Study Of Shelf Life And Quality Of Wild Walnut Oil

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Abstract
The objectives of this project to investigated the oxidation degree by effect on peroxide value and effect on rancidity by organoleptic evaluation. The quality of wild walnut oil was evaluated by measuring the peroxide value over a 90 days storage period. Wild walnut oil is excellent source of omega – 3 fatty acids and high polyunsaturated fat content. Wild walnut oil undergoes oxidative rancidity was caused by oxygen, due to that rancidity may cause in wild walnut oil. Storage temperature at room temperature effect on oxidation rate, which affects the oil with off-flavor formation. During the shelf life study, peroxide value was goes increasing from 1.27 to 18.68 meq/kg walnut oil. In addition, rancidity increases during that with an undesirable rancid taste makes wild walnut oil unacceptable for the consumer.

Keywords: Shelf life of wild walnut oil, peroxide value, sensory evaluation.

1. Introduction
Wild walnut (Juglans nigra) is a much-appreciated nut because of its good organoleptic characteristic (Lopez, Pique, Romero & Aleta, 1995), high levels of 18:1, 18:2, and 18:3 fatty acids (Crews et al., 2005; Zwarts, Savage, & McNeil, 1994; Sabate & Fraser, 1994), antihypertensive effects (Sabate & Fraser, 1994) and protective effect against cardiovascular disease. Currently, walnut is a crop of a high economic interest for the food industry. Walnut fruit contains high level of oil from 52 to 72 g/100g (Zwarts, Sevage & McNeil, 1999) and it is consumed, fresh or toasted, alone or in other edible products. Walnuts are well known because of its high polyunsaturated fatty acid (PUFA) content. The major fatty acid found in walnut are linoleic (0.8-15.4 g/100g), oleic (13.8-33.0/100g), and linolenic (8.0-15.4/100 g), an PUFA contents of the total fatty acids range from 57.3 to 76.6g /100g (Zwarts, Sevage & McNeil, 1999). Walnut oil contains approximately 7% saturated, 20% monounsaturated and 73% polyunsaturated fatty acids; these high levels of polyunsaturated fatty acids make walnuts prone to oxidation (Stark, McNeil, & Savage, 2000; Vergano et al., 1995) and may mean that the flour has a limited shelf-life, particularly considering that the large, flour surface area could expose the oil to much air. This could lead to increased oxidative changes during subsequent storage.

A higher intake of polyunsaturated fatty acids reduces blood pressure, total and LDL cholesterol. Such changes would have some positive effect in reducing the risk of coronary heart disease (Iso et al., 2002). However, high PUFA content limits the shelf life of the products due to susceptibility of PUFA to oxidation. Lipid oxidation is the most important quality parameter decreasing economic value of walnuts during storage. Oxidation resulting with an undesirable rancid taste makes walnuts unacceptable for the consumer (Jensen, Sorensen, Brockhoff, & Bertelsen, 2003). A number of storage experiments have been carried out on the storage of in-shell walnuts, walnut kernels and walnut oil. Temperature, light, moisture and exposure to oxygen have been found to be the main contributing factors to oxidation (Jan et al., 1988; Koyuncu & Askin, 1999; Mate, Salveit, & Krochta, 1996; Stark et al., 2000). Marcela Martinez et al. 2013 reported that fresh walnuts had peroxide value at 0 days (0.55±0.06 meq/kg oil), 90 days (22.5±0.06 meq/kg oil), 180 days (70.92±0.01 meq/kg oil) in light condition. Stark et al. (2000) found that walnut oil stored at room temperature (mean 24°C) in the dark, in sealed bottles, showed only small rises in peroxide levels after four months storage and remained an acceptable product in terms of its organoleptic properties.
Oxygen concentration is one of the most important extrinsic factors affecting lipid oxidation. The rate of oxidation is independent of oxygen concentration at very high oxygen partial pressures, while it is proportional to oxygen concentration at low oxygen partial pressures (Labuza, 1971). The availability of oxygen in a package can, to a certain extent, be controlled by the oxygen permeability of the packaging material (Jensen et al., 2003). Jensen et al. (2001) investigated the effect of storage conditions on walnut quality using transparent and aluminum coated plastic laminates to compare storage under light and in the dark. To accelerate lipid oxidation they flushed the packages with approximately 50% O\textsubscript{2} and 50% N\textsubscript{2} during packing before storage at two temperatures (5 and 21°C).

The objective of the present study was to investigate the effect of storage of wild walnut oil on peroxide value and rancidity by organoleptic properties.

2. METHODS & MATERIAL

2.1 Wild walnut oil
The wild walnut oil, which was extracted during this project investigation by using soxhlet method and evaluate the all phyco-chemical properties. Wild walnut oil containing high amounts of omega-3 fatty acid, with a yellowish colour. The wild walnut oil only filtered with filter paper. Wild walnut oil was packed in glass bottle and stored at optimum temperature. Physico-chemical characteristics of the wild walnut oil are shown in Table 1.

Table 1. Physico-chemical properties of wild walnut oil

<table>
<thead>
<tr>
<th>Properties</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Relative Density</td>
<td>0.98±0.0012</td>
</tr>
<tr>
<td>Refractive index</td>
<td>1.4704±0.00</td>
</tr>
<tr>
<td>Butyro meter</td>
<td>67.00±0.001</td>
</tr>
<tr>
<td>Peroxide value</td>
<td>67.00±0.23</td>
</tr>
<tr>
<td>Free fatty acid</td>
<td>0.252±0.06</td>
</tr>
<tr>
<td>Acid value (mg)</td>
<td>0.283±0.12</td>
</tr>
<tr>
<td>Iodine value</td>
<td>144.81±6.71</td>
</tr>
<tr>
<td>Saponification</td>
<td>196.46±7.14</td>
</tr>
<tr>
<td>Unsaponification</td>
<td>5.07±0.73</td>
</tr>
</tbody>
</table>

*Mean ± standard deviation

2.2 Peroxide value
Primary oxidation products – hydro peroxides- are determined by peroxide value measurement. Peroxide values of oils were measured by titration of liberated iodine with standardized sodium thiosulphate solution according to the AOAC official method 965.33 (AOAC 1990). Peroxide value was determined with known weight if oil dissolved in a mixture of acetic acid/ chloroform (3:2 v/v), and saturated solution of KI was then added. The liberated iodine was titrated with sodium thiosulphate solution (0.05 m) in the presence of starch as indicator.

2.3 Sensory evaluation
Organoleptic evaluation was performed for pasta salad with dressings of wild walnut oil, following their storage time, simultaneously with oxidative stability measurements. The analytical panel, consisting of 20 untrained members, was asked to make its evaluation based on rancidity. Panelists were chosen using the following criteria: ages between 18 and 60, non-smokers. For sample evaluation, the panelists were served a control reference sample, which is without dressing of wild walnut oil along with the test samples. The panelists were students and members of the staff of the Laboratory and were asked to score the samples by smelling and tasting them. The samples were served in (20 g in each plate) at room temperature with water and paper ballots on a tray. Panelists were instructed to consume the whole sample and rinse mouth with sparkling water (room temperature), in between sample evaluation. Rancidity grading was based upon a four-point intensity scale: 1 = no perception; 2 = weak; 3 = medium; 4 = extreme (Jellinek, 1984).

2.4 Statistical analysis
Statistical analysis was done on the data by analysis of variance (ANOVA) on Surface Response Methodology. RSM were used for multivariatative variance analysis test at (p<0.05). Whenever ANOVA indicated a significant, a pair-wise comparison of means by least significance difference (LSD) was carried out.

3. RESULT & DISCUSSION
Primary oxidation of oils was determined by measuring peroxide value. Changes in peroxide values of the wild walnut oil are shown in Figure 1.

![Chart Title](Fig 1 Effect of storage on Peroxide Value (meq/kg) of wild walnut oil)
Initially, there were little changes in the peroxide values of the wild walnut oil. The initial PV of fresh wild walnut oil was very low (1.03 meq O2/kg walnut oil). Namely, the peroxide value increased from approximately 1.04 to 8.35 (meq/kg) during storage of up to 45 days. However, after that, a rapid increase in peroxide value was observed and at end of the experiment (90th day of storage) that value had increased to 18.68 (meq/kg). According to Savage, McNeil, and Osterberg (2001) fresh walnuts had a PV ranging from 0.05 to 0.29 meq O2/kg walnut oil. Marcela Martínez et al. (2013) reported that fresh walnuts had peroxide value at 0 days (0.55±0.06 meq/kg oil), 90 days (22.25±0.06 meq/kg oil), and 180 days (70.92±0.01 meq/kg oil) in light condition. These values are higher than those of the present work in the range of 4.0 meq O2/kg walnut oil. Differences may be attributed to the accelerated conditions of storage (50% O2) used in the former work. Maté et al. (1996) studied the influence of O2 concentration and relative humidity on the rancidity of walnuts and reported PV between 1 and 2 meq O2/kg walnut oil for products stored at low O2 (<2.5%) and either 21% or 53% RH at 37°C. Respective PV for high O2 (21%) storage after 10 months were 5.0 meq O2/kg walnut oil at 21% and 13.5 meq O2/kg walnut oil at 53% RH. Peroxide values of walnuts packaged in low O2 environments are similar to those reported in the present study given the differences in experimental temperatures employed. 

The results indicate that for safe consumption of this wild walnut oil, a maximum of 45 days shelf life is suggested under storage conditions of domestic consumption. 

**Sensory studies**

Sensory evaluations have been carried out to characterize the salad dressings and to find a correlation with chemical analysis. The sensory evaluation of the salad dressings appears to be necessary since the increased shelf life of wild walnut oil. Results of assessment of flavor and taste of wild walnut oil are shown in Fig 2. As shown in Fig. 2, after 15 days of storage at room temperature, taste and flavor was change. The rating of the taste and flavor was goes on decreasing during the storage of wild walnut oil due to the rancidity of the oil was increases. This decrease in the rating the acceptance of wild walnut oil also goes on decreases.

**Fig. 2 Effect of storage on rancidity of pasta salad using wild walnut oil as dressing**

### 4. CONCLUSIONS

Wild walnut oil can be stored for up to 90 days in glass containers at optimum temperature. Under conditions of domestic consumption of wild walnut oil, little changes were observed in the peroxide values of the wild walnut oil. There is increase in PV value from 1.03 to 18.68 meq/kg of oil. A peroxide value of 8 meq/kg, an acceptability limit of oil for human consumption, was reached in 45 days for safe consumption of wild walnut oil. Rancidity was also goes increasing. Due to that, acceptability for consumer was decreases. Wild walnut oil, a maximum of 45 days shelf life is suggested.

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### References


