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The use of mixture design in optimization of biodegradable three-component composite film formulation of starch, zein and PVA based on physicochemical and mechanical properties

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ABSTRACT

Production and use of biodegradable films with appropriate physical and mechanical properties and replacing them with synthetic materials is an effective solution to reduce many environmental pollutions. In this study, with the aim of producing biodegradable consistent structure and durable composite films, starch, zein and PVA were mixed with different proportions according to mixture design and ternary composites were prepared. Physical and mechanical properties were compared and optimum formulation were determined. According to the results, adding PVA to starch improved the mechanical properties and adding zein to it reduced the water solubility of the composite films. The optimal formulation of the three-component composite was determined as %85 starch, %15 zein and %5 PVA with a desirability level equal to %98. The x-ray diffraction patterns of optimal film showed two specified peak at 2θ=17-19° and 2θ = 22-24°, this pattern indicated the amorphous and crystalline structure of the composite film. Finally, differential scanning calorimetry showed that the highest weight loss of composite film is 285/3-340°C equal to %48/2.

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1- Introduction

Biodegradable resins with the circulation of water, carbon dioxide, oxygen and nitrogen in the food chain are a suitable solution for reducing environmental pollution caused by the widespread use of synthetic resins and preserving non-renewable oil resources. Since a large amount of plastic and non-degradable waste is related to the consumption of packaged food products, in recent years much attention has been paid to research to find suitable biodegradable substitutes for synthetic resins in food product packaging [1, 2]. Natural resins that can be used in packaging production can be divided into four categories based on their chemical structure: proteins, polysaccharides, lipids and polyesters. These materials are used alone or in combination with each other to produce packaging films [3].

Biodegradable films, in comparison with synthetic resins that are commonly used in the packaging industry, due to weak internal bonds and hydrophilic nature, high solubility and poor mechanical properties, which show their use as food packaging materials. limits. Researchers have used various methods to improve these characteristics, including adding hydrophobic compounds, adding softeners, mixing with synthetic resins, and adding fillers with special properties [5, 4, 3].

Starch is used as a suitable raw material in the structure of many biodegradable films due to its high biodegradability, cheapness, availability, safety of consumption and easy application. In recent years, many researchers have used starch from different sources to produce films and coatings alone [6, 4, 2] or as a composite with other natural polymers and various compounds [10, 9, 8, 7]. investigated and determined the functional and mechanical properties of the resulting films.

The results of various researches have shown that mixing starch with proteins reduces its sensitivity to water. Among the proteins, zein is a suitable option for this purpose due to the presence of non-polar amino acids in its structure. Zein is a protein soluble in alcohol, it is hydrophobic and thermoplastic, and it is a byproduct of corn flour production [11]. The good barrier properties of zein have suggested it as a suitable coating for use in food packaging, so that in various studies, zein has been used alone in the production of packaging film [11, 12, 13, 14]. Zein is also used as a compound component for common plastics such as polypropylene [15].

PVA is the product of hydrolysis of polyvinyl acetate polymer, which is biodegradable and soluble in water. PVA has favorable mechanical properties such as tensile strength and elongation to breaking point, so mixing it with starch polymer improves the mechanical properties of starch film [16].

In this research, with the approach of developing and producing a biodegradable composite film with a stable and durable structure, the three-component starch-zein-PVA composite by extrusion and then molding, the production and physical and mechanical properties of the films were fully investigated and finally The optimal composite formulation was introduced.

2- Materials and methods

2-1- Materials

Corn starch was obtained from Glucosan, frankincense from local market, zein from Sigma, polyvinyl alcohol and other chemicals from Merck, Germany.

2-2- Preparation of three-part composite films

After performing the pre-treatments and determining the usable limits of the compounds for film formation, in order to determine the appropriate proportions of the film-forming components of the mixture design.[1] And Minitap software version 16.2.3 was used. The components of the plan include the percentage of starch (X_1), percentage of zein (X_2) and PVA percentage (X_3) They were. The ratio of components was expressed as a combination with the sum of 1 ($X_1+X_2+X_3=1$) (Table 1). To prepare the composite films, specific amounts of the three compounds were weighed according to the proportions determined in Table 1 and were completely mixed with each other in a dry form. 30% by weight/volume of glycerol was added as a plasticizer to all treatments. Three materials were mixed together by extrusion method and Brabander extruder machine with the temperature of the thermal areas of 128/129/128/130/130 degrees Celsius and 60 rpm propeller.

[1]- Mixture Design

Table 1. Composit films formulation based on mixture design.

Treatment	PVA(X_3)	Which (X_2)	Starch(X_1)
1	0.125	0.125	0.75
2	0.150	0.200	0.65
3	0.75	0.125	0.80
4	0.75	0.175	0.75
5	100	0.150	0.75
6	100	0.150	0.75
7	100	0.150	0.75
8	0.150	100	0.85
9	0.150	0.200	0.75
10	0.150	100	0.75
11	0.125	0.175	0.70
12	100	0.150	0.75
13	100	0.150	0.75

Then, 2 grams of extruded granules were soaked in 80 milliliters of water-ethanol mixture (50:50 ratio) for 24 hours. Then the resulting suspension was placed on a magnet heater with a temperature of 100 degrees for 2 hours to form a proper gel. Finally, the solution was uniformly spread on the glass plate and dried for 48 hours in the vicinity of air and ambient temperature to obtain the desired films.

Fig1. Starch-Zein-PVA extruded granules- A sample of composite film.

2-3- Film solubility in water

For this purpose, 3 x 2 cm pieces of the film were weighed with an accuracy of 0.0001 and placed in test tubes containing 80 cc of deionized water. The samples were stirred for 1 hour on a shaker at 50 rpm at a temperature of 25 degrees Celsius. Then the remaining parts of the film were dried in a 60 degree oven until reaching a constant weight and weighed again. The solubility percentage was calculated from the following equation [2].

$$Solubility\ percentage = \frac{(Initial\ weight - final\ weight)100}{initial\ weight}$$

4-2-Stability in acid and alkaline solutions

For this purpose, pieces with the same dimensions of the films were prepared and after weighing with an accuracy of 0.0001, they were placed in Erlenmeyer flasks containing 25 cc of 0.1 normal solutions of sodium hydroxide and hydrochloric acid, and continuously for one hour on the shaker. They were stirred at 50 rpm at a temperature of 25 degrees [17]. The remaining parts of the film were separated from the solution with the help of a paper filter and dried in a 60 degree oven until reaching a constant weight and then weighed. The degree of stability of the films in acidic and alkaline solutions was determined based on their solubility percentage in said solutions.

2-5- Crossing between light and transparency

The property of blocking visible light in films was measured using UV-Vis spectrophotometer CAMSPEC M550 made in England based on the following relationship. In this equation, A_{600} The amount of absorption at the wavelength of 600 nm and X is the average thickness of the film in millimeters. [18 and 19].

$$\text{Transparency}=\frac{A600}{X}$$

6-2- Color features

The color of the film samples was measured using the Hunterlab colorflex machine made in America and CIELAB values included $^*L$ (light), $^*a$ (red-green) and $^*b$ (yellow-blue) was recorded. The cellophane transparent plastic sample was selected as a control and the following formulas were used to obtain the total colorimetric difference (ΔE) for each sample compared to the control sample and the chroma index:

$$C=\sqrt{(a)^2+(b)^2}$$

$$\Delta E^*=\sqrt{(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2}$$

2-7- Mechanical characteristics

Features of tensile strength[2] (TS and the percentage of elongation to breaking point (%E) were measured based on the ASTM standard, D882-02 and with slight changes by the LLOYD tissue tester model RS 232 made by AMETEK, America, according to the following relationships. The samples were taken before The test was adjusted in terms of humidity. The distance between the two jaws before the test was set to 50 mm, the jaw movement speed was 50 min/mm and the load cell was set to 50 Newtons.

$$\%E=\frac{\text{Elongation at breaking point}}{\text{original length}}$$

$$\text{Tensile Strength}=\frac{\text{Maximum Load}}{\text{Cross sectional area of samples}}$$

which is Maximum load, the maximum force applied to the film in Newtons, and Cross-sectional area, the initial cross-sectional area of the film in square meters, Elongation at breaking point, the amount of expansion until the moment of rupture in millimeters, and original length, the initial length of the sample between two Jaw is (mm).

2-8- X-ray diffraction test (XRD[3])

Optimum sample of composite film using XRD analyzer (Unisantif, XMD-300) made in Switzerland with CuK $beam_a$ It was scanned at ambient temperature with a wavelength of λ = 1.5418 angstroms and an incident angle (2θ) of 5 to 50 degrees, and its X-ray diffraction spectrum was compared to the starch film as a control [20].

9-2- Analysis of thermal behavior of composite by thermogravimetric method (TGA[4])

The thermal analysis of the optimal film was performed in comparison with the starch film as a control using the BaHR analyze device model STA-503. Dry alumina powder (AL_2O_3) as a reference substance in taking thermograms[5] used. The weight of the sample was 12.53 milligrams and the weight of the reference material was 13.75 milligrams. The samples were heated from the ambient temperature to a maximum of 510 degrees Celsius at a rate of 10 degrees per minute, in an air atmosphere with a flow rate of 20 milliliters per minute [21].

3-Results and discussion

1-3-Results of determination of physical characteristics

The results of measuring physical properties including thickness, density, enzymatic sensitivity, solubility in water, stability in acid and alkaline solutions and WVP are shown in Table 2. Based on the results of data fitting with different models, all physical characteristics with high correlation coefficient were fitted with a specific quadratic model.

[2] tensile strength

[3] X-Ray Diffraction

[4] - Thermogarvimetric analysis

[5] - Thermogram

Table2: Mean of physical charactristic determination of starch/zein/PVA composits

Treatment	WVP	Solubility in NaOH (%)	Solubility in HCL (%)	Solubility in water (%)	Density (g/m^3)	Thickness (mm)
1	3.57	21.1	36.94	38.66	1.16	0.00013
2	6.53	100	41.98	46.52	1.24	0.00016
3	3.7	62.87	42.20	41.54	1.42	0.00011
4	4	100	42.79	41.59	1.31	0.00013
5	4.40	54.10	38.56	47.11	1.18	0.00013
6	4.62	53	36.46	35.23	1.17	0.00012
7	4.20	51.01	36.01	38.95	1.18	0.00012
8	4.21	60.92	32.10	34.90	1.29	0.00012
9	2.66	41.26	28.74	35.93	1.24	0.00013
10	4.1	30.44	40.89	45.19	1.39	0.00013
11	3.23	9.24	40.08	39.83	1.18	0.00010
12	4.30	54.54	38.37	37.08	1.18	0.00012
13	4.5	55	37.10	38.77	1.18	0.00011

According to the results of Table 2, with the increase in the percentage of zein in the formulation, the density of the films decreases, which can be justified as a result of the increase in the thickness of the films. Also, the prepared composite samples had solubility between 34-45%, which is acceptable (Table 2). According to Figure 2, increasing the percentage of zein decreased the solubility of the films and increasing the amount of PVA and starch in the formulation caused an increase in the solubility of the films. At the minimum amount of zein, increasing the amount of PVA more than starch has been effective in increasing the amount of solubility. In the minimum amount of starch, the solubility has increased by decreasing the amount of zein and increasing the percentage of PVA. Also, with the minimum amount of PVA, increasing the percentage of starch in the composite shows an increase in solubility. Zein constitutes more than 45-50% of the proteins in corn and is especially rich in the amino acids leucine (20%), glutamic acid (21-26%), alanine (10%) and proline (10%). The presence of these non-polar and hydrophobic amino acids in the structure of zein causes its insolubility in waterIt can [22]. For this reason, increasing the amount of zein in the composite will decrease the solubility, and on the other hand, starch and PVA, due to their polyoleic nature and as a result The high hydrophilic property increases the solubility of the composite in water.

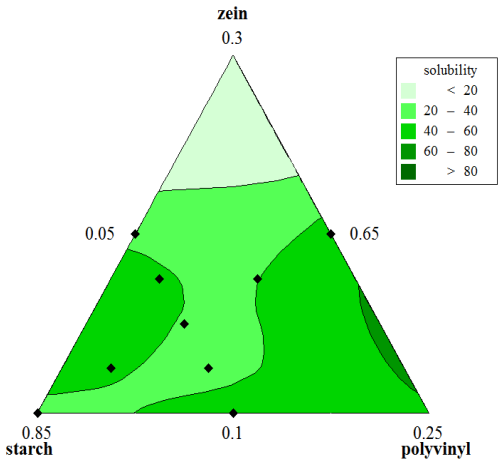

Fig2. The counter diagram of the effects of composite components on the solubility of the films in water

Based on the obtained quantitative results, starch/zein/PVA composite films are less stable in alkaline environment than in acidic environment and dissolve more, so that some samples such as sample 2 and 4 are completely dissolved in alkaline solution and their structure they lost Since the hydrolysis of starch increases in an acidic environment, increasing the percentage of starch in the composite formulation and vice versa decreasing the amount of zein increases the solubility of films in an acidic environment.
Ko et al reported in their research that PVA film is more unstable in alkaline environment than in acidic environment. They observed that in HCL and NH solution$_4$HSO$_4$, the crystal structure of PVA increases and the solubility of the film decreases [23]. Based on this, the increase in the solubility of composites in alkaline environment compared to acidic environment can be attributed to the change of PVA structure in acidic environment.

According to Figure 3, increasing the amount of zein in the composite formulation causes a decrease in WVP and increasing the amount of starch and PVA causes an increase in WVP in the samples. At the minimum amount of zein, as the percentage of PVA in the formulation increases, the amount of WVP will be higher. Since the presence of PVA in the composite composition makes the film smoother and more homogeneous and increases the solubility of the film, the WVP increases. On the other hand, in the minimum amount of PVA, the amount of WVP increases with the increase of starch. Since the composite starch is hydrophilic, an increase in the amount of WVP can be expected.

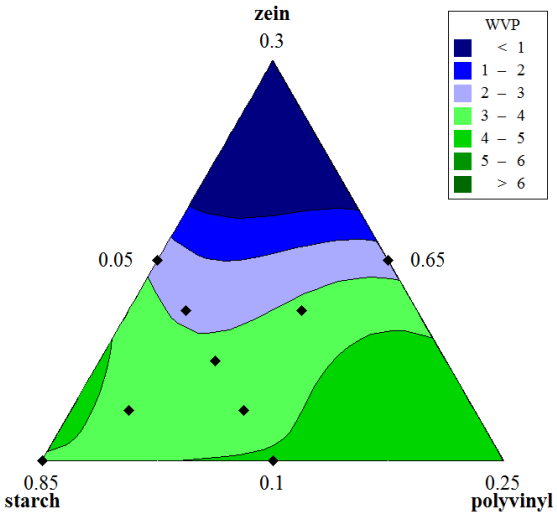

Fig3. The counter diagram of the effects of composite components on WVP of the films.

3-2- **Results of determination of color characteristics**

The results of measuring the color characteristics of composites including transparency, L, a, b, ΔE and chroma are shown in Table 3.

Table3: Mean of optical charactristic determination of starch/zein/PVA composits

Treatment	ΔE	C	b	a	L	transperancy
1	29.24	5.42	5.40	-0.51	34.27	5.99
2	57.20	12.58	12.54	-1.01	62.1	11.46
3	36.07	3.99	3.91	-0.83	41.47	8.74
4	41.02	8.37	8.27	-1.25	45.55	6.99
5	41.11	8.97	8.95	-0.44	46.01	6.81
6	31.67	6.99	6.96	-0.62	36.54	5.17

7	32.95	5.31	5.15	-0.59	38.27	6.65
8	17.75	1.51	1.46	-0.38	23.52	4.46
9	39.40	7.44	7.39	-0.89	44.27	8.05
10	35.27	4.96	4.80	-1.23	40.79	7.09
11	40.59	6.01	5.94	-0.93	45.94	8.99
12	30.02	7.11	7.09	-0.55	34.66	5.09
13	25.68	4.38	4.32	-0.64	30.88	5.04

Based on the results, the prepared composites had a brightness or L value equal to 23 to 60. By reducing the amount of zein and increasing the percentage of starch and PVA in the composite formulation, the brightness of the films increases. Also, at the minimum amount of zein, the higher the amount of PVA in the composite, the brighter the color of the samples, which is expected due to the transparency and high L value of PVA.

Since the value of a shows the degree of redness-greenness of the samples, positive numbers indicate redness and negative numbers indicate greenness of the samples. According to Table 4, all the prepared composites have a negative a value, in other words, they have green color. Increasing the amount of zein in the composite formulation tends to change the color of the samples from green to red. Also, increasing the amount of PVA and starch increases the amount of green tahrang. Similarly, b describes the degree of yellow-blueness of the color. According to Table 4, the positive b values show that all the tested composite samples have a yellow tint, which is caused by the effect of the zinc used in the formulations. Therefore, by increasing the amount of zein in the formulation, the amount of b increases.

According to Table 4, among the produced composites, sample number 8 showed the lowest colorimetric difference compared to the control sample (cellophane transparent plastic) and had the lowest ΔE. It should be mentioned that ΔE calculated for composites is in the range of 17-57, which comparison with starch film samples that had ΔE between 1.4-5.4, shows that the addition of zein and PVA to starch causes The increase in the overall color difference between the films and the sample is witnessed.

The results of the calculation of the amount of C indicate an increase in the purity or saturation of the color in the samples at the same time as the increase in the amount of zein in the composite formulation. The prepared films mostly had C less than 10, which was expected due to the existence of more than three different compounds in the formulation and as a result of its impurity compared to single part films. It should be noted that the individual films of starch had a C value of 66-78 (Table 4).

Transparency is a very important feature for films that are used as a cover or packaging for foodare used [24]. Also Transparency is an effective and efficient index in determining the information related to the size of particles dispersed in the polymer matrix, so that particles and granules larger than the visible wavelength block the path of light and increase the opacity of the film.[25]. Necessary It should be mentioned that the transparency is affected by the thickness of the film [26] and the thickness of the film should be carefully controlled in the study of transparency.

The results obtained from the test of the transparency of the composites by the spectrophotometer indicate that the transparency of the composite increases with the increase in the percentage of PVA and starch and the decrease in the amount of zein. As mentioned, transparency can be considered as an index to determine the size of the dispersed particles and the uniformity of the polymer matrix. Researchers have reported that in the mixed film of starch and zein, cracks are observed along the length of the mixed matrix, which is caused by the poor compatibility of the two substances. This lack of compromise affects the characteristics of the mixtureis [26]. so It can be said that in the mixture of starch, zein and PVA, zein is not well dispersed in the composite matrix due to poor compatibility with the other two materials. This lack of proper dispersion prevents the passage of light and creates turbidity in the film. Therefore, among the produced composites, the highest transparency is related to the films that have lower amounts of zein.

3-3- Results of determination of mechanical properties

Table 4 shows the results of measuring the mechanical properties of the films.

Table4: Mean of mechanical charactristic determination of starch/zein/PVA composits

Treatment	%E(mm)	TS(MPa)	Modulus of elasticity (MPa)
1	22.397	2.364	39.910
2	20.228	2.114	38.359
3	36.031	4.973	28.117
4	20.093	3.157	121.311
5	34.806	2.483	46.863
6	34.654	2.357	39.339
7	34.163	2.176	49.572
8	48.967	5.322	106.05
9	21.517	2.847	120.930
10	31.636	3.622	60.833
11	25.827	2.965	74.606
12	34.425	2.664	49.084
13	34.883	2.339	47.957

The obtained results showed that the mechanical properties of films differ based on the type of composite formulation used in their structure. According to Figure 4, increasing the amount of zein in the formulation leads to a decrease in the tensile strength of the composite. Also, in the minimum amount of zein, increasing the amount of starch has led to an increase in TS. In the minimum amount of starch, with increasing amount of PVA, the tensile strength of the composite has increased.

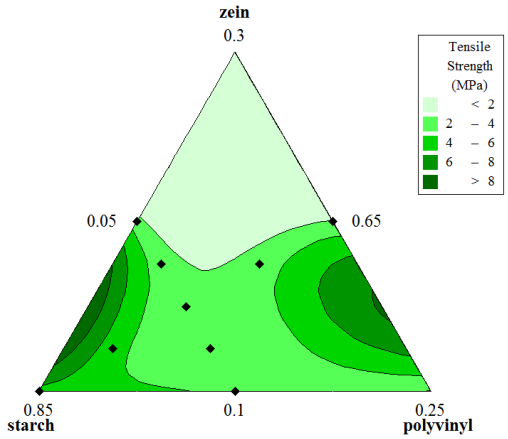

Fig4. The counter diagram of the effects of composite components on TS of the films

Young's modulus or elasticity is the ratio of stress to strain of solid materials in the elastic region (below the yield point). Since stress is pressure and strain is a dimensionless quantity, the modulus of elasticity is pressure and is measured in MPa. As can be seen in Figure 5, with the increase in the percentage of zein, the modulus of elasticity in the samples increased and the increase in the percentage of starch and PVA in the formulation decreases the amount of this property. The modulus of elasticity of the prepared composite samples is in the range of 28-121 MPa. It should be noted that the higher the elastic modulus of the film, the more force it needs to change the shape in the elastic region of the stress-strain curve, and in other words, it will have a higher yield strength.

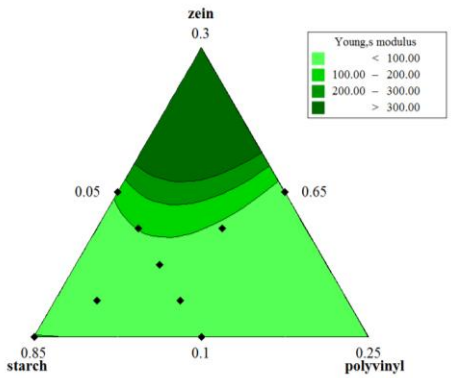

Fig5. The counter diagram of the effects of composite components on Modulus of elasticity of the films

Increasing the amount of zein in the formulation has caused an increase in the length of the composites. At the minimum amount of zein, increasing the percentage of starch and vice versa decreasing the amount of PVA has increased the length of the composites. Also, in the minimum amount of starch, increasing the percentage of PVA in the composite composition has led to a decrease in %E. At the minimum amount of PVA, the elongation has increased in purer samples (containing more zein or more starch) (Figure 6).

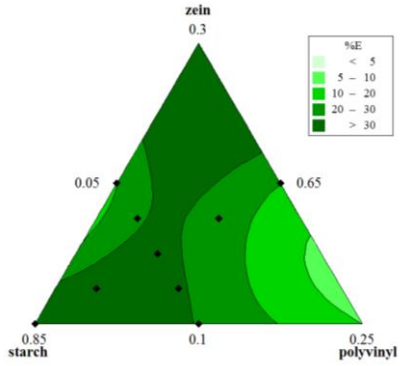

Fig6. The counter diagram of the effects of composite components on %E of the films

The obtained results regarding the mechanical properties of the produced composites are consistent with the results of other researchers. Based on the results of their morphological studies on films produced by thermal molding of corn starch and zein, the researchers reported that as a result of condensation of zein in the film structure, a macromolecular network was formed, which causes the starch granules to stick together. As a result of this adhesion, the mechanical properties of the film such as tensile strength are affected and reduced. These researchers concluded that the size of the zein particles is effective on the structure and thermodynamic properties of the starch/zein mixture, so that with the smaller size of the zein particles, the structure of the film will be more uniform and its mechanical properties will be better [27]..

In their research on the production of starch/zein compound by applying shear forces, Habich et al. reported that in fact, the weak adhesion and incompatibility of the two substances zein and starch to each other increases the percentage of polymer elongation. This is caused by the non-uniform deformation mechanism of zein and starch under shear forces, which creates cracks along the matrix of the composition. In other words, the internal friction of the starch polymer is reduced as a result of the presence of zein and the increase in length in the polymer, but on the other hand, a decrease in TS or tensile strength will be observed due to the gaps created [28].

It should be noted that the strength of the polymer is also influenced by plasticizers, so that the tensile strength decreases with the increase in the amount of plasticizer. In this research, the amount of plasticizer (glycerol) used is fixed and the difference in the mechanical properties of the composites is exclusively attributed to the difference in the compounds in their formulation and the reaction of these compounds with each other under the shear forces applied in the extrusion process.

4-3- Fitting the best model to describe the data

Tables 2, 3 and 4 results They showed a mixed design study. Both independent and dependent variables were fitted with specific linear, quadratic, cubic and quartic models. According to Cornell et al.'s description, the best data fitting model is the model that has the lowest standard deviation.[6], the lowest predicted sum of squares[7], and the highest correlation coefficient (R^2) and the lack of fit is meaningless [29]. Based on this, for the responses of thickness, solubility in water, WVP, L, a, chroma, transparency, ΔE and tensile strength, the special quartic model was recognized as the best model. The regression coefficients and correlation of the data of the fitted model for some of the answers mentioned are shown in Table 5.

[6]- Standard deviation

[7]- Predicted sum of squares

5-3- The results of optimization of composite formulation

Because the desired properties of the final film include low water solubility, low WVP, high transparency, E was low and tensile strength was high, by determining the acceptable range for each characteristic, the optimal formulation including 85% starch, 10% zein and 5% PVA was determined. The desirability of the determined formula (D) was calculated as 98%.

6-3- Investigation of X-ray diffraction of composite film

According to the results of the X-ray diffraction test (Figure 7), most of the structure of the produced starch film is amorphous and a relatively distinct peak can be seen only in the range of θ2 times 17-24º. In the composite film, due to the fact that starch forms the majority of the structure, the amorphous pattern of starch can be observed without change in many parts, but in the range of θ2 equal to 17-19º and 22-24º certain peaks have been created in the composite structure, therefore. It can be said that the structure of the composite is amorphous and crystalline in some parts, and this change can be attributed to the presence of PVA in it.

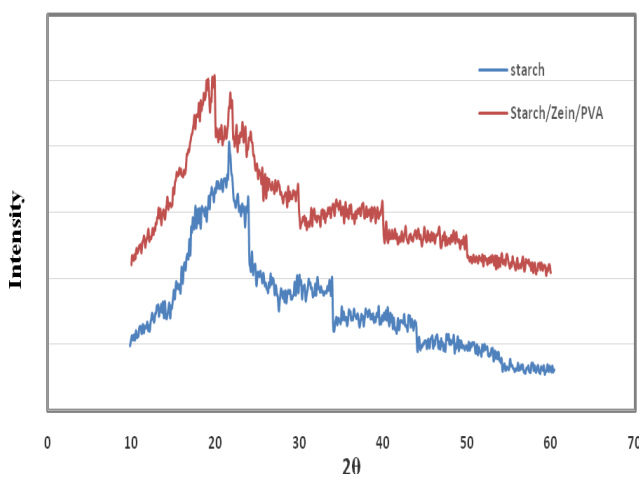

Fig 7. XRD of composite film in compare with starch film.

3-7- Examination of the thermal behavior (TGA) of the composite film

TGA measures the amount and rate of change in weight of a sample as a function of temperature or time in one Determines the controlled atmosphere. This information is used to determine the thermal or oxidation stability of materials and to identify their compositional properties. This method can analyze substances that lose weight or gain weight as a result of decomposition, oxidation or loss of volatile substances such as moisture. This technique is especially suitable for studying polymeric materials such as thermoplastics, thermosets, elastomers, composites, films, fibers and coatings.[30].

The TGA method is especially used to determine information regarding the compositional analysis of multicomponent materials, thermal stability, oxidation stability, determination of shelf life, analysis of decomposition kinetics, the effects of the reactive atmosphere on materials, moisture content and volatile compounds, etc.[31].

Temperature map or TG curve of starch film and composite film in air atmosphere is shown in Figure 8. The amount of weight loss in different stages was calculated by drawing a tangent on the curve and determining the Y point from the equation of the line in different stages. As can be seen, in the starch film, the highest weight loss occurred in the temperature range of 296-325 degrees Celsius and the sample lost more than 69.1% of its weight. After that, in the range of 325 to 500 degrees Celsius, 14.64% weight loss is observed in the samples.

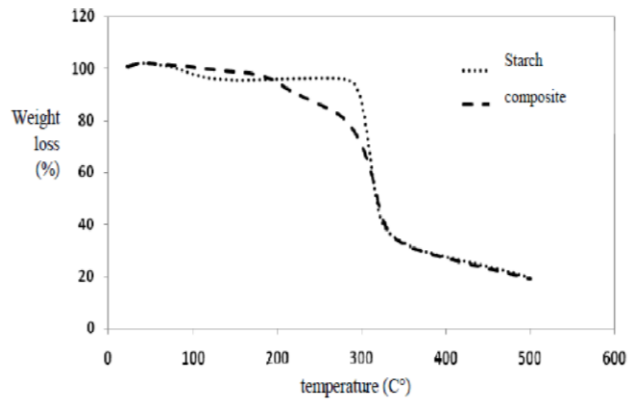

Fig8. TG of compsite and starch film.

In the sample of the composite film, changes in the structure of the composite due to heat have occurred in a more gradual manner compared to the starch film. The starting temperature of weight loss changes in the samples is 181.3 and from this temperature to 285.23, 16.2% weight loss has occurred in the sample. The highest weight loss of the composite is observed in the temperature range of 285-340 degrees Celsius and equivalent to 48.2%. From the temperature of 340 to 500 degrees Celsius, a 13.5% weight loss has occurred in the samples.

From the examination of the thermal behavior of the composite film, it can be said that the designed formulation is stable up to the temperature range of 180 degrees Celsius and does not undergo thermal

decomposition, because the beginning of the temperature changes of the composite occurred at higher temperatures. Also, the more gradual changes in the structure of the composite compared to starch can be described due to the simultaneous presence of three compounds of starch, zein, and PVA and the reactions of these compounds with each other. The weight loss of the film samples in different stages can be attributed to the exit of moisture, the thermal decomposition of the polymer chain and the release of volatile compounds, the cross-linking reaction between molecules, and the destruction of amino groups in the zein structure [21].

4 - Conclusion

In total, the investigation of the results of the composite film made of starch with zein and polyvinyl alcohol showed that the resulting composite has acceptable physical and mechanical properties, so that the presence of PVA significantly improves the mechanical properties of the film, and zein, in Reducing the solubility of starch in water is effective. Also, for mixing these materials and producing films, the extrusion method can be used, which is a common method for producing films in an industrial way. The FTIR examination of the composite film compared to the pure films showed that a spectrum of all constituent components can be seen in it, which is a good indication of the composition of the components. The results of the X-ray diffraction test also showed that the structure of the composite is amorphous and crystalline in parts. Investigating the thermal behavior of the composite film indicated that the designed formulation is stable up to the temperature range of 180 degrees Celsius and does not undergo thermal decomposition, because the temperature changes of the composite begin at higher temperatures. This feature is important from the industrial point of view of film production.

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مقاله علمی_پژوهشی

استفاده از طرح مخلوط در بهینه سازی فرمولاسیون فیلم کامپوزیت سه جزئی زیست‌تخریب‌پذیر حاوی نشاسته، زئین و پلی وینیل الکل برپایه ویژگی های فیزیکوشیمیایی و مکانیکی

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چکیده

تولید فیلم‌های زیست‌تخریب‌پذیر دارای خواص فیزیکی و مکانیکی قابل قبول و جایگزینی آنها با بسپارهای سنتزی در بسته بندی محصولات غذایی، راه حلی موثر برای کاهش بسیاری از آلودگی‌های محیطی است. در این پژوهش با هدف تولید فیلم زیست-تخریب‌پذیر با ساختار پایدار و با دوام، نشاسته، زئین و PVA با نسبت‌های مختلف تعیین شده با استفاده از طرح مخلوط با یکدیگر ترکیب و کامپوزیت‌های سه‌جزئی تهیه شد. خواص فیزیکی و مکانیکی کامپوزیت‌ها مورد مقایسه قرار گرفته و فرمولاسیون بهینه تعیین گردید. بر اساس نتایج به دست آمده، افزودن PVA به نشاسته، سبب بهبود ویژگی‌های مکانیکی و افزودن زئین به آن، سبب کاهش حلالیت در آب فیلم‌های حاصل گردید. فرمولاسیون بهینه کامپوزیت سه جزئی به صورت ۸۵٪نشاسته، ۱۵٪ زئین و ۵٪ PVA با میزان مطلوبیت معادل ۹۸٪ تعیین شد. الگوی پراش پرتو ایکس کامپوزیت بهینه نشان داد در محدوده 2θبرابر ^{o}۱۹-۱۷ و ^{o}۲۴-۲۲ پیک‌های مشخص در ساختار کامپوزیت ایجاد شده، بنابراین ساختار کامپوزیت به صورت آمورف و در بخش‌هایی کریستالی است. بر اساس نتایج بررسی دمانگاشت فیلم کامپوزیت بهینه، بیشترین افت وزنی در محدوده دمایی ۳۴۰-۲۸۵/۳ درجه سانتیگراد و معادل ۴۸/۲٪ بود.