

## Physicochemical and Quality Characteristics of Cold and Hot Press of *Nigella sativa* L Seed Oil Using Screw Press

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### ABSTRACT

Screw pressed is one of the most important mechanical methods for oil extraction from *N. sativa* seed. The physicochemical and quality characteristics of the oil were studied after pressing at cold and hot (50°C until 100°C) temperatures. The results obtained from free fatty acid (FFA) and acid value (AV) found to be lowest at respectively 100°C (0.19% and 0.39%) and highest at 80°C (0.24% and 0.47%). The value of specific extinction coefficient (K<sub>232</sub>) value was constant for all temperatures (2.82-2.83) and K<sub>270</sub> value observed the lowest at 50°C (1.48) and highest at 100°C (1.61). Carotenoid content was the lowest at 50°C (1.95 mg/kg) than 100°C (2.46 mg/kg) and SN value found lowest at 50°C (132.75 mg KOH/g oil) and highest at 70°C (198.21 mg KOH/g oil). Viscosity was the lowest at 60°C (63.80 mPaS) than 100°C (71.47 mPaS) and PV measured the highest at 80°C (342.37 meq O<sub>2</sub>/kg oil) and lowest at 50°C (204.58 meq O<sub>2</sub>/kg oil). Chlorophyll and density measured from 1.97 to 2.50 mg/kg and 0.93 to 0.98 g/cm<sup>3</sup> respectively. The color was green and yellow with increasing temperatures. Sensory analysis panelists were liked moderately of oil pressed at 60°C.

**Key words:** *N. sativa* seed oil, physicochemical, cold and hot press temperatures, screw pressed.

### INTRODUCTION

*Nigella sativa* L is a vegetal spice belongs to the *Ranunculaceae* family, commonly known as black cumin seed. *Nigella sativa* is native to the Mediterranean region and cultivated into other parts of the world such as Asia, North Africa & Arabian Peninsula. The seeds of *N. sativa* have several therapeutic effect such as prevention of cancer, antihypertensive effect [26] anti-inflammatory, analgesic [1] and antihistaminic action [21]. The oil extracted from to seed of *Nigella sativa* used in Egyptian system of medicine. *N. sativa* seed oil is used as an antimicrobial [55], anti-inflammatory [45], anti-oxidant activity [53,2813,14] anti-tumour activity [13], anti-cancer [39], gastroprotective [25,24]. Besides, this the seed also used as a spices and cosmetic products.

*N. sativa* seed oil has been produced by solvent and mechanical extraction methods. Solvent extraction method is capable of removing nearly 90% from the seeds but the equipment required for this methods are generally too expensive and there is the inherent danger of fire and explosion in terms of the solvent used [72,4,15]. Screw pressed is the one of mechanical method for oil extraction [49]. The extracted from screw pressed method is often higher

in quality as compared to solvent-extracted method due to higher oxidative stability and lower nonhydratable phospholipids [53,70]. Screw pressed method did not extensively replaced the solvent extraction in processing of commodity oilseeds, because screw presses recover a lower proportion of the oil [70,47]. However, the screw press method does not have high extraction efficiencies as compare to solvent extraction methods [63,16]. Many factors e.g. applied pressure, heating temperature, moisture conditioning of sample, particle size, and heating time are play important role in order to determine the extraction efficiency [35,3]. Atta [10] found that the crude oil by cold press is more stable to auto-oxidation rancidity than crude oil by solvent and significant difference in physicochemical of oil. Mechanical screw pressing method is most popular method in the world to separate oil from vegetable oilseeds on small scale. Because it required low investments, and easily operated, [61].

Until now there is no study available on the effect of temperatures on quality of screw-pressed *N. sativa* seed oils. Therefore, the objective of this research to analyze the physicochemical and quality characteristics of screw-pressed *N. sativa* seed oils at six different temperatures.

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## Materials And Methods

### Raw Material:

The samples of *N. sativa* seeds, derived from India were procured from a supplier, Abdul Ghafar Enterprise, Malaysia. The *N. sativa* seed were sieved to remove dust, sand and other foreign matters. The samples were kept in vacuumed plastics and stored at room temperature until further processing.

### Preparation of *N. sativa* L seeds oil:

About of 350 g seeds were pressed for 4 to 5 min to achieve a steady flow of oil and meal before processing samples. Upon achieving steady operation, triplicate of 3000 g sample was poured into the hopper and pressed at six different temperatures ( $100\pm 3^\circ$ ,  $90\pm 3^\circ$ ,  $80\pm 3^\circ$ ,  $70\pm 3^\circ$ ,  $60\pm 3^\circ$ , and  $50\pm 3^\circ$  °C). The yield of oil was stored away from light in a dark container (wrapped with aluminum foil), stored in a chiller (+4 °C) for 48 hours to let the oil to settle foreign materials. After 48 hours, the oil was centrifuged at 3500 rpm to remove other fine particles in the oil, flushed with Nitrogen gas and then kept in a freezer (-18 °C). The oils was weighed and calculated with the formula:

$$\text{oil yield (\%)} = \frac{m_o}{m_s} \times 100 \%$$

Where:  $m_o$  – weight of oil extract,  $m_s$ – weight of samples

### Physicochemical characteristic of *N. sativa* L seeds oil:

#### Free Fatty Acid (FFA):

Free fatty acids of *N. sativa* seeds oils were determined according to AOCS recommended practice Ca 5a-40 [9].

#### Acid value (AV):

The acid value (AV) was converted from free fatty acid with a conversion factor (1.99 as oleic acid).

$$\% \text{ Acid value} = \% \text{ FFA (as oleic)} \times 1.99$$

#### $K_{232}$ and $K_{270}$ specific extinction coefficient:

$K_{232}$  and  $K_{270}$  extinction coefficients were measured from absorbance at 232 nm and 270 nm, respectively, with UV spectrophotometer (SECOMAN) using 1 % solution of oil in cyclohexane and a path length of 1 cm [12].

#### Saponification Number (SN):

Determination of oilseeds saponification number was used AOCS recommended practice Cd 3-25 [9].

#### Pigment content of *N. sativa* seeds oil:p

Oil was weighed accurately 7.5 g oil and dissolved in cyclohexane up to final volume of 25 ml. Chlorophylls and carotenoids contents were calculated from the absorption spectra of the oils. The absorption at 670 nm was usually considered to be related to the chlorophylls fraction, pheophytin is

being its major component. The dominant pigment in the carotenoids fraction was lutein and the absorption was measured at 470 nm. Thus chlorophylls and carotenoids contents were expressed as mg of pheophytin and lutein per kg of oil, respectively [34,44].

The levels of pigments are obtained as follow:

$$[\text{Chlorophylls}] = \frac{A_{670} \times 10^6}{613 \times 100 \times \text{density}} \text{ mg/kg}$$

$$[\text{Carotenoids}] = \frac{A_{470} \times 10^6}{2000 \times 100 \times \text{density}} \text{ mg/kg}$$

#### Density of *N. sativa* seeds oil:

The density of liquid is required to determine the power required for pumping. The analysis was carried out using Pycnometer method recommended practice 41a-2 [9].

#### Determination viscosity of *N. sativa* seeds oil:

Viscosity of the oil samples was measured with a vibration (Oscillation) viscometer (AND VIBROVISCOMETER SV-10). The principle of surface loading whereby the surface of an immersed probe generates a shear wave that dissipates in the surrounding medium. The measurements depend on the ability of the surrounding fluid to damp the probe vibration [57]. Measurements were performed at 25°C with a plastic-plate at 0.3~10.000 mPaS.

#### Peroxide Value (PV):

Peroxide values represent the amount of hydroperoxides or peroxides groups, and identically with initial products of lipid oxidation. The changes of the procedure may change the results because this method is empirical and difficult to obtain sufficient quantities from foods that are low in fat [52]. The method included AOCS recommended practice Cd 8-53 [9].

#### Color of *N. sativa* seeds oil:

Color of *N. sativa* seeds oil was evaluated using a CIELab Minolta spectrophotometer CM 3500d. The 15 ml sample was pipette into a sample cup, and color values were obtained using a D6 5/10 °C(daylight 65 illuminant/10°C observer/set) with color scale coordinates :  $L^*$ ,  $a^*$  and  $b^*$ .  $L^*$  were represented the difference between light ( $L^* = 100$ ) and dark ( $L^* = 0$ ). The component  $a^*$  was represented the difference between green ( $-a^*$ ) and red ( $+a^*$ ) and component  $b^*$  was represented the difference between blue ( $-b^*$ ) and yellow ( $+b^*$ ) (Sahin *et al.*, 2006). The method followed according AOCS with Cc 13b-45 [9].

#### Mineral contents of *N. sativa* seeds Oil:

Some of the minerals content of *N. sativa* seedsoil and meal present such as sodium, calcium, manganese, nickel, cuprum, cadmium, iron, lead, and zinc were analyzed by with Perkin-Elmer Analyst 100 with induction coupled plasma atomic emission (ICPAES) spectroscopy [7]. The sample (oil was 1 g)

was digested (ETHOS 900 Milestone, microwave Lab station) with 6 ml concentrated Nitric acid and 1 ml acid peroxide (30 %) until a transparent solution was obtained. The instrument was calibrated with known standards and the samples were analyzed at corresponding wavelengths.

#### *Sensory analysis:*

##### *Description of panel sensory:*

The panelists divided two group that commonly used and not used to consume of *N. sativa* seeds oil (n= 40). First of group (n= 25) are randomly panel from other country e.g. Indonesia, Ghana, Sri Lanka, and Malaysia which randomly not used consume of *N. sativa* seeds oil. This group was students and staff at Universiti Sains Malaysia with ages between 21-45 years old. The second group are panelists (n = 15) were consumer society of *N. sativa* seeds oil from Kedah (Malaysia) with ages between 31-65 years old. This group was recruited on the basis of their previous experience in descriptive sensory analysis, interest, availability and consumption of *N. sativa* seeds oil at least once a day. The test of panels was conducted between 11:00 A.M. and 1:00 P.M. on the first day and 2:00 P.M. and 5:00 P.M on the second day.

Samples evaluations, 30 ml of the *N. sativa* seeds oil were placed into transparent plastic cups with lids coded with 3 digit random numbers. Samples consisting of different temperatures extraction of screw press at cold press (50°, 60°, 70° C) and hot press(80°, 90° and 100°C). Samples were presented with water and paper ballots on a plastic tray. Panelists were instructed to consume the whole sample and rinse their mouths with water between samples to minimize any residual effect [31,74].

According to Aminah [6] stated that 7 point scale is suitable for sensory analysis. Hedonic scale is a rating scale method used in sensory evaluation. Here's an example:where 1 = dislike very much, 2 = dislike moderately, 3 = dislike slightly, 4= neither like nor dislike, 5 = like slightly, 6 = like moderately, and 7 = like very much, according to the acceptability of color, odor, taste, viscosity, bitterness and overall quality.

##### *Statistical Analysis:*

The effects of temperatures on physicochemical, sensory analysis, antioxidant and nutritional properties of *N. sativa* seeds oil and meal were analyzed using ANOVA one way and Tukey's test with two replicates. All of the statistical was performed at the 5 % significance level were presented as mean  $\pm$  standard deviation (M $\pm$ SD) using SPSS 12.0 for windows [50].

## **Result and Discussion**

### *Physicochemical characteristics of N. sativa seeds oil:*

Free fatty acid (FFA) measurement is directly correlated with the acid value as shown in Table 1.

FFA and acid values of oil extracted at 80 °C was significantly different (p<0.05) with those at 50 °C, 60 °C, and 100 °C. However, FFA and Acid value of oil extracted at 100 °C showed the values were lowest when compared to temperature at 80 °C. Both conditions are due to effect of high temperature extraction, inhibiting hydrolysis process during oil extraction. Hydrolysis process so occurring in the oil is generally caused by water content and lipaseenzymes. When seeds are extracted, the existing water in the seeds will come out with the oil and react with triglycerides to form FFA and glycerol [62]. Therefore, the values of FFA and acidity production were higher at low extraction temperature and the quality of oil decreases because oxidation readily happens in these conditions.

These result was lowest than comparing with FFA in red raspberry and jatropha oil are 1.32 % and 1.03 % found by Šuuroviet *et al* [66] and Salimon *et al.* [59]. Šuurovi *et al* [66] told that FFA and acid value were showing condition the seeds before and during the processing oil extraction. The non-refining oil must have  $\pm$  2 % of FFA. Atta [10] found that FFA and AV of the solvent extraction were lower than cold pressed extraction of *N. sativa* seeds oil. The value of FFA was 11.0 % as oleic used by cold press and 6.7 % by solvent extraction.

However, Dandik *et al.* [23] reported that the low temperature may give a higher value of FFA and causing the disposition to the hydrolysis action catalyzed by native lipase in ground seeds. Acid value allows the standards of Codex Alimentarius Commission [5] for edible oil at 4% as oleic acid. Despite the high value obtained data lower temperature, hence compared to standard values for edible oils the value is still below standards.

Specific extinction usually determined by  $K_{232}$  and  $K_{270}$ .  $K_{232}$  value measured by the presence of conjugated dienoic acid in the oil is an indication of primary oxidation products at 232-234 nm. Some researchers have used absorption at 268-270 nm to measure conjugate trienes as well as ethylenicdiketones and conjugate ketodienes and dienals arising as secondary oxidation products [47]. Farmer *et al* [27] reported that the extent of double bond displacement correlated with the degree of oxidation occurring in unsaturated oil. Value of  $K_{232}$  and  $K_{270}$  represent primary and secondary oxidation component in oils. From the Table 1, it shows that the value of  $K_{232}$  at 50 °C and 60 °C was significantly different (p<0.05) with temperature at 90 °C.

The values of  $K_{270}$  at 50 °C was significantly different (p<0.05) with other temperatures, except temperature at 60 °C. Primary and secondary oxidation is increased by the increasing the temperatures of heating. White *et al.* [69] said that double bond can also change to form conjugated

dienes during the hydrogenation or deodorization of oil if the temperature rises above 245 °C.

**Table 1:** Physicochemical and quality characteristics of *N. sativa* seeds oil pressed at different temperatures.

Temperature	50 °C	60 °C	70 °C	80 °C	90 °C	100 °C
Physicochemical characteristics						
Free Fatty Acid (as oleic %)	0.20±0.01 <sup>ab</sup>	0.20±0.02 <sup>ab</sup>	0.23±0.01 <sup>bc</sup>	0.24±0.00 <sup>c</sup>	0.23±0.01 <sup>abc</sup>	0.19±0.00 <sup>a</sup>
Acid Value (as oleic %)	0.41±0.01 <sup>ab</sup>	0.42±0.01 <sup>ab</sup>	0.423±0.02 <sup>ab</sup>	0.47±0.01 <sup>c</sup>	0.45±0.03 <sup>bc</sup>	0.39±0.01 <sup>a</sup>
K <sub>232</sub> (%)	2.82±0.01 <sup>a</sup>	2.82±0.00 <sup>a</sup>	2.83±0.00 <sup>ab</sup>	2.83±0.00 <sup>ab</sup>	2.83±0.00 <sup>b</sup>	2.83±0.01 <sup>ab</sup>
K <sub>270</sub> (%)	1.48±0.00 <sup>a</sup>	1.50±0.02 <sup>ab</sup>	1.53±0.01 <sup>b</sup>	1.57±0.02 <sup>c</sup>	1.61±0.02 <sup>d</sup>	1.610±0.01 <sup>d</sup>
SN (mg KOH/g oil)	132.75±10.02 <sup>a</sup>	172.44±17.46 <sup>abc</sup>	198.21±16.33 <sup>c</sup>	193.64±17.70 <sup>bc</sup>	183.91±15.35 <sup>bc</sup>	154.56±13.80 <sup>ab</sup>
Chlorophyll (mg/kg)	1.97±0.19 <sup>a</sup>	2.27±0.20 <sup>a</sup>	2.47±0.05 <sup>a</sup>	2.50±0.32 <sup>a</sup>	2.22±0.14 <sup>a</sup>	2.09±0.21 <sup>a</sup>
Carotenoid (mg/kg)	1.95±0.05 <sup>a</sup>	2.043±0.02 <sup>a</sup>	2.25±0.05 <sup>b</sup>	2.32±0.07 <sup>bc</sup>	2.34±0.07 <sup>bc</sup>	2.46±0.12 <sup>c</sup>
Density (g/cm <sup>3</sup> )	0.93±0.04 <sup>a</sup>	0.98±0.00 <sup>b</sup>	0.98±0.00 <sup>b</sup>	0.98±0.00 <sup>b</sup>	0.98±0.00 <sup>b</sup>	0.98±0.00 <sup>b</sup>
Viscosity (mPaS)	64.53±0.31 <sup>ab</sup>	63.80±0.61 <sup>a</sup>	66.23±1.29 <sup>b</sup>	66.47±0.81 <sup>b</sup>	69.53±0.31 <sup>c</sup>	71.47±0.45 <sup>c</sup>

Means in the same row with different superscripts are significantly different (p<0.05)

These result of K<sub>232</sub> and K<sub>270</sub> was higher than *N. sativa* seed oil from Tunisian (1.07 and 0.27) and Iranian (0.74 and 0.18) found by Cheikh-Rouhouet *al.* The values of K<sub>232</sub> and K<sub>270</sub> were related to data from Besbeset *al* [19] which found that value increase the Rancimat time from 2.30 to 13 and 0.5 to 2, respectively. The value of K<sub>232</sub> was higher due to sensitivity to heating during extraction processing to form higher content of primary oxidation of date seed oils and virgin olive oils. Because product of primary oxidation is unstable under heating it would be degraded causing of secondary oxidation and absorption at 270 nm. It shows that value of secondary products of oxidation was lower than primary products oxidation. Proving that *N. sativa* seeds oil has a good resistance against heating condition [68,19].

The saponification number (SN) increased with heating temperature of 50 °C to 70 °C and decreased from 80 °C to 100 °C (Table 1). This is because temperature is one way to eliminate the compounds of other impurities and allow the natural process of saponification. Comparing the SN value to other oilseed e.g. rapeseed oil (184 mg KOH/g oil), pumpkin oil (199.3 mg KOH/g oil), safflower oil (184.2 mg KOH/g oil), coconut oil (247.9 mg KOH/g oil), melon seeds oil (979 mg KOH/g oil), palm kernel oil (732 mg KOH/g oil) and castor seeds oil (137 mg KOH/g oil), the value of SN from palm kernel and melon seeds oil was higher than other oilseeds [11,56,48].

The higher value of suggests that the oilseed contain high molecular weight fatty acids with low level of impurities and can be used in soap making industry [36]. This shows that the quality of the oil decreases due to the process of oxidation and hydrolysis [62]. The value of SN is higher at 70 °C because the heat may have denatured the fat-hydrolyzing enzyme with higher degree of denaturation in oil extracted from *N. sativa* seed. The lower SN may be due to the presence of residual water which may have aided hydrolysis resulting in lower amount of mg KOH.

Carotenoids significantly increased at temperature of 50 °C to 70 °C and of 100 °C (Table 1). The increase in temperature was significantly different (p<0.05) with carotenoid at temperature at 50 °C with 70 °C and 100 °C. Chlorophyll was insignificantly different (p<0.05) with increase in temperatures. A similar result was obtained by Luaceset *al* [38], who observed the main pigment compounds in the virgin olive oil ( $\beta$ -carotenoid and chlorophyll) treated with heat-temperatures. The heat-treatment caused increased both of  $\beta$ -carotenoid and chlorophyll. But there is no relation was found between chlorophyll compound and the treatment temperatures [38].

The pigments decreased the quality of oils due to its interaction with light making it easily oxidized. The  $\beta$ -Carotene is easily oxidized to epoxy-carotenoids, which are colorless and oxidation products of  $\beta$ -Carotene can induce autocatalytic oxidation in oils [34]. Chlorophyll is converted to colorless derivatives by reacting with peroxyradicals produced during oxidation [37]. The  $\beta$ -carotenoid in *N.sativa* seeds oil was higher than oilseed from Parsley (0.783 – 0.989 mg/kg), and lower than Mullein (1.121 mg/kg) and pumpkin seeds oil (5.957 mg/kg) [51]. The chlorophyll was more found in the canola oil (4-30 mg/kg), and rapeseed (5-55 mg/kg) but in refined bleach and deodorized (RBD) of canola oil the value less than 0.025 mg/kg and trace in the soybean oil [32].

Density of *N. sativa* seeds oil in the table 1 show that at temperature 50 °C the value was significantly different (p<0.05) with other temperatures. This result was in contrast with density in *Slovene Camelinasativa* oil was decreased (0.9207 g/cm<sup>3</sup> to 0.9041 g/cm<sup>3</sup>) with increasing of temperature (20 °C to 50 °C) [2]. The density of liquid oil is less than that of most other food components, and so there is a decrease in density of food as its fat content increases. Thus the lipid content of foods can be determined by measuring their density.

The viscosity value decreased at 50 °C to 60 °C and increased again at 70 °C until 100 °C (Table 1).

There was significantly different ( $p < 0.05$ ) between temperature at 60 °C with others temperature, except at 50 °C. Value at 70 °C and 80 °C was significantly different ( $p < 0.05$ ) with 80 °C and 100 °C. The increase in viscosity value during heating is in correlation with the formation of dimers and polymers due to the increase of carbon chain length and also related to the difference in saturated fatty acids with the consequence of a higher melting points. In most cases, an increase in temperature will lower the viscosity of a material, but sulphur, which form polymers, is an exception. Viscosity is another physical characterization which mostly depends on temperature and on to some extent the compositional difference of vegetable oils. Viscosity of *N. sativa* seed oil was higher than Malaysia *Jatropha curcas* seeds oil (36 cPs), Nigeri *J. curcas* seeds oil (17-52 cPs), and soybean oil (50.09 cPs) [32,59].

The peroxide values of the *N. sativa* seed oil from 50 °C (204.58 meq O<sub>2</sub>/kg oil) to 100 °C (324.52 meq O<sub>2</sub>/kg oil) did not show any significant difference ( $p < 0.05$ ), except between 50 °C (204.58 meq O<sub>2</sub>/kg oil) with 80 °C (342.37 meq O<sub>2</sub>/kg oil) the value becomes significantly different ( $p < 0.05$ ) (Fig. 1). The peroxide value was increased from 30 mmol O<sub>2</sub>/kg become 120 mmol O<sub>2</sub>/kg oil due to different temperatures [66].

Peroxide values are one other content from primary products (hydroperoxides) of lipid oxidation in oilseeds. These values are usually used as an indicator for the initial stages of oxidation process. However, in time, some hydroperoxides form rapidly during storage. Because of these reasons, Sherwin [60] stated that peroxide values do not necessarily

indicate the actual extent of lipid oxidation. Nevertheless, secondary products of lipid oxidation (aldehydes) are less easily destroyed than peroxide values (hydroperoxides) during heating processing [20]. It has relation with K<sub>232</sub> as a secondary product of lipid oxidation.

GoerlichPharma International [29] reported that the peroxide value of *N. sativa* seeds oil rose rapidly immediately after production. However, because of the residues of essential mock oxygen of the fatty oil bound as peroxide, it caused the peroxide value to not be a suitable measure for rancidity. The peroxide value of *N. sativa* seeds from Egypt was found to be <120 meq O<sub>2</sub>/kg oil [29]. Cheikh-Rouhouet *al* [22] reported values of 5.65 meq O<sub>2</sub>/kg (Tunisian) and 4.35 meq O<sub>2</sub>/kg (Iranian).

The difference in the values was resulted due to the source of the plant and the condition in which the plants grow and effects of processing. In addition, Pike [52] said that the disadvantage of this method is the requirement of 5-g oil sample size. It is difficult to obtain sufficient quantities from foods with low fat and empirical and this method can changes the result with any modification.

The CIELAB color measurement method was determined by its color coordinates: L\* represent the difference between light (L\* = 100) and dark (L\* = 0), a\* represent the difference between green (-a\*) and red (+a\*) and b\* represent the difference between blue (-b\*) and yellow (+b\*). The *N. sativa* seed oil was lighter by the increase of heat temperature at 70 °C and become less or dark at the temperature of 100°C (Table 2).

**Table 2:** Color (CIELAB L\* a\* b\*) value *N. sativa* seeds oil pressed at different temperatures.

Color	L*	a*	b*
Temperature (°C)			
50	28.59±0.35 <sup>ab</sup>	-4.51±0.46 <sup>b</sup>	9.41±0.16 <sup>a</sup>
60	31.08±0.33 <sup>bc</sup>	-4.51±0.03 <sup>b</sup>	10.58±0.18 <sup>ab</sup>
70	31.98±0.45 <sup>c</sup>	-4.73±0.11 <sup>a</sup>	11.26±0.21 <sup>bc</sup>
80	31.74±0.12 <sup>bc</sup>	-4.67±0.07 <sup>ab</sup>	12.02±0.44 <sup>bc</sup>
90	31.18±0.52 <sup>bc</sup>	-4.64±0.03 <sup>ab</sup>	12.40±0.41 <sup>c</sup>
100	25.82±2.72 <sup>a</sup>	-4.73±0.07 <sup>a</sup>	9.36±1.46 <sup>a</sup>

Means in the same column with different letters are significantly different ( $p < 0.05$ ).

The darkness may be due to the complicated process during processing, interactions with fatty acids, dimers, polymers and other minor compounds present in the oil and effect of increased heat [71]. Significant difference ( $p < 0.05$ ) were found between 70 °C and 100 °C (for value of L\*), temperature of 50 °C, 60°C with 70 °C and 100°C (for value of a\*) and temperature of 50 °C and 100 °C with 90 °C (for value of b\*). The color of the *N. sativa* seed oil was commonly more greenness and yellowness by the increasing of temperature. Color of *N. sativa* seeds oil was darker and more yellowish than Malaysian rubber seeds oil (L\* = 33.98; a\* = 0.86; and b\* = 0.47) (Bashar *et al.*, 2009). Compared the color with other oilseeds e.g. castor seeds, coconut seeds, melon seeds, palm kernel seeds oil, the color

were golden yellow, pale yellow, golden yellow, and pale yellow [48].

Mineral contents of *N. sativa* seeds oil are shown in Table 3. Table 3 shown values increased with decline in temperature but it was not significant different ( $p < 0.05$ ) of increase pressing temperature. It was shown for iron, zinc, lead, cadmium increase with increase the temperature pressing. Calcium only found at temperature 50 °C and 100 °C in small quantity. Some minerals were not found at certain temperatures, e.g. nickel only was found at 60 °C, 80 °C and 100 °C. Values increased with increase in pressing temperature (Table 3).

The data shows *N. sativa* seeds oil has highest contents of iron, lead, manganese and zinc in ranges 4585.40 x10<sup>-3</sup>-2853.80 x10<sup>-3</sup> mg/L, 571.20 x10<sup>-3</sup>-

3030.50  $\times 10^{-3}$  mg/L, 133.30  $\times 10^{-3}$ -193.60  $\times 10^{-3}$  mg/L, 345.40  $\times 10^{-3}$ -2710  $\times 10^{-3}$  mg/L, respectively. Mineral contents in NSM were high iron, lead, manganese and zinc in ranges 78.25-119.80 mg/100 g, 10.45-30.38 mg/100g, 18.0-19.44 mg/100 g,

16.56-28.40 mg/100 g, respectively. It was similar to soybean meal and cotton seeds meal whereby, Imorouet *al* [33] found that iron, zinc, calcium and phosphorus were higher in content in that meal.

**Table 3:** Mineral contents from *N. sativa* seeds oil pressed at different temperatures.

Temperature Mineral	50 °C	60 °C	70 °C	80 °C	90 °C	100 °C
Sodium	7.00±0.00 <sup>a</sup>	7.60±0.00 <sup>a</sup>	6.20±0.00 <sup>a</sup>	4.90±0.00 <sup>a</sup>	10.20±0.00 <sup>a</sup>	8.70±0.00 <sup>a</sup>
Calcium	0.40±0.04 <sup>a</sup>	nd	nd	nd	nd	5.80±0.01 <sup>a</sup>
Manganese	nd	193.60±0.03 <sup>a</sup>	187.60±0.03 <sup>a</sup>	133.30±0.00 <sup>a</sup>	184.00±0.00 <sup>a</sup>	178.40±0.00 <sup>a</sup>
Nickel	nd	129.0±0.02 <sup>a</sup>	nd	64.40±0.04 <sup>a</sup>	nd	133.70±0.05 <sup>a</sup>
Cuprum	43.10±0.01 <sup>a</sup>	34.70±0.01 <sup>a</sup>	17.30±0.00 <sup>a</sup>	3.90±0.00 <sup>a</sup>	nd	nd
Cadmium	272.80±0.09 <sup>a</sup>	386.10±0.01 <sup>a</sup>	421.50±0.07 <sup>a</sup>	361.60±0.01 <sup>a</sup>	430.70±0.44 <sup>a</sup>	389.60±0.01 <sup>a</sup>
Iron	3831.00±0.24 <sup>a</sup>	3569.10±0.15 <sup>a</sup>	2844.40±1.00 <sup>a</sup>	2853.80±0.19 <sup>a</sup>	3866.40±0.30 <sup>a</sup>	4585.40±0.07 <sup>a</sup>
Lead	nd	1290.60±0.21 <sup>a</sup>	571.20±1.00 <sup>a</sup>	1344.50±1.21 <sup>a</sup>	3030.50±0.32 <sup>a</sup>	2542.90±0.11 <sup>a</sup>
Zinc	667.80±0.54 <sup>a</sup>	1099.00±0.10 <sup>a</sup>	1146.30±0.93 <sup>a</sup>	345.40±0.03 <sup>a</sup>	2644.10±0.18 <sup>a</sup>	2710.00±0.04 <sup>a</sup>

Data are represented in M ( $\times 10^{-3}$ )  $\pm$  SD (mg/L). N =2. nd was not detected. Different superscript in the same row represent significant difference (p<0.05).

The value of lead, iron and zinc increased with decline of heat temperature of the screw press. The screw press machine is made out of steel and at elevated temperatures can be catalyzed to breakdown and appear in the oil and meal in minute quantities. Friction inevitably fuels this process as the metal parts come into contact at high temperature causing, also, some level of lipid oxidation in the presence of oxygen. Crude oil has impurities, like heavy metal, minerals and need to be refined to produce safe product for human consume. Likewise meal seeds, for safe consumption, are suggested be processed using cold pressing rather than hot press for *N. sativa* seeds.

#### Sensory analysis:

Sensory analysis is the most important parameter for acceptability of a product from consumers. Some factors influencing sensory evaluation and habitual on panelist to consume the product e.g. origin state, healthy or ages of panelist, origin of product, cultivar, agricultural practices, methods of harvests, transport, technological operation of producing the product and storage the products [41].

Sensory analysis of *N. sativa* seed oil was ranked on the basis of 6-point hedonic scale for

various attributes like color, odor, taste, viscosity, bitterness and overall acceptability. Sensorial analysis of panelist that used to consume of *N. sativa* seed oil was shown in Table 4. Analyses of the samples show that temperature 50 °C, 60 °C and 100 °C were significantly differs (p<0.05) in color. Sample at 100 °C has more dislikes with panelists. Samples at 60° were liked moderately than 70 °C. The odor of sample is preferably at temperature of 60 °C and liked moderately than others.

In terms of taste, panelists preferred the sample at 60 °C, like slightly at 50° and dislike moderately sample at 90°C. Panelists disliked very much such sample at 100 °C and is significantly different (p<0.050) with sample at 60 °C. Panelists preferred viscosity from sample at 50° and 60 °C. The viscosity was significantly different (p<0.05) between 50°, 60 °C with 90° and 100 °C. Bitterness of the samples was noticeably at 80° until 100 °C than sample at 60 °C. For overall acceptability, the panelist agreed on the sample at 60 °C (4.80) was like moderately. Sample at 100 °C was significantly disliked moderately by panelists. Evaluation of samples exposes that oil produce at 100 °C was significantly different in color, odor, taste, bitterness and overall acceptability (Table 4).

**Table 4.** Sensory analysis of panelist that used to consume *N. sativa* seeds oil (pressed at different temperatures), n = 15

Temperature	50°C	60°C	70°C	80°C	90°C	100°C
Sensory Analysis						
Color	3.20±1.57 <sup>b</sup>	5.20±0.94 <sup>c</sup>	3.73±1.53 <sup>b</sup>	3.27±1.62 <sup>b</sup>	2.80±1.32 <sup>ab</sup>	1.47±1.06 <sup>a</sup>
Odor	3.60±1.12 <sup>ab</sup>	5.07±1.16 <sup>b</sup>	3.13±1.64 <sup>a</sup>	4.00±1.3 <sup>ab</sup>	3.47±1.69 <sup>a</sup>	2.67±1.54 <sup>a</sup>
Taste	3.87±1.25 <sup>cd</sup>	4.80±0.56 <sup>d</sup>	3.13±1.12 <sup>bc</sup>	2.27±1.62 <sup>ab</sup>	1.80±1.14 <sup>a</sup>	1.47±0.74 <sup>a</sup>
Viscosity	4.53±0.99 <sup>b</sup>	4.53±1.30 <sup>b</sup>	3.60±1.72 <sup>ab</sup>	3.73±1.75 <sup>ab</sup>	2.53±1.69 <sup>a</sup>	2.93±1.87 <sup>ab</sup>
Bitterness	3.00±1.85 <sup>a</sup>	2.93±1.22 <sup>a</sup>	4.13±1.36 <sup>ab</sup>	4.93±1.44 <sup>b</sup>	4.87±1.77 <sup>b</sup>	5.53±0.83 <sup>b</sup>
Overall	4.60±0.74 <sup>bc</sup>	4.80±0.78 <sup>c</sup>	3.40±1.45 <sup>ab</sup>	3.27±1.03 <sup>a</sup>	2.53±0.83 <sup>a</sup>	2.27±1.44 <sup>a</sup>

Means in the same row with different superscripts are significantly different (p<0.05)

But different evaluation among panelist commonly consume of *N. sativa* seeds oil with not commonly consume (Table 5). Panelist was evaluated all of sample were not significantly different (p<0.05) with temperatures on color, odor,

taste and bitterness. But on the viscosity, it was significantly different (p<0.05) among sample at 90° and 100 °C with others. Overall acceptability of panelist not consume of *N. sativa* seeds oil was like slightly (4.16) sample of 60 °C than sample of 90 °C.

**Table 5:** Sensory analysis of panelist that not used to consume *N. sativa* seeds oil (pressed at different temperatures).

Temperature	50°C	60°C	70°C	80°C	90°C	100°C
Sensory Analysis						
Color	4.12±1.05 <sup>a</sup>	4.44±1.08 <sup>a</sup>	4.52±1.08 <sup>a</sup>	4.24±1.09 <sup>a</sup>	3.60±1.26 <sup>a</sup>	3.64±1.28 <sup>a</sup>
Odor	4.04±1.27 <sup>a</sup>	3.88±0.97 <sup>a</sup>	4.08±0.91 <sup>a</sup>	4.16±0.98 <sup>a</sup>	3.80±1.04 <sup>a</sup>	3.72±1.24 <sup>a</sup>
Taste	3.68±1.14 <sup>a</sup>	3.48±1.01 <sup>a</sup>	3.40±1.38 <sup>a</sup>	3.36±1.18 <sup>a</sup>	3.28±0.84 <sup>a</sup>	3.16±1.31 <sup>a</sup>
Viscosity	4.12±0.97 <sup>b</sup>	4.24±0.88 <sup>b</sup>	4.16±0.85 <sup>b</sup>	4.32±0.98 <sup>b</sup>	3.28±1.14 <sup>a</sup>	3.52±1.12 <sup>ab</sup>
Bitterness	3.04±1.02 <sup>a</sup>	3.32±1.14 <sup>a</sup>	3.24±1.33 <sup>a</sup>	3.24±0.93 <sup>a</sup>	3.00±1.04 <sup>a</sup>	2.92±1.44 <sup>a</sup>
Overall	3.96±0.79 <sup>bc</sup>	4.16±0.78 <sup>c</sup>	3.80±1.26 <sup>abc</sup>	3.76±1.13 <sup>abc</sup>	3.08±0.95 <sup>a</sup>	3.20±1.35 <sup>ab</sup>

Means in the same row with different superscripts are significantly different ( $p < 0.05$ )

Assessment of color on sensory analysis related to premises color analysis is done by instrument. Table 2 shows that increase of the extraction temperature would make oil become darker, greenish, and bluish. An objective assessment of the panelists proved that oil in the temperature of 60 °C and 70 °C are brighter and yellowish just like *N. sativa* seed oil that were sold in the market. Many consumers preferred the bright color, transparent but close to its natural color of oil. No more chemical refining, bleaching or further process purification with chemical addition was done. The application of mechanical, chemical and heat in processing oilseed or vegetable oil extraction may affect the color of the sample [68].

*N. sativa* seeds oil has a strong odor, spicy like fennel flowers and has a sticky consistency [30]. The higher oil extraction temperature caused the stronger spicy smell and closer to a burning smell. This is caused due to the seeds of *N. sativa* containing high protein, carbohydrate, double bonds of carbon and unsaturated fatty acids. The heat temperature would be denatured composition of protein, degraded of carbohydrate, breakdown the double bonds become a long chain carbon or saturated fatty acid and oxidized the unsaturation fatty acids [71]. The color of meal become darker charred and the smell was very strong similar with the *N. sativa* seed oil.

The sensory analysis of taste and bitterness are similar of which higher temperature caused dislike in taste because of higher bitterness value (Table 4 and 5). Conclusion from all panelists preferred oil sample pressed at 60 °C than sample at 100 °C. Similar to a virgin olive oil sensory where different sensory notes described the characteristic of the virgin olive oil and the high score shows a balance between main sensory perceptions and the consumer appreciation [43]. Sultan *et al.* [64] stated that flavor is the major priorities which were influenced by temperature, moisture, in contact-air, light and presence of antioxidants.

The vegetable oil or oilseed would be characterized by a high nutritional value in addition to a high oxidative stability making it appropriate to be applied in high temperature [41]. *N. sativa* seed oil has majority of compounds e.g. oleic, linoleic, linolenic and palmitic acids [10,64,22]. The unsaturation of fatty acid e.g. oleic becomes an

importance in nutritional value because it could lower LDL.

Increasing HDL and linolenic acid was responsible for unpleasant room-odor during deep-fat frying in olive oil [41]. Unsaturated fatty acids such as oleic, linoleic, linolenic in oil and fat become oxidized during the processing. Lipid oxidation cause adverse flavors and aromas, compromises the nutritional quality of fats and oil and lead to the production of toxic compound [42].

#### Conclusion:

Values of  $K_{232}$ ,  $K_{270}$ , carotenoid, SN, peroxide and viscosity were significantly different ( $p < 0.05$ ) at different temperature except chlorophyll. Similarly with the FFA and acid values which showed the lowest value at the high temperature due to inhibition of the hydrolysis process during oil extraction at higher temperatures. Color of *N. sativa* seed oil was commonly more green than yellow with increasing temperature. Overall test of sensory analysis showed panelists liked moderately oil pressed at 60°C.

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