

## THE EFFECT OF ROASTING PROCESS ON FATTY ACID COMPOSITION AND PHENOLIC CONTENT OF WHOLE FLAXSEED, FLAXSEED FLOUR AND FLAXSEED MEAL FLOUR

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### Abstract

In this study, the change in fatty acid composition, secoisolariciresinol diglucoside (SDG) lignan, phenolic and flavonoid contents of whole flaxseed, flaxseed flour and flaxseed meal flour were investigated during the roasting process at 180°C for 5, 10 and 15 min. The level of  $\alpha$ -linolenic acid in flaxseed flour and flaxseed meal flour significantly ( $P < 0.05$ ) decreased 1.1 times (from 58.10 to 55.48%) and 1.4 times (from 56.13 to 40.17%) after the roasting process at 180°C for 15 min, respectively. Whole flaxseed had the highest SDG lignan content after the roasting process at 180°C for 5, 10 and 15 min. Besides, free flavonoid and free phenolic content of whole flaxseed significantly ( $P < 0.05$ ) increased 4.5 and 2.4 times after the roasting process at 180°C for 5 min, respectively.

**Keywords:** Flaxseed, roasting,  $\alpha$ -linolenic acid, SDG lignan, phenolics, flavonoids.

## TANE KETEN TOHUMU, KETEN TOHUMU UNU VE KETEN TOHUMU KÜSPESİ UNUNUNUN YAĞ ASİDİ KOMPOZİSYONU VE FENOLİK BİLEŞİK İÇERİĞİNE KAVURMA İŞLEMİNİN ETKİSİ

### Öz

Bu çalışmada, tane keten tohumu, keten tohumu unu ve keten tohumu küspesi ununun yağ asidi kompozisyonu ile SDG lignan, fenolik ve flavonoid içeriğininin 180°C sıcaklıkta 5, 10 ve 15 dak kavurma işlemleri sırasındaki değişimi araştırılmıştır. Keten tohumu ununun ve keten tohumu küspesi ununun  $\alpha$ -linolenik asit düzeyleri 180°C'de 15 dak kavurma işleminden sonra istatistiksel ( $P < 0.05$ ) açıdan önemli düzeyde sırası ile 1.1 kat (%58.10'dan %55.48'e) ve 1.4 kat (%56.13'den %40.17'ye) azalmıştır. Tane keten tohumu 180°C'de 5, 10 ve 15 dak kavurma işlemlerinden sonra en yüksek SDG lignan içeriğine sahiptir. Ayrıca, tane keten tohumunun serbest flavonoid ve serbest fenolik içeriği 180°C'de 5 dak kavurma işleminden sonra istatistiksel ( $P < 0.05$ ) açıdan önemli düzeyde sırası ile 4.5 and 2.4 kat artmıştır.

**Anahtar kelimeler:** Keten tohumu, kavurma,  $\alpha$ -linolenik asit, SDG lignan, fenolikler, flavonoidler.

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## INTRODUCTION

Flaxseed (*Linum usitatissimum* L.) has health promoting effects because of its valuable nutrients. Especially phytoestrogenic (1), anticarcinogenic (2), antioxidative (1) and cardioprotective (3) effects of flaxseed were attributed to secoisolariciresinol diglucoside (SDG) lignan, phenolic acids, flavonoids and  $\alpha$ -linolenic acid. The main lignan, SDG is varied between 5.87 and 23.84 mg/g in defatted flaxseed flour (4). The percent of  $\alpha$ -linolenic acid in flaxseed is 5.5 times higher than that in the next-highest sources, walnuts and canola oil (5) and vary between 47.00-60.42% of total fatty acids (6). Flaxseed is also a rich source of phenolic acids and flavonoids (4).

Flaxseed is increasingly used as functional ingredient in food products because of these valuable bioactive compounds. Some uses of flaxseed include ready-to-eat breakfast cereals, cakes, biscuits, crackers, bagels, energy bars, salad dressings, breakfast drinks and soups. Besides, flaxseed flour is used commercially in breads and cookies. The hulls and meals were found to be higher in SDG lignan, thus these by products were also used as a functional food ingredient in breakfast cereals and bakery products (7). Especially dry heating process such as roasting, baking and extrusion have important effects on lipid quality and phenolic content of oilseeds, cereals and legumes. In the literature, there are heat treatment studies such as phenolics in roasted sesame seeds (8), fatty acids and phenolics in roasted peanut (9, 10), fatty acids in roasted sunflower seeds (11), proanthocyanidin and total phenolics in roasted cocoa beans (12), isoflavones in roasted and puffed soybean (13).

The fatty acid composition of flaxseed oil is known to consist of high levels of  $\alpha$ -linolenic acid followed by linoleic and oleic acid. The use of flaxseed oil for edible purposes, particularly as cooking oil, has been limited because of its instability, but it can be used as salad oil (14). On the other hand, nowadays, various flaxseed products are used as a functional ingredient in functional foods and generally, heat treatment is used during production of these foods as bakery products. In the literature, there are two studies about the influence of toasting on nutritious of flaxseed (15, 16). However, no studies about the

effect of roasting process on fatty acids and phenolic compounds of flaxseeds and their products have been reported. Therefore, in this study, fatty acid composition, trans fatty acids, SDG lignan, phenolic acids and flavonoids in flaxseed products (whole flaxseed, flaxseed flour and flaxseed meal flour) were investigated during roasting process in a conventional oven at 180°C for different heating times (0, 5, 10 and 15 min).

## MATERIALS AND METHODS

### Samples and roasting process

An oil-type brown flaxseed (TR 77705) cultivar (*Linum usitatissimum* L.) was used in this study and it was supplied from National Gene Bank of Aegean Agricultural Research Institute in İzmir, Turkey. These flaxseeds were stored at 4°C after cleaning. In this study, three flaxseed products as whole flaxseed, flaxseed flour and flaxseed meal flour were used. Flaxseed flour was obtained by grinding (250  $\mu\text{m}$ <) of whole flaxseed using a coffee grinder (Bosch, KM 13). Flaxseed meal was prepared using a screw press. This operation was done by feeding a screw press (6 inch) at 80°C. Flaxseed meal was also ground (250  $\mu\text{m}$ <) with a coffee grinder to obtain flaxseed meal flour just before the extraction.

Roasting process of whole flaxseed, flaxseed flour and flaxseed meal flour were occurred in a conventional oven (Teba High-01, Inox) at 180°C for 5, 10, and 15 min. In this study, 500 gram batch was used for each roasting process and three replication was applied for each roasting time. For all flaxseed products, each replication was extracted separately and then each of the extracted oils and defatted flaxseed samples were analyzed independently. Each flaxseed product was roasted using two aluminum pan at the same time in the oven for each replication and the batch was not stirred during the roasting process to prevent the temperature changes in the oven and also the thickness of the batch was very low.

### Reagents and standards

The chemicals and reagents used in the study were n-hexane, sodium carbonate, hydrochloric acid, ammonium acetate, acetonitrile (Merck);

potassium hydroxide (Supelco); sodium hydroxide, sulphuric acid (J. T. Baker), methanol, Folin-ciocalteu phenol reagent, luteolin, standard mixture fatty acid methyl esters (Sigma-Aldrich); ferulic acid, 2-aminoethyl dipheylborinate (Fluka) and SDG (secoisolariciresinol diglucoside) lignan standard (Bosco, Hong Kong). All the chemicals and solvents were analytical or HPLC grade.

#### Determination of fatty acid composition

Each flaxseed sample was defatted with n-hexane using magnetic stirrer at 20°C for 1 h and the extract was filtered through rough filter paper. This process was repeated two times and n-hexane was removed with a rotary evaporator. Fatty acid composition of the flaxseed oil samples were determined using gas chromatography of fatty acid methyl esters (FAME). FAME were prepared according to the method of AOAC (17). FAME was quantified on an Agilent 5890N gas chromatograph, (Agilent Technologies Inc., Wilmington, DE, USA) and a flame ionization detector. Separation was carried out on a DB23 capillary column (30m\*250 µm, J. W. Scientific) with a film thickness of 0.25 mm. The FAME in n-hexane (2 µL) was injected into the column with a split ratio of 100:1. The injector and detector temperature were set at 250°C. The column temperature was programmed from 30 to 150°C at 20.0°C/min and then to 235°C at 6.0°C/min and was held at 230°C for 20 min (18). Identification of fatty acids was carried out using a reference standard mixture fatty acid methyl esters (Sigma-Aldrich), which is a ready to use standard.

#### Determination of SDG lignan content

The procedure of Özkaynak Kanmaz and Ova (19) was followed. 1 g of flaxseed samples were added into 25 mL flasks and then mixed with 4 mL of methanol, 1 mL of distilled water, and 5 mL of 2 mol/L NaOH. The flasks were sealed and shaken at room temperature on an orbital shaker for 1 h. Each hydrolysate was subsequently acidified with the addition of 5 mL of 1 mol/L H<sub>2</sub>SO<sub>4</sub>. The mixtures were then centrifuged at 11,000xg for 10 min and the supernatants were collected. 0.4 mL liquid phase from the supernatant was added to each of two microcentrifuge tubes, and then mixed with 0.6 mL of 100% methanol. The solution

was allowed to sit for 30 min at room temperature before re-centrifuging for 5 min at 11,000xg in order to precipitate and remove water-soluble polysaccharides and proteins. The supernatant was then filtered through a 0.45 µm filter and analysed by HPLC. Analysis of SDG lignan was performed using a HPLC-MS/MS system (API 4000) equipped with a Waters Model 600 pump, a 717 plus autosampler, an Agilent 1100 degasser, and a 996 photodiode array detector. The chromatographic separation of SDG lignan was carried out using a Zorbax Eclipse XDB-C18 extend with a guard column, 150 mmx2.1mmx5µm column (Agilent). The column was thermostated at 40°C. The injection volume was 5µL. The mobile phase consisted of 0.05 mmolL<sup>-1</sup> ammonium acetate in water (solvent A), and 0.05 mmolL<sup>-1</sup> ammonium acetate in acetonitrile (solvent B). The solvent flow was 0.2 mL/min, and a linear gradient elution was followed with 2% B for 4.50 min, and 90% B from 4.50 to 8.50 min, and 2% B 8.50 to 13.30 min. SDG lignan was identified and quantified by comparison to SDG lignan standard.

(r value: 0.9989, accuracy: 3.25%, recovery: 88-107%, level of detection (LOD): 0.0016 ppm, level of quantification (LOQ): 0.005 ppm).

#### Determination of free and esterified phenolics

Free phenolics from 1 g of flaxseed samples were extracted with 45 mL of 80% aqueous methanol in a shaker bath set at 40 °C for 90 min and filtered. Then, the flasks was allowed to cool to room temperature and diluted to a 50 mL volume with distilled water. The procedure of Özkaynak Kanmaz (20) was followed for the extraction of esterified phenolics and spectrophotometric determination of phenolics. Esterified phenolics from 1 g of flaxseed sample were extracted with 30 mL of 1.2 M HCl in 80% aqueous methanol (v/v) in a shaker bath set at 80°C for 1 h. The extract was cooled, filtered and diluted to a 50 mL volume with distilled water. Total phenolic acid contents of these samples were determined by spectrophotometrically using Folin-Ciocalteu phenol reagent. One millilitre of solutions were mixed with 0.5 mL of Folin-ciocalteu phenol reagent. After 3 min, 15 mL of Na<sub>2</sub>CO<sub>3</sub> (7 g/100 mL) solution was added. The solutions were mixed and diluted to 25 mL with distilled water. The extinction was measured

after 1 h at 725 nm against a reagent blank. Ferrulic acid served as a standard for preparing the calibration curve at five point.

#### **Determination of free and esterified flavonoids**

The procedure of Özkaynak Kanmaz (20) was followed for the spectrophotometric determination of free and esterified flavonoids. About 1 mL of the free and esterified phenolic extracts were mixed with 100  $\mu$ L of 1% 2-aminoethyl-diphenylborate solution, and then these solutions were diluted to a 10 mL volume with 80% methanol. Luteolin served as a standard for preparing the calibration curve at five point with assay solution in 80% methanol.

#### **Statistical analysis.**

Analyses were performed in triplicate. Results were expressed as means  $\pm$  standard deviation. One-way analysis of variance, least significant difference (LSD) and univariate analysis of variance was applied using SPSS statistical package. The statistical significance was evaluated at  $P < 0.05$  level.

## **RESULTS AND DISCUSSION**

### **Fatty acid composition of roasted flaxseed products**

The fatty acid composition of whole flaxseed had minor changes whereas, a significant ( $P < 0.05$ ) change was determined in the fatty acid composition of flaxseed meal flour during the roasting process at 180°C in this study (Table 1). Manthey et al. (21) also reported that lipid stability is a major concern when using milled flaxseed as a food ingredient. Besides, Oomah and Sitter (22) reported that flaxseed hull oils consisted minor amounts of free fatty acids between 1.4 and 2.6%.

In this study,  $\alpha$ -linolenic acid of roasted whole flaxseed was found to be more stable than roasted flaxseed flour at 180°C for 5, 10 and also 15 min. The level of  $\alpha$ -linolenic acid in flaxseed flour and flaxseed meal flour significantly ( $P < 0.05$ ) decreased 1.1 times (from 58.10 to 55.48%) and 1.4 times (from 56.13 to 40.17%) during the roasting process at 180°C for 15 min (Table 1). Both Manthey et al. (21) and also Schorno et al. (23) reported that

$\alpha$ -linolenic acid is mostly affected by oxidation because autoxidation reaction rate increases with the number of double bonds present in a fatty acid. However, Epaminondas et al. (15) reported that the level of  $\alpha$ -linolenic acid in whole flaxseed did not vary with toasting process at 160°C for 15 min. On the other hand, the percentage of oleic acid in flaxseed meal flour significantly ( $P < 0.05$ ) increased 1.6 times (from 17.08 to 27.61%) during the roasting process at 180°C for 15 min whereas, the level of oleic acid in whole flaxseed was not affected with roasting process in this study. Also, the ratio of n-6/n-3 in flaxseed meal flour increased 1.3 times (from 0.28 to 0.37) during the roasting process at 180°C for 15 min (Table 1).

The level of saturated fatty acids (SFA) in flaxseed flour and flaxseed meal flour significantly ( $P < 0.05$ ) increased 1.2 times (from 8.28 to 10.07%) and 1.4 times (from 9.45 to 13.52%) during the roasting process at 180°C for 15 min, respectively. However, a significant ( $P < 0.05$ ) decrease was determined as 1.3 times (from 71.86 to 55.05%) for polyunsaturated fatty acids (PUFA) content of flaxseed meal flour with 15 min of roasting at 180°C (Table 1). On the other hand, after the roasting process at 180°C for 5 min, PUFA fraction in unroasted flaxseed flour decreased from 72.96 to 70.72%. Bozan and Temelli (24) also reported that the oxidation process mainly involved the degradation of PUFA and the generation of free radicals. It was also reported that the toasting process exposed the lipids and other constituents to oxidative processes, especially for golden flaxseeds and also the formation of oxidation products lead to a higher thermal stability of the toasted seeds when submitted to new thermal processes (16). Besides, Yoshida et al. (9) reported that roasting time had a significant effect on fatty acids of peanut oil and significant differences in fatty acid distribution of peanut seeds were obtained for 20 and 30 min of roasting process. On the other hand, different results were obtained about the change of PUFA/SFA ratio in flaxseed products during the roasting process in this study. PUFA/SFA ratio in flaxseed meal flour decreased 1.9 times (from 7.60 to 4.07) after the roasting process at 180°C for 15 min whereas, for 5 min, 1.2 times (from 7.60 to 8.90) increase was found in

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Table 1. Fatty acids and ratios of nutritional interest obtained during roasting at 180°C for the three products of flaxseed.

Whole flaxseed				
Roasting time (min)				
Fatty acids (%)	0	5	10	15
Palmitic	4.67±0.07 <sup>ay</sup>	5.25±0.18 <sup>by</sup>	5.32±0.23 <sup>by</sup>	4.61±0.08 <sup>az</sup>
Stearic	3.61±0.09 <sup>ax</sup>	3.48±0.12 <sup>cy</sup>	3.52±0.34 <sup>ay</sup>	3.55±0.26 <sup>ay</sup>
Oleic	17.60±0.32 <sup>ax</sup>	17.55±0.29 <sup>az</sup>	17.82±0.53 <sup>ay</sup>	17.54±0.45 <sup>az</sup>
Linoleic	14.86±0.20 <sup>ay</sup>	14.91±0.09 <sup>ax</sup>	14.97±0.23 <sup>ay</sup>	14.89±0.19 <sup>ay</sup>
α-Linolenic	58.28±1.47 <sup>ax</sup>	58.10±1.01 <sup>ax</sup>	57.75±1.19 <sup>bx</sup>	57.46±0.87 <sup>bx</sup>
Σ TRANS	0.25	0.36	0.36	0.25
Oleic	0.02	0.01	0.01	0.02
Linoleic	0.06	0.05	0.05	0.05
α-Linolenic	0.17	0.30	0.30	0.18
SFA	8.28	8.73	8.84	8.16
PUFA	73.14	73.01	72.72	72.35
P/S	8.30	8.36	8.23	8.87
n-6/n-3	0.26	0.26	0.26	0.26
Flaxseed flour				
Roasting time (min)				
Fatty acids (%)	0	5	10	15
Palmitic	4.67±0.07 <sup>dy</sup>	7.02±0.16 <sup>bx</sup>	6.72±0.05 <sup>cx</sup>	7.31±0.09 <sup>ax</sup>
Stearic	3.61±0.09 <sup>ax</sup>	2.85±0.08 <sup>bz</sup>	2.78±0.11 <sup>bz</sup>	2.76±0.27 <sup>cz</sup>
Oleic	17.60±0.82 <sup>bx</sup>	18.29±0.54 <sup>ax</sup>	17.84±0.67 <sup>by</sup>	17.88±0.28 <sup>by</sup>
Linoleic	14.86±0.20 <sup>ay</sup>	14.81±0.07 <sup>ay</sup>	15.00±0.21 <sup>ay</sup>	14.98±0.28 <sup>ax</sup>
α-Linolenic	58.10±1.47 <sup>ax</sup>	55.91±0.91 <sup>bz</sup>	55.59±1.39 <sup>bx</sup>	55.48±1.13 <sup>by</sup>
Σ TRANS	0.25	0.20	0.28	0.50
Oleic	0.02	0.00	0.02	0.03
Linoleic	0.06	0.07	0.06	0.09
α-Linolenic	0.17	0.13	0.20	0.38
SFA	8.28	9.87	9.50	10.07
PUFA	72.96	70.72	70.59	70.46
P/S	8.81	7.17	7.43	7.00
n-6/n-3	0.26	0.27	0.27	0.27
Flaxseed meal flour				
Roasting time (min)				
Fatty acids (%)	0	5	10	15
Palmitic	6.70±0.15 <sup>bx</sup>	4.61±0.08 <sup>cz</sup>	4.60±0.19 <sup>cz</sup>	7.05±0.15 <sup>ay</sup>
Stearic	2.75±0.11 <sup>cy</sup>	3.60±0.21 <sup>bx</sup>	3.97±0.07 <sup>bx</sup>	6.47±0.12 <sup>ax</sup>
Oleic	17.08±0.45 <sup>cy</sup>	17.68±0.39 <sup>cy</sup>	18.93±0.25 <sup>bx</sup>	27.61±0.43 <sup>ax</sup>
Linoleic	15.73±0.14 <sup>ax</sup>	14.91±0.17 <sup>bx</sup>	15.21±0.49 <sup>ax</sup>	14.88±0.28 <sup>by</sup>
α-Linolenic	56.13±1.22 <sup>ay</sup>	56.08±1.21 <sup>ay</sup>	56.05±0.87 <sup>ax</sup>	40.17±1.09 <sup>bz</sup>
Σ TRANS	0.43	0.20	0.25	0.36
Oleic	0.01	0.01	0.00	0.08
Linoleic	0.12	0.06	0.06	0.10
α-Linolenic	0.30	0.13	0.19	0.18
SFA	9.45	8.21	8.57	13.52
PUFA	71.86	72.99	71.26	55.05
P/S	7.60	8.90	8.32	4.07
n-6/n-3	0.28	0.27	0.27	0.37

Values are means±standard deviations of three (n=3) measurements

<sup>abcd</sup> Values with different superscript letters within a row for each flaxseed product are significantly different at  $P<0.05$

<sup>xyz</sup> Values with different superscript letters within a row for each time between flaxseed product are significantly different at  $P<0.05$

SFA: saturated fatty acids, PUFA: polyunsaturated fatty acids, P/S: Polyunsaturated fatty acids/saturated fatty acids, n-6: linoleic acid, n-3: α-linolenic acid

PUFA/SFA ratio for flaxseed meal flour with roasting. However, for flaxseed flour, PUFA/SFA ratio decreased 1.2 times (from 8.81 to 7.17) after the roasting process at 180°C for 5 min (Table 1).

In this study, trans fatty acids (trans oleic, trans linoleic, trans  $\alpha$ -linolenic) formation in unroasted and roasted flaxseed products were also analyzed. Trans  $\alpha$ -linolenic acids were significantly ( $P<0.05$ ) higher than trans oleic and trans linoleic acids. The highest level of total trans fatty acids in unroasted flaxseed meal flour and roasted flaxseed flour were observed as 0.43 and 0.50% after the roasting process at 180°C for 15 min, respectively (Table 1).

### Phenolic content of roasted flaxseed products

Naturally occurring phytochemicals such as polyphenols play an important role in the protection of nuts and oilseeds against fat deterioration (10). Essential fatty acids in flaxseed oil are highly susceptible to oxidation and therefore it has a very short shelf life. However, in flaxseed, lipids are protected against oxidation by various mechanisms, for example, the presence of antioxidants such as lignans (18). In most cereal grains, the majority of phenolic compounds are bound to cell wall components in the hull (25). Also, flaxseed hulls were used as a source of lignans because of their highest lignan content (26).

In this study, the results showed that the roasting process had a significant ( $P<0.05$ ) effect on SDG lignan, phenolics and flavonoids of flaxseed products. Besides, roasting process did not improve the amount of SDG lignan in flaxseed products compared to phenolics and flavonoids and all unroasted flaxseed products had the highest SDG lignan content. SDG lignan in roasted whole flaxseed was found to be more stable than

flaxseed flour and also, whole flaxseed had the highest content of SDG lignan after the roasting process at 180°C for 5, 10 and 15 min (Table 2). SDG lignan amount in whole flaxseed and flaxseed flour significantly ( $P<0.05$ ) decreased 1.3 times (from 23.84 to 19.13 mg/g defatted sample in DW) and 1.7 times (from 23.84 to 14.21 mg/g defatted sample in DW) after the roasting process at 180°C for 15 min, respectively whereas, flaxseed meal flour did not exhibit a significant ( $P<0.05$ ) decrease with roasting process. In the literature, Shahidi et al. (27) also reported that the effect of heating process on the sesamin content of coated seeds was low and the decrease generally did not exceed 20% of the original values. Lee and Lee (13) also found that total isoflavones of soybeans decreased 25.46% after 21 min of roasting at 200°C.

Also, SDG lignan amount in whole flaxseed and flaxseed flour significantly ( $P<0.05$ ) decreased 1.2 times (from 23.84 to 20.21 mg/g defatted sample in DW) and 1.3 times (from 23.84 to 18.04 mg/g defatted sample in DW) after the roasting process at 180°C for 5 min, respectively whereas, SDG lignan content of flaxseed meal flour did not change significantly ( $P<0.05$ ) in this study. However, SDG lignan content of whole flaxseed exhibited a significant ( $P<0.05$ ) decrease as 1.3 times (from 23.84 to 17.83 mg/g defatted sample in DW) after pressing process by feeding a screw press at 80°C (Table 2). SDG lignan is diglycosylated form of secoisolaricresinol (SECO) and SDG are linked by 3-hydroxy-3-methyl-glutaryl units (HMG), derived from 3-hydroxy-3-methyl-glutaric acid (HMGA), to form an ester-linked biopolymer. An alkaline treatment can break the SDG-HMG link and releases free SDG and also, to obtain the aglycone form of the SECO, SDG must undergo an acidic or an enzymatic treatment in order to break the glycosidic link (28). In this study, the change in SDG lignan of

Table 2. SDG lignan content obtained during roasting at 180°C for the three products of flaxseed.

Roasting time (min)	SDG lignan (mg/g defatted sample in DW)			
	0	5	10	15
Whole flaxseed	23.84±0.21 <sup>ax</sup>	20.21±0.18 <sup>bx</sup>	20.13±0.73 <sup>bx</sup>	19.13±0.21 <sup>cx</sup>
Flaxseed flour	23.84±0.21 <sup>ax</sup>	18.04±0.87 <sup>by</sup>	16.31±2.85 <sup>cy</sup>	14.21±0.30 <sup>dz</sup>
Flaxseed meal flour	17.83±0.13 <sup>ay</sup>	17.76±0.35 <sup>ay</sup>	17.65±0.19 <sup>by</sup>	17.60±0.11 <sup>by</sup>

Values are means±standard deviations of three (n=3) measurements

<sup>abcd</sup> Values with different superscript letters within a row are significantly different at  $P<0.05$

<sup>xyz</sup> Values with different superscript letters within a column are significantly different at  $P<0.05$

flaxseed with pressing process could be explained with the high effect of the pressure on the SDG-HMG ester link and glycosidic link of SDG lignan during screw pressing process of flaxseed. In the literature, it was also reported that glycosides were unstable at higher temperatures and longer extraction times under the high pressure during subcritical water extraction (29).

SDG lignan and other phenolic compounds in flaxseed are present in bound forms with both glucosidic and ester bonds. Phenolic compounds at high concentrations in flaxseed were hydroxycinnamic acid glucosides as ferulic acid glucoside and *p*-coumaric acid glucoside. Flaxseed also contain *p*-coumaric, *o*-coumaric, ferulic, *p*-hydroxybenzoic, gentisic, vanillic, and sinapic acids in free and/or bound forms. (30, 31). After pressing process by feeding a screw press at 80°C, free and esterified phenolic content of whole flaxseed significantly ( $P<0.05$ ) increased 3.2 times (from 249.33 to 784.78 mg ferulic acid/100 g defatted sample in DW) and 1.6 times (from 959.87 to 1522.69 mg ferulic acid/100 g defatted sample in DW), respectively (Table 3). Also, free and esterified flavonoid content of whole flaxseed significantly ( $P<0.05$ ) increased 5.0 times (from 16.41 to 82.74 mg luteolin/100 g defatted sample in DW) and 2.7 times (from 11.58 to 31.47 mg luteolin/100 g defatted sample in DW) after pressing process by feeding a screw press at 80°C, respectively (Table 4). These results could be explained with degradation of SDG lignan because of screw pressing and also heating. In the literature, it was suggested that there was a stronger binding between

hydroxycinnamic acid glucosides and HMG complex than SDG-HMG (32). These results could be also explained with the high effect of the pressure on the hydroxycinnamic acid glucosides and HMG complex during screw pressing process of flaxseed. Beejmohun et al. (32) reported that flaxseed had high amount of *p*-coumaric acid glucoside (3.7 mg/g) and ferulic acid glucoside (4.1 mg/g). In the literature, glycosides were reported to become unstable at higher temperatures (29). Also, after screw pressing process, the high increase in phenolics could be explained with breaking of the interactions between phenolic compounds and cell wall polysaccharides as cellulose, hemicellulose and pectin because natural bioactive compounds such as phenolics- especially flavonoids, are present in different forms, interacting with the cell wall polysaccharides (33). Also, it could be suggested that the releasing of free phenolics and free flavonoids from the cell wall with screw pressing process was easier than esterified phenolics and esterified flavonoids.

Besides, free phenolics in whole flaxseed and flaxseed flour exhibited a significant ( $P<0.05$ ) increase as 2.4 and 1.6 times after the roasting process at 180°C for 5 min, respectively and also, esterified phenolics of whole flaxseed and flaxseed flour significantly ( $P<0.05$ ) increased 1.8 and 1.7 times under these roasting process conditions, respectively (Table 3). Also, free flavonoids of whole flaxseed and flaxseed flour significantly ( $P<0.05$ ) increased 4.5 and 6.0 times after the roasting process at 180°C for 5 min, respectively (Table 4). The increase in free and

Table 3. The change in free and esterified phenolics of roasted flaxseed products at 180°C.

Roasting time (min)	Free phenolics (mg ferulic acid/100 g defatted sample in DW)			
	0	5	10	15
Whole flaxseed	249.33±11.10 <sup>ax</sup>	597.10±29.92 <sup>ax</sup>	575.74±35.75 <sup>ax</sup>	555.08±28.81 <sup>by</sup>
Flaxseed flour	249.33±11.10 <sup>ax</sup>	408.30±22.33 <sup>bx</sup>	417.04±25.90 <sup>bx</sup>	841.66±60.43 <sup>ax</sup>
Flaxseed meal flour	784.78±39.32 <sup>by</sup>	441.07±27.44 <sup>by</sup>	426.89±36.38 <sup>by</sup>	431.25±24.84 <sup>bx</sup>
Roasting time (min)	Esterified phenolics (mg ferulic acid/100 g defatted sample in DW)			
	0	5	10	15
Whole flaxseed	959.87±38.20 <sup>by</sup>	1677.68±99.65 <sup>ax</sup>	1677.68±79.65 <sup>ay</sup>	1665.06±100.07 <sup>ax</sup>
Flaxseed flour	959.87±38.20 <sup>by</sup>	1667.24±87.03 <sup>ax</sup>	1670.14±82.84 <sup>az</sup>	1446.12±76.79 <sup>bx</sup>
Flaxseed meal flour	1522.69±77.51 <sup>cx</sup>	1661.66±79.93 <sup>ay</sup>	1688.60±102.16 <sup>ax</sup>	1598.54±57.23 <sup>by</sup>

Values are means±standard deviations of three (n=3) measurements

<sup>abc</sup> Values with different superscript letters within a row are significantly different at  $P<0.05$

<sup>xyz</sup> Values with different superscript letters within a column are significantly different at  $P<0.05$

Table 4. The change in free and esterified flavonoids of flaxseed products during roasting at 180°C.

Roasting time (min)	Free flavonoids (mg luteolin/100 g defatted sample in DW)			
	0	5	10	15
Whole flaxseed	16.41±0.86 <sup>cy</sup>	73.16±3.91 <sup>ay</sup>	52.28±2.20 <sup>bz</sup>	50.54±2.62 <sup>bz</sup>
Flaxseed flour	16.41±0.86 <sup>cy</sup>	98.29±7.31 <sup>ax</sup>	56.84±2.39 <sup>by</sup>	103.08±6.38 <sup>ax</sup>
Flaxseed meal flour	82.74±4.25 <sup>ax</sup>	70.40±4.23 <sup>by</sup>	59.44±3.14 <sup>cx</sup>	72.61±3.55 <sup>by</sup>
Roasting time (min)	Esterified flavonoids (mg luteolin/100 g defatted sample in DW)			
	0	5	10	15
Whole flaxseed	11.58±0.66 <sup>cy</sup>	16.82±0.28 <sup>bz</sup>	19.12±0.95 <sup>az</sup>	17.80±0.45 <sup>bz</sup>
Flaxseed flour	11.58±0.66 <sup>cy</sup>	26.16±0.69 <sup>by</sup>	30.78±1.83 <sup>ay</sup>	28.64±1.79 <sup>ay</sup>
Flaxseed meal flour	31.47±1.38 <sup>cx</sup>	40.78±2.15 <sup>ax</sup>	37.00±1.10 <sup>bx</sup>	36.82±1.37 <sup>bx</sup>

Values are means±standard deviations of three (n=3) measurements

<sup>abc</sup> Values with different superscript letters within a row are significantly different at  $P<0.05$

<sup>xyz</sup> Values with different superscript letters within a column are significantly different at  $P<0.05$

esterified phenolics and flavonoids after the roasting process could be explained with breaking of binds in the structure of SDG lignan complex and also other polyphenolic compounds in flaxseed with heat treatment at high temperature as 180°C. Also, the increase in phenolics could be explained with breaking of the interactions between phenolic compounds and cell wall polysaccharides such as cellulose, hemicellulose and pectin because of the high temperature as 180°C during the roasting process. Jannat et al. (8) also reported that total phenolic content of sesame significantly increased during roasting process until 200°C and 20 min. However, De Brito et al. (12) found a 57% decrease of total phenolic content after toasting of cocoa beans at 150°C for 30 min. Cämmerer and Kroh (10) also reported that total polyphenol content of peanut only slightly decreased with heat treatment at 170°C.

## CONCLUSION

Consequently, the roasting process and roasting time had significant ( $P<0.05$ ) effects on the fatty acids, SDG lignan, phenolics and flavonoids of flaxseed products. It was shown that the roasting process did not improve the amount of SDG lignan in flaxseed products compared to phenolics and flavonoids so, all unroasted flaxseed products had the highest content of SDG lignan. Roasted whole flaxseed was the most nutritional flaxseed product because of the highest SDG

lignan and  $\alpha$ -linolenic acid level compared to roasted flaxseed flour and roasted flaxseed meal flour. So, whole flaxseed was suggested to be used as a functional ingredient in the functional foods especially as bakery products and other heat processed food products in the food industry.

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