Preparation and storage stability of flaxseed chutney powder, a functional food adjunct

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Abstract Flax (*Linum usitatissimum* L.) chutney powder (FSCP), a palatable functional food adjunct was prepared by mixing roasted and powdered flaxseeds with other selected spice ingredients. The protein content of these powders was 24.2% and 23.4% in flaxseed powder (FSP) and FSCP respectively. Total polyphenol content of FSP and FSCP was 439 and 522 mg/100 g respectively. The free fatty acid content of FSCP increased from 0.38 to 1.03 after 6 months storage. The critical moisture content for FSP and FSCP was 10.2 and 13.5%, which were equilibrated at 82 and 68% RH respectively and the ERH studies indicated both the powders are non-hygroscopic in nature. Overall sensory quality of FSCP served with cooked rice scored 'good' (7.4) even after 6 months of storage.

Keywords Flaxseed · Processing · Flaxseed chutney powder · Fatty acid composition · Sorption isotherm · Sensory quality

Introduction

Flaxseed (*Linum usitatissimum* L.) belongs to the family of *Linaceae* and has recently gained good attention as a functional food due to the presence of omega 3- fatty acid and fibre. 100 g of ground flaxseed supplies about 450 kcal energy, 41 g fat, 28 g fiber and 20 g protein (Anonymous 2008). Flaxseed is the richest dietary source of the lignan,

secoisolariciresinol (SECO), a diphenolic compound when ingested by humans influences a wide range of biological systems that keep humans healthy (Muir 2010). γ -Glutamyl transpeptidase levels are increased abnormally high during inflammation, in alcoholics with abscess and degenerative disease conditions including tumorigenesis. Flaxseed chutney diet showed significant reduction in both γ -glutamyl transpeptidase level (52%) and micro-nuclei formation (47%) in rats (Shakir and Madhusudhan 2007). Commercial flaxseed hull oils were evaluated for physicochemical characteristics and antioxidant properties. Yield of oil ranged from 9–28% depending on extraction method. It was reported that the crude lipid contained 92.5% neutral lipids, 3.1% phospholipids, 2.4% acidic lipids and 2.1% free fatty acids. Oil extracted from flaxseed hull using supercritical CO₂ showed highest antioxidant capacity and the resultant defatted meal contained the highest (53 mg/g) secoisolariciresinol diglucoside which is an antioxidant phytochemical (Oomah and Sitter 2009).

Prasad et al. (1998) studied the antiatherogenic activity of lignans present in flaxseed from the reduction in hypercholesterolemic atherosclerosis due to a decrease in serum total cholesterol and LDL-cholesterol. Vitamin E-deficient diets containing 5 to 20% ground flaxseed or 2.75% ethyl linolenate protected mice against the malarial parasite *Plasmodium yoelii* as shown by decreased parasitemia and enhanced survival (Orville et al. 1991). Addition of flaxseed into processed products is on the rise to extract the beneficial aspects of high α -linolenic acid content and the lignans present. Incorporation of flaxseed at 10, 15, and 20% to corn tortillas exhibited a high amount of total unsaturated fatty acids between 26.3 and 30.1% (Rendon-Villalobos et al. 2009). The α -linolenic acid content of raw and cooked beef patties increased as the

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level of flaxseed flour increased. The PUFA/SFA ratio in the control with 10% fat increased from 0.04 to 0.62 in the raw beef patties with 15% flaxseed flour. Similarly the n-6/n-3 ratio in the control with 10% fat decreased from 5.76 to 0.36 in the raw beef patties with 15% flaxseed flour (Elif Bilek and Turhan 2009). Addition of flaxseed and flaxseed oil in bread making has more effectively retained moisture and softness than in control during 6 days storage at room temperature (Terhi et al. 2006). Flaxseed at 30–50% substitution for flour greatly enhanced the nutritional qualities of some nutrients without affecting the overall acceptability of bakery products. The sensory scores of breads containing 30% and muffins containing 50% flaxseed flour were acceptable. The linolenic acid, fiber and folate contents were improved by 28 g, 16 g and 387 mg dietary folate equivalents (DFE), respectively, in 30% flaxseed bread compared to control. In 50% flaxseed muffins compared to control, α -linolenic acid, fiber and folate contents were improved by 21 g, 17 g and 341-mg DFE, respectively (Elina and Vijay 2009). The preparation of a high energy product by using barley, flaxseeds and teff, and a binding agent admixture consisting of corn syrup, honey, rapeseed oil and ground sunflower seeds has been described in a patent (Shifferaw and Wolde 2000). An extruded food product was prepared by using linseed, wherein grains are ground, impregnated with water by steaming, made into to paste form, and extruded (Well 2003). Susheelamma (1989) studied the polysaccharide preparations from raw and roasted flaxseed in the preparation of idly-type steamed puddings and found that the porous texture of the pudding was stabilized satisfactorily by the polysaccharides, which can satisfactorily replace the black gram polysaccharide. Oomah and Mazza (1993) concluded that flaxseed can be incorporated in a wide range of potential products.

In India, a variety of *chutneys* and *pickles* in large volumes based on vegetables, pulses and spices are consumed along with rice and breakfast items like *chapatti*, *idly*, *dosa*, and *vada*. Literature is available on development and standardization of several food adjuncts namely traditional chutneys instant chutneys and chutney powders based on the various raw materials available during different seasons (Balaswamy et al. 2005; Satyanarayana et al. 2001; Balaswamy et al. 2004; Prabhakara Rao et al. 2005; Jyothirmayi et al. 2006; Narsing Rao et al. 2008). Interestingly, there was no literature available on utilization of flaxseed in such food adjuncts. Hence, the main objective of the present study was to prepare high protein chutney powder with considerable quantities of dietary ingredients (α -linolenic acid) and evaluated for sensory quality, shelf stability of the product in metalised polyester polyethylene (MPE) pouches.

Materials and methods

Flaxseed, red chilly (*Capsicum annuum*), cumin (*Cuminum cyminum*), black gram (*Phaseolus mungo*), Bengal gram (*Cicer arietinum*), curry leaf (*Murraya koenigii*), garlic (*Allium sativum*), tamarind (*Tamarindus indica*), refined sunflower (*Helianthus annuus*) oil and common salt were purchased from local market. Chemicals used in this study were procured from SD Fine Chemicals Ltd, Mumbai, India.

Preparation of Flaxseed Chutney Powder (FSCP) Flaxseeds procured from market were thoroughly cleaned manually to remove dust, husk, stones and other foreign material. The pre-cleaned flaxseeds and other ingredients were separately roasted with little sunflower oil on a frying pan for 5 min at 80 ± 2 °C to remove raw flavor. After allowing flaxseed and other ingredients cool to room temperature, they were separately ground in a laboratory mixer (Kenstar, Mumbai) to get a powder to pass through BS30 (500 μ).

Flaxseed powder (FSP) 50 parts was mixed with other ingredients namely black gram powder (4 parts), bengalgram powder (2.5 parts), *jeera* (cumin) 2.5 parts, curry leaf powder (5.5 parts), garlic 5.5parts, tamarind 7 parts, red chilly powder (10 parts) along with common salt in required quantities (13 parts) and blended in a laboratory mixer to obtain flaxseed chutney powder (FSCP). The recipe for the preparation of FSCP was standardized in several trials by varying the quantities of flaxseed powder and other ingredients in various proportions (25:75, 30:70, 45:55, 50:50 and 60:40) in order to yield a palatable product.

Proximate composition FSP and FSCP were analyzed for proximate composition namely, moisture, total ash, acid insoluble ash, crude fat, crude protein and crude fiber according to the standard methods described by Ranganna (1986). The carbohydrate content was calculated by difference. Salt, calcium and iron contents were estimated according to a standard AOAC method (1995). Total polyphenol content was determined by the method suggested by Sadasivam and Manickam (1997).

Extraction of fat and its analysis FSP and FSCP were extracted in a soxhlet apparatus with hexane for a period of 16 h to extract the fat. The solvent extract was distilled using rotary vacuum evaporator (Jain Scientific Glass Works, New Delhi, India) at 50 °C to collect the total fat. The fat was analysed for free fatty acid content (FFA) as per standard method (Ranganna 1986).

Fatty acid methyl esters (FAMES) were prepared by saponification and methylation using 2% sulphuric acid in methanol. The esters were extracted into ethyl acetate and washed free from acid and dried over anhydrous sodium

sulphate. The dried esters were further concentrated to 2 ml for quantification in a Gas chromatograph (GC) and gas chromatograph—mass spectrometer (GC-MS).

Gas chromatography and gas chromatography mass spectrometry The GC-FID analyses were performed with an Agilent 6850 series gas chromatograph equipped with a flame ionization detector (FID). The chromatographic conditions for GC-FID were adjusted according to the class of compound. For FAME, a DB-225 capillary column (30 m, 0.25 mm i.d.) was used. The column temperature was initially maintained at 160 °C for 2 min, increased to 180 °C at 6 °C/min, maintained for 2 min at 180 °C, then further increased to 230 °C at 4 °C/min and finally maintained for 10 min at 230 °C. The carrier gas was nitrogen at a flow rate of 1.5 mL/min. The injector and detector temperatures were maintained at 230 °C and 250 °C, respectively, and the split ratio was 50:1.

The GC-MS analyses was performed with an Agilent (Palo Alto, USA) 6890 N gas chromatograph equipped with an HP-5 MS capillary column (30 m, 0.25 mm i.d.; 0.25 µm of film thickness and connected to an Agilent 5973 mass spectrometer operating in the EI mode (70 eV; m/z 50–550; source temperature 230 °C; quadrupole temperature 150 °C). The column temperature was initially maintained at 200 °C for 2 min, increased to 300 °C at 4 °C/min, and maintained for 20 min at 300 °C. The carrier gas was helium at a flow rate of 1.0 mL/min. The inlet temperature was maintained at 300 °C and the split ratio was 50:1. Structural assignments were based on interpretation of mass spectrometric fragmentation and confirmed by comparison of retention times as well as fragmentation pattern of authentic compounds and the spectral data obtained from the Wiley and NIST libraries.

Equilibrium Relative Humidity (ERH) studies FSP and FSCP were exposed to different relative humidity conditions such as 10, 30, 50, 70, 90 using standard normal solutions of sulphuric acid and 100% using distilled water in glass desiccators (Landrock and Procter 1951) at room temperature (25 ± 2 °C). Both the powders were exposed to the above conditions by keeping 5 g of sample in different petri plates and moisture absorbed by the sample was determined at regular intervals until they attained constant weight. The samples were observed critically for adverse changes like lump formation, discoloration and mold growth during the study. Moisture sorption isotherms of samples were plotted by drawing graphs of relative humidity (RH) versus equilibrium moisture content (EMC).

Packaging and storage studies FSP and FSCP were packed in metallized polyester (2.5 µ)/polyethylene (7.5 µ) (MPE) laminated pouches and stored at room

temperature (25 ± 2 °C) over a period of 6 months. The FSP and FSCP samples were subjected to chemical and sensory analysis at bi-monthly intervals.

Sensory analysis FSCP was mixed with cooked rice at 2.5–10% along with sunflower oil (10% on FSCP basis). FSCP was analyzed for sensory quality by serving to a group of 10 semi-trained panelists for 5 attributes namely appearance, colour, flavour, taste and overall acceptability. The panelists were earlier made to acquaint with various chutney powders available in the market. The panelists were asked to score based on Hedonic scale with a maximum score of 9 for 'like extremely' and minimum of 1 for 'dislike extremely' (Amerine et al. 1965).

Statistical analysis All the experiments were done in triplicate and average values were computed with standard deviation (SD). Sensory scores were analysed statistically by ANOVA using SPSS 15.0 to evaluate the significance at $P < 0.05$.

Table 1 Proximate, mineral, total polyphenol content and fatty acid composition of flaxseed flour (FSP) and flaxseed chutney powder (FSCP)

Parameter	FSP	FSCP
Moisture (%)	5.4±0.48	5.7±0.19
Total ash (%)	2.4±0.33	13.7±0.14
Sodium chloride (%)	NA	12.7±0.21
Acid insoluble ash (%)	0.11±0.03	0.12±0.02
Crude protein (% N×6.25)	24.2±0.76	23.4±0.85
Crude fat (%)	40.4±1.32	25.4±0.78
Crude fibre (%)	9.2±0.61	7.1±0.62
Carbohydrates (% by difference)	18.4±2.91	24.7±1.08
Energy (Kcal/100 g)	534.0±1.52	420.8±4.73
Calcium (mg/100 g)	41.5±1.34	55.1±0.80
Iron (mg/100 g)	1.5±0.10	1.9±0.20
Phosphorous (mg/100 g)	409.2±3.86	336.0±5.29
Total polyphenols (mg/100 g)	438.5±1.49	522.3±8.06
Fatty acid composition ^a		
16:0	7.1	8.2
18:0	7.8	7.5
Saturated	14.9	15.7
18:1	25.2	28.7
20:1	1.1	0.3
Mono Unsaturated	26.3	29.0
18:2	11.5	17.2
18:3	47.3	38.1
Polyunsaturated	58.8	55.3

$n=3$

NA Not applicable

^a values are mean of duplicate analyses

Fig. 1 Moisture sorption isotherms of flaxseed powder and flaxseed chutney powder

Results and discussion

Preparation of FSCP and proximate composition The standardized recipe for the preparation of FSCP consisted of roasted and ground flaxseed powder and other ingredients in the ratio of 50: 50 which yielded a highly acceptable product. Flaxseed (1,000 g) on roasting yielded 906 g FSP which resulted in a final product yield of 1,812 g of FSCP when mixed with other ingredients.

The proximate composition of FSP and FSCP were reported in Table 1. They possess higher protein content (23–24%). Calcium and iron contents are higher in FSCP (55, 2 mg/100 g) than the FSP (42 and 1.5 mg/100 g). The total polyphenols were found to be 438 and 522 mg/100 g in FSP and FSCP samples respectively. The crude fat contents were 40.4% and 25.4% in FSP and FSCP respectively. It was reported that the major fatty acid in

the flax oil was α -linolenic acid, comprising 58.3% of total fatty acids (Berrin and Feral 2008). The fatty acid composition of FSP showed that the major fatty acids were 18:3, 18:2 and 18:1 followed by 16:0 and 18:0 in the flaxseed powder (Table 1). It was found that there was a slight increase in 18:1 and 18:2 and a marginal decrease in the α -linolenic acid (18:3) and other fatty acids in FSCP over the FSP due to the addition of different other ingredients during the preparation of chutney powder.

Moisture sorption isotherms Moisture sorption isotherms of FSP and FSCP samples were presented in Fig. 1. The FSP had an initial moisture content (IMC) of 5.39%, which equilibrated at 64% RH at room temperature. Similarly the FSCP had an IMC of 5.72%, and equilibrated at 50% RH. The critical moisture contents were calculated as 10.22% and 13.49%, which equilibrated at 82% and 68% RH for FSP and FSCP respectively. Hence both the powders are non hygroscopic in nature and found to be stable at higher humidity conditions.

Changes in FFA and total polyphenols FFA content of FSP and FSCP was presented in Table 2. A marginal increase in FFA was observed in both the powders during the storage period of 6 months. A similar trend was also noticed by Satyanarayana et al. (2001) during the storage of instant chutneys from *pudina* (*Mentha spicata*) and *gongura* (*Hibiscus* sp.). In milled flaxseed an increasing trend in FFA content was observed from 0.30% to 1.58% during storage for 128 days (Malcolmson et al. 2000). The increase in FFA may be due to the oxidation of unsaturated fatty acids present in powders. The polyphenol content in FSCP was almost constant (522–528 mg/100 g) during the storage period. Strandas et al. (2008) observed that phenolic glucosides ranged between 15 and 157 mg/100 g dry bread

Table 2 Changes in free fatty acid, peroxide value, total polyphenols content and sensory scores of flaxseed chutney powder during storage at room temperature ($28 \pm 2^\circ\text{C}$)

Parameter	Storage period (Months)			
	0	2	4	6
Free fatty acids (% oleic acid on extracted fat basis)	0.38 $\pm$ 0.07	0.44 $\pm$ 0.09	0.79 $\pm$ 0.06	1.0 $\pm$ 0.31
Total polyphenols (mg/100 g)	522.0 $\pm$ 4.35	524.0 $\pm$ 5.29	525.0 $\pm$ 7.81	528.0 $\pm$ 3.46
Sensory scores				
Appearance	8.4 $\pm$ 0.52	8.2 $\pm$ 0.54	8.0 $\pm$ 0.33	7.8 $\pm$ 0.26*
Colour	8.3 $\pm$ 0.54	8.1 $\pm$ 0.52	8.1 $\pm$ 0.52	7.9 $\pm$ 0.21
Flavour	8.1 $\pm$ 0.52	7.6 $\pm$ 0.39	7.5 $\pm$ 0.41*	7.0 $\pm$ 0.24*
Taste	8.5 $\pm$ 0.47	8.4 $\pm$ 0.46	8.4 $\pm$ 0.46	8.1 $\pm$ 0.52
Overall quality	8.4 $\pm$ 0.46	8.3 $\pm$ 0.42	8.0 $\pm$ 0.24	7.4 $\pm$ 0.57*

$n=3$

*Indicates the significant difference in the row from 0 months at $P<0.05$

when FSP was incorporated into bread. A marginal increase in polyphenol content (502–542 mg/100 g) was observed in apple juice during 30 days storage (Zhang et al. 2008). As polyphenols are complex natural constituents consisting of cinnamates, anthocyanins, flavanols etc. the stability depends on the type of compounds present in the material. Del Caro et al. (2004) observed that changes in various classes of polyphenols in prunes were affected to different proportions during drying and storage.

Sensory quality during storage It was observed that the 5 g of FSCP per 100 g rice was found to be optimum for sensory evaluation studies. The overall sensory quality of FSCP on 0 day was 8.4 (excellent), and after 6 months the score was 7.4 (good). Studies on addition of flaxseed flour at the level of 15% was good and at higher levels up to 25% it was found satisfactory in bread (Mente 2008). It was observed that the colour of the FSCP is retained well, and scores for the characteristic flavour decreased significantly during the storage period. Marginal variation was observed in the sensory scores during the storage period (Table 2). There was a significant difference in overall sensory quality at $P < 0.05$.

Conclusion

The studies revealed that the preparation of flaxseed chutney powder can be scaled up to small scale level and can be used as nutritional and functional food adjunct. The product had high protein and considerable amounts of essential n-3 fatty acids along with minerals. The product was shelf stable for 6 months in metallised polyester polyethylene laminated pouches at room temperature without detrimental sensory attributes. EMC—RH data also confirmed that FSCP was non-hygroscopic in nature. The FSCP had a characteristic mouth feel with good flavour of the spice ingredients and taste when consumed with cooked rice.

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