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# The qualitative analysis of some types of oils used in alimentation

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***Abstract***

Lately, several types of oils have been made available on the market to complete the range of edible oils in our country. These include walnut oil and grape seed oil, which are distinguished particularly by the special olfactory-gustatory qualities and the nutritional principles obtained only due to the cold pressing. One of the major disadvantages of using the studied oils is that they rapidly oxidize in contact with air, leading to storage under special and short term conditions, and they lose rapidly their properties by heating.

This study aims to present the qualitative analysis of the main quality indices of the studied oil samples (acidity index and peroxide index) over a 5-month period, the samples being stored at room temperature in contact with the air (a part of the samples) and in natural light, conditions specific also for the domestic kitchens.

***Keywords***: nut and grape seed oil, qualitative analysis

## 1. Introduction

Since most of the time the oils purchased for household consumption when unsealed until their first use remain in contact with the air for a longer period of time (depending on the rate of use by the consumer), it becomes necessary to analyse them in order to determine when these oils meet no longer the qualitative expectations imposed by the consumer safety standards into force.

Fats have an energetic role in living organisms, being important in the fight against cold, contribute to the absorption of fat-soluble vitamins, elimination of the bile, improve the taste qualities of food and determine saturation [1, 5, 6, 12] .

In general, edible vegetable oils obtained by cold pressing abound in nutrients and unsaturated fatty acids, which distinguishes them from other types of oils made in our country both in terms of composition and storage conditions [15, 7] .

Recent studies on the properties of grape seed oil have determined that it has curative properties due to the high content of polyunsaturated fatty acids (ω and ω-6), natural antioxidants such as vitamin E (α, β, γ) and polyphenols (proanthocyanidins), which demonstrates the increased biological value of this product [8, 9,14] .

Both grape seed oil and walnut oil are a rich source of antioxidants (vitamin E) and essential fatty acids. From the essential fatty acids that are absolutely essential to the human body, the linoleic acid has been identified in high amount in the grape seed oil, which has positive effects on the cardiovascular, circulatory, immune system, contributing to the maintenance of the elasticity of the blood vessels, the lowering of the cholesterol level, prevention of high blood pressure, effective fight against free radicals [3, 4, 11, 13, 10].

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From this point of view, special attention is paid to these types of oil, much appreciated in recent years due to the increased antioxidant potential, which is a source of health for the body, provided that the rules of Stas/Standard regarding storage conditions and consumption are followed.

## 2. Materials and Methods

In order to conduct a qualitative analysis of the two types of studied oils (grape seed oil and walnut oil), the acidity index and the peroxide index were determined for a period of 5 months after unsealing the containers.

The acidity index is the amount of KOH, in mg, required to neutralize the free fatty acids in one gram of fat.

Samples of liquid, homogeneous and clear oils at room temperature are analysed as they are, without any transformation. The oil dissolved in ethanol is titrated with 0.1 N NaOH solution in the presence of phenolphthalein.

The acidity is expressed in % oleic acid and is calculated according to the related calculation formula.

The peroxide index shows the degree of rancidity of a fat and it is expressed by the number of milliliters of 0.002 N sodium thiosulfate consumed per gram of fat.

The oil is titrated in acetic acid and chloroform, with potassium iodide solution. The liberated iodine is titrated with a sodium thiosulfate solution with known titre, according to the current Stas/Standards into force. Peroxides have the property of decomposing potassium iodide by liberating the iodine. It is neutralized with n/100 sodium thiosulphate in the presence of starch indicator solution until clarification. The peroxide index can be expressed in milliequivalents per kilogram of product, in micrograms of active oxygen per 1 gram of product, in millimoles of peroxide per 1 kilogram of product. Two determinations are conducted from the same sample for analysis, and as a result the arithmetic mean is taken into consideration if the difference between them does not exceed 0,5 milliequivalents per kilogram of product.

The organoleptic analysis (appearance, odour, taste, colour) of the studied oils was carried out under normal laboratory conditions at a constant temperature of 20°C and under natural light.

To determine the appearance, the oil is placed in a Berzelius glass beaker so as to cover half of the container surface. It is brought to the eye level and it is examined in the presence of natural light. It will be seen whether the analysed oil is or is not turbid and whether it contains or not sediment. For odour analysis, it is inhaled quickly from Berzelius glass beaker covered for several seconds. The intensity of the raw material from which the studied oils originated was observed. The taste was appreciated by tasting the sample at a temperature of 20-25°C, trying to observe the nature of the raw material from which the samples originated and whether there is a strange taste: altering, pungent, bitter or rancid. The colour was observed in natural light. Ascertained aspects are transferred to the laboratory register [2].

## 3. Results and Discussions

In this study, four laboratory samples consisting of two samples of walnut oil and two samples of grape seed oil were made. Considering that in domestic use, the opened oil usually remains in contact with the air, two samples of intact content were formed, these being unsealed and noted with $PN_{500}$ and $PS_{500}$. The other two samples were formed from the initial samples (500 ml) from which 250 ml were taken out. The oil samples formed from the remaining oil in the container (in contact with the air) were marked with $PN_{250}$ and $PS_{250}$. In order to establish the consumption time after unsealing the containers, the main quality indices (acidity index and peroxide index) were observed for a period of 5 months.

The data obtained were recorded in a register and compared with the current requirements into force for each category of oil. Although the storage temperature was 6-8°C, there was observed that after 2 months of unsealing, the values of the halved oil samples (in contact with air) were much altered compared to the oil samples taken from full containers (the initial sample). Table 1 shows a continuous increase in the peroxide index of the initial sample until the 5th month of determination when a significant increase of the peroxide index in the walnut oil mass is observed. The recorded values in the initial sample of walnut oil (and 5.3 meq active $O_2$/kg for $PN_{250}$, 5.3 meq active $O_2$ / kg for $PN_{500}$ respectively) show that the oil samples are fresh. The peroxide index records a faster increase in the mass of packed oil in a 500-ml container, that was halved ($PN_{250}$ and $PS_{250}$ samples).

If the initial walnut oil samples recorded a peroxide index of 5.3 meq active $O_2$/ kg, after 4 months of storage the peroxide index value increases a lot for $PN_{250}$ (12.8 meq active $O_2$/kg) in comparison to $PN_{500}$ which recorded 7.4 meq active $O_2$/kg.

***Table 1.*** The determination of peroxide index IP (meq O2 activ/kg)

| Sample oil | Storage Period | | | | | |
|---|---|---|---|---|---|---|
| | Initial sample Pi | 1 month | 2 months | 3 months | 4 months | 5 months |
| $PN_{500}$ | 5,3 | 5,7 | 6,2 | 6,8 | 7,4 | 8,8 |
| $PN_{250}$ | 5,3 | 7,9 | 8,8 | 11,5 | 12,8 | 15,6 |
| $PS_{500}$ | 4,4 | 5,1 | 6,0 | 6,7 | 7,2 | 8,6 |
| $PS_{250}$ | 4,4 | 6,2 | 7,9 | 9,9 | 11,2 | 12,8 |

In which: Pi - initial sample after unsealing the PET; $PN_{250}$ - 250 ml cold pressed nut oil in contact with air; $PN_{500}$ - 500 ml cold pressed nut oil; $PS_{250}$-250 ml cold pressed grape seed oil in contact with air; $PS_{500}$ – 500 ml cold pressed oil from grape seed.

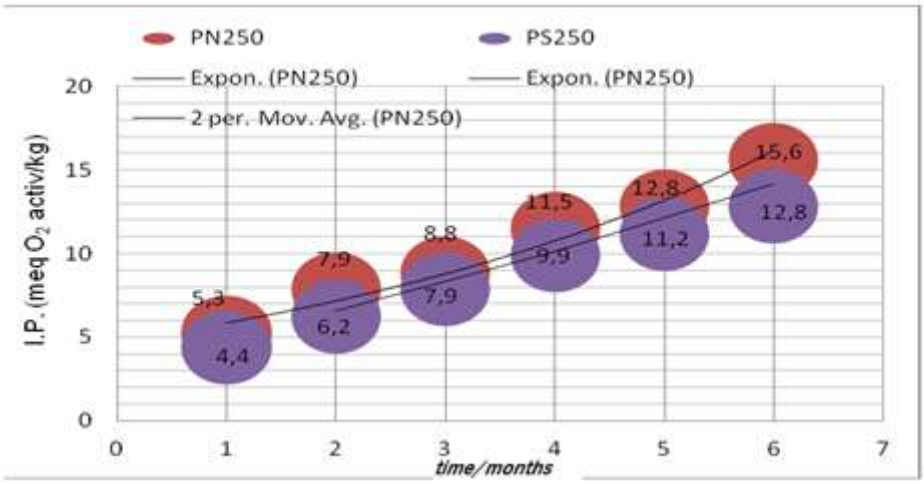


***Figure 1.*** The determination of peroxide index (meq active $O_2$/kg) in oil samples kept in contact with air ($PS_{250}$ and $PN_{250}$)

***Table 2.*** The determination of the acidity index IA (mg KOH/g)

| Oil sample | Storage Period | | | | | |
|---|---|---|---|---|---|---|
| | Initial sample Pi | 1 month | 2 months | 3 months | 4 months | 5 months |
| $PN_{500}$ | 0,7 | 1,0 | 1,5 | 2,2 | 2,8 | 3,5 |
| $PN_{250}$ | 0,7 | 1,3 | 1,9 | 2,8 | 3,9 | 4,2 |
| $PS_{500}$ | 0,9 | 1,1 | 1,3 | 1,8 | 2,2 | 2,8 |
| $PS_{250}$ | 0,9 | 1,2 | 1,5 | 2,1 | 2,9 | 3,3 |

In which: Pi - initial sample after unsealing the PET; $PN_{250}$ - 250 ml cold pressed nut oil in contact with air; $PN_{500}$ - 500 ml cold pressed nut oil; $PS_{250}$-250 ml cold pressed grape seed oil in contact with air; $PS_{500}$ – 500 ml cold pressed oil from grape seed, m- month.

For samples kept in contact with air (250 ml) from the third month of storage, the peroxide index value exceeds the maximum value indicated by the current standard for walnut oil of 10 meq active $O_2$/kg. This shows that the oxidation process of the oils taken into consideration for the analysis starts from the 3rd month of storage. The idea of halving the content of the analysed containers is in line with the domestic use of the oil.

After 5 months of storage, although the value of the peroxide index for $PN_{500}$ increases, the oil partially changes its colour, has no sediments (as noted at $PN_{250}$), the odour being slightly pungent, compared to the heavy odour of the $PN_{250}$ oil sample.

Analyzing the grape seed oil with the two samples that were formed just like in the case of the walnut oil, a lower peroxide index is observed compared to that of the walnut oil. Thus starting from 4.4 meq active $O_2$/kg, the peroxide index reached after 5 months of storage a value of 12.8 meq active $O_2$/kg, proving an advanced degree of oxidation due to the prolonged contact with the air, therefore the product is altered and becomes inappropriate for human consumption ($PS_{250}$).

Analyzing comparatively the 250 ml samples from both the analyzed oils, a higher peroxide index in walnut oil (PN250) was observed throughout the analysis period in comparison to $PS_{250}$. The peroxide index values indicate a good oil for consumption of up to 3 months (excluding the walnut oil that exceeded the standard value of 10 milligrams of active $O_2$/kg reaching 11.5 meq active $O_2$/kg).

Although the grape seed oil values are lower compared to $PN_{250}$, after 5 months of storage the samples are altered and cannot be used in alimentation, the values being above the limit required by the standards.

Regarding the analysed oils packed in 500 ml unsealed containers, the value of the peroxide index increases constantly over the study period and can be consumed throughout the storage period, with the peroxide index not exceeding the standard value.

In general terms, the oil acidity is the percentage of free fatty acids present in oil indicating the degree of oil degradation.

Table 2 shows a constant increase in acidity from the moment of unsealing the containers until the completion of analyses.

Although the growth is constant, an upward trend is observed especially at the $PN_{250}$ samples which at the end of the determination period exceed the value indicated in the Stas/Standards, expressing it in mg KOH/g (4 mg KOH/g).

The optimum for walnut oil samples kept in contact with air in terms of acidity is no more than 2 months (1.9 mg KOH/g for $PN_{250}$ and 1.5 mg KOH/g for $PS_{250}$ respectively), the values recorded in the 3rd and 4th month indicating an oil at the beginning of degradation, the taste and smell being degraded (especially noticeable during the 4th month at sample $PN_{250}$).

An acidity at the initial sample for grape seed oil samples, expressed in mg KOH/g is observed and it is higher than that recorded in walnut oil samples, but during storage, the increase is constant reaching 5 months at a value of acidity lower than that of walnut oil kept in contact with air. The samples of 500 ml have a normal acidity, although it increases continuously during the study; the grape seed oil samples record a lower acidity than $PN_{500}$ at the end of storage ($PS_{500}$ after 5 months reaches an acidity value of 2.8 mg KOH/g compared to 3.5 mg KOH/g recorded at $PN_{500}$ (Figure 2).

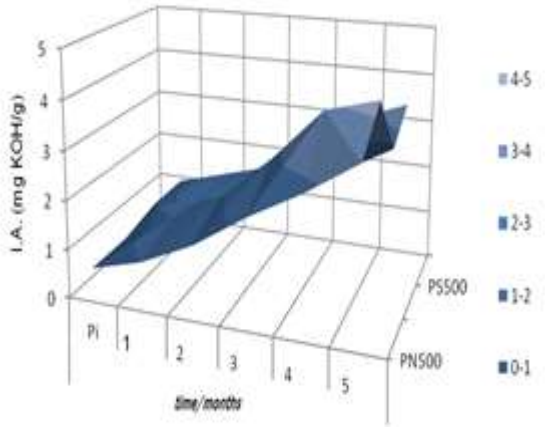


***Figure 2.*** The determination of the acidity index (mg KOH/g)

During the study period, the acidity of the $PS_{250}$ and $PS_{500}$ samples increased constantly but not with the same intensity as for the $PN_{250}$ and $PN_{500}$ samples.

## 4. Conclusions

The assessment of the degree of oxidative alteration of the analysed oils is one of the most important analysis to be carried out on the oil samples obtained by cold pressing.

The oxidative alteration takes place faster in the case of nut oil packed in 250 ml containers in contact with air than the oil packed in 500 ml containers. The longer the storage period is, the higher the degree of oxidation of the analysed oils is, with higher values recorded by the oil stored in contact with the air.

If the walnut oil peroxide index value indicates a shelf life of up to 2 months ($PN_{250}$), the grape seed oil can be consumed for up to 2 months and a half -

3 months (when it has a maximum value of Ip according to the quality standard).

In this respect, the consumption of the analysed oils kept in contact with the air is recommended for a maximum period of 2 months and the consumption of the oils packed in 500 ml glass container is recommended for 3-4 months, because after this period the oxidative degradation phenomenon gradually appears.

Although the peroxide index values recorded by the grain seed oil are lower compared to the values recorded by walnut oil, after 4 months of storage the samples kept in contact with the air are qualitatively altered and cannot be used in alimentation.

The determination of the acidity index of the analysed oils shows that oil samples stored in 500 ml containers at the end of the storage period record normal values for both categories of oils.

Although the growth is constant, the acidity trend is more pronounced particularly at the whole walnut oil samples compared to grape seed oil samples that are not kept in contact with air, at the end of the determination period (5 months) the values are reaching the limit.

The optimal acidity value in walnut oil samples kept in contact with air is up to 2 months for consumer safety, higher at $PN_{250}$ compared to $PS_{250}$, the values recorded in the $4^{th}$ month of analysis indicating a beginning of alteration of the oil, the taste and smell being altered (particularly noticeable at sample $PN_{250}$).

During the study period, the acidity of the $PS_{500}$ and $PN_{500}$ samples increased constantly but not with the same intensity as for the $PN_{250}$ and $PS_{250}$ samples.

As a result, according to the obtained data, a higher risk of losing quality is represented by the $PN_{250}$ walnut oil (oil kept in contact with the air) followed closely by the $PS_{250}$ grape seed oil which besides the oxidative alteration and high acidity also showed a coarse sediment during the middle of the storage period, which will bring a change in colour and smell, thus a qualitative alteration.

It is recommended in domestic use to purchase the two types of studied oils (cold pressed nut oil and grape seed oil) in small containers (up to 250 ml) and to consume them in a period of 3 months after unsealing the containers which are being kept under appropriate storage conditions.

**Compliance with Ethics Requirements.** Authors declare that they respect the journal's ethics requirements. Authors declare that they have no conflict of interest and all procedures involving human / or animal subjects (if exist) respect the specific regulation and standards.

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