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Characterization and quality assessment of mechanically and solvent extracted Niger (*Guizotia abyssinica*) Seed oil

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Abstract
In the present study an attempt was made to evaluate physico-chemical and nutritional quality of mechanically and solvent extracted niger seed oil. SE exhibited higher yield of niger seed oil (39.34%) as compared to ME (29.23%). There was a significant variation observed in iodine value, acid value, peroxide value saponification value, free fatty acids, color and viscosity whereas, refractive index and specific gravity did not differ between niger seed oils produced by both the methods. The fatty acid profile of SE and ME oils showed marginal variation in palmitic acid. Oleic and linoleic acid showed considerable variations for both the oils. Higher total tocopherol contents were observed in ME oil (309.64 mg kg-1) as compared with SE oil (74.69 mg kg-1), respectively. Overall, it could be concluded that ME niger seed oil has superior nutritional characteristics compared with SE niger seed oil which might be attributed to the mild extraction conditions employed during mechanical extraction.

Keywords: GC, HPLC, mechanical oil extraction, Niger seed oil, tocopherol

1. Introduction
Niger seed (*Guizotia abyssinica* (L. f.) Cass.) belongs to the *Compositae* family. Ethiopia and India are the two major niger seed producing countries in the world. Besides Ethiopia and India, the Food and Agriculture Organization reports that other production areas include Nepal, Myanmar, Bangladesh and several countries in eastern and central Africa. Niger seed is used as a condiment and minor oilseed crop in India amounting to a production of 102,000 tons of seeds and 20,000 tons of oil during 2012-13 [1].

Niger seed oil has linoleic acid (C18:2) as the principal fatty acid (65.7-68.5 %, weight percent of total lipid). Oleic acid (C18:1) was the second major unsaturated fatty acid (5.4-7.5 %). Niger contains two major saturated fatty acids [palmitic (9.6-10 %) and stearic (7.6-8.1 %)]. The above fatty acids represent 91-97% of the fatty acid present. Palmitoleic, linolenic, arachidic, eicosenoic, behenic, erucic and lignoceric acids constituted less than 1% each [2].

The oil content of niger seed ranges of 40–44 %. Niger seed oil, like sunflower and safflower oils, contains high content of omega-6 PUFA i.e. linoleic acid (63-75%) [3]. Dietary fats and oils, rich in linoleic acid, have been reported to prevent cardiovascular disorders such as coronary heart disease, atherosclerosis, as well as high blood pressure. Also linoleic acid derivatives serve as structural components of the plasma membrane and as precursors of some metabolic regulatory compounds [2].

The tocopherols (737-847 ppm) and sterols (4,000-4,200 ppm) compositions of Ethiopian and Indian niger seed oils and reported a-tocopherol (*95 % of total tocopherols) and b-sitosterol (*42 % of total sterols) as the major tocopherol and sterol [4, 5]. Tocopherols are essential for protection of polyunsaturated fatty acid (PUFA) in plants and animals against oxidative deterioration. They exert their antioxidant effect by numerous biochemical and biophysical mechanisms, including scavenging active oxygen species and free radicals, and through action as efficient chain terminators in lipid autoxidation reactions [6].

Two main processes mostly employed for oil extraction from seeds on industrial scale are mechanical and solvent extraction. Solvent method, involving the use of *n*-hexane or petroleum ether as extraction solvent, although gives higher oil yield but a higher temperature employed in this method may cause undesirable effects on the quality of oil [7]. In mechanical oil extraction, although with lower oil yield, is advantageous with regard to mild operational temperature conditions, process safety and product quality [8].

In a view of the huge demand and consciousness about the consumption of functional, nutritional and healthy oil, the present study was therefore framed with the main objective to evaluate and compare the physicochemical parameter and nutritional quality of quality of mechanically and solvent extracted niger seed oil.

2. Materials and Methods

2.1 Extraction of niger oil

The oil from the niger seeds was extracted through mechanical extraction. The solvent extraction was done by method as described in (AOAC, 2002) [9] using food grade hexane as solvent. The traces of hexane were removed using rotary flash evaporator. The extracted oil was stored in dark place at room temperature. The oil extracted was assessed for different physical and chemical parameters.

2.2 Physical and chemical Characteristics of oils

Colour (L*, a*, b* values) of the oil was determined by using Hunter Colour Flex Meter as described by Park *et al* [10]. Refractive index was determined as method described by Pearson [11]. The specific gravity was expressed in terms of ratio of density of oil to water. A Brookfield Viscometer Model DV-E was used to measure the viscosity of extracted oil [12]. Viscosity was determined to at constant speed of 100 rpm and at constant temperature with a spindle number S-62 and it was expressed in terms of (mPas). The chemical properties such as the iodine value (IV), acid value (AV), Peroxide value (PV), saponification value (SV), and free fatty acids (FFA) of the extracted oils were analyzed according to AOAC standard methods [13].

2.3 Fatty acid composition of niger seed oil

Fatty acid composition of the oil was determined using Gas chromatography of FAMEs (Fatty Acid Methyl Esters) with Flame Ionization Detector by AOCS Official Method Ce 1h-05 [14]. The oil (10–20 mg) was saponified for 1 hr with 1 ml of methanolic KOH (0.7 N) at 60 ^{0}C, followed by neutralisation with 1 ml of methanolic HCl (0.7 N). The resulting free fatty acids were extracted in hexane and evaporated to dryness. The fatty acids were methylated using boron trifluoride (14% in methanol) and 0.2 ml benzene. The FAME was extracted in hexane, washed with water and evaporated to dryness. Fatty acid analysis was performed using a gas–liquid chromatograph (Shimadzu, GC-14B, Shimadzu Corporation, Japan) fitted with a fused silica capillary column (BP 21: 30 m length, 0.30 mm i.d., 0.50 µm film thickness). The GC was equipped with a flame ionization detector, Clarity Lite 420 integrator and at isothermal conditions. The column temperature was set at 220 ^{0}C, the injector temperature at 230 ^{0}C and the detector temperature at 240 ^{0}C. Nitrogen gas was used as the carrier gas with a flow rate of 1 ml/min. Individual fatty acids in the oil were identified by comparison with the retention times of standard fatty acid methyl esters.

2.4 Total tocopherol

Niger seed oil (1.0 g) was saponified using 4 ml of 5% ethanolic pyrogallol (w/v) and 1 ml KOH (100%) and boiling in a water bath for 3 min. Samples were then cooled, 30 ml distilled water was added and the mixture was extracted three times with diethyl ether. The combined extracts were washed with water to neutralize and remove fatty acid soaps. The extract was dried with anhydrous sodium sulphate and evaporated to dryness under a vacuum at 40 ^{0}C. The residue was dissolved in 1.0 ml ethanol and 4.0 ml of benzene and dried under a stream of nitrogen. The quantification of tocopherol of Niger seed oil was performed by using HPLC method [15] using a Agilent 1100 Series HPLC system equipped with a fluorescence detector and Phenominix C18, column (250×4.60 mm, 5 µm particle size). The excitation wavelength used was 290 nm and the emission wavelength was 330 nm. An isocratic elution program was employed using a mobile phase containing methanol. The flow rate was 1.0 ml/min. Tocopherol peak was identified by comparison to the retention time of known reference standards.

3. Result and Discussion

3.1 Physical properties of extracted oil

The physical properties such as colour, refractive index, specific gravity were determined and represented in Table-1.

Table 1: Percent yield and physical properties of Niger seed oil extracted by different methods

Parameters	Extraction Method	
	ME*	SE**
Total Yield (%)	29.23	39.34
Colour L	53.61	54.48
a*	-4.05	-4.66
b*	26.57	21.41
Refractive Index (Abbes, 27 ◦C)	1.4679	1.4671
Specific gravity mg/ml	0.918	0.917
Viscosity (cp)	45.6	31.5

ME*- mechanical extracted SE**- solvent extracted
*Each value is a mean of three determinations.

The oil yield of solvent extraction was more as compared to mechanical extraction. The niger seed shows the oil yield of i.e. 29.23% and 39.34% by mechanical and solvent method respectively. Similar results have been reported earlier [16-18].
Most oils are yellow-red or amber liquids due to the presence of chlorophylls and carotenoids. Often lighter color has been associated with better quality oils, especially for salad oils and shortenings. Colour is also one of physical property which determines the adulteration of oil or fat [17]. The physical properties of niger oil has colour values such as L* value (53.61), (54.48), a* value (-4.05), (-4.66) and b* value (26.57), (21.41) for mechanical extraction and solvent extracted oil respectively. These values are in correlated with results earlier findings [17].
There was very negligible difference in case of refractive index and specific gravity of mechanical and solvent extracted oil. Refractive index is used mainly to measure the change in unsaturation as the fat or oil is hydrogenated. The refractive index of oils depends on their molecular weight, fatty acids chain length, degree of unsaturation and degree of conjugation [20].
It shows that the viscosity values for mechanical extracted oil 42.50 (mPas) is higher than solvent extracted oil 37.83 (mPas) at 25 ^{0}C, this might be due to the dissolved solids and impurities in mechanical extracted oil.

3.2 Chemical properties of extracted oil

The chemical properties such as acid value, iodine value, peroxide value, saponification value and free fatty acid were studied and represented in Table-2.

Table 2: Chemical properties of Niger oil extracted by different methods

Parameters	Extraction Method	
	ME*	SE**
Iodine value (g of I_2/100g of oil)	128.4	126.3
Acid value (mg KOH/g of oil)	6.20	8.27
Peroxide value ($meqO_2$/kg oil)	2.60	4.20
Saponification value (KOH/g of oil)	190.8	188.5
Free fatty acid (as oleic acid %) (g/100g of oil)	3.12	4.16

ME*- mechanical extracted SE**- solvent extracted
*Each value is a mean of three determinations.

Iodine value is a measure of unsaturated fatty acid content of fat. Higher iodine value indicates lower degree of saturation and vice versa (Dim, 2013). It is observed from Table-2 that the Iodine value (IV) of mechanically extracted niger oil is 128.4 g of I_2/100g of oil whereas IV for solvent extracted niger seed oil is 126.3 g of I_2/100g of oil. The (IV) of extracted oils agreed with previous reports [21]. The Acid value which is a measure of the free fatty acids in oil, was found to be 6.20 and 8.27 mg KOH/g of oil for mechanically extracted and solvent extracted niger oil respectively. The results of acid value are in accordance with (IS-3490) [22].

Peroxide value (PV) is a measure of oxidation during storage and the freshness of lipid matrix (Atinafu and Bedemo)[23]. The (PV) for mechanically extracted and solvent extracted niger seed oil was 2.60 and 4.20g of meqO_2/kg oil respectively. It reveals that the mechanical extraction is better for yielding good quality oil due to mild operational conditions [17]. The results of peroxide value are in accordance with (IS-3490) [22]. Saponification value is an indication of the molecular weights of triglycerides in oil [24]. The results showed that (SV) of 190.8 and 188.5 KOH/g of oil for mechanically extracted and solvent extracted niger seed oil. The SV of the oils agreed well with the literature [17].

The value for free fatty acids (FFA) was 3.12 and 4.16g/100g of oil for mechanically extracted and solvent extracted niger seed oil respectively. The FFA of the oils agreed well with earlier finding [17]. The FFA content was higher in solvent extracted oil because FFA is readily soluble in polar solvents [17].

3.3 Fatty acid profile and total tocopherol of extracted oil

The data pertaining to fatty acid and total tocopherol content of extracted oil are given in Table-3.

Table 3: Fatty acid profile of niger seed oil extracted by different method

Fatty Acids		Extraction Method	
		ME*	SE**
Saturated Fatty Acids		**Percent Concentration**	
Lauric acid	(C12:0)	ND	0.09
Myristic acid	(C14:0)	0.04	0.10
Palmitic acid	(C16:0)	7.61	7.25
Heptadeconoic acid	(C17:0)	0.05	ND
Stearic acid	(C18:0)	6.52	5.61
Arachidic acid	(C20:0)	0.56	ND
Behenic acid	(C22:0)	0.95	0.80
Lignoceric acid	(C24:0)	0.71	0.59
Unsaturated Fatty Acids			
Monounsaturated fatty acids			
Palmitoleic acid	(C16:1)	0.12	0.11
Oleic acid	(C18:1n9)	34.69	30.59
Eicosenoic acid	(C20:1)	0.10	ND
Polyunsaturated fatty acids			
Linoleic acid	(C18:2n6)	48.36	54.86
Gamma linolenic acid	(C18:3n6)	0.15	ND
Alpha linolenic acid	(C18:3n3)	0.08	ND
Total Tocopherol (mg/100g of oil)		309.64	74.69

ME*- mechanical extracted SE**- solvent extracted ***ND-not detected
*Each value is a mean of three determinations.

Overall, there was marginal difference observed for the fatty acid composition of the extracted oil obtained by mechanical and solvent extraction method. It is observed from present finding that among the saturated fatty acids the palmitic acid concentration was found to be higher as compared to the other saturated fatty acid followed by the stearic acid. The palmitic acid concentration was observed in case of niger seed oil was 7.61 and 7.25 percent in case of solvent and mechanical extracted oil respectively. The findings of present experiment were more or less similar to that of the earlier results [25].

In mono-unsaturated fatty acids, oleic acid concentration was found to be highest. The oleic acid concentration was found to be 34.69 and 30.59 percent in case of mechanical and solvent extracted niger seed oil respectively. The present findings regarding the mono-unsaturated fatty acid is almost comparable with the concentration reported earlier [3]. In regards of polyunsaturated fatty acids linoleic acid (C18:2n6) was predominant niger oil. The niger seed oil shows a linoleic acid concentration of 48.36 and 54.84 percent for mechanical and solvent extracted oil respectively which was much lower than previous reports [17]. Niger oil extracted by mechanical method shows a concentration of 0.08 percent alpha linolenic acid. The present findings are more or less similar [17].

The highest tocopherol content was found in mechanically extracted niger seed oil. The total tocopherol content of mechanically and solvent extracted niger seed oil were 309.64 and 74.66 mg/100g of oil respectively. The lower value for solvent extracted niger seed oil is due to the high temperature employed during solvent extraction [18]. The tocopherol content of extracted niger seed oils were found to be low as compared to the literature reports by Dutta *et al.* [4], Marini *et al.* [5] and Ramadan and Morsel [26] but higher than Bhatnagar and Gopala Krishna [21].

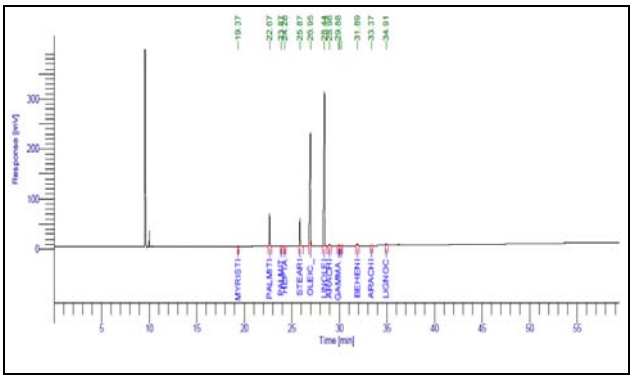

Fig 1: Fatty acids chromatogram of mechanically extracted lniger oil

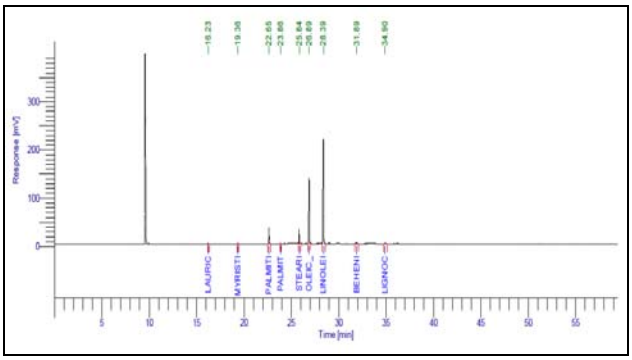

Fig 2: Fatty acids chromatogram of solvent extracted niger oil

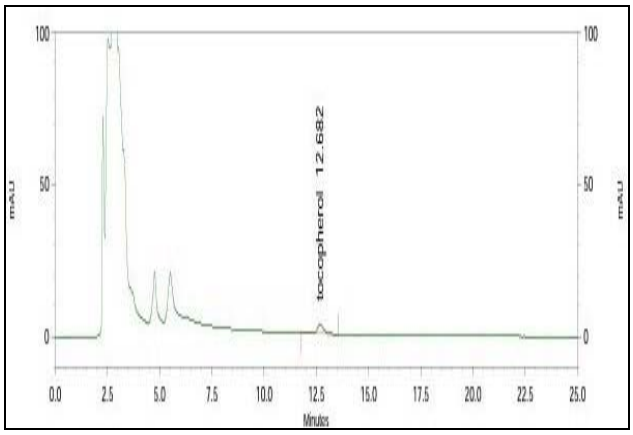

Fig 3: Chromatogram of tocopherol of mechanically extracted niger seed oil

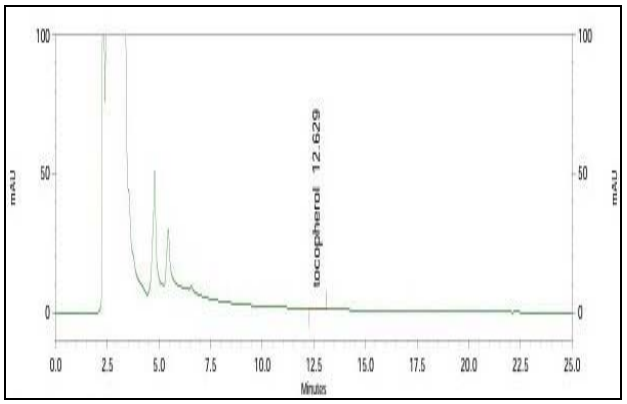

Fig 4: Chromatogram of tocopherol of solvent extracted niger seed oil

4. Conclusion

Due to lower peroxide value and higher total tocopherol content, it showed that high stability and superior quality oil was extracted by mechanical extraction method. Although the mechanical extraction gives lower yield, is advantageous with regard to mild operational temperature conditions, process safety and product quality.

5. Acknowledgements

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