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ANALYTICAL METHODS FOR ANALYZING SOME OILS USED IN COSMETICS

BY

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Abstract. In this paper three different oils have been analysed: sesame, olive and jasmine. For their analyses standard methods for oils and fats described by American Oil Chemists Society (AOCS, 1993) and pharmacopoeia assays (Brazilian pharmacopoeia, 2010), such: index of acid, saponification, iodine, peroxides, determination of potential of hydrogen (pH), and density. Those analyses are important because they indicate the quality and authenticity of the oil.

The results showed that the oil extracted from sesame and olive has acceptable characteristics and is of good quality. For all of those the saponification index, the acidity and peroxidation value have been determined.

Keywords: index of acid; index of saponification; index of peroxide; olive; jasmine; sesame oils.

1. Introduction

This article presents the use of titration in order to analyse the quality indicators for fat vegetable oils. Oils are obtained with an yield of 30-50% from different seeds, fruit (olives) and are used in pharmaceutical industry as a solvent of the soluble drug substances, as well as in the preparation of ointments, emulsions.

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Three oil samples (olive, jasmine, and sesame) were analysed and several chemical characteristics were determined, namely: saponification value, the acid value and peroxide index of these oils that can be used in cosmetics. These results were compared with the values obtained from standards tables and were found to be consistent with them.

2. Experimental

Determination of the saponification number

It is the amount in milligrams of potassium hydroxide required to saponify 1 g of oil or fat. The method recommended by the AOCS (AOCS, 1993) was applied.

Materials: Hydrochloric acid, 0.5 N, Potassium hydroxide alcoholic solution, 0.5N, Phenolphthalein, alcoholic solution, 1%, Benzene or anhydrous toluene.

Methods

• Weigh 2 g of oil into a 250 mL conical flask.

• Add 25 mL of KOH 0.5 N.

• Attach to a condenser and heat on a water bath for 30 min.

• Add 5 drops of phenolphthalein and titrate with hot 0.5 N HCl solution until discoloration. Perform a blank without oil, then titrate exactly the same conditions for the establishment of the alkaline solution of potassium hydroxide.

For the calculation of the saponification number, the following formula has been used:

$$I_s = \frac{28.052(V - V_1)}{M} \quad (1)$$

where: 28.052 mL = mg KOH corresponding to 1mL 0.5N HCl;

V = mL 0.5N HCl used in the blank titration;

V_1 = mL 0.5N HCl used in titration of the sample of oil;

M = grams of oil taken for determination.

To calculate the correction factor of 0.5 N HCl, I used three Erlenmeyer flasks were used, each of them having the following solutions:

-10 mL standard solution of borax ($Na_2B_4O_7$ x 10 H_2O) 0.5 N

-2-3 drops of methyl red indicator and we titrated with 0.5 N HCl until color change from yellow to red, giving the following results:

$$F = V_1 / V_2 \quad (2)$$

where: V_1 is the volume of the standard solution; V_2 – volume used for titration.

F_1 = 10 mL / 10.2 = 0.9803

F_2 = 10 mL / 10.2 = 0.9803

F_3 = 10 mL / 10.3 = 0.9708

$F = F_1 + F_2 + F_3 / 3 = 0.9803 + 0.9803 + 0.9708 = 0.9771$

Next we applied the previous described procedure was applied for the three samples of oil (olive, jasmine and sesame), and the results are listed in Table 1.

Table 1
The Saponification Number for the Three Oil Samples

Oil	V_1(mL) HCl 0.5 N	Oil Mass (g)	V(mL) HCl 0.5 N	Saponification value mg KOH/1g sample
olives	10.5	2	10.9	5.44
jasmine	9.7	2	10.9	16.44
sesame	10.6	2	10.9	4.11

Determination of acidity

This index is expressed as the number of milligrams of potassium hydroxide required to neutralize the free acids of 1 g of sample (Bejan *et al*., 2006).

Materials: Potassium hydroxide, 0.1 N; Ethanol-benzene mixture 1:2; Phenolphthalein, alcoholic solution, 1%;

Experimental procedure

In a conical flask 10 g of the oil, 40 mL of the mixture ethanol-benzene neutralized and few drops of phenolphthalein are added. The solution is then titered with 0.5 N KOH solution, until the color persists for about 1 min.

If the mixture becomes cloudy, the dish is gently heated on a boiling water bath.

The acid number is determined by the following formula:

$$Ia = (5.6104\ V)/M \tag{3}$$

where: V = mL titration using KOH of 0.1 N,
M = grams of oil,
5.6104 = mg KOH for 1 mL HCl of 0.1N.

To determine the correction factor of 0.1 N KOH, three Erlenmeyer flasks were used, in each being added the following (Bulgariu, 2010):

- 5mL $H_2C_2O_4$ standard 0.1 N;
- indicator methyl red; and I titrated with 0.1 N KOH until the colour change from red to obtain yellow colour, achieving the following results:

$$F = V_1 / V_2, \tag{4}$$

where: V_1 is the volume of the standard 5 mL;
V_2 = volume used for titration.
$F_1 = 5\ mL / 5.2 = 0.9615$

F_2 = 5 mL / 5.1 = 0.9803

F_3 = 5 mL / 5.2 = 0.9615

F = F_1 + F_2 + F_3 / 3 = 0.9615 + 0.9803 + 0.9615 = 0.9677

By working with the three samples of oil (olive, jasmine, and sesame oils), and using the procedure describes above, were obtained the results presented in Table 2 were obtained.

Table 2

Determination of the Acid Value

Oil	V(mL) KOH 0.1 N	Oil mass (g)	Acid value (mg KOH/1g oil)
olives	3.5	10	1.9
jasmine	3.8	10	2.06
sesame	4.8	5	5.21

Determination of peroxide

The peroxide value is the volume of sodium thiosulphate 0.002 N used in the titration of 1 g of oil.

Materials**:** Acetic acid conc., Chloroform; Potassium iodide; Sodium thiosulfate 0.01 N and 0.002 N; Starch, fresh solution, 1%;

Methods

In a heat-resistant tube, 1g of oil is weighted. Add 1 g of KI and 20 mL of a mixture of glacial acetic acid and chloroform 2:1. The tube is heated on a low flame until boiling, after which and the temperature is maintained for about 30 sec. After cooling the tube content was transferred into a vial containing 3 mL of distilled water and titrate with 0.01 N sodium thiosulfate to light yellow coloration. Add 1 mL of starch solution and the solution is titrated again with the sodium thiosulfate. 0.01 N until a persistent blue color appear. At the same time a control sample with oil is performed, making the titration with sodium thiosulphate 0.002 N solution. The peroxide index is calculated using the following formula (Bejan *et al*., 2006):

$$I_p = \frac{5(V - V_1)}{M} \quad (5)$$

where: V = mL $Na_2S_2O_3$ 0.01N, consumed in the titration of the sample oil;

V_1 = mL $Na_2S_2O_3$ 0.002 N consumed in the blank titration;

M = grams of oil taken for analysis;

5 = conversion factor of 0.01 N solution in 0.002 N;

By working with all the three samples of oil (olive, jasmine, and sesame oils), were obtained the results showed in Table 3.

Table 3
Determination of Peroxide Value

Oil	V(mL) $Na_2S_2O_3$ 0.01N	Oil mass (g)	V_1(mL) $Na_2S_2O_3$ 0.002N	Peroxide value, I_p (mL/g)
olives	10.5	1	4.35	30.75
jasmine	7.5	1	4.35	15.75
sesame	9	1	4.35	23.25

Discussion

Chemical characteristics varied with the type of oil used, as follows:

• Index of saponification (specific fatty acid composition of oil) is much higher for the jasmine oil than for olive oil and the sesame oil;

• The acid value (which is the amount of KOH required to neutralize the fatty acids in 1 gram of fat) is twice as large for the sesame oil compared to the other samples;

• The highest index of peroxide is observed for the olive oil.

3. Conclusions

When the molecular weight of the fatty acids is bigger, it requires a smaller amount of KOH; so saponification gives indications on the chemical composition of the oil searched;

The fat must have a very low acidity. If the acidity is high, it denotes a process of hydrolysis or that the refining process was not properly carried out.

Peroxide index shows the degree of rancidity of fats. Polyunsaturated fats containing acids have higher oxidation capacity (olive oil over other oils studied).

All the parameters that have been determined are in accordance with the standard 13531/2008 (Food Products, Determination of peroxide index, 2008).

High unsaponifiable matters content (1.76%) guarantee the use of the oils in cosmetics industry (Brazilian Pharmacopoeia, 2010).

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METODE ANALITICE DE ANALIZĂ A UNOR ULEIURI UTILIZATE ÎN COSMETICĂ

(Rezumat)

În această lucrare au fost analizate trei uleiuri diferite: ulei de susan, ulei de măsline și de iasomie. Metodele de analiză standard pentru uleiuri și grăsimi descrise de American Oil Chemists Society (AOCS, 1993) și testele din farmacopee (Brazilian Pharmacopoeia, 2010), cum ar fi indicele de aciditate, saponificare, iod, peroxizi, determinarea pH-ului și a densității sunt importante, deoarece acestea indică autenticitatea și calitatea uleiului. Rezultatele au arătat că uleiurile extrase din măsline, susan, iasomie au caracteristici acceptabile și sunt de bună calitate. În laborator s-au determinat indicele de saponificare, indicele de aciditate și indicele de peroxid al probelor de ulei mai sus amintite, iar rezultatele au fost prezentate în acest articol.