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Effect of *Nigella sativa* oil addition on post frying quality of commercial cooking oils

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Abstract

The quality of *Nigella sativa* seeds was evaluated in terms of percent moisture, thousand seeds weight and bulk density. Solvent extraction method was used for extraction of *Nigella sativa* oil. In order to reduce the post frying degradation of cooking oils, *Nigella sativa* oil was added in commercial cooking oils was added at varying levels (0 – 1000 ppm). The free fatty acid was estimated till seven times frying. Oil at the concentration of 800 and 1000 ppm is shown to have strong protective effects against lipid peroxidation of commercial oil during frying condition. The Pearson's correlation suggested strong negative correlation between amount of *Nigella sativa* oil and free fatty acids till the seventh deep frying cycle.

Keywords: Antioxidant, FFA, frying cycle, Nigella sativa,

1. INTRODUCTION

Lipid oxidation is a significant challenge for the food and cosmetic industries. This is pertinent when the lipid substrates consist of unsaturated or polyunsaturated fatty acids (PUFA) that are prone to oxidation. The efficacy of antioxidants is contingent upon their chemical reactivity (as radical scavengers or metal chelators), interactions with food constituents, environmental factors (such as pH and concentration), and the physical positioning of the antioxidant within food systems (Lucas et al., 2010).

Oxidation generates unattractive flavors and odors, diminishes the nutritional content of oils, and results in the formation of hazardous chemicals. Lipids participating in the oxidation process comprise unsaturated fatty acids, including oleic, linoleic, linolenic, and long-chain polyunsaturated fatty acids (PUFAs); also, other unsaturated lipids, such as sterols, are subject to oxidation. The oxidation rate of fatty acids escalates with higher unsaturation levels and diminishes in the presence of lipid-soluble antioxidants (Warner & Knowlton, 1997).

The oxidation of consumable fats and oils can be regulated through the application of antioxidants, employing processing methods that reduce the depletion of tocopherols and other antioxidants, deactivating prooxidant metals and enzymes, limiting exposure to oxygen, heat, and light, hydrogenating polyunsaturated fatty acids, and utilizing inert gas or vacuum packaging to eliminate atmospheric oxygen (Miraliakbari & Shahidi, 2008).

Vegetable oils experience hydrolysis, oxidation, and polymerization during storage and heat processing. The flavors associated with deep frying result from breakdown products of linoleic acid, and their strength can be reduced by frying food in oil with a low linoleic acid level (Chang et al., 2020). Corn, sunflower, and

soybean oils, characterized by elevated quantities of polyunsaturated fatty acids (PUFA), are the predominant oils utilized for cooking and frying. These oils, however, are not particularly appropriate for frying due to their increased susceptibility to oxidation at extreme temperatures (Anwar et al., 2007).

Certain high-oleic oils, such as canola or olive oil, may be utilized for frying due to their stability at elevated temperatures. Nonetheless, their elevated expense limits its widespread application. Consequently, the utilization of more stable, cost-effective frying oils would be advantageous. To address the issue of inadequate oxidative stability (OS) in conventional oils, methods to diminish the unstable polyunsaturated fatty acid (PUFA) content and enhance natural antioxidants were explored. One method to enhance the oxidative stability of these oils is by combining them with oils that possess high oleic acid content and elevated quantities of antioxidants.

The demand for broadly applicable and readily accessible bioactive lipids and natural antioxidants is increasing. Kalonji (*Nigella sativa* L.) seed oils have been integral to supplementary diets globally, with their usage gaining popularity in non-producing nations. Among contemporary sources of edible oils, these seed oils are noteworthy and may significantly contribute to human nutrition and health due to their unique fatty acid composition and the presence of substantial quantities of fat-soluble bioactives (Ramadan et al., 2003). *Nigella sativa* is an annual herbaceous plant that belongs to the Ranunculaceae family. Furthermore, components of N. sativa seeds have been used to make functional cosmetic and nutritional supplement products. *Nigella sativa* oil is abundant in important fatty acids, bioactive phytosterols, and tocopherols (Ramadan, 2007).

Combining various vegetable oils can enhance the concentrations of bioactive lipids and natural antioxidants in the mixtures, resulting in superior quality oils with customized physicochemical features and enhanced nutritional value at reasonable costs. Recently, the production and commercialization of blended oils, comprising traditional edible oils combined with unconventional oils, has been authorized (Ramadan et al., 2008). The present study characterized the kalonji seeds grown in Pakistan and prepared blend of non conventional black cumin seed oil and commercial cooking oil. The effect of this blending at different concentrations and frying cycles was evaluated. This approach has not been reported in the literature earlier.

2. MATERIALS AND METHOD

2.1. Materials

Nigella sativa seeds, potatoes and unbranded cooking oil were procured from a local market in Karachi, Pakistan. Moreover, two branded cooking oils were purchased from a supermarket. First brand (MC) cooking oil was a blend of canola, soya beans and sun flower seeds oils while the second brand (SC) was a mixture of soyabean oil and canola oil. Analytical grade reagents were employed.

2.2. Physical properties of seeds

Randomly selected three seeds samples were used to evaluate the physical properties. The seeds were cleaned manually to remove all foreign materials such as stones, chaff, immature and broken seeds. The moisture content was analysed using the AOAC method No. 984.25 (Horwitz, 1925).

For seed index, 1000 seed were counted by hand and mass was measured in grams by using a digital balance having an accuracy of 0.001 g (Sharma et al., 2011). The 1000 seeds were poured in a 250 mL graduated cylinder with a constant rate then the volume was noted. Bulk density was calculated by taking the ratio of 1000 seeds mass and volume.

2.3. Extraction of Nigella sativa oil

Cleaned *Nigella sativa* seeds were ground by using a grinder (AG 640 Delux Grinder, ANEX)

prior to extraction of oil through Soxhlet extraction method. n-hexane was employed as the solvent and extraction was carried out for 6 hours. Afterwards, the solvent was evaporated by using rotary evaporator (BUCHI, Rotavap R-200) at 90 °C for 30 min and the oil was stored in a brown colour glass bottle.

2.4. Frying process

Fresh potatoes were peeled and sliced to a uniform thickness of 2.5 mm using a mechanical slicer. The potato slices were stored at room temperature, immersed in distilled water, and subsequently blotted dry with tissue paper before being weighed into 100 g batches for frying. Cooking oil was added to a fryer (AG 2012, ANEX) the temperature was initially raised to 60°C. At this point, varying concentrations of *Nigella sativa* oil (400 ppm, 800 ppm, and 1000 ppm) were incorporated into Meezan oil, Soya Supreme oil, and Local oil, respectively. To ensure proper dissolution of the *Nigella sativa* oil, the mixture was stirred continuously for 10 minutes. A control sample was similarly maintained without the addition of *Nigella sativa* oil. Afterwards, the temperature of the oil was increased to 180°C and held for 20 minutes before frying commenced. Frying was carried out in batches of 100 g of raw potato chips, which were fried for 2.5 minutes with a 20-minutes interval between batches, over a period of seven consecutive days. Throughout the frying process, the fryer remained uncovered. After each frying session, oil samples were collected in sealed bottles for subsequent analysis. Oil quality tests were conducted immediately following frying. The fryer was then covered, allowing the oil to cool naturally, and the procedure was repeated the next day. No fresh oil was added to the frying vessel during the experiment.

Table 1. Amount of added Nigella sativa extract added in branded and non branded oil samples.

Samples	Concentration of *Nigella sativa* extract (ppm)
SC	0
S400	400
S800	800
S1000	1000
MC	0
M800	800
LC	0
L800	800

2.5. Estimation of free fatty acid

The free fatty acid (FFA) percentage in fried oil samples was determined following the official method of the American Oil Chemists' Society (AOCS Ca 5a-40, 2017), with slight modifications according to the procedure described by Moufakkir et al. Briefly, 10 g of fried oil was combined with 2 mL of phenolphthalein solution and a few drops of 0.1 N sodium hydroxide (NaOH). The mixture was then diluted with 50 mL of ethanol and stirred until a pale pink color was observed. Then, the solution was titrated with 0.25 N NaOH, and the volume of NaOH used in titration was recorded as Vs. For the control, the titration was repeated using a blank solution, with the volume of NaOH recorded as Vb. The FFA percentage was calculated using the following equation:

$$\%FFA=((Vb-Vs)mL\ of\ NaOH\times N\times 28.14)/m\times 100$$

where ,
m = mass of the oil sample in grams,
N = Normality of NaOH,
Vs = Volume of NaOH used during titration of the oil sample (mL),
Vb = Volume of NaOH used in the blank titration (mL).

2.6. Statistical analysis

The SPSS software (version17, 263 SPSS Inc., USA) was used for statistical analysis of experimental data. ANOVA was used to calculate significant differences between the mean values (n= 03) and Duncan's test at $p \leq 0.05$ was employed. Moreover, Pearson's correlation was studied at $P< 0.01$ between concentration of *Nigella sativa*oil and number of frying.

3. RESULTS AND DISCUSSION

3.1. Physical properties of Nigella sativa seeds

To optimize the design of equipment for harvesting, handling, storage, and other processes involving seeds, it is essential to have a comprehensive understanding of their physical attributes (Altuntaş et al., 2005). The physical properties of *Nigella sativa* seeds were studied in terms of moisture content, seed index, seed volume and bulk density (Table 2). The moisture content specifies potential stability of seeds during the storage period. The average *Nigella sativa* seed percent moisture at the time of the experiment was found to be 6.83±0.31% (Table 2).

The weight of 1000 grains is a critical indicator of general grain quality and significantly influencing germination of seed, its viability, seedling development, and overall plant performance (Afshari et al., 2011). The seed index was found to be 3.34 ±0.48g (Table 2). Pakistani *Nigella sativa* seeds was found to have higher seed index than Egyptian *Nigella sativa* seeds, reported by Atta, (2003). The seed index for poppy seeds and flax seeds was found 0.5 g and 6.6 g, respectively (Krajewska et al., 2016). According to Deivasigamani & Swaminathan, (2018), mass and size of 100 and 1000 seeds could differ between different crops, among varieties within the same crop, and even from one year to the next or from one field to another within the same variety.

Bulk density is a crucial physical property, as it directly influences the storage capacity and efficiency of transport systems (Sharma et al., 2011). The bulk density of *Nigella sativa* seed was found to be 0.515 g/cm^3 (Table 2). The bulk density was thus lower than that of Egyptian *Nigella sativa* seeds (Atta, 2003), sunflower seeds (Sacilik et al., 2007) and fenugreek seeds (Altuntaş et al., 2005) whereas, it was higher than Jatropha curcas seeds (Herak et al., 2013).

Table 2. Physical Characteristics of Nigella sativa seeds

Characteristics	Determined values	Values reported in literature[1]
Moisture content (%)	6.83±0.31	7.0±0.5
Seed index (g)	3.34 ±0.48	2.21±0.14
Seed volume (cm3)	5.96±0.57	2.76±0.10
Bulk density (g/cm3)	0.56 ± 0.02	0.80±0.01

[1](Atta, 2003)

3.2. Effect of Nigella sativa oil on post frying oil quality

The major reactions that deteriorate an oil's quality during frying could be classified into

hydrolysis, oxidation and polymerization which causes the production of FFAs, aldehydes, ketones, acids, and other degradation products (Bazina & He, 2018). The FFA content indicates the amount of potassium hydroxide (KOH) needed to neutralize the FFA in 1 g of oil. The formation of FFAs occurs through the hydrolysis of triacylglycerols and the decomposition of hydroperoxides at elevated temperatures, particularly in the presence of air and moisture (Guo et al., 2016). The FFA level of all samples upto 7th cycle of frying potatoes ranged between 0.092 to 1.39%. The lower increment in FFA levels of oils could be due to the smaller of potatoes (100 g) in 3 L of oil, which consequently caused little hydrolytic degradation. Pakistan Standard Quality Control Authority (PSQCA) requires that fryer oil should be discarded when FFA levels in the oil exceed 2%. All oils samples were within the limit set by PSQCA. The regulatory guidelines on maximum FFA levels in frying oil are 1.0% for the Iranian National Standardization Organization and United States Department of Agriculture (Esfarjani et al., 2019).

The percentage of FFA increased with the increase in number of frying in control samples and *Nigella sativa* oil containing samples (Table 3). The high frying temperature and exposure to air occurred during frying which promoted the hydrolysis and oxidation of triglycerides consequently increased the percentage of FFA in all samples. However, percent increase in FFA (%) of *Nigella sativa* oil containing samples was lower than their respective control samples. Interestingly, FFA observed for S1000, S800 and M800 after each frying was lower than S400 and samples without *Nigella sativa* oil. Moreover, no significant difference was observed between the post FFA percentages of S1000, S800 and M800 suggesting the 800ppm addition is the optimum level of *Nigella sativa* oil in the present study. Enhanced oxidative stability of sunflower seed oil after blending with black cumin seed oil was also observed by Ramadan, (2013). Furthermore, the difference in FFA of oil blends without *Nigella sativa* oil (LC, SC and MC) could be due to the differences in sources and ratios of oils present in the blends. The local market oil was not labeled however, MC was a blend of canola, soyabean and sun flower seeds oils while SC was a mixture of soyabean and canola oil according to the information given at package label

3.3. Correlation between amount of Nigella sativa extract and free fatty acids

The Pearson's correlation of frying times and quantity of *Nigella sativa* extract in cooking oil samples is summarized in Table 4. A strong negative correlation was observed for number of frying and the amount of *Nigella sativa* oil upto seven frying time by deep frying potato samples. It suggested the inverse relation of free fatty acid formation during frying with the amount of extract. However, the correlation of number of frying is strong and positive with free fatty acids. Febrianto et al., (2019) also observed increased FFA content with number of frying by using chicken, flour based food and cat fish samples.

Table 3. Effect of Nigella sativa extract on free fatty acids after seven frying times

Samples	Post frying free fatty acids (%)						
Number of Frying	1st	2nd	3rd	4th	5th	6th	7th
SC	0.169 ± .003e	0.205 ± .006c	0.382 ± .009e	0.503 ± .006d	0.752± .002e	0.789 ± .003c	1.199 ± .002e
S400	0.113 ± .002b	0.159 ± .006b	0.319 ± .009d	0.390 ± .006c	0.390 ± .002c	0.389 ± .002b	0.450± .002b
S800	0.097 ± .002a	0.112 ± .006a	0.112 ± .009a	0.169 ± .006a	0.188± .003a	0.188± .002a	0.226 ± .001a
S1000	0.091 ± .002a	0.095 ± .006a	0.112 ± .009a	0.169 ± .006a	0.178 ± .002a	0.185 ± .002a	0.225± .002a
MC	0.152 ± .006d	0.189 ± .006c	0.321 ± .009d	0.405 ± .006b	0.698± .023d	0.817± .031c	0.971± .045d
M800	0.092 ± .003a	0.111 ± .006a	0.155 ± .009b	0.170 ± .006a	0.179 ± .005a	0.191± .007a	0.244± .009a
LC	0.171 ± .007e	0.259 ± .006d	0.463 ± .009f	0.691 ± .006e	0.903 ± .011f	0.942± .039d	1.39 ± .024f
L800	0.130 ± .005c	0.197 ± .006c	0.224 ± .009c	0.267 ± .006b	0.292 ± .025b	0.389± .011b	0.515± .024c

Table 4. Pearson's correlation of frying times and amount of Nigella sativa extract

	F1	F2	F3	F4	F5	F6	F7
Amount of added oil	-.894**	-.789**	-.917**	-.892**	-.957**	-.948**	-.918**
F1		.932**	.897**	.899**	.953**	.968**	.974**
F2			.898**	.906**	.879**	.895**	.902**
F3				.974**	.941**	.921**	.925**
F4					.956**	.928**	.945**
F5						.991**	.987**
F6							.983**

4. CONCLUSIONS

This study presents a unique methodology that combines kalonji (*Nigella sativa*) seed oil, with commercially available cooking oils often utilized for frying in domestic and small-scale industrial settings. This novel combination was evaluated at two concentration levels, 800 and 1000 ppm of *Nigella sativa* oil, and its effect on the stability of frying oils was assessed. The findings indicated that these mixtures were exceptionally successful, preserving their frying quality and stability for as much as seven deep frying cycles. The research underscores the exceptional capacity of kalonji oil to mitigate oxidation and degradation, prevalent challenges during repeated frying, so presenting a practical and sustainable approach to enhancing oil longevity.

The hotel industry, which has a strong demand for consistent and inexpensive frying oils, is one such area where our results may find substantial use. By adding kalonji oil to commercial cooking oils, restaurants, caterers, and small food businesses may increase the frying oils' oxidative stability, cut down on waste, and boost the nutritional value of their fried dishes. This value addition offers a viable path toward both financial and environmental gains in the food sector, and it is consistent with current trends toward healthier and more sustainable food production practices.

Ethical Approval

None.

Declaration of conflicting interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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Author Contribution

Concept: ZL, MFA. Design: ZL, MFA. Data collecting: AM, WA, SAA Statistical analysis: AM. Literature review: AM, WA, SAA Writing: ZL Critical review: ZL, MFA

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