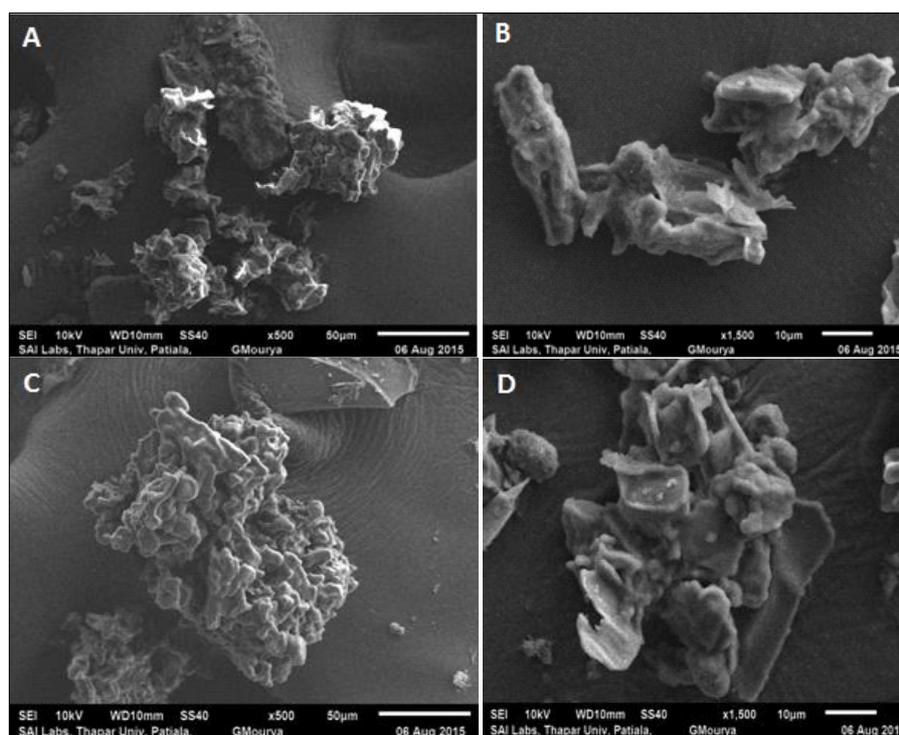


**Fig 1:** Fatty acid profile of raw and roasted linseed flour

### 3.4 Scanning electron microscopy (SEM)

Structural differences between raw and roasted linseed flours were evident as shown in Fig.2. The morphological analysis showed irregular shaped particles of raw linseed flour (Fig. 2, A-B) whereas roasting resulted in formation of a cluster like

structure. Microscopic observations (Fig. 2, C-D) of the linseed flour reveal the established organization of linseed flour in form of clusters, which may be due to rupturing of cell after roasting releasing the oil content which bind powder and form cluster.

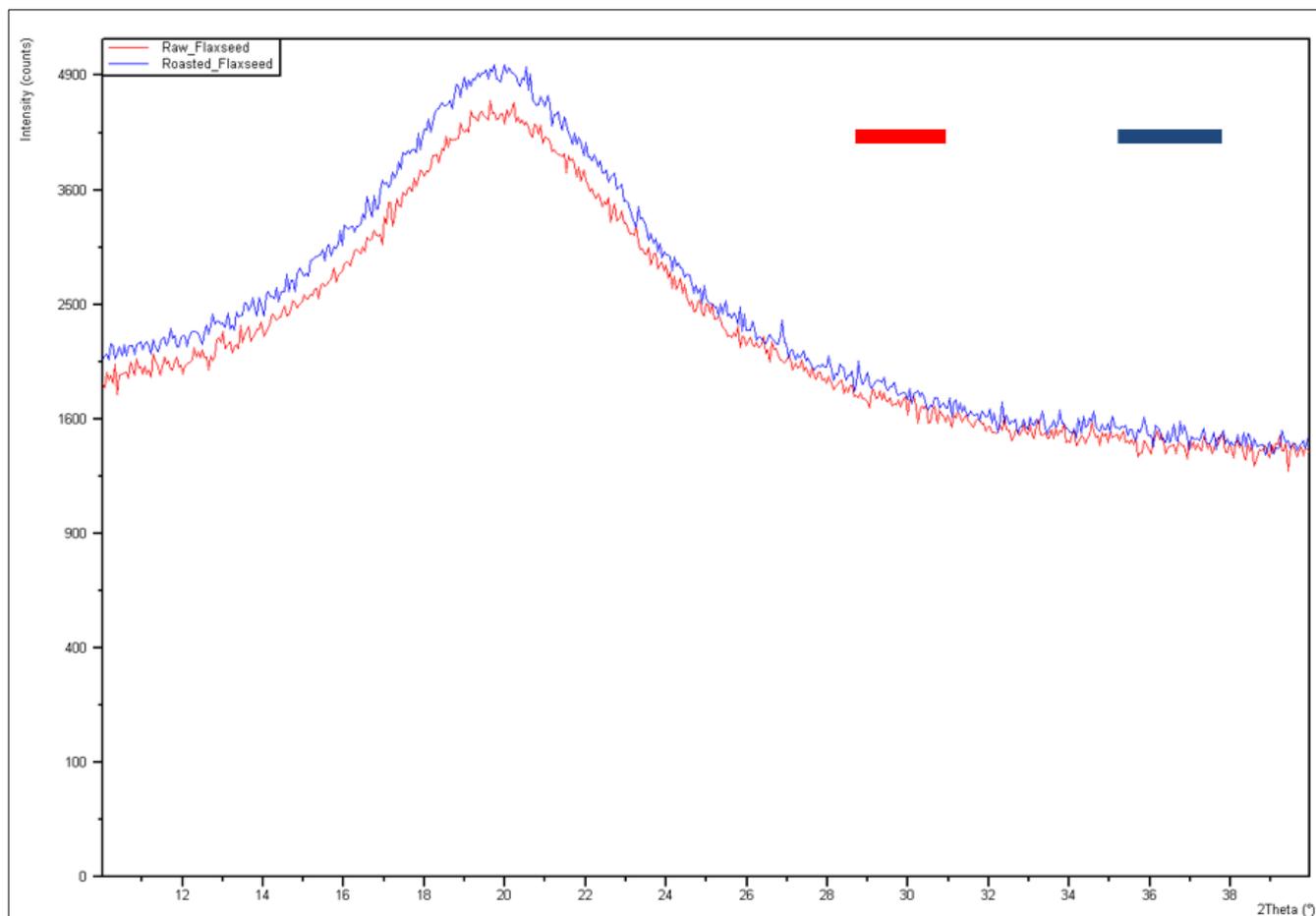


**Fig 2:** SEM micrographs of Linseed Flour: (A, B) top two images representing raw linseed flour at magnification level of 500 $\times$  and 1,500  $\times$  while the last two micrographs (C, D) are representing roasted linseed flour at 500 $\times$  and 1,500 $\times$ .

### 3.5 X-ray diffraction analysis

Diffuse and large peaks in an XRD profile indicates amorphous materials where as sharp and defined peaks are set for crystalline one <sup>[17]</sup>. Fig. 3 shows the XRD profile of raw

and roasted linseed flour. Graph clearly shows the amorphous nature of powder with a distinctive broad peak. The percentage of crystallinity of raw linseed flour was found to be 14.43% that changed to 15.92% after roasting treatment.



**Fig 3:** X-ray diffraction (XRD) pattern of Raw and Roasted linseed flour

### 4. Conclusions

The physico-chemical and functional properties along with fatty acid profile were significantly ( $p < 0.05$ ) influenced by the roasting treatment. An increase in the fatty acid profile of roasted linseed flour was noticed as compared to unroasted raw linseed flour. Roasted linseed flour consists of more protein, fat, ash and energy value whereas, having less moisture, carbohydrate and fibre content than raw linseed flour. Water absorption capacity (WAC) of the raw linseed flour (1.99 g/g) was found more as compare to roasted linseed flour (1.79 g/g) whereas, after roasting, water solubility index (WSI) and oil absorption capacity (OAC) of roasted linseed flour was found 11.51% and 1.99 g/g, higher than raw linseed flour i.e. 8.95 % and 1.66 g/g, respectively. The morphological analysis showed irregular shaped particles in raw linseed flour whereas roasting resulted in the formation of cluster like structures in roasted linseed flour sample. XRD profile revealed diffused and large peaks of raw and roasted linseed flour indicating the presence of amorphous materials within its matrix. Roasted linseed flour in comparison to raw linseed flour having more value and useful in functional food applications.

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