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Isolation of Oleic Acid from Virgin and Extra Virgin Olive Oil and Study Their Physico-Chemical Properties

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ABSTRACT:
The iodine number for Extra virgin olive oil and oleic acid is high in oleic acid has greater resistance to oxidation and degradation. The peroxidation of the oil is primary arising because of the oxidation process, high temperature and visibility to light. The lower this number the longer the oil will retain the shelf life and will delay the possibility of rancidity, while high peroxide number usually indicates poor processing, and that the quality of the oil is low. Setting a low peroxide number standard for olive oil means that it will be more stable. The peroxide number of different grades of olive oil and oleic acid were measured. It was observed that Extra virgin olive oil and oleicacid have more saponification number than virgin olive oil because they have a relatively fewer number of carboxylic functional groups per unit mass of the fat as compared to short chain fatty acids (has long chain fatty acids).
Keywords: *Olive oil,*

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I. INTRODUCTION:

Olive oil is a liquid obtained from olives (the fruit of Olea europaea; family Oleaceae), a traditional tree crop of the Mediterranean Basin. The oil is produced by pressing whole olives. It is commonly used in cooking, whether for frying or as a salad dressing [1]. It is also used in cosmetics, pharmaceuticals, and soaps, and as a fuel for traditional oil lamps, and has additional uses in some religions. There is limited evidence of its possible health benefits [2]. The olive is one of three core food plants in Mediterranean cuisine; the other two are wheat and grapes. Olive trees have been grown around the Mediterranean since the 8th millennium BC [3].

The types of olive oil are extra virgin, virgin, refined, olive pomace and lampante oils. Olive oil is composed mainly of triacylglycerols (triglycerides or fats) and contains small quantities of free fatty acids (FFA), glycerol, phosphatides, pigments, flavor compounds, sterols, and microscopic bits of olive [4]. Triacylglycerols are the major energy reserve for plants and animals [5]. Chemically speaking, these are molecules derived from the natural esterification of three fatty acid molecules with a glycerol molecule. The glycerol molecule can simplistically be seen as an "E-shaped" molecule, with the fatty acids in turn resembling longish hydrocarbon chains, varying (in the case of olive oil) from about 14 to 24 carbon atoms in length [6].

Oleic acid is a fatty acid that occurs naturally in various animal and vegetable fats and oils. It is an odorless, colorless oil, although commercial samples may be yellowish. In chemical terms, oleic acid is classified as a monounsaturated omega-9 fatty acid, abbreviated with a lipid number of 18:1 cis-9. It has the formula CH_3-$(CH_2)_7$-CH=CH-$(CH_2)_7$-COOH[7]. The name derives from the Latin word oleum, which means oil. It is the most common fatty acid in nature[4]. The salts and esters of oleic acid are called oleates [8].

The aim of the research is to isolate oleic acid from olive oil. The objectives are as follows: (i) To estimate the iodine number, saponification number, peroxidenumber, free fatty acid number, Acid number and ester number present in virgin and extra virgin olive oils; and (ii) To estimate the iodine number, saponification number, peroxidenumber, free fatty acid number, Acid number and ester number in oleic acid produced from virgin and extra virgin olive oils.

II. MATERIALS AND METHODS:

Materials:

Virgin and extra virgin olive oils were collected from local supermarket in Salalah, Oman. All the other chemicals, reagents, equipments and instruments are used from the various laboratories of Chemical Engineering Section, University of Technology and Applied Sciences, Salalah.

Methods:

Isolation of oleic acid from olive oil [9]:

- Take 10 mLolive oil in beaker.
- Add 10 mL of 3N sodium hydroxide, then stirring.
- Add 10 mL of 3N lead acetate, mix well.
- Separate the precipitate by filtering.
- Dry it in oven.
- Add 10 mL of ether and mix well.Then, filter.
- Filtrate is evaporated.
- Residue is heated in 1N concentrated HCl.
- Then,oleic acid layer will form.

Acid number:

- Place 5.0 g of fat or oil in a dried conical flask.
- Add 25 mL of absolute ethanol and add (2-3) drops of phenolphthalein.
- Heat with shaking in water bath (65%) for 10 mins, and then cool.
- Titrate the solution against 0.1 N KOH until pink color appears (end point).
- Record your observations.
- Calculate the acid number (AV) and free fatty acid (%FFA) using below equation:

Acid number = mL of KOH x N x 56/Mass of sample

Free fatty acid value = Acid value x 0.503

Iodine number:

- Weight approximately 0.25g of the oil into a 250mL Conical Flask.
- Add 10 mL of chloroform.
- Add 30mL of Hanus Solution and close the flask completely by pare film, the leave the solution for 40 min with shaking continuously.
- Add 10mL of 15% potassium iodide solution and then shake.
- Add 100 mL of distilled water.
- Titrate the iodine solution against 0.1 N sodium thiosulphate till yellow color formed, then add 2 – 3 drops of starch where blue solution formed and then continue with titration till the color is disappeared.
- Do same above procedure but without sample (volume of $Na_2S_2O_3$ at end point represents B) (Volume (mL) of $Na_2S_2O_3$ at end point represents S)
- Calculate the iodine number as follows:

Iodine number = (B – S) x Normality of $Na_2S_2O_3$ x 0.127 g/meq x 100/Mass of sample

Peroxide number:

- Take 1 gram of oil and 1 gram of KI and 20 mL of solvent mixture.
- Boiling for 30 seconds.
- Transfer sample into conical flask contains 20 mL of 5% KI and 20 mL +20 mL of water wash.
- Titrate the sample with 0.002 N $Na_2S_2O_3$ till light yellow color appears.
- Add 0.5 mL starch solution.
- Continue titration till bule color disappears.

Peroxide number = (B-S) *N *1000/Mass of sample

Saponification Number:

- Weigh approximately 2 g of the fat or oil into a 250 mL conical flask.
- Add 25 mL of alcoholic potassium hydroxide solution (0.5 N).
- Attach a reflux condenser and heat the flask contents on a boiling water bath for 1 hour with occasional shaking.
- While the solution is still hot, add 3 drops of phenolphthalein indicator and titrate the excess potassium hydroxide with the 0.5 N hydrochloric acid (VmL of hydrochloric acid at end point represents S).
- Do same above procedure but without sample (VmL of hydrochloric acid at end point represents B).
- Calculate the saponification number by using the following formula:

Saponification number = 56 x (B – S) x Normality of HCl/Mass of sample

Ester Number (EV) = Saponification Number (SV) – Acid Number (AV)

% glycerin = Ester Numberx 0.054664

III. RESULTS AND DISCUSSION:

Acid number:

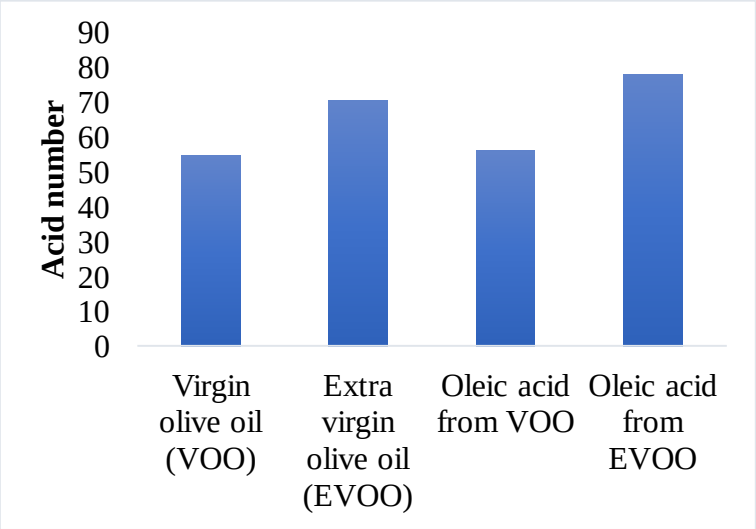

Figure 1. Acid number of virgin and extra virgin olive oils, and oleic acid produced from virgin and extra virgin olive oils.

The acid number(AN) *is* the number that expresses, in milligrams the quantity of potassium hydroxide required to neutralize the free acids present in 1 g of the substance. The acid number may be overestimated if other acid components are present in the system, *e.g.*,amino acids or acid phosphates. The acid number is often a good measure of the breakdown of the triacylglycrols into free fatty acids, which has an adverse effect on the quality of many lipids.Extra virgin olive oil has more acid number than virgin olive oil becauseas oil-fats rancidify, triglycerides are converted into fatty acids and glycerol, causing an increase in acid number. It has more carboxylic acid groups. It has more a monounsaturated omega-9 fatty acid, so Extra virgin olive oil high in oleic acid has greater resistance to oxidation and degradation.

Free Fatty Acid number:

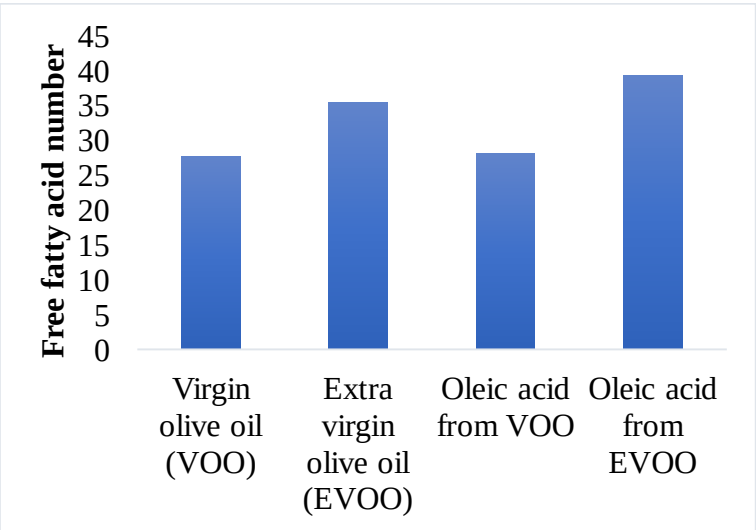

Figure 2. Free fatty acid value of virgin and extra virgin olive oils, and oleic acid produced from virgin and extra virgin olive oils.

The limit for free fatty acid in extra virgin olive oil is 0.8% (0.8g per 100g). Our standard for high quality olive oil is an FFA score below 0.4%. A low FFA is desirable. Free fatty acid speaks to the condition of the fruit at the time of crush. The higher the FFA, the greater the indication of poor-quality fruit such as damaged, bruised, overripe, insect infestation, overheating during production or too much of a delay between harvest and crush. The increase of free acidity in olive oil is due to free fatty acids that are released from triglycerides. The greater the oleic acid, the freer fatty acid.

Iodine Number (IN)

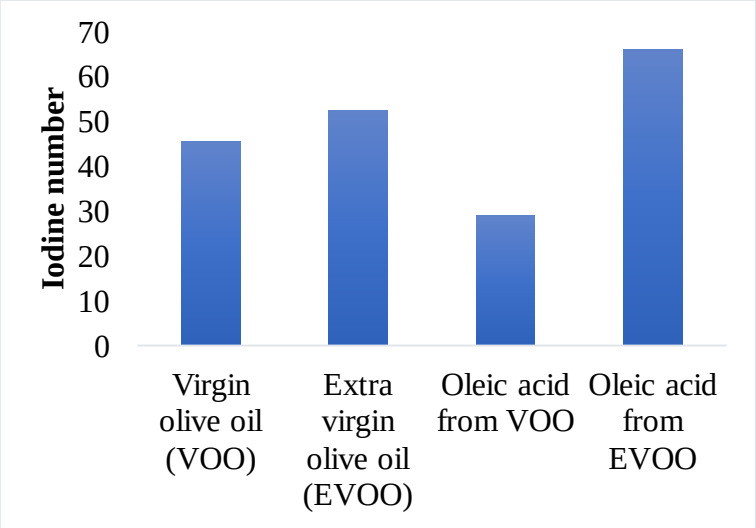

Figure 3. Iodine number of virgin and extra virgin olive oils, and oleic acid produced from virgin and extra virgin olive oils.

The iodine number (IN) gives a measure of the average degree of unsaturation of a lipid: the higher the iodine number, the greater the number of C=C double bonds. The iodine number is expressed as the grams of iodine absorbed per 100g of lipid. Iodine number (IN) is directly proportional to the degree of unsaturation (No of double bonds.) and inversely proportional to the melting point (M.P.) of lipid. An increase in IN indicates high susceptibility of lipid to oxidative rancidity due to high degree of unsaturation. One of the most used methods for determining the iodine number of lipids is "Hanus method". The lipid to be analyzed is weighed and dissolved in a suitable organic solvent, to which a known excess of iodine chloride is added. Some of the IBr reacts with the double bonds in the unsaturated lipids, while the rest remains:

$$R\text{-}CH{=}CH\text{-}R + IBr_{excess} \rightarrow R\text{-}CHI\text{-}CHBr\text{-}R + IBr_{remaining}$$

The amount of IBr that has reacted is determined by measuring the amount of IBr remaining after the reaction has gone to completion ($IBr_{reacted} = IBr_{excess} - IBr_{remaining}$). The amount of IBrremaining is determined by adding excess potassium iodide to the solution to liberate iodine, and then titrating with a sodium thiosulfate ($Na_2S_2O_3$) solution in the presence of starch to determine the concentration of iodine released:

$IBr_{remaining} + 2KI \rightarrow KBr + KI + I_2$

I_2 + starch + $2Na_2S_2O_3$ (blue) $\rightarrow$ 2NaI + starch + $Na_2S_4O_6$ (colorless)

The greater the iodine number, the more unsaturation and the higher the susceptibility to oxidation.

Saponification Number

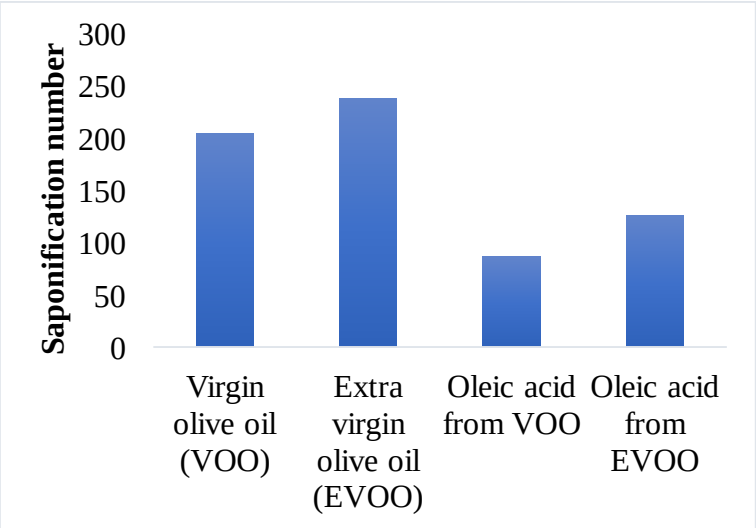

Figure 4. Saponification number of virgin and extra virgin olive oils, and oleic acid produced from virgin and extra virgin olive oils.

The saponification number is the number of mg of potassium hydroxide required to neutralize the free acids and to saponify the esters in 1 g of the substance. The saponification number is a measure of the average molecular weight of the triacylglycerols in a sample. Saponification is the process of breaking down a neutral fat into glycerol and fatty acids by treatment with alkali. The smaller the saponification number the larger the average molecular weight of the triacylglycerols present i.e., Saponification number is inversely proportional to the mean molecular weight of fatty acids (or chain length).

Extra virgin olive oil has more saponification number than virgin olive oil because they have a relatively fewer number of carboxylic functional groups per unit mass of the fat as compared to short chain fatty acids (has long chain fatty acids).

Ester Number

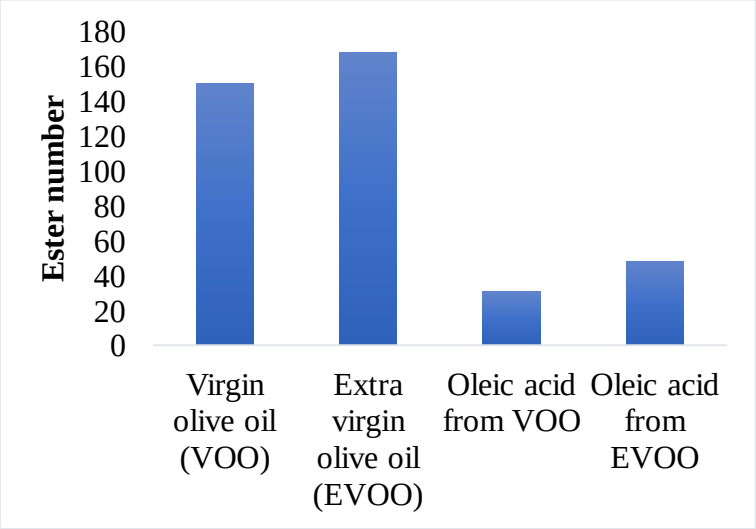

Figure 5. Ester number of virgin and extra virgin olive oils, and oleic acid produced from virgin and extra virgin olive oils.

The ester number is defined as the mg of KOH required to react with glycerin (glycerol / or glycerin) after saponify one gram of fat. It is calculated from the saponification number and the acid number:

Ester Number = Saponification Number – Acid Number

The ester number is more in extra virgin oil this is because of the ester number shows the amount of alkali consumed in the saponification of ester and is possible identify and differentiate the fats with this number.

Peroxide number:

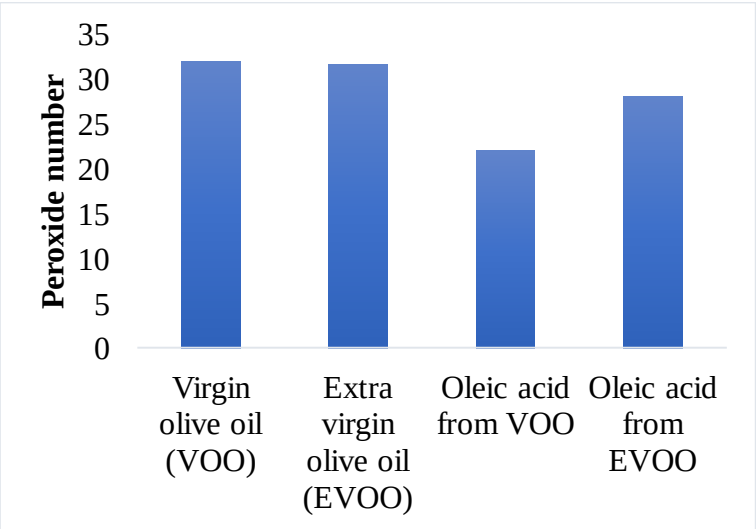

Figure 6. Peroxide number of virgin and extra virgin olive oils, and oleic acid produced from virgin and extra virgin olive oils.

The maximum peroxide number for extra virgin olive oil is 20. High quality extra virgin olive oil should have a peroxide number below 12. A very low peroxide number is desirable. The peroxide level starting point of an oil when it is first pressed is determined by the extraction process and storage conditions of the oil. Unsaturated free fatty acids react with oxygen and form peroxides, which generate volatile substances responsible for a typical musty/rancid bad oil smell. So, an elevated level of peroxide indicates an oil has been damaged by free radicals and is beginning to go rancid. These reactions are accelerated by high temperature, light, and oxygen exposure.Extra virgin olive oil has more peroxide number than virgin olive oil because it has more rancidity.

Glycerin percentage:

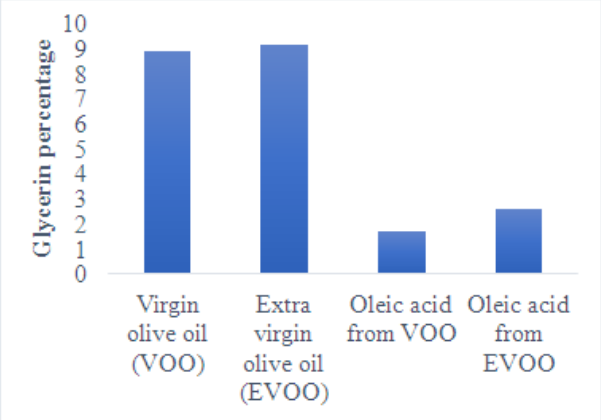

Figure 7. Glycerin percentage of virgin and extra virgin olive oils, and oleic acid produced from virgin and extra virgin olive oils.

Physically, glycerin is a water-soluble, clear, almost colorless, odorless, viscous, hygroscopic liquid with a high boiling point. Chemically, glycerin is a dihydric alcohol, capable of being reacted as an alcohol yet stable under most conditions.Glycerin number is more in extra virgin oil because extra virgin oil Glycolipids are composed of glycerol and fatty acids.

IV. CONCLUSION:

The iodine number for Extra virgin olive oil and oleic acid is high in oleic acid has greater resistance to oxidation and degradation. The peroxidation of the oil is primary arising because of the oxidation process, high temperature and visibility to light. The lower this number the longer the oil will retain the shelf life and will delay the possibility of rancidity, while high peroxide number usually indicates poor processing, and that the quality of the oil is low. Setting a low peroxide number standard for olive oil means that it will be more stable. The peroxide number of different grades of olive oil and oleic acid were measured. It was observed that Extra virgin olive oil and oleicacid have more saponification number than virgin olive oil because they have a relatively fewer number of carboxylic functional groups per unit mass of the fat as compared to short chain fatty acids (has long chain fatty acids).

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