Food Chemistry 368 (2022) 130777

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Impact of microwave roasting on physicochemical properties, maillard reaction products, antioxidant activity and oxidative stability of nigella seed (*Nigella sativa* L.) oil

Kanchan Suri [a], Balwinder Singh [b,*], Amritpal Kaur [a,*]

[a] Department of Food Science and Technology, Guru Nanak Dev University, Amritsar, Punjab 143005, India
[b] P.G. Department of Biotechnology, Khalsa College, Amritsar, Punjab 143002, India

ARTICLE INFO

Keywords:
Nigella seed oil
Roasting
Fatty acids
Pigments
MRPs
Oxidative stability

ABSTRACT

In this study, oils extracted from nigella seeds (NS) subjected to microwave roasting at 180, 360, 540 and 720 W for 5 and 10 min were evaluated for quality and stability characteristics. The impact of microwave powers on oil yield, pigment content, Maillard reaction products (MRPs), radical scavenging activity (RSA), color, viscosity, total phenolic content (TPC), oxidative stability index (OSI) and fatty acid composition (FAC) of oil was studied. TPC, RSA, OSI, MRPs, viscosity, chlorophyll and carotenoid contents were higher in oil of NS heated at 720 W for 10 min while maximum oil yield and low acid value was observed for NS heated at 540 W for 10 min. FAC was slightly influenced by microwave roasting and FTIR spectra exhibited a minor difference in intensities of peaks at 3009, 2925, 2854, 1745 and 1161 cm^{-1}. The results of the study indicated that microwave roasting improves NS oil quality characteristics.

1. Introduction

The herbal plants with medicinal potentials have gained attention in human diet for the treatment of acute and chronic diseases (Farzaneh & Carvalho, 2015). *Nigella sativa* L. (nigella, kalonji, black seed or black cumin) is a well-known medicinal plant of the *Ranunculaceae* family that has received recognition for the treatment of various ailments (Mukhtar, Qureshi, Anwar, Mumtaz, & Marcu, 2019; Gholamnezhad, Havakhah, & Boskabady, 2016). It is endemic to the Mediterranean basin and is commonly cultivated as a spice or herbal medicine in Saudi Arabia, South Europe, Syria, Turkey, India, Pakistan and Iran (Mukhtar et al., 2019). This plant has a rich historical background and is regarded as a miracle medicinal herb for its biologically active constituents and health benefits (Mukhtar et al., 2019; Gholamnezhad et al., 2016).

Nigella seeds (NS) are traditionally used for the treatment of asthma, bronchitis, cough, chest congestion, fever, infertility, inflammation, diarrhoea, dysentery and flatulence (Gholamnezhad et al., 2016; Ahmad et al., 2013). NS have oregano-like quality with slight bitterness and a warm, toasted-onion flavour. They are commonly utilized as food ingredients in cheese, pickles, pastry, pasta and bakery products (Ahmad et al., 2013). NS contains protein (20–27%), carbohydrate (23.5–33.2%) and oil (25–32%) with a good amount of fatty acids (FAs) like linoleic, linolenic and oleic acids (Moghimi, Farzaneh, & Bakhshabadi, 2018; Al Juhaimi, Uslu, & Özcan, 2018). Some prior studies revealed that NS oil can play a protective and therapeutic role in human health and nutrition (Mazaheri, Torbati, Azadmard-Damirchi, Savage, 2019a; Suri, Singh, Kaur, Yadav, & Singh, 2019a; Ahmad et al., 2013). NS can be used as novel source of edible oils for potential health benefits (Saxena et al., 2017). NS oil exhibits antioxidant, anti-inflammatory, antimicrobial, anti-allergic, analgesic, immunomodulatory, antitumor, anti-hyperlipidemic, hepatoprotective, cardioprotective and antidiabetic effects (Mazaheri et al., 2019a; Mukhtar et al., 2019; Gholamnezhad et al., 2016; Ahmad et al., 2013). It is a good source of bioactive constituents and is rich in essential FAs (Mazaheri et al., 2019a; Suri et al., 2019a).

Oilseeds are preheated ahead of oil extraction to increase oil yield, coagulate protein, inactivate enzymes, impart aroma and flavor to the oils (Zou, Gao, He, & Yang, 2018). Preheating or roasting, a conventional method employed for oilseeds influences the composition and quality attributes of extracted oils (Suri, Singh, Kaur, & Singh, 2019b; Suri, Singh, Kaur, Yadav, & Singh, 2020). Roasting treatment significantly influences the phenolic content and antioxidant activities of oils. Maillard reaction products (MRPs) generated during preheating of oilseeds improves the antioxidant activity of oils (Al Juhaimi, Özcan, Ghafoor, & Babiker, 2018). Among different pretreatments of oilseeds,

* Corresponding authors.
E-mail addresses: bbs171@rediffmail.com (B. Singh), amritft33@yahoo.co.in (A. Kaur).

https://doi.org/10.1016/j.foodchem.2021.130777
Received 9 April 2021; Received in revised form 3 August 2021; Accepted 4 August 2021
Available online 6 August 2021

microwave roasting is a simple, appropriate and efficient method to obtain high-quality oils. Microwave preheating is an efficient processing method since the energy is dispersed and consumed immediately and equally throughout the oilseeds (Suri, Singh, Kaur, Yadav, & Singh, 2020). Moreover, microwave roasting amplifies the release of valuable nutraceuticals and favorable oil components from oilseeds (Al Juhaimi et al., 2018; Fathi-Achachlouei, Azadmard-Damirchi, Zahedi, & Shaddel, 2019; Suri et al., 2020). The study of oils extracted from microwave heated seeds is extremely important. Recent studies on microwave pretreatments of chia seed (Özcan, Al-Juhaimi, Ahmed, Osman, & Gassem, 2019), flaxseed (Suri et al., 2020), rapeseed (Rękas, Wroniak, & Ścibisz, 2017), cashew nut (de Carvalho, de Figueiredo, de Sousa, de Luna, & Maia, 2018), walnut (Zhou, Fan, Chu, & Pei, 2016), milk thistle (Fathi-Achachlouei et al., 2019) and apricot kernel (Al Juhaimi et al., 2018) reported improved extraction of valuable nutraceuticals and favorable oil components.

Considering the available literature, there are many studies on nutritional composition and health benefits of NS while limited studies reported the impact of microwave roasting on NS oil quality. The microwave roasting of NS may effect the fatty acid composition (FAC), pigments, MRPs, oxidative stability index (OSI) and antioxidant properties of oil and the study of these attributes are of extreme importance. The present study, therefore, aimed to insight the impact of microwave power (180, 360, 540 and 720 W) and roasting duration (5 and 10 min) on NS oil quality and stability characteristics. This study explored changes in yield, physicochemical, antioxidant properties, FAC, pigments, MRPs, OSI, viscosity and FTIR spectra of oil extracted from microwave roasted NS. This study was conducted to optimize microwave roasting conditions for the improvement of NS oil quality and support the potential uses of microwave roasted NS oil in different food products.

2. Materials and methods

2.1. Materials

The commercial variety of nigella seeds (NS) grown in November 2019 and harvested in February 2020 at Ratlam, Madhya Pradesh were procured from a local shop (Amritsar, India), manually cleaned and stored in airtight polybags at 4 °C. The certified FAME standard and 5-hydroxymethylfurfural of Sigma-Aldrch (St. Louis, MO, USA) and chemicals of analytical grade were used.

2.2. Roasting and oil extraction

NS were heated in a microwave oven (LG, India) at 2450 MHz frequency with four power adjustments (180 W, 360 W, 540 W and 720 W) for 5 and 10 min. For each pre-treatment, 250 g of NS were spread in a glass petri plate (120-mm diameter). The microwave pretreatments were selected based on initial trials. After roasting, seeds were cooled (at 25 °C) and stored in polybags (at 4 °C) till further use. The moisture contents (MC) of NS heated at 180, 360, 540 and 720 W for 5 min were 5.83, 5.22, 4.60 and 3.80%, respectively while NS heated for 10 min showed MC of 5.42, 4.63, 4.28 and 3.24%, respectively. The MC of raw (unroasted) NS was 6.25%. NS oil was extracted using a mechanical screw expeller at a temperature below 50 °C from unroasted (control) and microwave roasted NS. After extraction, the oil samples were centrifuged (12000 rpm) for 10 min to remove solid impurities and stored in sealed dark-colored bottles at −20 °C till analysis. The extracted NS oil was weighed and calculated to estimate the oil yield (%).

2.3. Colour

The a* (+a* value for redness and -a* value for greenness), b* (+b* value for yellowness and -b* value for blueness), and L^* (0 to 100, 0 for darkness and 100 for lightness) values of NS oil samples were monitored using Ultra Scan VIS (Hunter Associates, USA) colour spectrophotometer.

2.4. Pigments

The pigments (chlorophylls and carotenoids) of unroasted and microwave roasted NS oil were estimated by dissolving in cyclohexane as reported elsewhere (Suri et al., 2020). The values of absorbance for carotenoids (at 470 nm) and chlorophylls (at 670 nm) were recorded on a spectrophotometer (Agilent Technologies, USA) and contents were estimated using the below-mentioned equations.

$$Chlorophylls\ content\ (mg/kg) = \left(Abs_{670} \times 10^6\right)/(613 \times 100 \times\ density)$$

$$Carotenoids\ content(mg/kg) = \left(Abs_{470} \times 10^6\right)/(2000 \times 100 \times\ density)$$

2.5. Total phenolic content (TPC)

TPC of NS oil was estimated using the procedure described elsewhere (Suri et al., 2020). 0.1 mL oil sample taken in the amber glass test tube was mixed with *n*-hexane (1 mL). Then, 10% sodium carbonate solution (3 mL) and ten-fold diluted Folin-Ciocalteu reagent (0.75 mL) was mixed, homogenized and the solution was placed for 90 min at ambient temperature. The absorbance was recorded at 710 nm using a spectrophotometer and TPC was measured as μg of gallic acid equivalent (GAE)/mL of NS oil.

2.6. Radical scavenging activity (RSA)

RSA was estimated by analyzing the ability of NS oil to scavenge DPPH radical. The solution of NS oil (1 mL) prepared using ethyl acetate was vortexed with 4 mL of 0.1 mM DPPH and left incubated for 30 min (in dark). The absorbance of oil sample was recorded at 515 nm on UV–vis spectrophotometer and RSA (% inhibition in DPPH) was measured according to the previous report (Suri et al., 2020) using the below-mentioned equation.

$$\mathrm{RSA}(\%) = \left[1 - \frac{A_{30}}{A_c}\right] \times 100$$

Where A_{30} represents the absorbance of the oil samples after incubation (30 min) and A_c represents the absorbance of DPPH (control) solution.

2.7. Maillard reaction products (MRPs)

2.7.1. Browning index (BI)

The BI of unroasted and microwave roasted NS oil was measured by dissolving in chloroform (1:20 w/v) as reported by Suri et al (2020). The absorbance was measured at 420 nm using a UV–Vis spectrophotometer to represent the non-enzymatic BI of oils.

2.7.2. 5-Hydroxymethylfurfural (HMF):

HMF was extracted from unroasted and microwave roasted NS oil and detected with a 1260 HPLC system (Agilent) according to the method given by Suri et al (2019a). In brief, 0.5 g of NS oil was added to 1 mL methanol (70%), vortexed (1 min) and centrifuged (10,000 rpm) for 5 min. The upper phase was separated and the process repeated under the same conditions three times. Then, the combined upper phase was diluted by adding methanol (70%) to 5 mL, filtered (syringe filter, 0.45 μm) and analyzed using HPLC. HMF quantification (mg/kg) was carried out by acquiring and comparing the peak observed at 285 nm with the standard.

2.8. Chemical properties

The acid, p-anisidine and peroxide (AV, *p*-AV and PV) values of unroasted and microwave roasted NS oil were estimated using Ca 5a-40, Cd 18–90 and Cd 8b-90 methods, respectively of American Oil Chemists' Society (American Oil Chemists' Society, 2017; American Oil Chemists' Society, 1997).

2.9. Oxidative stability Index (OSI)

The unroasted and microwave roasted NS oil was studied for OSI using Rancimat (Metrohm, Switzerland) according to the procedure given by Suri et al (2020). In Brief, 3 g of NS oil heated to 120 ± 1.6 °C in a tube was exposed to air inflow of 20 L/h and OSI was determined as induction period and reported in hours.

2.10. Viscosity

The viscosity of unroasted and microwave roasted NS oil was determined by MCR-102 Rheometer (Anton-Paar, Austria) using procedure described elsewhere (Kaur, Singh, Kaur, & Singh, 2019). The shear rate in the range of 0.1 to 200 s^{-1} using cone plate (CP40) geometry at 30 °C was used for measurement of viscosity and results reported in mPa s.

2.11. Fatty acid composition

The raw and microwave roasted NS oil was studied for FAC by gas chromatography (GC) using the Ce-1 h-05 method (American Oil Chemists' Society, 1997) with minor modifications (Suri et al., 2020). The FA methyl esters were analyzed using Agilent GC (7820A) fitted with FID and DB-WAX column using procedure reported elsewhere (Suri et al., 2019a). The FAs were identified and quantified as a relative percentage (g/100 g) by relating time of retention and peak area with FA methyl esters standard.

2.12. FTIR spectroscopy

FTIR spectra of NS oils were recorded using Vertex-70 FTIR Spectrometer (Brucker, Germany) equipped with ATR and Brucker OPUS software as reported by Suri et al (2020). FTIR spectra ranged from 500 to 4000 cm^{-1} were acquired (64 scans/sample) at a resolution of 4 cm^{-1}. The spectra in triplicate for each NS oil sample was collected, averaged and analyzed.

2.13. Statistical analysis

All experiments were repeated three times and data presented as a mean value of triplicate ± standard deviation. Two-way ANOVA was applied to evaluate the impact of different microwave power and roasting time on NS oil quality characteristics. The relation between different attributes of NS oil was determined on Minitab (14.12.0 version) software using pearson correlation and principal component analysis.

3. Results and discussion

3.1. Oil yield

The oil yield of raw (unroasted) and microwave roasted NS is presented in Table 1. The oil yield from raw NS was 23.4%. Saxena et al (2017) recorded oil yield ranging from 14.7 to 27.0% in twenty-three genotypes of Nigella collected from different regions of India. Another study by Acar et al (2016) reported 20.13% oil yield from raw NS. The higher oil yield (28.0 to 36.4%) was reported by Matthaus & Özcan (2011) in fourteen NS samples collected from Germany and Turkey. F value revealed the greater significant effect of roasting time than the microwave power on NS oil yield ($P \leq 0.005$; Table S1). The oil yield of microwave roasted NS varied from 23.4 to 38.1%, with a maximum after heating at 540 W for 10 min among different microwave treatments. The oil yield was improved by increasing microwave power from 180 to 540 W and declined by increasing microwave power to 720 W. Microwave heating changes cell wall structure, increases porousness and

Table 1
Effect of microwave roasting on oil yield, physicochemical properties, pigments, total phenolic content, radical scavenging activity, Maillard reaction products and oxidative stability index of NS oil.

Parameters	Unroasted (raw)	180 W (5 min)	180 W (10 min)	360 W (5 min)	360 W (10 min)	540 W (5 min)	540 W (10 min)	720 W (5 min)	720 W (10 min)
Oil Yield (%)	23.4 ± 0.10^{a}	26.5 ± 0.10^{b}	30.9 ± 0.10^{c}	32.1 ± 0.06^{c}	37.5 ± 0.10^{e}	37.6 ± 0.10^{e}	38.1 ± 0.10^{e}	35.40 ± 0.02^{d}	33.88 ± 0.07^{d}
a*	0.53 ± 0.07^{d}	−0.08 ± 0.03^{b}	−0.10 ± 0.01^{b}	−0.14 ± 0.01^{b}	−0.18 ± 0.01^{b}	−0.31 ± 0.06^{a}	−0.40 ± 0.02^{a}	−0.36 ± 0.01^{c}	−0.43 ± 0.02^{c}
b*	1.36 ± 0.01^{d}	0.85 ± 0.01^{c}	0.79 ± 0.02^{c}	0.59 ± 0.02^{b}	0.44 ± 0.04^{b}	0.42 ± 0.02^{b}	0.23 ± 0.01^{a}	0.39 ± 0.01^{b}	0.21 ± 0.02^{a}
L*	23.83 ± 0.02^{e}	23.54 ± 0.04^{d}	23.46 ± 0.08^{cd}	23.42 ± 0.09^{c}	23.40 ± 0.01^{c}	23.32 ± 0.03^{c}	23.21 ± 0.06^{b}	23.23 ± 0.03^{b}	22.98 ± 0.04^{a}
Carotenoids (mg/kg)	2.48 ± 0.02^{a}	2.55 ± 0.03^{b}	2.64 ± 0.01^{b}	2.99 ± 0.14^{b}	3.06 ± 0.12^{c}	3.21 ± 0.02^{c}	3.78 ± 0.03^{d}	3.52 ± 0.09^{d}	6.61 ± 0.03^{e}
Chlorophylls (mg/kg)	3.04 ± 0.02^{a}	4.70 ± 0.02^{b}	4.87 ± 0.02^{b}	5.53 ± 0.07^{c}	5.86 ± 0.05^{c}	6.04 ± 0.01^{d}	7.05 ± 0.03^{e}	6.43 ± 0.06^{d}	9.01 ± 0.05^{f}
TPC (μg GAE/mL)	100.02 ± 1.94^{b}	88.19 ± 0.96^{a}	81.27 ± 0.94^{a}	131.64 ± 1.72^{c}	160.52 ± 0.69^{d}	154.94 ± 1.71^{d}	185.37 ± 0.88^{f}	174.22 ± 1.87^{e}	208.4 ± 2.01^{g}
RSA (% DPPH inhibition)	60.21 ± 0.19^{b}	59.38 ± 0.31^{a}	58.66 ± 0.23^{a}	61.60 ± 0.30^{b}	65.72 ± 0.16^{c}	64.12 ± 0.04^{c}	68.11 ± 0.08^{d}	69.58 ± 0.11^{d}	75.22 ± 0.40^{e}
BI (Abs_{420nm})	0.240 ± 0.001^{a}	0.257 ± 0.009^{a}	0.273 ± 0.019^{a}	0.294 ± 0.037^{a}	0.339 ± 0.023^{b}	0.344 ± 0.028^{b}	0.359 ± 0.023^{c}	0.441 ± 0.003^{d}	0.633 ± 0.020^{e}
HMF (mg/kg)	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.53 ± 0.02^{b}	0.34 ± 0.02^{a}	0.79 ± 0.01^{c}	1.77 ± 0.01^{d}	2.08 ± 0.01^{e}
AV (mg KOH/g)	3.84 ± 0.04^{f}	3.18 ± 0.04^{d}	2.91 ± 0.03^{d}	2.69 ± 0.04^{c}	1.97 ± 0.04^{b}	1.53 ± 0.04^{a}	1.30 ± 0.02^{a}	3.17 ± 0.04^{d}	3.23 ± 0.03^{e}
PV ($meqO_2$/Kg)	5.91 ± 0.06^{e}	7.57 ± 0.17^{g}	8.17 ± 0.04^{h}	8.54 ± 0.09^{i}	6.07 ± 0.10^{f}	5.74 ± 0.21^{d}	5.32 ± 0.03^{c}	4.20 ± 0.02^{b}	3.18 ± 0.03^{a}
p-AV	1.42 ± 0.08^{a}	2.01 ± 0.13^{b}	2.43 ± 0.07^{b}	2.65 ± 0.10^{b}	2.81 ± 0.03^{b}	2.90 ± 0.04^{b}	3.44 ± 0.07^{c}	6.00 ± 0.10^{d}	6.61 ± 0.05^{d}
OSI (h)	6.84 ± 0.06^{a}	7.22 ± 0.08^{b}	8.45 ± 0.05^{c}	9.16 ± 0.06^{c}	10.21 ± 0.03^{d}	10.93 ± 0.10^{e}	11.42 ± 0.07^{e}	10.99 ± 0.06^{e}	13.95 ± 0.08^{f}
Viscosity (mPa.s)	40.26 ± 0.06^{a}	41.50 ± 0.02^{b}	43.62 ± 0.02^{d}	43.37 ± 0.04^{c}	43.18 ± 0.07^{c}	42.75 ± 0.07^{c}	44.89 ± 0.04^{d}	48.51 ± 0.02^{e}	51.69 ± 0.06^{f}

Data values with the same alphabetic superscript in a row do not vary significantly ($P < 0.05$) among microwave roasting treatments. AV = Acid Value; PV = Peroxide Value; *p*-AV = *p*-Anisidine Value; TPC = Total Phenolic Content; RSA = Radical Scavenging Activity; HMF = 5-Hydroxymethylfurfural; BI = Browning Index; OSI = Oxidative stability index.

permeability, thus eases more efficient extraction of oil from oilseeds (Fathi-Achachlouei et al., 2019; Suri et al., 2020). Microwave roasting had increased oil yield from apricot kernel and flaxseed in recent studies (Al Juhaimi et al., 2018; Suri et al., 2020). While the decline in oil yield observed at higher microwave power might be due to high moisture loss which increases brittleness, decreases plasticity and elasticity of oilseeds. The high brittleness may obstruct the release of oil during the extraction process (Suri et al., 2020). The oil yield displayed a significant positive ($p < 0.05$) correlation with microwave roasting ($r = 0.782$) as shown in Table 3. A similar positive correlation of flaxseed oil yield with microwave roasting was reported by Suri et al (2020).

3.2. Oil color

The a*, b* and L* values of raw (unroasted) and microwave roasted NS oil are presented in Table 1. The raw (unroasted) NS oil exhibited a*, b* and L* values of 0.53, 1.36 and 23.83, respectively. The oil obtained from unroasted NS was light and bright in color compared to oil from microwave roasted NS as indicated by b* and L* values. The color values depend on the concentration and type of pigments present in the edible oils. It influences consumer perception and is widely used in food industries to directly relate the quality of products. The extraction of chlorophylls and carotenoids gives the green and yellow color to oils (Suri et al., 2019). The -a* value increased while b* and L* value declined by increasing microwave power and roasting time. A greater significant ($P \leq 0.005$) effect of roasting time than the microwave power on a*, b* and L* values of NS oil was observed (Table S1). The oil of microwave roasted NS at 720 W for 10 min exhibited the lowest L*, a* and b* (22.98, −0.43 and 0.21, respectively) values among roasting treatments. The increase in -a* value might be related to enhanced extraction of chlorophyll pigments in NS oil after microwave roasting. While, a decline in b* and L* values (darkening of oils) relates with MRPs formation and enhanced release of color pigments from roasted oilseeds (Suri et al., 2019a; Suri et al., 2019b; Suri et al., 2020). A similar variation in color of roasted pistachios oil was observed by Rabadán, Gallardo-Guerrero, Gandul-Rojas, Álvarez-Ortí, & Pardo, (2018). The L*and b* values exhibited highly significant ($p < 0.005$) negative correlation ($r = -0.947$ and -0.930, respectively) while a* value showed significant ($p < 0.05$) negative correlation ($r = -0.857$) with microwave roasting (Table 3). Suri et al. (2020) also presented the significant negative correlation of b* and L* values of flaxseed oil with microwave roasting.

3.3. Chlorophylls and carotenoids

The level of carotenoids and chlorophylls in unroasted and microwave roasted NS oil is given in Table 1. The level of carotenoids and chlorophylls in unroasted NS oil was 2.48 and 3.04 mg/kg, respectively. The previous study by Kiralan, Özkan, Bayrak, & Ramadan (2014) reported lower chlorophylls (0.30 to 0.81 mg/kg) and carotenoids (0.18 to 0.40 mg/kg) content in oil extracted from NS using different extraction methods. While another study by Mazaheri, Torbati, Azadmard-Damirchi, & Savage (2019b) reported higher carotenoids (3.79 mg/kg) in NS oil. The difference in chlorophylls and carotenoids contents might be due to differences in genotype, maturity stage, harvesting time and processing method used for oil extraction. Microwave power and roasting time significantly influenced the level of chlorophyll and carotenoid pigments in NS oil ($P < 0.05$). In microwave roasted NS oil, the chlorophylls and carotenoids content varied from 4.70 to 9.01 mg/kg and 2.55 to 6.61 mg/kg, respectively. F value revealed the greater significant effect of roasting time than the microwave power on chlorophylls and carotenoids contents of NS oil ($P \leq 0.005$, Table S1). The maximum increase in carotenoids and chlorophylls was observed in oil of NS roasted at 720 W for 10 min. Similar results of changes in pigment contents with microwave roasting were reported for flaxseed (Suri et al., 2020), rapeseed (Rękas et al., 2017) and milk thistle (Fathi-Achachlouei et al., 2019) oils. The chlorophyll and carotenoid pigments are mainly present in a complex form with proteins. During microwave treatment, the complex of pigments and bound proteins disintegrate, which results in more accessibility and extraction of pigments in oils. These results concur with previous studies reporting the enhanced release of chlorophyll and carotenoid pigments in oils after microwave roasting (Rękas et al. 2017; Mazaheri et al., 2019a). The chlorophylls of NS oil exhibited a highly significant positive ($p \leq 0.005$) correlation with microwave roasting ($r = 0.933$) and negative correlation with b* ($r = -0.961$) and L* ($r = -0.984$) values as shown in Table 3. The carotenoids of NS oil exhibited a significant positive ($p \leq 0.05$) correlation with microwave roasting ($r = 0.788$) and negative correlation with b* ($r = -0.739$) and L* ($r = -0.823$) values. Similarly, the positive correlation of microwave roasting with the level of chlorophylls and carotenoids of flaxseed oil was reported by Suri et al (2020).

3.4. TPC

TPC of raw (unroasted) and microwave roasted NS oil is presented in Table 1. TPC in raw NS oil was 100.02 μg GAE/mL. The previous study reported higher phenolic content (129 to 212 μg GAE/mL) in oil obtained from seeds of twenty-three Nigella genotypes (Saxena et al., 2017). TPC showed significant variation in oil extracted from NS roasted using different microwave power and durations. A greater significant effect of roasting time than the microwave power on TPC of NS oil was observed ($P \leq 0.005$; Table S1). TPC of NS oil was decreased by roasting at low microwave power (180 W) and increased by roasting at high microwave power. The highest TPC (208.4 μg GAE/mL) was recorded in oil of NS heated at 720 W for 10 min. A comparable effect of roasting treatments on TPC was presented for the almond kernel (Lin et al., 2016) and wheat germ (Zou et al., 2018) oils. Microwave roasting may destroy the cell structure of NS and release bound phenolics thus increases TPC in oil. Yang et al (2013) reported a significant positive correlation of microwave pre-treatment time with TPC in microwave roasted rapeseeds oil. Chemical alteration and release of phenolic compounds from bound structures at higher microwave roasting powers increases TPC in oil (Suri et al., 2020). TPC exhibited highly positive significant ($p \leq 0.005$) correlation with microwave roasting and chlorophylls ($r = 0.934$ and 0.878, respectively) while significant positive correlation with carotenoids ($r = 0.783$, $p \leq 0.05$) of NS oil (Table 3). Yang et al (2013) also observed positive correlation of microwave roasting with TPC in rapeseed oil.

3.5. RSA

The RSA (% DPPH inhibition) of raw and microwave roasted NS oil is shown in Table 1. A significant positive impact of microwave power and roasting time on the RSA of NS oil was observed. The RSA of unroasted NS oil was 60.21% while it varied from 58.66 to 75.22% for microwave roasted NS oil. The previous study by Kiralan et al. (2014) recorded RSA varying from 61.69 to 78.45% for oil extracted from raw NS using different extraction methods. A greater significant effect of roasting time than the microwave power on RSA of NS oil was observed ($P \leq 0.005$; Table S1). The lowest and highest RSA was recorded for oils of NS roasted at 180 W for 10 min and 720 W for 10 min, respectively. Similarly, an increase in RSA by increasing microwave power was reported for the apricot kernel (Al Juhaimi et al., 2018) and flaxseed (Suri et al., 2020) oils. Similar improvements in RSA were recorded for a roasted walnut (Gao, Liu, Jin, & Wang, 2018) and wheat germ (Zou et al., 2018) oils. The higher RSA of oils from NS heated at 720 W for 10 min relates with higher TPC (208.4 μg GAE/mL). Phenolic compounds have importance in the shelf life of oil as they mainly determine the resistance of the oil to auto-oxidation (Cheikh-Rouhou et al., 2007). RSA displayed a highly positive significant ($p \leq 0.005$) correlation with microwave roasting, TPC and carotenoids ($r = 0.930$, 0.951 and 0.893, respectively) of NS oil as shown in Table 3. A similar significant positive

correlation was observed between TPC and RSA of microwave roasted chia seed (Özcan et al., 2019) and flaxseed (Suri et al., 2020) oils.

3.6. MRPs

The BI of unroasted and microwave roasted NS oil is presented in Table 1. The unroasted NS oil exhibited a BI of 0.240. The BI was slightly elevated by increasing microwave power and roasting time indicating a higher extent of Maillard reaction in roasted NS oil. Similar changes in BI of microwave roasted flaxseed oil were reported by Suri et al (2020). A greater significant effect of roasting time than the microwave power on BI of NS oil was observed ($P \leq 0.005$; Table S1). The highest BI (0.633) was noted in oil of NS heated at 720 W for 10 min. A comparable effect of roasting on BI was recorded for peanut (Suri et al., 2019b), wheat germ (Zou et al., 2018) and almond kernel (Lin et al., 2016) oils. The reducing sugars interact with free amino acids or lipid oxidative products and produce MRPs during heating and their release while extraction increases BI of oils (Zou et al., 2018). BI presented a highly positive significant ($p \leq 0.005$) correlation with RSA, microwave roasting and TPC ($r = 0.948$, 0.874 and 0.834, respectively) indicating that the increment in BI with roasting also increases the antioxidant activity of NS oil (Table 3). The scavenging activity, reducing power and metal ion chelating properties of MRPs (melanoidins and reductones) might contribute in increasing RSA (Zhou et al., 2016; Fu et al., 2020). A similar correlation of BI with RSA of flaxseed oil was reported by Suri et al (2020).

The formation of HMF, a Maillard reaction product intermediate in unroasted and microwave roasted NS oil was estimated (Table 1). The HMF formation was not observed in oil of unroasted NS and those roasted at 360 W (5 min) and 180 W (5 and 10 min). The oil of NS heated at 360 W for 10 min had low HMF content (0.53 mg/kg). The microwave power showed a greater significant effect than the roasting time on the level of HMF in NS oil ($P \leq 0.005$, Table S1). HMF level significantly increased in NS oil by increasing microwave power from 540 to 720 W, with the highest level (2.08 mg/kg) observed for those heated at 720 W for 10 min. A similar effect on HMF content was presented for roasted wheat germ (Zou et al., 2018), peanut (Suri et al., 2019b) and flaxseed (Suri et al., 2020) oils. HMF exhibits favorable biological effects such as anti-allergic, antioxidative, anti-inflammatory, hepatoprotective, anti-hypoxic, anti-hyperuricemic and anti-sickling effects (Li et al., 2015). HMF content exhibited highly positive significant correlation ($p \leq 0.005$) with RSA, BI and microwave roasting ($r = 0.949$, 0.941 and 0.888, respectively) while positive significant ($p \leq 0.05$) correlation with TPC ($r = 0.831$) of NS oil (Table 3). Thus, higher HMF content improves the antioxidant activity of NS oil. The positive correlation observed between BI and HMF indicate Maillard reaction in microwave roasted NS oil.

3.7. Chemical properties

3.7.1. Acid value (AV)

AV acts as a measure of decomposition reactions going on in oil. AV of raw and microwave roasted NS oil is presented in Table 1. The AV of 3.84 mg KOH/g was observed for unroasted NS oil. The previous report by Mazaheri et al. (2019a) recorded higher AV (5.20 mg KOH/g) for raw NS oil. The microwave roasted NS oil showed AV from 1.30 to 3.23 mg KOH/g, the lowest observed for those heated at 540 W for 10 min (Fig. S1a). A greater significant effect of roasting time than the microwave power on AV of NS oil was observed ($P \leq 0.005$; Table S1). The AV of NS oil decreased gradually with an increase in microwave power from 180 to 540 W and then increased at 720 W. The decrease in AV at low microwave power (180 to 540 W) could be attributed to the gradual increase in the inactivation process of oxidative enzymes. While an increase in AV at higher microwave power (720 W) might be due to the cleavage of the existing bond between glycerol and fatty acids and the release of free fatty acids in oil. AV of unroasted and microwave roasted NS oil was lower than the permissible limit (4 mg KOH/g) recommended by codex alimentarius committee for oils (FAO/WHO, 2009). AV is an important edible oil quality parameter that determines triglyceride hydrolysis and the production of free fatty acids in oils (Tenyang et al., 2017). Our results concur with a recently published work on microwave treatment (at 800 W for 4 min) reporting a decline in AV (from 4.24 to 2.16 mg KOH/g) of milk thistle seed oil (Fathi-Achachlouei et al., 2019). Similar results of decrease in AV were recorded for the roasted sesame (Ji, Liu, Shi, Wang, Wang, 2019) and wheat germ (Zou et al., 2018) oils. As shown in Table 3A, a highly significant negative correlation ($p \leq 0.005$) of AV with OSI ($r = -0.984$) was observed. This correlation suggests that oxidative stability increases with a decline in AV of microwave roasted NS oil.

3.7.2. PV

PV measures the formation of peroxides due to degradation in oils. The PV of raw and microwave roasted NS oil is presented in Table 1. The oil extracted from raw (unroasted) NS exhibited a PV of 5.91 meqO_2/kg. Mazaheri et al. (2019a) recorded a PV of 5.60 meqO_2/kg for raw NS oil. The PV of microwave roasted NS oil ranged between 3.18 and 8.54 meq O_2/kg, with the lowest for those heated at 720 W for 10 min (Fig. S1b). Microwave power showed a greater significant impact than the roasting time on PV of NS oil ($P \leq 0.005$; Table S1). PV reported for microwave roasted and unroasted NS oil was lower than the limit (10 meqO_2/kg) recommended by codex alimentarius committee for oils (FAO/WHO, 2009). The PV was increased by roasting at low microwave power (180 and 360 W) and decreased by roasting at high microwave power (540 and 720 W). The initial increase in PV of microwave roasted NS oil might be due to an attack of free radicals on unsaturated FAs and accumulation of peroxides. At higher microwave power and roasting time, the primary oxidation products (peroxides) generated may get decomposed and reduced to secondary oxidation products (aldehydes, ketones, epoxy-hydroxy, and epoxy-hydroperoxy) due to their instability. An earlier study recorded a similar trend in PV of sesame seed oil with the extension of roasted time and temperature (Ji et al., 2019). The decline in PV of NS oil might be related to an increase in TPC while roasting at high microwave power and roasting time (720 W for 10 min). A similar decline in PV with an increase in TPC of oil obtained from microwave roasted milk thistle seeds was reported by Fathi-Achachlouei et al (2019). PV exhibited a significant negative ($p \leq 0.05$) correlation with microwave roasting and OSI ($r = -0.758$ and -0.740, respectively) as shown in Table 3. Moreover, a highly significant negative ($p \leq 0.005$) correlation of PV with RSA and TPC ($r = -0.888$ and -0.803, respectively) was also observed. Özcan et al. (2019) also showed a similar negative correlation of PV with the antioxidant activity of oil obtained from microwave roasted chia seeds.

3.7.3. p-AV

The *p*-AV indicates production of secondary oxidative products in oils due to lipid oxidation (Kaur, Singh, Kaur, & Singh, 2020). The *p*-AV of raw and microwave roasted NS oil is presented in Table 1. The raw NS oil had *p*-AV of 1.42. The study conducted by Mazaheri et al. (2019b) recorded *p*-AV of 1.06 for raw NS oil. The *p*-AV of NS oil increases with an increment in microwave power and roasting time. This indicates the formation of secondary oxidative products in microwave roasted NS oil. The microwave roasted NS oils exhibited *p*-AV in the range of 2.01 to 6.61, with the highest value for those heated at 720 W for 10 min (Fig. S1c). The increase in *p*-AV relates with the degradation of primary hydroperoxides into non-volatile carbonyl compounds during the microwave roasting process. Hashemi et al (2017) also noted an increment in *p*-AV of oil extracted from microwave-treated novel oilseed sources. Similar results of increment in *p*-AV were reported for oils extracted from roasted rapeseed (Rękas et al., 2017) and sesame seeds (Tenyang et al., 2017). A highly significant positive ($p \leq 0.005$) correlation of *p*-AV with microwave roasting ($r = 0.901$) and negative ($p \leq 0.005$) correlation with PUFAs ($r = -0.928$) of NS oil was observed (Table 3).

Similar relation of roasting treatment and PUFAs with *p*-AV of sesame oil was reported by Tenyang et al (2017).

3.8. OSI

OSI indicates development of lipid oxidative products and the deterioration of oil during storage, heating and frying (Kaur et al., 2020). OSI of unroasted NS oil was 6.84 h. However, the previous study by Kiralan et al. (2014) reported lower OSI (3.48 h) for oil extracted from NS by cold pressing. Microwave roasting significantly affected the OSI of NS oil as shown in Table 1. A greater significant impact of roasting time than the microwave power was noticed on the OSI of NS oil ($P \leq 0.005$; Table S1). OSI of microwave roasted NS oil ranged from 7.22 to 13.95 h, with a maximum for NS heated at 720 W for 10 min (Fig. S1d). The improved OSI may be due to an increase in the level of phenolic antioxidants, chlorophylls and carotenoids in microwave roasted NS oil. The previous study by de Carvalho et al. (2018) reported microwave heating (820 W for 1 min) enhances the release of phenolic compounds and increases the OSI of cashew nut oils. Similar results were observed in previously published reports on microwave roasted rapeseed (Rękas et al., 2017) and flaxseed (Suri et al., 2020) oils. OSI presented a highly positive significant ($p \leq 0.005$) correlation with microwave roasting, chlorophylls, TPC, carotenoids, BI and HMF ($r = 0.966, 0.966, 0.936, 0.873, 0.905$ and 0.839, respectively) content of NS oil as shown in Table 3. The above correlations indicate that pigments, phenolics and MRPs have a role in improving the OSI of NS oil. Zhou et al (2016) has also related an increase in OSI of microwave roasted walnut oils with Maillard reaction and its antioxidant products (melanoidins). A similar positive correlation of microwave roasting with OSI was observed for rapeseed (Yang et al., 2013) and linseed (Suri et al., 2020) oils.

3.9. Viscosity

The viscosity of raw (unroasted) and microwave roasted NS oil is presented in Table 1. The oil of raw NS had a viscosity of 40.26 mPa s. The viscosity observed for NS oil was higher compared to the previous report (Cheikh-Rouhou et al., 2007). This might be due to the difference in the proportion of long-chain SFAs (C20:0 and C24:0) as they contribute significantly to the viscosity of oils (Kaur et al., 2020). The viscosity of NS oil increases with the rise in microwave power and roasting time. A greater significant impact of roasting time than the microwave power was observed on the viscosity of NS oil ($P \leq 0.005$; Table S1). The viscosity of microwave roasted NS oils ranged from 41.50 to 51.69 mPa s, with the maximum for NS heated at 720 W for 10 min. This is in agreement with Özcan et al. (2019) who reported an increment in viscosity of roasted chia seed oils with an increase in microwave power from 180 to 900 W. A comparable impact of roasting on the viscosity of pistachio oils was presented by Rabadán et al (2018). The increment in viscosity of microwave roasted NS oils might be related to changes in the proportion of FAs (saturated FAs increased and unsaturated FAs decreased) as shown in Table 2. The unsaturated FAs have a lower dynamic viscosity than the saturated FAs due to their freely filled structures (Kaur et al., 2019). The formation of polymers and dimers during microwave heating also contributes to increasing the viscosity of oil Özcan et al. (2019). As shown in Table 3, the viscosity of NS oil showed a highly significant positive correlation ($p \leq 0.005$) with microwave roasting ($r = 0.878$) and SFAs ($r = 0.869$) and negative correlation ($p \leq 0.005$) with PUFAs ($r = -0.907$).

3.10. FAC

Table 2 shows FAC of oil from raw (unroasted) and microwave roasted NS. The major saturated FAs present in NS oil were palmitic (C16:0) and stearic (C18:0) while oleic (C18:1n9c), linoleic (C18:2n6c) and eicosadienoic (C20:2n6c) were the major unsaturated FAs. Myristic (C14:0), arachidic (C20:0), eicosanoic (C20:1n9c) and lignoceric (C24:0) acids were detected in a very small amount in NS oil. The dominant FAs of NS oil were linoleic (57.39%) and oleic (23.98%) acids and their contents are comparable with previous reports (Matthaus & Özcan, 2011; Al Juhaimi, Matthaeus, Ghafoor, ElBabiker, & Ozcan, 2016; Al Juhaimi et al., 2018). The unroasted NS oil had higher proportions of polyunsaturated (PUFAs) followed by monounsaturated

Table 2
Effect of microwave roasting on fatty acid composition of NS oil.

Fatty acids	Unroasted (raw)	180 W (5 min)	180 W (10 min)	360 W (5 min)	360 W (10 min)	540 W (5 min)	540 W (10 min)	720 W (5 min)	720 W (10 min)
C14:0 (Myristic)	0.17 ± 0.01^{a}	0.18 ± 0.01^{ab}	0.19 ± 0.01^{b}	0.20 ± 0.01^{c}	0.20 ± 0.01^{c}	0.21 ± 0.01^{d}	0.22 ± 0.01^{e}	0.24 ± 0.01^{f}	0.26 ± 0.01^{g}
C16:0 (Palmitic)	11.63 ± 0.01^{a}	11.81 ± 0.01^{b}	11.84 ± 0.01^{b}	11.99 ± 0.01^{c}	12.09 ± 0.06^{d}	12.14 ± 0.04^{d}	12.25 ± 0.01^{e}	12.17 ± 0.01^{e}	12.32 ± 0.01^{f}
C18:0 (Stearic)	2.86 ± 0.01^{a}	2.89 ± 0.01^{b}	2.92 ± 0.01^{bc}	2.95 ± 0.01^{c}	2.96 ± 0.01^{cd}	2.98 ± 0.01^{d}	3.01 ± 0.02^{e}	3.04 ± 0.01^{ef}	3.09 ± 0.01^{f}
C20:0 (Arachidic)	0.20 ± 0.01^{a}	0.20 ± 0.01^{a}	0.20 ± 0.01^{a}	0.20 ± 0.01^{b}	0.22 ± 0.01^{b}	0.22 ± 0.01^{b}	0.24 ± 0.01^{c}	0.24 ± 0.01^{c}	0.27 ± 0.01^{d}
C24:0 (Lignoceric)	0.25 ± 0.01^{a}	0.26 ± 0.01^{ab}	0.29 ± 0.02^{c}	0.29 ± 0.01^{c}	0.29 ± 0.01^{c}	0.30 ± 0.02^{cd}	0.32 ± 0.00^{de}	0.33 ± 0.01^{e}	0.38 ± 0.01^{f}
C16:1 (Palmitoleic)	0.21 ± 0.01^{b}	0.22 ± 0.01^{c}	0.22 ± 0.01^{c}	0.22 ± 0.01^{c}	0.22 ± 0.01^{c}	0.20 ± 0.01^{a}	0.20 ± 0.01^{a}	0.20 ± 0.01^{a}	0.20 ± 0.01^{a}
C18:1n9c (Oleic)	23.98 ± 0.01^{e}	23.91 ± 0.03^{e}	23.77 ± 0.02^{d}	23.63 ± 0.03^{c}	23.60 ± 0.03^{c}	23.52 ± 0.02^{b}	23.45 ± 0.01^{b}	23.42 ± 0.01^{b}	23.38 ± 0.01^{a}
C20:1n9c (Eicosanoic)	0.39 ± 0.01^{d}	0.38 ± 0.01^{cd}	0.37 ± 0.01^{c}	0.36 ± 0.01^{bc}	0.36 ± 0.01^{bc}	0.35 ± 0.01^{b}	0.34 ± 0.01^{ab}	0.33 ± 0.01^{a}	0.32 ± 0.01^{a}
C18:2n6c (Linoleic)	57.39 ± 0.01^{f}	57.25 ± 0.02^{e}	57.13 ± 0.03^{d}	57.06 ± 0.03^{d}	56.99 ± 0.01^{c}	56.96 ± 0.01^{c}	56.91 ± 0.03^{c}	56.73 ± 0.03^{b}	56.64 ± 0.03^{a}
C20:2n6c (Eicosadienoic)	2.57 ± 0.01^{f}	2.56 ± 0.01^{ef}	2.54 ± 0.02^{e}	2.52 ± 0.02^{de}	2.47 ± 0.01^{c}	2.46 ± 0.01^{c}	2.45 ± 0.02^{c}	2.42 ± 0.01^{b}	2.39 ± 0.01^{a}
C18:3 n3c (γ-Linolenic)	0.25 ± 0.01^{e}	0.25 ± 0.01^{e}	0.23 ± 0.01^{d}	0.22 ± 0.01^{c}	0.22 ± 0.01^{c}	0.22 ± 0.01^{c}	0.21 ± 0.01^{b}	0.21 ± 0.01^{b}	0.19 ± 0.01^{a}
SFAs	15.10 ± 0.03^{a}	15.34 ± 0.02^{b}	15.44 ± 0.03^{b}	15.62 ± 0.02^{c}	15.75 ± 0.01^{cd}	15.84 ± 0.03^{d}	16.03 ± 0.01^{e}	16.02 ± 0.02^{e}	16.32 ± 0.01^{f}
MUFAs	24.58 ± 0.01^{e}	24.51 ± 0.05^{e}	24.36 ± 0.02^{d}	24.22 ± 0.04^{c}	24.18 ± 0.02^{c}	24.07 ± 0.01^{b}	23.99 ± 0.03^{a}	23.95 ± 0.01^{a}	23.90 ± 0.01^{a}
PUFAs	60.21 ± 0.01^{f}	60.08 ± 0.02^{e}	59.94 ± 0.04^{d}	59.83 ± 0.04^{d}	59.71 ± 0.02^{c}	59.68 ± 0.02^{c}	59.62 ± 0.04^{c}	59.40 ± 0.03^{ab}	59.29 ± 0.03^{a}

Data values with the same alphabetic superscript in a row do not vary significantly ($P < 0.05$) among microwave roasting treatments. SFAs = Saturated Fatty acids, MUFAs = Monounsaturated Fatty Acids, PUFAs = Polyunsaturated Fatty Acids.

Table 3
Pearson correlation coefficients of various parameters of unroasted and microwave roasted NS oil.

	Roasting	L*	b*	OSI	PV	*p*-AV	Chlorophylls	Carotenoids	TPC	RSA	BI	HMF	MUFAs	PUFAs	SFAs
Oil Yield	0.782*														
L*	−0.947**														
a*	−0.857*														
b*	−0.930**														
AV				−0.984**											
PV	−0.758*														
p-AV	0.901**														
Chlorophylls	0.933**	−0.984**	−0.961**												
Carotenoids	0.788*	−0.823*	−0.739*												
TPC	0.934**				−0.803**		0.878**	0.783*							
RSA	0.930**				−0.888**			0.893**	0.951**						
BI	0.874**								0.834**	0.948**					
HMF	0.888**	−0.806*	−0.721*		−0.886**	0.977**	0.818*	0.837**	0.831*	0.949**	0.941**				
MUFAs	−0.986**														
PUFAs	−0.991**					−0.928**									
SFAs	0.989**														
OSI	0.966**				−0.740*		0.966**	0.873**	0.936**		0.905**	0.839**	−0.952**	−0.957**	
Viscosity	0.878**													−0.907**	0.869**

*p < 0.05; **p < 0.005, PV = Peroxide Value, AV = Acid Value, *p*-AV = *p*-Anisidine value, OSI = Oxidative stability Index, TPC = Total Phenolic Content, RSA = Radical Scavenging Activity; HMF = 5-Hydroxymethylfurfural, MUFAs = Monounsaturated Fatty Acids, PUFAs = Polyunsaturated Fatty Acids, SFAs = Saturated Fatty acids, BI = Browning Index.

(MUFAs) and saturated (SFAs) fatty acids (60.21, 24.58 and 15.10%, respectively). Rudzińska, Hassanein, Abdel–Razek, Ratusz, & Siger (2016) reported 61.3% PUFAs, 22.1% MUFAs and 16.3% SFAs in raw NS oil. Kiralan et al (2014) also recorded similar contents of FAs in cold-pressed NS oil. The high proportion of PUFAs and a low proportion of SFAs make NS oil a nutraceutical component for functional foods (Kiralan et al., 2014; Suri et al., 2019). FAC of NS oil was slightly changed by increasing microwave power (from 180 to 720 W) and time (from 5 to 10 min). Roasting time displayed a greater significant impact than the microwave power on MUFAs, PUFAs and SFAs levels of NS oil ($P \leq 0.005$; Table S1). The proportion of MUFAs (23.90%) and PUFAs (59.29%) was slightly decreased while SFAs content increased (16.32%) in NS oil by microwave roasting at 720 W for 10 min (Table 2). The degradation of unsaturated FAs at higher microwave power and roasting time might change the proportion of FAs (Suri et al., 2020). Similar results were recorded for microwave roasted flaxseed (Suri et al., 2020), milk thistle (Fathi-Achachlouei et al., 2019) and apricot kernel (Al Juhaimi et al., 2018) oils. A highly significant negative correlation ($p \leq 0.005$) of microwave roasting with MUFAs ($r = -0.986$) and PUFAs ($r = -0.991$) while a positive correlation ($p \leq 0.005$) with SFAs ($r = 0.989$) was observed (Table 3). Similar correlations of microwave roasting with unsaturated and saturated FAs of flaxseed oil were observed by Suri et al. (2020). Moreover, OSI displayed a highly significant negative ($p \leq 0.005$) correlation with MUFAs and PUFAs (r = -0.952 and −0.957, respectively) indicating that the higher level of unsaturated FAs contributes to lower OSI of oils (Table 3). Neđeral et al (2012) also observed a negative correlation of OSI with PUFAs of roasted pumpkin seed oil.

3.11. FTIR spectroscopy

IR spectra of oils extracted from microwave roasted and unroasted NS were acquired in the mid-infrared region of 3500–500 cm^{-1}. The presence or absence of functional groups was confirmed in two regions (3100–2800 and 1800–700 cm^{-1}) of the FTIR spectra as described in previous studies of oils (Suri et al., 2020; Kaur et al., 2020). The three peaks observed in first region (at wavenumbers of 3008 cm^{-1}, 2929 cm^{-1} and 2854 cm^{-1}) and eight peaks in second region (at wavenumbers of 1745 cm^{-1}, 1651 cm^{-1}, 1465 cm^{-1}, 1371 cm^{-1}, 1237 cm^{-1}, 1161 cm^{-1}, 1095 cm^{-1} and 725 cm^{-1}) are shown in FTIR spectra of NS oil (Fig. 1A). The shoulder peak noted at 3008 cm^{-1} (attributed to =C—H (cis) stretching (symmetric) vibration), sharp peak at 2929 cm^{-1} (designated to –C—H (CH_2) stretching (asymmetric) vibration), sharp shoulder peak at 2854 cm^{-1} (attributed to –C—H (CH_2) stretching (symmetric) vibration), sharp peaks at 1745 cm^{-1} (assigned to –C═O (ester) stretching vibration), a small peak at 1651 cm^{-1} (*cis*-olefins C═C stretching vibration), peak at 1465 cm^{-1} (designated to bending vibration of –C—H (CH_2)), a small peak at 1371 cm^{-1} (attributed to –C—H (CH_2) bending (symmetric) vibration), peak at 1161 cm^{-1} and shoulder peaks at 1237 cm^{-1} and 1095 cm^{-1} (designated to C—O stretching vibration) and peak at 725 cm^{-1} (attributed to *cis*–HC = CH − bending (out-of-plane) vibration and $–(CH_2)_n–$ rocking vibration). The peaks observed in the first region (3100–2800 cm^{-1}) are mainly attributed to hydrogen bond stretching vibrations of *cis*-olefinic double-bonds and aliphatic CH_2 groups of triglycerides (Kaur et al., 2020). While the peaks of the second region (1800–700 cm^{-1}) relate with conjugated bonds of triglycerides, bending and stretching vibrations of aliphatic groups of triglycerides and generation of oxidative products (Suri et al., 2020).

Fig. 1B shows IR spectra of raw (unroasted) and microwave roasted NS oil. The visual investigation of IR spectra of microwave roasted and unroasted NS oils does not show any marked difference. While comparing peak intensities, a slight change was noticed in certain IR spectral peaks of microwave roasting NS oils. A minor increment in the intensities of two peaks (2854 cm^{-1} and 2929 cm^{-1}) and a slight decline in the intensity of peak at 3008 cm^{-1} were noticed in oil of microwave roasted NS (Fig. 1B). The 3008 cm^{-1} peak is associated with *cis*-olefinic groups of unsaturated FAs while peaks at 2854 cm^{-1} and 2929 cm^{-1}

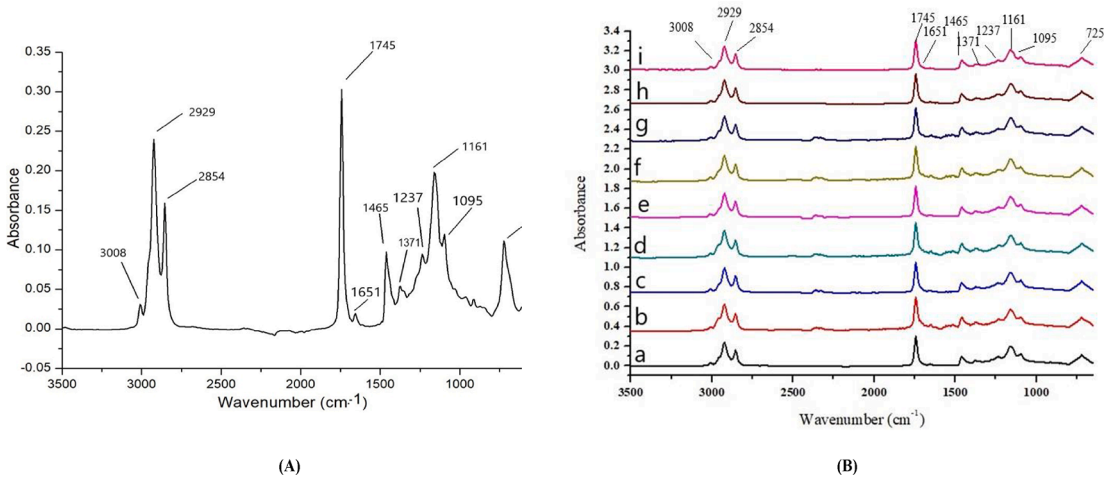

Fig. 1. (A) FTIR spectrum of NS oil at room temperature (25 °C) and (B) FTIR spectra of unroasted (raw) and microwave roasted NS oil (a: unroasted; b: 180 W for 5 min; c: 180 W for 10 min; d: 360 W for 5 min; e: 360 W for 10 min; f: 540 W for 5 min; g: 540 W for 10 min; h: 720 W for 5 min; i: 720 W for 10 min) NS oils.

relates to the level of saturated FAs (CH stretching vibration of aliphatic groups) in oils. The level of saturated FAs (stearic, palmitic and myristic acids) increased while the content of unsaturated FAs (eicosadienoic and eicosanoic acids) decreased in NS oil by increasing microwave power and roasting time (Table 2). Similar relation of saturated and unsaturated FAs with intensities of peaks at 2854 cm^{-1}, 2929 cm^{-1} and 3008 cm^{-1} were reported elsewhere (Suri et al., 2020; Kaur et al., 2020). The oil from NS roasted at a microwave power of 720 W for 5 and 10 min showed an increase in the intensity of peak at 1745 cm^{-1} compared to unroasted oil. Similarly, the peak near 1161 cm^{-1} was also affected by microwave roasting and its intensity increased slightly at higher microwave power and roasting time. A similar increase in the intensity of peaks at 1745 and 1161 cm^{-1} with microwave roasting was reported for flaxseed oil (Suri et al., 2020). The two peaks (1745 cm^{-1} and 1161 cm^{-1}) could be related to the formation of secondary oxidative products at higher microwave power and roasting time as indicated by changes in *p*-AV of NS oil (Table 1). The intensities of peaks noted at 725 cm^{-1}, 1095 cm^{-1}, 1237 cm^{-1}, 1371 cm^{-1}, 1465 cm^{-1} and 1651 cm^{-1} for oil of NS roasted at 720 W for 10 min exhibited no major variation in comparison with unroasted NS oil. Suri et al (2020) also reported similar results for these peaks at high microwave power and roasted time in flaxseed oils. Suri et al (2019a) also reported no change in intensity of these peaks in oils of infrared and dry air roasted black cumin seeds.

3.12. Principal component analysis (PCA)

The impact of microwave roasting (power and time) on physicochemical properties, MRPs, FAC, oxidative stability and pigments of NS oil was studied using multivariate analysis. The results of PCA were presented in loading and score (Fig. 2A and B) plots. The relative contribution of principal components (PC) was determined by only considering eigenvalues greater than one. Table 4 shows variables that accounted for total variability of 93.3% for PC1 and PC2 (eigenvalue > 1) in the data set. PC1 was highly interrelated with parameters such as L* (0.259), b* (0.252), *p*-AV (−0.247), chlorophylls (−0.261), carotenoids (−0.253), TPC (−0.253), RSA (−0.259), BI (−0.252), HMF (−0.244), OSI (−0.265), MUFA (0.258), PUFA (0.265) and SFA (−0.267) and viscosity (−0.246) with total variance of 76.5%. While, a* (0.537), AV (0.528), oil yield (−0.359) and PV (−0.224) mainly accounted for PC2 with total variance of 16.8%.

The score plot shows three groups of NS oils based on the microwave power and roasting time (Fig. 2A). The gradual shift in the arrangement of NS oils can be seen from the extreme right to the left side of the score plot with an increment in microwave power and roasting time. The first group (I) on the right zone of the score plot is constituted by NS oils of three roasting treatments (180 W for 5 and 10 min and 360 W for 5 min). This group represents microwave roasted NS oils that showed an increase in PV while the decline in TPC and RSA. The second group (II) on the lower left zone of the score plot includes three microwave roasting treatments (360 W for 10 min, 540 W for 5 and 10 min). This group

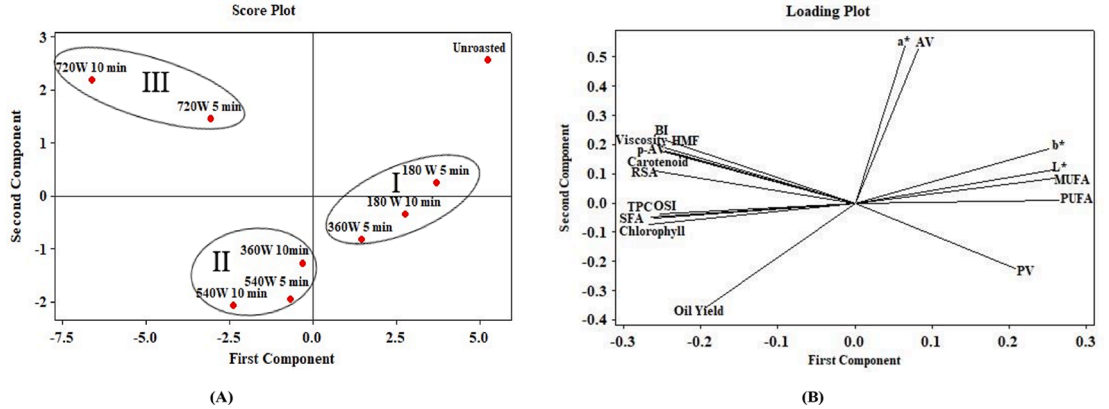

Fig. 2. Principal component analysis score plot (A) and loading plot (B) describing the relationship among different properties of NS oil at microwave powers of 180, 360, 540 and 720 W for 5 and 10 min.

Table 4
Principal components for illustrating the interpretation in Fig. 2.

Variable	PC1	PC2
Oil Yield	−0.195	−0.359
L*	0.259	0.085
a*	0.065	0.537
b*	0.252	0.186
AV	0.082	0.528
PV	0.209	−0.224
p-AV	−0.247	0.189
Chlorophylls	−0.261	−0.051
Carotenoids	−0.253	0.171
TPC	−0.253	−0.036
RSA	−0.259	0.110
BI	−0.252	0.179
HMF	−0.244	0.215
MUFA	0.258	0.010
PUFA	0.265	0.010
SFA	−0.267	−0.073
OSI	−0.265	−0.047
Viscosity	−0.246	0.179

PV = Peroxide Value, AV = Acid Value, *p*-AV = p- Anisidine value, TPC = Total Phenolic Content, RSA = Radical Scavenging Activity; HMF = 5-Hydroxymethylfurfural, MUFAs = Monounsaturated Fatty Acids, PUFAs = Polyunsaturated Fatty Acids, SFAs = Saturated Fatty acids, OSI = Oxidative stability Index, BI = Browning Index.

represents an increase in TPC, RSA, carotenoids, BI and formation of HMF and decrease in AV and PV of oils. The third group (III) on the upper left zone of the score plot is constituted by two high microwave roasting conditions (720 W for 5 and 10 min). This group represents higher HMF, BI, OSI, RSA, AV, *p*-AV and viscosity while lower PV compared to other microwave roasted NS oils. The oil from unroasted NS showed a typical behavior in comparison to oils from microwave roasted samples and was characterized by lower pigments and OSI while higher AV and color (a*, b* and L*) values. OSI was closely related with TPC, SFAs, chlorophylls, carotenoids, BI, and HMF content as shown in loading plot (Fig. 2A), indicating the contribution of these variables in increasing oxidative stability of NS oil. However PV, MUFAs and PUFAs showed opposite relation with OSI indicating the negative role of these variables in the oxidative stability of NS oils. The microwave roasted NS oil with higher contents of pigments, TPC, SFAs, HMF, and BI while lower PV and PUFA content exhibited higher oxidative stability.

4. Conclusion

The microwave powers and roasting time had a significant impact on oil yield, OSI, antioxidant activity and other quality attributes of NS oil. The roasting of NS at higher microwave power resulted in more leaching of phenolics, chlorophylls and carotenoids in oil. The low PV and high TPC, RSA, OSI, MRPs, chlorophyll and carotenoid contents were recorded for oil from NS roasted at 720 W for 10 min. However, the high oil yield and low *p*-AV was observed in oil from NS roasted at 540 W for 10 min. The higher viscosity and *p*-AV of microwave roasted NS oil was compensated by a significant increase in OSI, TPC, pigments and RSA at higher microwave power and roasting time (720 W for 10 min). A slight change in the level of SFAs, PUFAs, MUFAs and formation of oxidative products in microwave roasted NS oils correlate with the changes in the intensities of FTIR peaks. The oil obtained from microwave roasted NS has extended shelf life, higher OSI and antioxidant activity with many possible uses in food processing and pharmaceutical industries. Based on the results, it can be concluded that microwave roasting at 720 W for 10 min would be ideal for the improvement of NS oil quality as well as stability characteristics.

CRediT authorship contribution statement

Kanchan Suri: Investigation, Data curation, Validation, Writing – original draft. **Balwinder Singh:** Conceptualization, Formal analysis, Resources, Writing - review & editing. **Amritpal Kaur:** Conceptualization, Investigation, Funding acquisition, Project administration, Resources, Formal analysis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors gratefully acknowledge SERB for providing financial support in the form of women excellence award (SB/WEA/09/2017) and research project (EEQ/2020/000565).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2021.130777.

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