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# Active packaging and increasing shelf life of virgin olive oil using biodegradable film based on polyvinyl alcohol modified with silver chloride nanoparticles and spirulina

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## ABSTRACT

In this study, biodegradable film was prepared from polyvinyl alcohol. Silver chloride nanoparticles and spirulina algae powder were used to modify the physicochemical structure and create antioxidant activity in the film. The surface morphology of the film was investigated. Polyvinyl alcohol film and its composites were used for packaging virgin olive oil. D-optimal design was used to investigate the effect of packaging type and storage time on the chemical, color and sensory characteristics of the oil. Chemical characteristics (acidity, peroxide value, refractive index and total phenol), sensory and color characteristics were investigated. The response surface method was used to investigate the effect of independent variables on the oil as well as the created mathematical models. The obtained results showed that in polyvinyl alcohol film modified with silver chloride particles and polyvinyl alcohol modified with silver chloride and spirulina particles, the presence of these particles on the surface of the film was completely evident. The results of the chemical, sensory and color analysis of the oil showed that the chemical, sensory and color quality of the oil decreased with the increase in the storage time of the oil in all packages. Peroxide value as the most important indicator of the quality of oils in normal packaging increased from 1 to 7 (mEq O2/Kg oil) during 30 days of storage, while in oil packaged with polyvinyl alcohol modified with silver chloride and spirulina algae, this increase was from 1 to 2 (mEq O2/Kg oil). In general, oils packaged with polyvinyl alcohol modified with silver chloride and spirulina algae showed the least quality changes compared to normal packaging, which indicates the ability of these films to control the quality and increase the shelf life of virgin olive oil.

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# 1. Introduction

Olive oil is widely appreciated due to its organoleptic properties and is consumed fresh or as a flavoring in prepared foods. However, the nutritional value of virgin olive oil is related to its overall composition, including various chemicals such as fatty acids, aliphatic alcohols, and triterpenes. , phytosterols, tocopherols and phenolic compounds, however, the role of phenolic compounds is generally accepted as the main factor. The quality of olive oil is influenced by storage conditions, packaging materials and exposure to oxygen or light, which determines the commercial shelf life of virgin olive oil [1-3].

During olive oil storage, the involved reactions include hydrolysis and oxidation in the form of autoxidation and photooxidation depending on the presence of light, single or triple oxygen, prooxidants, etc. On the other hand, phenolic compounds, which are the main factors of olive oil stability, protect it from autoxidation and photooxidation during storage. Oxidation is the main process affecting the quality of olive oil, because some unstable compounds that can change the sensory and nutritional characteristics of virgin olive oil are produced during oxidation. The rate of oxidative degradation of virgin olive oil is not only affected by its chemical composition, but its storage conditions also affect the rate of oxidative degradation of olive oil. Therefore, packaging by protecting the product from oxygen and light is very important in preserving it. The quality and shelf life of edible oil is limited by its oxidation, and this is the main factor for quality deterioration of virgin olive oil during its storage. Oxidation causes the oil to become rancid and unfit for human consumption. Light, temperature, atmospheric oxygen, the amount of natural antioxidants and oxygen dissolved in virgin olive oil during bottling are among the factors that are very effective on its oxidation level during storage [4-6]. In recent years, the use of biodegradable and environmentally friendly polymers as an alternative to petroleum polymers has attracted the attention of many researchers. One of the most important biopolymers to replace petroleum polymers is polyvinyl alcohol [7-10].

Nano materials are used as fillers in film preparation due to their suitable mechanical properties. Nanoparticles are mainly used to provide high tensile strength, elongation at break point, thermal resistance and appropriate electrical conductivity in the preparation of biodegradable composites, and these materials also have antibacterial properties. Silver nanoparticles and their compounds are often used as antibacterial agents due to their antibacterial ability against a wide range of bacteria. Silver ions released by silver nanoparticles damage the cell membrane of bacteria, prevent protein synthesis and cause the death of bacteria. When silver nanoparticles are used as antibacterial agents to prepare antibacterial film or polymer, the release concentration of silver ions can be controlled by modifying silver nanoparticles [13-11].

Silver chloride is a white crystalline solid that is famous for its low solubility in water. It occurs naturally as the mineral chlorargyrite, and when exposed to sunlight or heated, it decomposes into silver and chlorine, in which case specimens turn blackish gray or purple in color. Silver chloride is used as an antimicrobial agent in some personal deodorizing products and also for long-term preservation of drinking water in water tanks [14 and 15].

Blue-green spirulina (microalgae) is a source of high biological value because it is a rich source of protein, vitamins, especially vitamin A and vitamin B12, and rich in minerals, especially iron. It is also rich in essential fatty acids, especially gammalinolenic acid, which is very important in the synthesis of prostaglandins. Spirulina contains many compounds that have antioxidant activity both in vitro and in vivo, such as carotene, alpha-tocopherol, phycocyanin, xanthophyll, and phenolic compounds. Spirulina contains complete protein with a high digestibility index, so that its protein ratio is up to 60%. This substance contains all essential amino acids and is one of the nutritious and rich sources of beta-carotene, gamma-linolenic fatty acid, B vitamins, minerals, trace elements, enzymes and chlorophyll as well as other valuable nutrients such as carotenoids, sulfolipids, glycolipids, phycocyanin. , superoxide dismutase, RNA and DNA. Also,

spirulina can be used as a substitute for coloring agents [16-18].

In general, active biodegradable polymers containing antibacterial nanoparticles, bioactive substances, antioxidant pigments have the ability to be widely used in the packaging of different foods and increase the shelf life of foods and show the conditions of food storage and packaging in an intelligent way. they give] 19-21[.

Considering the things mentioned above and the vulnerability of virgin olive oil in normal packaging, which happens due to the rapid oxidation of this oil, in this study, polyvinyl alcohol film modified with silver chloride nanoparticles and spirulina algae was used for active packaging and Extending the shelf life of virgin olive oil was used. The high importance of spirulina as an antioxidant substance as well as the photochromic property of silver chloride, in this study, an active film that has both antioxidant/antibacterial and photochromic properties was prepared. The obtained results showed the very positive effect of polyvinyl alcohol film modified with silver chloride nanoparticles and spirulina algae in increasing the shelf life and controlling the quality of virgin olive oil during the storage period.

# 2- Materials and methods

## 2-1- Chemicals

Virgin olive oil was obtained from Daneh Khak Salamat Company (Urmia, Iran). Silver chloride with a purity of 99% from Parsian Merck Novin, spirulina algae powder from Spiro Bushehr, polyvinyl alcohol, glycerol, calcium nitrate, sodium carbonate and other chemical compounds used from Merck (Germany) and Aldrich (USA) were prepared

## 2-2- Preparation of polyvinyl alcohol/silver chloride/spirulina film

In order to prepare composite films, first, an aqueous solution of polyvinyl alcohol with a concentration of 2% was prepared, and silver chloride nanoparticles (0.15% of dry matter) and spirulina algae powder (0.2% of dry matter) were manually dissolved and homogenized in the film solution. Then a uniform solution was obtained by ultrasound. Next, the solution was poured into polystyrene plates with a diameter of 10 cm, and after 48 hours at a temperature of 25 degrees, the films were dried and then separated and stored in zipped bags (Figure 1-a).

### 2-2-1- Microstructure studies of films (SEM[1])

The microstructural analysis of the films was done using FE-SEM model Sigma VP (ZEISS company, Germany). First, the samples were coated with gold and then the analysis was done with a scanning electron microscope.

## 2-3- oil packaging with polyvinyl alcohol film

20 ml jars with silicone lids were used for oil packaging. For this purpose, the films prepared in dimensions of 10 x 10 cm were cut into squares and physically covered the inner wall of the glass bottle. Then 18 ml of olive oil was added to it so that the film was placed in the space between the oil and the glass wall. Finally, a silicone lid was placed and sealed in such a way that no air could penetrate into the glass (Figure 1-b).

1. Scanning electron microscopy

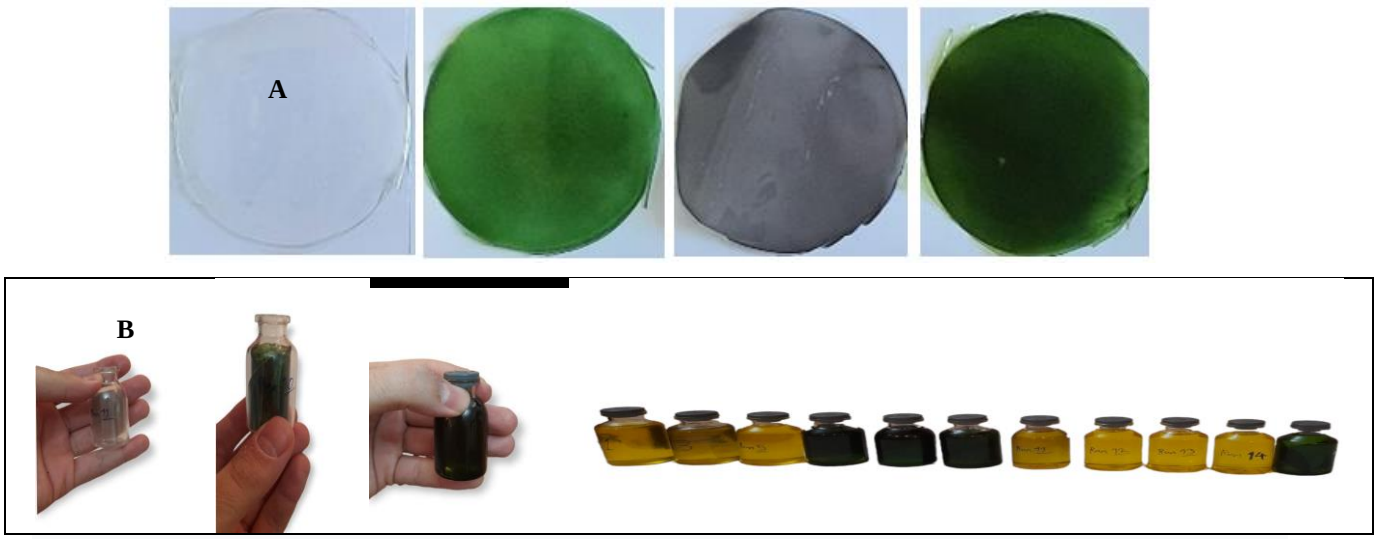


**Fig 1** Examples of prepared films (A) and packaging of virgin olive oil with prepared films (B)

## 2-4- oil tests

### 2-4-1- Acidity

Method No. (AOCS, 1993) Cd 3-63 was used to measure acidity. To perform this test, first mix 5 grams of oil with 20-30 ml of ethanol or another neutral alcohol and titrate by adding a few drops of phenolphthalein with 0.1 normal interest until a pink color appears. The amount of acidity was obtained from the following equation [22].

$$\mathbf{A} = \frac{\mathbf{28.2 \times N \times 100 \times V}}{\mathbf{1000 \times W}} \mathbf{\times 100}$$

In the above relationship, N: Normality of the consumed profit, V: Volume of the consumed profit in milliliters, W: Sample weight in grams, A: Free fatty acids in terms of oleic acid in 100 grams of sample.

### 2-4-2- Refractive index

To measure the refractive index of oil samples, these samples are placed in a refractometer at a temperature of 25 degrees Celsius, and their refractive index is measured according to the standard method. Cc 7-25(AOCS, 1993) was registered] 23[.

### 2-4-3- Add peroxide

Peroxide value was measured according to the AOCS method, No. Cd8-53. 5 grams of olive oil were poured into a 250 ml Erlenmeyer flask. 30 milliliters of acetic acid/chloroform solution with a ratio of 3:2 was added to Arlene and Arlene was shaken until the oil was dissolved. Then, 0.5 milliliters of saturated potassium iodide solution was added to Erlene and it was placed in the dark for one minute. Then 30 ml of distilled water was added to Erlenmeyer flask and 0.5 ml of 1% starch indicator solution was added. The final solution was titrated with 0.01 normal sodium thiosulfate until the water color disappeared. For control titration, all the above steps were performed without adding oil sample. Finally, the peroxide number is calculated according to the following formula [24].

$$PV = \left(\frac{S-B}{W}\right) PV = \left(\frac{S-B}{W}\right) \times N \times 1000$$

PV: Peroxide number in milliequivalents of oxygen per kilogram of oil sample
S: amount of tetrazole used in the sample
B: The amount of tetrazol used in the control
N: normality of tetrazol consumed in terms of equivalents per liter
W: weight of oil sample in grams

### 2-4-4- Total phenol content

Determining the amount of total phenolic compounds Total phenolic content was determined by colorimetric method and using Fulin Ciocalto reagent. For this purpose, one gram of each sample was mixed with three milliliters of methanol:water solution (90:10 ratio) and stirred for 4 minutes, and then placed in a centrifuge at 3000 rpm for 5 minutes, and 20 microliters of The upper phase of the methanolic extraction was mixed with 2.8 ml of water and 0.5 ml of Folin's reagent, and after 5 minutes, one ml of 10% sodium carbonate was added to

the above mixture and placed in a dark place for one hour at room temperature. After this period, the absorbance of the samples was read at room temperature with an ultraviolet spectrophotometer at 765 nm. Gallic acid (zero to 1000 micrograms per milliliter) was used to draw the standard curve, and the total phenolic content was reported as milligrams of gallic acid per kilogram of sample.

### 2-4-5-sensory properties

Sensory characteristics of oil samples, including color, texture, aroma, and overall score, were evaluated by 20 evaluators in the form of a 5-point descriptive hedonic test (1- very low, 2- low, 3- medium, 4- high, 5- very high). .

### 2-4-6-color properties

A colorimeter (Colorimeter Minolta model CR-410 Japan) was used to measure the surface color. First, the device was calibrated by a standard white screen. Then the oil was placed on a white paper and their color was measured using a colorimeter. This device measures four wavelengths in the visible range. The results include three dimensions of color with quantitative indices L*, a* and b*, which respectively represent brightness (from L=0 for black to L=100 for white), green to red (a=-60 for green). up to 60a = for red) and blue to yellow (from -60b = blue to 60b = for yellow).

## 5-2- Statistical analysis

In this study, to study the effect of packaging type and storage time on the chemical, sensory and color properties of virgin olive oil, the di-optimal statistical design according to Table 1 was used. For this purpose, 5 types of packaging include 1- normal packaging, 2- packaging with pure polyvinyl alcohol film, 3- packaging with polyvinyl alcohol film modified with silver chloride, 4- packaging with polyvinyl alcohol film modified with spirulina and 5- Packaging with polyvinyl alcohol film modified with silver chloride and spirulina was used. The storage time was 30 days and the tests were examined on the 1st, 15th and 30th day. The response level method was used to investigate the relationship between independent variables (type of packaging and storage time) and dependent variables and modeling. Data analysis was done at 95 probability level and curves and mathematical models were drawn using Design Expert-10 software.

**Table 1** List of experiments based on D-Optimal design

| Run | Factor A: Storage time (Day) | Factor B: Packaging type |
|---|---|---|
| 1 | 30 | Control |
| 2 | 1 | PVA/Sp* |
| 3 | 15 | PVA/AgCl |
| 4 | 1 | PVA |
| 5 | 30 | PVA |
| 6 | 1 | PVA/AgCl/Sp |
| 7 | 1 | PVA/AgCl |
| 8 | 30 | PVA/Sp |
| 9 | 15 | PVA/AgCl/Sp |
| 10 | 15 | PVA/Sp |
| 11 | 30 | PVA/AgCl |
| 12 | 1 | Control |
| 13 | 15 | Control |
| 14 | 15 | PVA |
| 15 | 30 | PVA/AgCl/Sp |

* Polyvinyl alcohol (PVA); Spirulina (Sp)

# 3. Results and Discussion

## 3-1- Studying the surface morphology of the film

The surface morphology of polymers and composites can be investigated and analyzed with SEM techniques. Figure 2 shows SEM images of polyvinyl alcohol film and its composites. According to the SEM results, the pure polyvinyl alcohol film has a regular structure without pores and surface cracks, which indicates the coherent and compact structure of the film. In polyvinyl alcohol film modified with silver chloride particles and polyvinyl alcohol modified with silver chloride and spirulina particles, the presence of these particles on the surface of the film is quite evident. In these films, the particles are distributed in the form of grains and almost uniformly. The size of silver chloride particles in these two films is estimated to be around 30 to 100 nm. In polyvinyl alcohol film modified with spirulina and polyvinyl alcohol modified with silver chloride and spirulina particles, the presence of spirulina particles on the surface of

the film is also evident. Also, in these 2 films, it seems that spirulina caused bumps and depressions on the surface of the film.

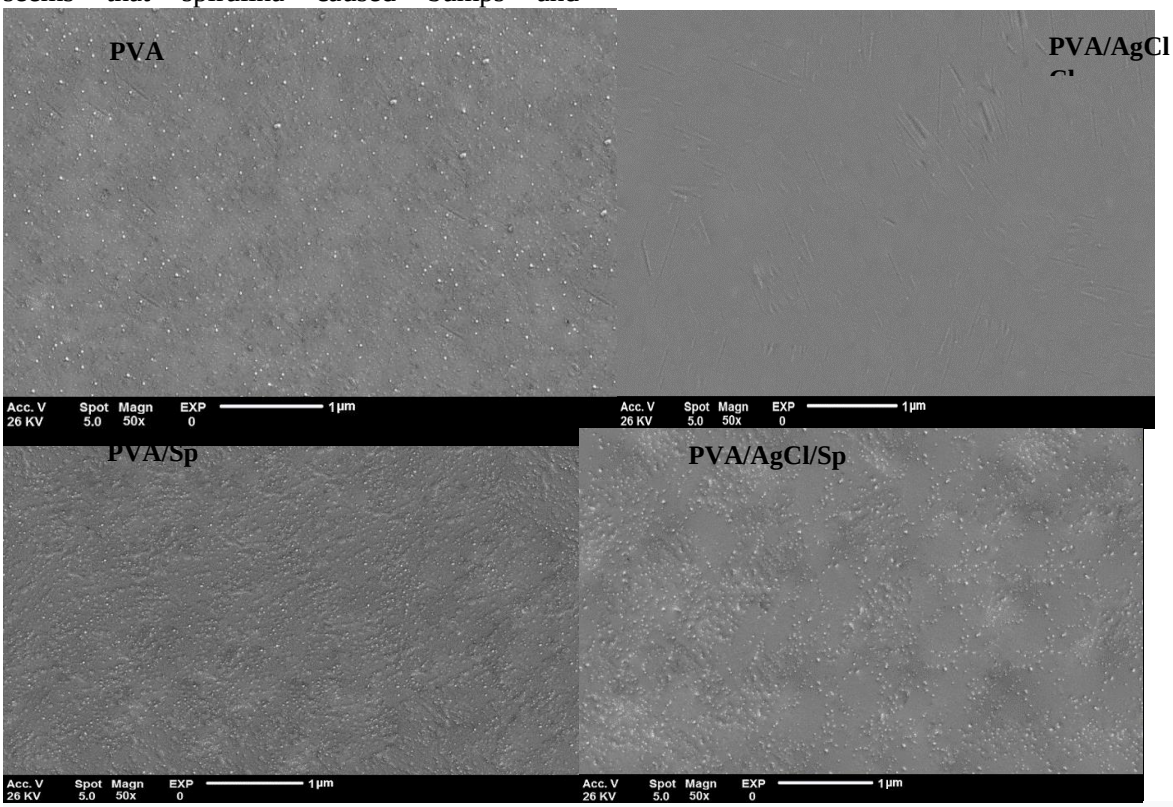


**Fig 2** SEM images of polyvinyl alcohol film and its composites

## 2-3-Acidity, refractive index, peroxide number and total phenol content

Linear interaction curves are a graphical representation of the relationship between two numerical and non-numerical variables that are used in two dimensions. Considering that in this study oil storage time is considered as a numerical variable and the type of packaging is considered as a non-numerical variable, Figures 3 to 5 and Tables 2 to 4, linear interaction curves and mathematical models (along with the regression coefficient and modified regression coefficient) show the relationship between some physicochemical, sensory and color properties and independent variables (storage time and type of packaging) in virgin olive oil.

The acid index is the number of milligrams of potassium required to neutralize the free fatty acids in one gram of fatty substance. In the acid index, the strength of the acid is important and the type of fatty acid is not important. But acidity determines how many grams of free fatty acids are present in 100 grams of fatty substances. The acidity value is a common parameter in the characteristics of fats and oils and is defined as grams of free fatty acids in 100 grams of oil (in terms of oleic acid, which is the main The most fatty acid present in the oil is expressed). An increase in the amount of acidity in a sample of oil or fat indicates the hydrolysis of triglycerides. Such a reaction occurs with the action of lipase enzyme and indicates inappropriate production and storage conditions (for example, high temperature, relative humidity and tissue damage).

Among the ways to identify and detect organic compounds is the use of the physical constant of the refractive index of light in an unknown liquid. The speed of light transmission is different in different environments. The deviation of the light radius when crossing the boundary between two different environments is one of the direct and visible results of this difference. The deviation of light is due to the change in its speed. The angle of refraction of light (angle of deviation) depends on the density

of the medium, the type of molecules present, the temperature of the medium and the wavelength of the light. If a medium such as air is chosen as a scale and the refraction angles are measured with a uniform wavelength at a uniform temperature, then the measured angles are one of the properties of the secondary medium in the distance and can be considered as a The specific characteristic of the body used in that environment.

Peroxide is the primary product of the oxidation of fatty substances, and in general, the higher the degree of unsaturation of oils, the more volatile aldehyde and columnar substances are formed, as well as short-chain fatty acids (the second and third products) that cause the unpleasant smell and taste of fatty substances. They are effective, they are created. Therefore, although the produced peroxide is not directly responsible for the unpleasant smell and taste in fatty substances, it is indicative of the progress of oxidation. Therefore, the higher the peroxide value, the higher the oxidation of the oil and the lower quality of the oil.

As it is clear from the interaction curve of acidity, refractive index and peroxide number with storage time and type of packaging, as well as the mathematical model reported in Table 2, all three of these factors increase with the increase of oil storage time. The increase in acidity, refractive index and peroxide number with the passage of time indicates the oxidation of olive oil with the passage of time and the reduction of oil quality. By comparing the effect of the type of packaging on the acidity, refractive index and peroxide number of the oil, it is determined that the greatest increase in these factors or the greatest decrease in oil quality is related to the normal packaging and the oils packaged with polyvinyl alcohol films modified with chloride. Silver and polyvinyl alcohol modified with spirulina and silver chloride show the least changes in acidity, refractive index and peroxide number, which silver chloride and spirulina have prevented oil oxidation. Due to the strong antioxidant property of spirulina and the photocatalytic property of silver chloride, these two substances prevent the oxidative erosion of oil.

Polyphenols are a group of flavonoids that exist as antioxidant substances in oils, essential oils and other plant compounds. These compounds are one of the quality indicators of olive oil as anti-cancer compounds. The higher the phenol content of the oil, the higher its antioxidant properties and the better the quality of the oil.

By examining the interaction curve of the total phenol content and also examining the related mathematical model, it was found that with the passage of time, the total phenol content has decreased in all oil packages, but the amount of this decrease in the oils packaged with polyvinyl alcohol films modified with Silver chloride and polyvinyl alcohol modified with spirulina and silver chloride have the lowest amount. This result shows that silver chloride and spirulina prevent the oxidation of total phenols and the destruction of these antioxidant compounds and preserve the quality of virgin olive oil.

Malherbi et al. (2022) prepared starch film modified with plant essential oils and used it for active packaging of olive oil. The results of their research showed that the chemical and oxidative properties of olive oil in active packaging are more stable than conventional packaging. The results of their research are completely consistent with the results of the current research [25]. Nogueira et al. (2022) have investigated the effect of active biodegradable package made of bean flour and acai seed extract on the quality of olive oil. They showed that the use of bioactive materials in the structures of packaging polymers can lead to an increase in the shelf life of olive oil, and the results of their research confirm the results of the present research [26].

**Table 2** Mathematical models and regression coefficients of the relationship between storage time and type of packaging and acidity, refractive index, peroxide value and total phenol content of the oil

| **Acidity (mg KOH/g oil)** | | **Refractive index** | | **PV(mEq $O_2$/Kg oil)** | | **Total Phenol (mg GAE/kg)** | |
|---|---|---|---|---|---|---|---|
| Coefficient | Factor | Coefficient | Factor | Coefficient | Factor | Coefficient | Factor |
| 0.372225 | | 1.776758 | | 3.155865 | | 163.2959 | |
| 0.1165 | * A | 0.0939 | * A | 1.302668 | * A | -34.3496 | * A |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 0.111763 | * B[1] | 0.070667 | * B[1] | 1.429124 | * B[1] | -35.9353 | * B[1] |
| -0.01338 | * B[2] | -0.00775 | * B[2] | -0.21124 | * B[2] | 5.490926 | * B[2] |
| -0.04415 | * B[3] | -0.03744 | * B[3] | -0.65325 | * B[3] | 16.76485 | * B[3] |
| 0.002304 | * B[4] | 0.019711 | * B[4] | 0.131235 | * B[4] | -5.14297 | * B[4] |
| 0.089594 | * AB[1] | 0.059425 | * AB[1] | 1.288799 | * AB[1] | -32.6568 | * AB[1] |
| -0.01971 | * AB[2] | -0.00485 | * AB[2] | -0.24303 | * AB[2] | 5.373646 | * AB[2] |
| -0.03779 | * AB[3] | -0.03673 | * AB[3] | -0.59381 | * AB[3] | 15.43707 | * AB[3] |
| 0.010955 | * AB[4] | 0.017786 | * AB[4] | 0.117725 | * AB[4] | -4.52868 | * AB[4] |
| -0.06273 | * A^2 | -0.06186 | * A^2 | -0.64273 | * A^2 | 17.86955 | * A^2 |
| $R^2$= 0.979 Adj$^2$=0.928 | | $R^2$= 0.990 Adj$^2$=0.967 | | $R^2$= 0.992 Adj$^2$=0.975 | | $R^2$= 0.995 Adj$^2$=0.983 | |

*A: Storage time and B: Packaging type

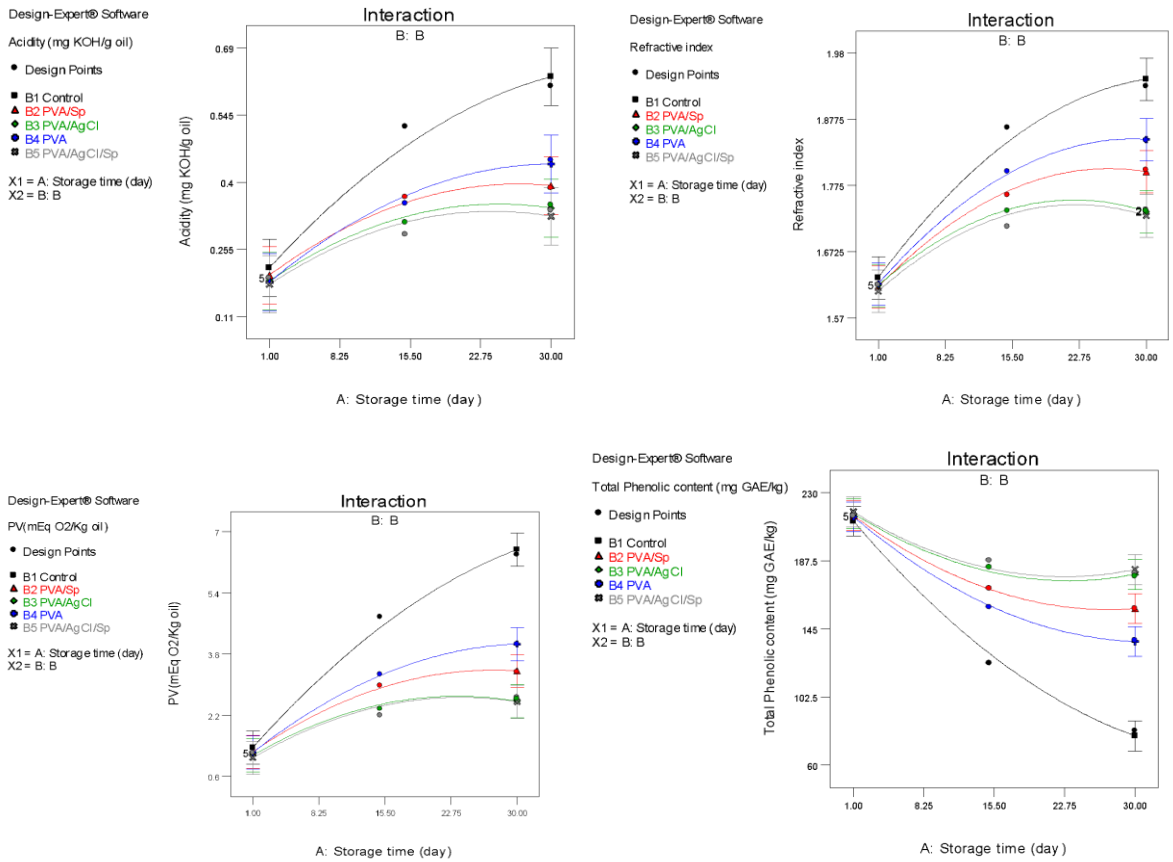


**Fig 3** Linear interaction curves of the effect of storage time and type of packaging on acidity, refractive index, peroxide value and total phenol content

## 3-3- Sensory properties

One of the tools naturally provided to humans is the five senses. By using the sense of touch, the quality of a material can be recognized. You can make sure that the food is not spoiled by tasting it. About recognizing the quality of olive oil, the five senses can help us to recognize its quality. Above all, the taste and smell of this oil can be a good criterion for evaluation. In this study, taste, color, texture and overall score are used as sensory properties to determine the quality of olive oil. According to the interaction curves of

the sensory factors in Figure 4 and the mathematical models presented in Table 3, the sensory factors have decreased with the increase in storage time, which indicates the decrease in oil quality with the passage of time. Although these sensory changes have not been very noticeable. The biggest decrease is observed in the sensory factor of the total score, which indicates that the total sensory score of this oil decreases with the passage of time from the point of view of the real consumers of the oil. Although the reduction of sensory factors is observed in all oil packaging, the most sensory changes were related to the normal packaging (without active polyvinyl alcohol film). Polyvinyl alcohol films containing silver chloride and spirulina have an effective and significant role in controlling the sensory characteristics of olive oil during storage, so that the overall acceptance of the oil packaged with polyvinyl alcohol/silver chloride on the day of Siam shows a difference with the first day. he does not give. This result shows the very important effect of silver chloride in preventing oxidative spoilage of olive oil.

Martillanes et al (2017) impactpackingactive on the stability of sensory characteristics of olive oil have been investigated. They showed that the antioxidant compounds can preserve the sensory characteristics and flavor of the oil, and their results confirm the results of the present research.]27[.Rababa and colleagues (2011) have investigated the effect of the type of packaging (active and inactive) on the sensory characteristics of olive oil. The results of their research confirm the results of the present study [28].

**Table 3** Mathematical models and regression coefficients of the relationship between storage time and type of packaging and flavor, color, texture and total score of the oil

| **Flavor** | | **Color** | | **Texture** | | **Total Score** | |
|---|---|---|---|---|---|---|---|
| Coefficient | Factor | Coefficient | Factor | Coefficient | Factor | Coefficient | Factor |
| 4.510238 | | 4.838399 | | 4.932647 | | 4.59794 | |
| -0.26932 | * A | -0.13926 | * A | -0.05975 | * A | -0.17924 | * A |
| -0.34667 | * B[1] | -0.04 | * B[1] | -0.06667 | * B[1] | -0.30309 | * B[1] |
| 0.02 | * B[2] | -0.00667 | * B[2] | 0.066667 | * B[2] | 0.033677 | * B[2] |
| 0.253333 | * B[3] | 0.026667 | * B[3] | 0 | * B[3] | 0.20206 | * B[3] |
| -0.11333 | * B[4] | -0.00667 | * B[4] | 0 | * B[4] | -0.03356 | * B[4] |
| | | | | | | -0.26886 | * AB[1] |
| | | | | | | 0.029873 | * AB[2] |
| | | | | | | 0.179239 | * AB[3] |
| | | | | | | -0.01953 | * AB[4] |
| $R^2$= 0.782 $Adj^2$=0.662 | | $R^2$= 0.686 $Adj^2$=0.512 | | $R^2$= 0.668 $Adj^2$=0.657 | | $R^2$= 0.972 | $Adj^2$=0.921 |

*A: Storage time and B: Packaging type

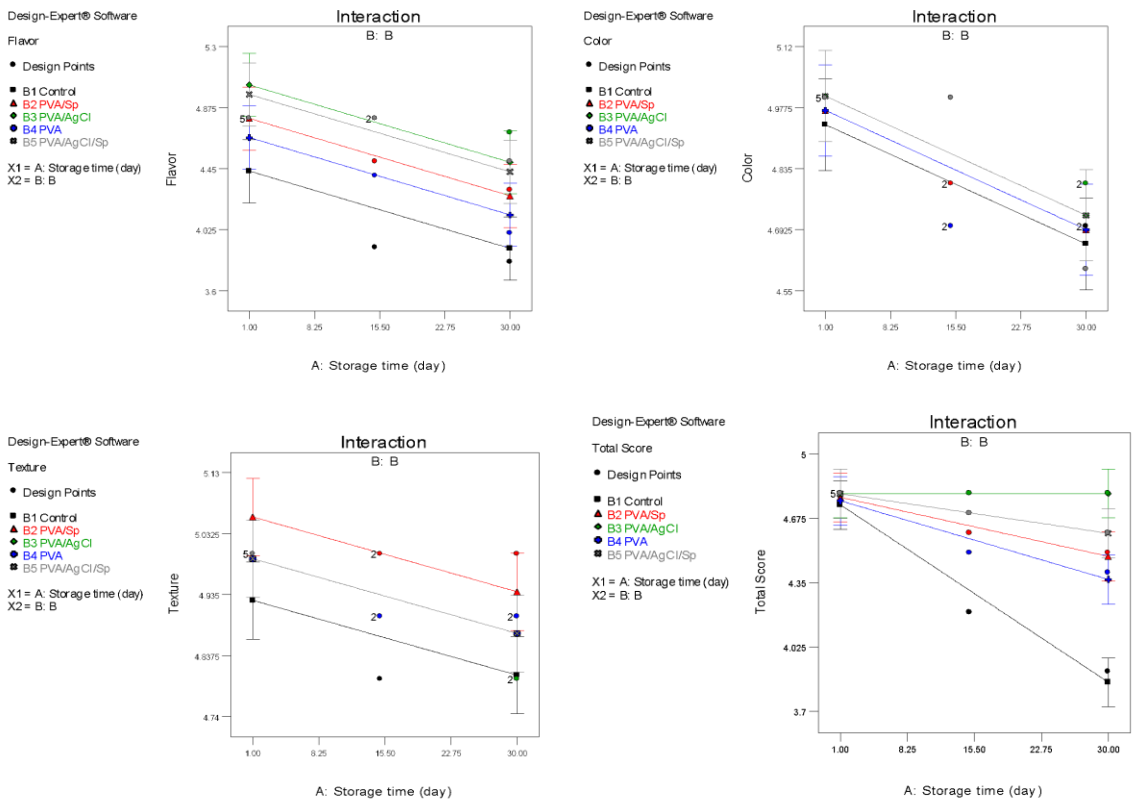


**Fig 4** Linear interaction curves of the effect of storage time and type of packaging on flavor, color, texture and total score of the oil

## 3-4- color properties

Colors are one of the characteristics of foods that are perceived by the sense of sight and are very effective in terms of consumer acceptance, because almost all foods, from the time they are raw to the time they become a complete food, have an acceptable color for consumption. are known as Color and taste of food are related in many cases. From the point of view of the food industry, it is essential to use colors to create new products or to improve products, and for this reason, the use of colors in food products is increasing day by day.Food coloringIt is directly related to stimulating the appetite and attracting the attention of consumers. The color we see clearly suggests the flavor we will taste, for example, the soft pink color represents the strawberry flavor inBeverage productsIs. The color also indicates the quality of the food and shows the high or low quality of the food. The activity of a group of microorganisms in food, to the extent that it makes the consumption of these substances dangerous in terms of health, can be associated with color changes in many cases. In this sense, according toFood coloringIn some cases, the color can be an indicator of the freshness or oldness of a food item. Along with taste, shape and size, color is one of the most important sensory characteristics of food, and the consumer first evaluates quality by color.

According to the oil transparency curve (Table 4), with the passage of time, the transparency of the oil shows a noticeable decrease in normal packaging, while the oils packaged in the presence of active films show a smaller decrease in transparency. The a* factor (negative values indicate greenness and positive values indicate redness of the oil) has moved towards more positive values over time, which indicates that olive oil has lost its green color over time. The b* factor curve (negative values indicate blue and positive values indicate yellowness of the oil) also shows that over time this factor has become more positive, which confirms the change in the color of virgin olive oil from green to yellow. In general, the lowest color changes in all three factors L*, a* and b* are related to the oil packed with polyvinyl alcohol film modified with silver chloride and spirulina.

**Table 4** Mathematical models and regression coefficients of the relationship between storage time and type of packaging and oil color factors

| **L*** | | **a*** | | **PV(mEq $O_2$/Kg oil)** | |
|---|---|---|---|---|---|
| Coefficient | Factor | Coefficient | Factor | Coefficient | Factor |
| 92.37344 | | 0.032642 | | 0.426338 | |
| -0.76629 | * A | 0.20063 | * A | 0.181193 | * A |
| -0.83122 | * B[1] | 0.213395 | * B[1] | 0.215239 | * B[1] |
| 0.118884 | * B[2] | -0.03949 | * B[2] | -0.03996 | * B[2] |
| 0.377514 | * B[3] | -0.089 | * B[3] | -0.10386 | * B[3] |
| -0.07311 | * B[4] | 0.015559 | * B[4] | 0.02693 | * B[4] |
| -0.74727 | * AB[1] | 0.19627 | * AB[1] | 0.185086 | * AB[1] |
| 0.140275 | * AB[2] | -0.05617 | * AB[2] | -0.03171 | * AB[2] |
| 0.350543 | * AB[3] | -0.07886 | * AB[3] | -0.09289 | * AB[3] |
| -0.07597 | * AB[4] | 0.01799 | * AB[4] | 0.027308 | * AB[4] |
| 0.394956 | * A^2 | -0.10621 | * A^2 | -0.08259 | * A^2 |
| $R^2$= 0.992 Adj$^2$=0.975 | | $R^2$= 0.991 Adj$^2$=0.970 | | $R^2$= 0.989 Adj$^2$=0.962 | |

*A: Storage time and B: Packaging type

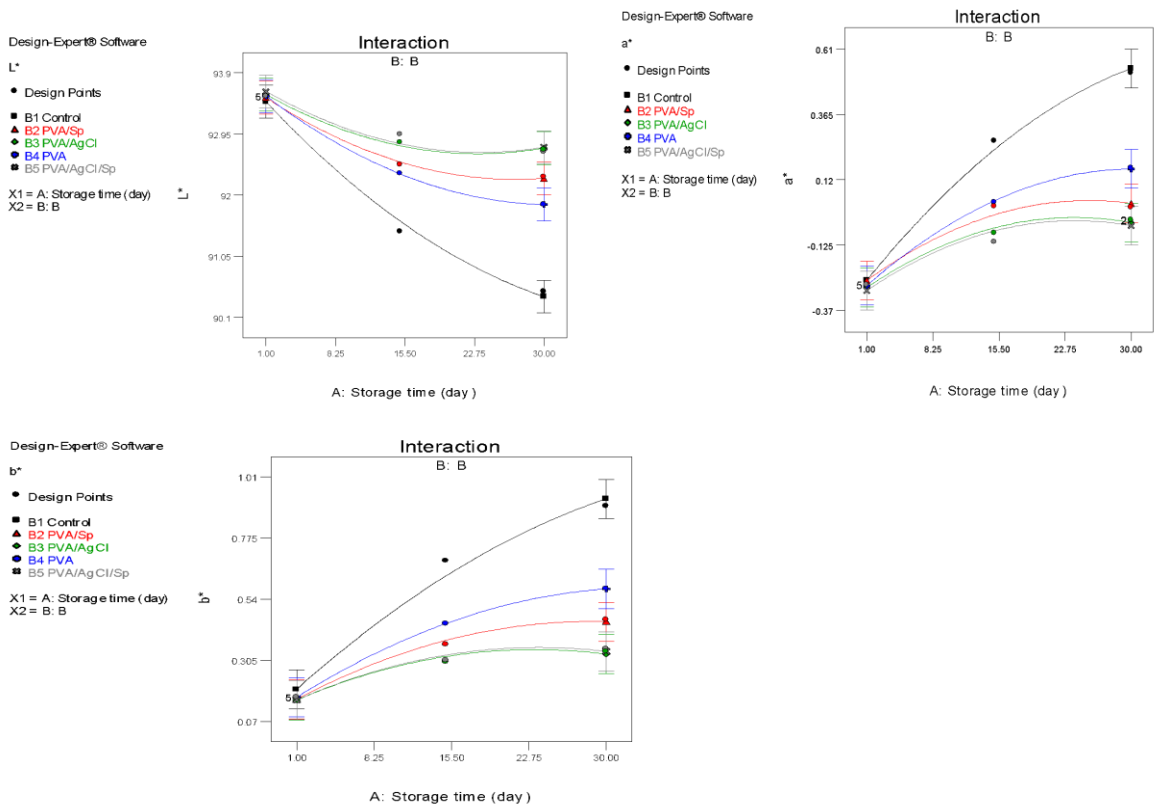


**Fig 5** Linear curves of the interaction of the effect of storage time and the type of packaging on the color characteristics of the oil

By examining the effect of silver chloride and spirulina in controlling the quality of olive oil (physicochemical, sensory and color properties), it was found that the effect of silver chloride nanoparticles was greater than spirulina, but when both of these materials were used simultaneously in the film structure, the effect was somewhat They have synergism and have increased the control power of oil quality. Also, by checking the regression models for all the measured properties, it was found that the regression coefficients and the adjusted

regression coefficients have high values, which indicates the suitability of the presented models to investigate the effect of storage time and type of packaging on the quality of virgin olive oil.
Stoll et al. (2017) have investigated the effect of biodegradable film activated with anthocyanin capsules on the quality characteristics of extra virgin olive oil during storage. The results of their research partially confirm the results of the present research [29]. Chavoshizadeh et al. (2020) have investigated the effect of active packaging on the color characteristics of oil. They showed that the color characteristics of the oil change during storage, that the use of active antioxidant polymers can control the color changes and make the oil color more stable. The results of their research fully confirm the results of this research [30].

## 4 - Conclusion

Due to the vulnerability of virgin olive oil in normal packaging, which happens due to the rapid oxidation of this oil, in this study, polyvinyl alcohol film modified with silver chloride nanoparticles and spirulina algae was used for active packaging and increasing the shelf life of virgin olive oil. became. The high importance of spirulina as an antioxidant substance as well as the photochromic property of silver chloride, in this study, an active film that has both antioxidant and photochromic properties was prepared. The obtained results showed that by comparing the effect of the type of packaging on the acidity, refractive index and peroxide number of the oil, it was found that the greatest decrease in the quality of the oil is related to the normal packaging and the oils packaged with polyvinyl alcohol films modified with silver chloride and Polyvinyl alcohol modified with spirulina and silver chloride showed the least changes in acidity, refractive index and peroxide value, which indicates that silver chloride and spirulina prevented oil oxidation. By examining the content of total phenol, it was found that with the passage of time, the content of total phenol decreased in all oil packages, but the amount of this decrease was in the oils packaged with polyvinyl alcohol films modified with silver chloride and polyvinyl alcohol modified with spirulina. And silver chloride has the lowest amount. This result shows that silver chloride and spirulina prevent the oxidation of total phenols and the destruction of these antioxidant compounds and preserve the quality of virgin olive oil. Overall acceptance of polyvinyl alcohol/silver chloride packaged oil on day 30 did not differ from day 1. This result shows the very important effect of silver chloride in preventing oxidative and microbial spoilage of olive oil. In general, the lowest color changes in all three factors L*, a* and b* are related to the oil packed with polyvinyl alcohol film modified with silver chloride and spirulina. By examining the effect of silver chloride and spirulina in controlling the quality of olive oil, it was found that the effect of silver chloride nanoparticles was greater than that of spirulina, but when both of these materials were used simultaneously in the film structure, there was a synergistic effect to some extent and the power of controlling the quality of the oil have increased Also, by checking the regression models for all the measured properties, it was found that the regression coefficients and the adjusted regression coefficients have high values, which indicates the suitability of the presented models to investigate the effect of storage time and type of packaging on the quality of virgin olive oil.

## 5- Resources

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مقاله علمی_پژوهشی

# بسته بندی فعال و افزایش ماندگاری روغن زیتون بکر با استفاده از فیلم زیست تخریب پذیر بر پایه پلی وینیل الکل اصلاح شده با نانوذرات کلرید نقره و اسپیرولینا

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**چکیده**

در این مطالعه فیلم زیست تخریب پذیر از پلی وینیل الکل تهیه گردید. از نانوذرات کلرید نقره و پودر جلبک اسپیرولینا برای اصلاح ساختار فیزیکوشیمیایی و ایجاد خواص آنتی اکسیدانی و فوتوکاتالیتیکی در فیلم استفاده شد. مورفولوژی سطحی فیلم مورد بررسی قرار گرفت. فیلم پلی وینیل الکل و کامپوزیت‌های آن برای بسته‌بندی روغن زیتون بکر استفاده شد. از طرح آماری دی-اوپتیمال برای بررسی تاثیر نوع بسته‌بندی و زمان نگه‌داری بر روی ویژگی‌های شیمیایی، رنگی و حسی روغن استفاده شد. ویژگی‌های شیمیایی(اسیدیته، عدد پرکسید، ضریب شکست و فنل کل)، ویژگی‌های حسی و رنگی مورد بررسی قرار گرفت. از روش سطح پاسخ برای بررسی تاثیر متغیرهای مستقل بر روی روغن و نیز مدل‌های ریاضی ایجاد شده استفاده شد. نتایج بدست آمده نشان داد،در فیلم پلی وینیل الکل اصلاح شده با ذرات کلرید نقره و پلی وینیل الکل اصلاح شده با ذرات کلرید نقره و اسپیرولینا، وجود این ذرات در سطح فیلم کاملا مشهود بود. نتایج آنالیز شیمیایی، حسی و رنگی روغن نشان داد با افزایش زمان نگه داری روغن در همه بسته‌بندی‌ها، کیفیت شیمیایی، حسی و رنگی روغن کاهش پیدا کرد. عددپراکسید به عنوان مهمترین شاخص کیفیت روغن‌ها در بسته‌بندی معمولی در طول ۳۰ روز نگه‌داری از ۱ تا ۷ (**mEq $O_2$/Kg oil**)افزایش یافت در حالیکه در روغن بسته‌بندی شده با پلی وینیل الکل اصلاح‌شده با کلرید نقره و جلبک اسپیرولینا این افزایش از ۱ تا ۲ ( **mEq $O_2$/Kg oil**) بوده است. در حالت کلی روغن‌های بسته‌بندی شده با پلی وینیل الکل اصلاح شده با کلرید نقره و جلبک اسپیرولینا کمترین تغییرات کیفی را نسبت به بسته‌بندی معمولی نشان دادند که نشان دهنده قابلیت این فیلم ها در کنترل کیفیت و افزایش ماندگاری روغن زیتون بکر می باشد.