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Preparation of biodegradable composite starch/tragacanth gum/Nanoclay film and study of its physicochemical and mechanical properties

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ABSTRACT

In this study, biodegradable composite starch/tragacanth gum/Nanoclay (St/TG/Clay) film was prepared. The basis of the film was wheat starch, which tragacanth gum was added at three levels (0.2 and 0.5% by w/w) and clay nanoparticles at three levels (0.1, 0.5 and 3% by w/w). Physicochemical properties of the film, including thickness, water solubility, moisture content, and vapor vapor permeability (WVP), were investigated. The mechanical properties of the films, including the tensile strength and elongation of the films, and the color characteristics of the films were examined. The results showed that adding tragacanth to the starch film increased the thickness, moisture content and permeability of the water vapor and reduced its solubility. The addition of tragacanth to the starch film increased tensile strength and elongation. The addition of tragacanth reduced the transparency of the films. Adding clay nanoparticles to starch film increased the water content, reduced water vapor permeability, and reduced solubility, but adding nanoparticles increased the thickness. Nanoclay increased the tensile strength of the films and reduced the elongation. The lightness of the films decreased with the addition of nano-clay, and the factor b, which indicated the yellowness of the film, increased. In general, it can be concluded that the addition of tragacanth and Nanoclay improves the physical and mechanical properties of starch film.

1. Introduction

Biodegradable polymers are a special type of polymer that decomposes after its intended purpose by the process of bacterial decomposition and leads to the formation of by-products such as CO_2, N_2, water, and mineral salts. These polymers are made naturally and artificially and are mainly composed of ester, amide and ether agents. Their decomposition properties and mechanism are determined by their exact structure. These polymers are often synthesized by compaction reactions, open loop polymerization, and metal catalysts. There are applications for degradable polymers [1 and 2]. Packaging materials made from environmentally friendly materials have been introduced in recent decades as green alternatives. Biodegradable films have attracted a lot of attention due to their environmentally friendly features, wide variety and availability, non-toxicity and low cost. Biodegradable polymers are often used to reduce the volume of packaging waste. There has also been significant efforts to replace petrochemicals with materials made from biodegradable components [3].

Starch is a white, soft, and tasteless powder that is produced by all green plants and is chemically a complex organic compound of carbohydrates insoluble in cold water, alcohol, or other solvents. It is widely used in human food, other animals and in industry. In terms of biochemistry, starch is a polysaccharide consisting of two types of carbohydrate deposits called

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amylose and amylopectin. The monomers of this polysaccharide are glucose units that bind together to form alpha 1 and 4 bonds. The simplest amylose starch is linear polymer. Betadine solution can be used to identify starch, which will change color to blue during the oxidation/reduction reaction of starch and iodine. Corn starch is a type of starch that is made from corn kernels. Corn starch is obtained from the seeds of corn seeds. Corn starch is a common food element used in thick sauce or soup and in making corn syrup and other sugars [4 and 5].

Tragacanth, also known as Gum tragacanth in the world, is the most important product of the tragacanth plant [6]. The most important suppliers of tragacanth in the world are Iran, Turkey and Syria, respectively. This medicinal and potent substance is available in different shapes and colors. Its colorless or white sheet is the best and most expensive product of tragacanth. As a medicinal substance, tragacanth has a decisive role in the manufacture of medicines and medicinal substances. In the pharmaceutical industry, this material is used for the preparation of mucilage, tragacanth powder, as well as for the preparation of various tablets [7]. Because tragacanth has medicinal properties, it is used as an emulsifier to keep water-soluble pharmaceuticals suspended. Tragacanth may also be added to volume-enhancing drugs. Other properties of tragacanth are used as an emollient and soothing in cosmetic products. Tragacanth is used as a suspension, emulsifier and adhesive in the pharmaceutical industry [8].

Nanoclay is a type of purified clay that at least is the nanometer size in one of the dimensions [9]. The chemical and physical properties of solids depend heavily on the size and shape of the microscopic particles that make them up. Nanoparticles have found many applications in a variety of fields, including medicine, pharmaceuticals, cosmetics, catalysts, food packaging, and the textile industry [10]. In addition to the mentioned applications, Nanoclay is also useful in protecting the environment. Their potential as adsorbents for volatile organic compounds, and organic/mineral pollutants is well documented in wastewater. This diversity in applications can be attributed to the adaptability of Nanoclay to change/modify. Nano-clays in combination with biodegradable polymers are very useful for modifying the mechanical properties of these polymers [11].

In this study, tragacanth gum and clay nanoparticles were used to improve the physicochemical and mechanical properties of biodegradable corn starch film. Due to the fact that starch film is mechanically brittle and easily dissolved in water and loses its special properties, the gelizing properties of tragacanth improve the mechanical behavior of starch film and increase the resistance of starch film in water. Clay nanoparticles also improve the mechanical properties of starch film and increase the film's resistance to water dissolution.

2. Material and Methods

2.1. Materials

Corn starch was prepared from Urum Aydan Sanat Co. (Iran, Urmia). Tragacanth was obtained from the Medicinal Plants Store (Urmia, Iran). Clay nanoparticles (with a particle size of 30 to 100 nanometers) were produced by Nanogiloguzak Company (Iran, Tehran). Magnesium nitrate, calcium chloride, and other chemical compounds used were produced by Merck (Germany) and Sigma-Aldrich (USA).

2.2. Preparation of St/TG/Clay film

To prepare the St/TG/Clay film, 2 g of starch in 100 ml of water at 70 ^{o}C was dissolved by a magnetic stirrer (RS3001, MLW, and Germany) at a speed of 2000 rpm. The tragacanth powder was then added to the solution in grams (according to Table 1 statistical design) and dissolved at 70 ^{o}C by a mechanical mixer (BH8, Iran) at 1500 rpm for 1 hour. Glycerol (40% weight of dry matter) was then added to the solution as a plasticizer and dissolved in the solution for 20 minutes under the same conditions. The Nanoclay powder (according to Table 1) was then added to the solution and dispersed by a mechanical mixer at 1500 rpm for 1 h. 25 ml of the final solution was poured into special plates with a diameter of 10 cm and dried for 48 hours at room temperature and the dried film was obtained. The dried film was stored in zippered bags at room temperature [12].

2.3. St/TG/Clay film tests

2.3.1. Thickness

The thickness of the films was measured randomly in 5 positions with a manual micrometer (MitutoyoCo, Japan) with a resolution of 0.001 mm and the mean values were used in the calculations.

2.3.2. Humidity content

To measure the humidity content of the film, firstly the film was weighed, then it was dried in the oven at 110 °C for 3 hours to reach the constant weight, and finally, the moisture content of the film was obtained from the following equation:

$$\text{Humidity content (\%)} = (M1-M2)/M2\times100 \qquad (1)$$

Where M1 is the initial weight of the film and M2 is the film weight after drying [13].

2.3.3. Solubility

Solubility in water was measured according to standard method and expressed as the percentage of dry matter of the film dissolved in water after 24 hours of immersion in water. The films (2×2 cm) were weighed, then weighed films were immersed in 30 ml of distilled water and stirred for 3 hours. Then the films were taken and were dried in an oven at 105 °C for 24 hours. Solubility percent in water films was calculated according to the following equation:

Solubility (%) = $(W_i - W_f)/W_i \times 100$ (2)

Where, W_i and W_f are the initial weight and final weight of the film samples, respectively.

2.3.4. Water vapor permeability (WVP)

Water vapor permeability (WVP) test was performed by the following method; Glass vials with an average diameter of 2 cm and width of 4.5 cm were used to determine WVP.

The films were cut into 2.5 cm diameter discs and then adhered to the vial exit's lid containing 3 g of water-free CaSO4. The vials were weighed with all their contents and placed in a desiccator containing saturated K2SO4 (K2SO4 saturated solution provides 97% constant relative humidity at 25 °C).

Vial weight changes were measured every 6 hours for four days. The amount of water vapor transmitted from the films was determined by the weight of the vials. The weight gain curves of the vials were plotted over time and after calculating linear regression, the slope of the resultant line was calculated. By dividing the slope of the line corresponding to each vial to the entire film surface exposed to water vapor, water vapor transmission rate (WVTR) and water vapor permeability (WVP) were calculated according to following equations [14].

WVTR=Slope/A (3)

WVP= (WVTR×L)/ΔP (4)

Where; WVTR is the water vapor transfer rate (g/m2h), L: film thickness (m), A: film surface area (m2), ΔP: relative water vapor pressure difference (Pa), WVP: Water vapor permeability (g/mhPa).

2.3.5. Mechanical properties

The mechanical properties of the films were measured by the texture analyzer instrument (Zwick/Roell Model FR010; Germany). Standard method number ASTM (2010) D 882-10 was used for this purpose. Prior to the mechanical tests, all samples were incubated for 24 hours at 55% relative humidity (calcium nitrite saturation), then three samples of each film were cut (0.5×8 cm).

The films were placed between the two jaws of the instrument. Elongation at break (MPa) was measured with the device and the following equation was used to calculate the tensile strength (TS) of the films [15]:

TS=F/(w×d) (5)

Where, TS is tensile strength (Pa), F is force (N), d is film thickness (μm) and w is film width (m).

2.3.6. Color properties

Color of films was measured by a colorimeter (Minolta, CR-410; Japan). The color properties including L, a and b were recorded. L indicates the brightness (from L=0 for black to L=100 for white), a indicates green to red (a=-60 for Green to a=60 for red) and b indicates blue to yellow (from b=-60 blue to b= 60 for yellow) [16].

2.4. Statistical properties

In this study, to investigate the effect of tragacanth gum and clay nanoparticles on the physical, chemical and mechanical properties of the film (thickness, moisture, solubility, water vapor permeability, tensile strength, elongation and color properties), a central compound design (CCD) was used (Table 1). Data analysis was performed at the 95 % probability level with the Design Expert-10 software.

Table 1. List of experiments based on a central composite design (CCD)

Run	TG (%)	Clay (%)
1	2.5	3
2	2.5	1.5
3	2.5	1.5
4	2.5	1.5
5	5	3
6	5	1.5
7	5	0
8	2.5	1.5
9	2.5	0
10	0	0
11	0	3
12	2.5	1.5
13	0	1.5

3. Result and discussions

The response surface method (RSM) was used to investigate the effect of tragacanth gum and clay nanoparticles on the physical, chemical and mechanical properties of the film (thickness, moisture, solubility, water vapor permeability, tensile strength, elongation and color properties). This method is a set of mathematical methods that determines the relationship between one or more response variables to several independent variables. Mathematical relationships, mathematical models, and regression coefficients between the responses and the independent variables (tragacanth gum effect and clay nanoparticles) were calculated and reported in Table 2.

3.1. Thickness and humidity

Fig. 1 shows 3-Dimensional plots and perturbation curves of the effect of TG and Clay on the thickness and humidity content of starch film. Thickness is very important in films and polymers, because the thickness of the film affects the mechanical properties, solubility, strength and permeability of the film to various gases. Especially when the film is used for food packaging, the film thickness is very important because there is a significant relationship between film thickness and food quality control. For example, high-thickness films with low permeability to water vapor significantly increase the ability to control the quality of sensitive food products.

Table 2. Mathematical models and regression coefficients between responses and independent variables

Response	Equation	R^2	AdjR^2
Thickness (μm)	==+41.5+7.8*TG (%)+6.2*Clay (%)	0.985	0.982
Humidity (%)	=+37.6+4.80*TG (%)+2.2*Clay (%)	0.996	0.995
Solubility (%)	=+29.80-2.74*TG (%)-1.79*Clay (%)+0.13*TG (%)*Clay (%)-0.09*TG $(\%)^2$-0.03*Clay $(\%)^2$	0.998	0.997
WVP (×10^{-5} g/m.s.Pa)	=+5.2+0.76*TG (%)-1.55*Clay (%)+0.03*TG (%)*Clay (%)+0.11*TG $(\%)^2$+0.21*Clay $(\%)^2$	0.982	0.968
TS (MPa)	=+4.61+0.70*TG(%)+1.44*Clay (%)-0.16*TG (%)*Clay (%)+0.15*TG $(\%)^2$+0.10*Clay $(\%)^2$	0.995	0.991
EB (%)	=+51.10+4.195*TG (%)-6.34*Clay (%)+0.33*TG (%)*Clay (%)+0.32*TG $(\%)^2$+0.46*Clay $(\%)^2$	0.989	0.982
L	=+79.06-3.86*TG (%)-2.77*Clay (%)	0.980	0.976
a	=1.43+1.53*TG (%)+1.88*Clay (%)	0.978	0.974
b	=+4.65+1.33*TG (%)+1.66*Clay (%)	0.980	0.976

As the thickness curves show, the addition of tragacanth and Nanoclay to the starch film increases its thickness. The effect of tragacanth on increasing film thickness is greater than that of Nanoclay. By adding 5% by weight of tragacanth to the film, the thickness of the film has increased by more than 100%. Due to the fact that tragacanth has a gel-like property and can absorb moisture and swell, increasing the thickness of the film in the presence of tragacanth was expected. Clay nanoparticles increase the interval between polymer chains and increase its thickness by placing them in the spaces between the starch polymers. In the film that had the highest amount of tragacanth and Nanoclay, the highest thickness of the film was observed.

Starch film humidity increased significantly with increasing tragacanth and Nanoclay. However, the effect of nanoparticles on increasing film moisture was much lower than that of tragacanth. As mentioned, tragacanth has a high moisture absorption property, and by absorbing water molecules, it can increase the moisture content of the film. The reason for the increase in film moisture in the presence of Nanoclay is probably due to the fact that hydrogen bonds are formed between water molecules and Nanoclay and the film moisture increases. The film showed the highest amount of tragacanth and the highest amount of Nanoclay, the high moisture content. Saberi and her colleagues have prepared a compound film of starch and guar gum and have examined the effect of guar gum on the thickness and moisture of the film, which their results are in line with the results of the present study [17].

3.2. Solubility and WVP

Fig. 2 shows 3-Dimensional and contour plots of the effect of TG and Clay on the solubility and WVP of starch film. Water solubility and water vapor permeability (WVP) are very important characteristics of biodegradable polymers. Since the structure of most food products is water-based and considering that the presence of moisture in food products creates the ground for microbial and algal spoilage of food products, so the solubility of biodegradable films and permeability to water vapor are factors to consider when choosing a packaging film. The proper WVP of a packaging film depends on the type of food product and its storage conditions.

As the results show, adding tragacanth to the starch film greatly reduces its solubility. Reducing the solubility of starch film in the presence of tragacanth is due to the fact that tragacanth contains a substance called bassorin, which is insoluble in water and reduces the solubility of the film in water. Nanoclay have also somewhat reduced the solubility of starch film. The reducing of solubility in the presence of clay nanoparticles is due to this fact that clay strengthens chemical bonds between polymer starch resins and makes them physically strong in the water.

According to the results, tragacanth has increased the permeability of starch film to water vapor. As mentioned earlier, due to the fact that tragacanth has the ability to create a gel, it causes the starch film to swell and creates a gap between the polymer chains of the starch, which allows the vapor molecules to pass through, thus increasing WVP. Unlike tragacanth, Nanoclay has reduced WVP. Clay nanoparticles are placed in empty spaces between starch polymers and prevent the passage of water vapor molecules, thus reducing WVP.

Santacruz et al. (2015) examined the effect of chitosan on the physical properties and solubility of starch film, the results of which confirmed the results of the present study [18]. Schmidt et al. (2013) examined the effect of stearic acid as a modifier of the structure film and concluded that the modifiers reduce the WVP of the film, which is consistent with the results of the present study [19].

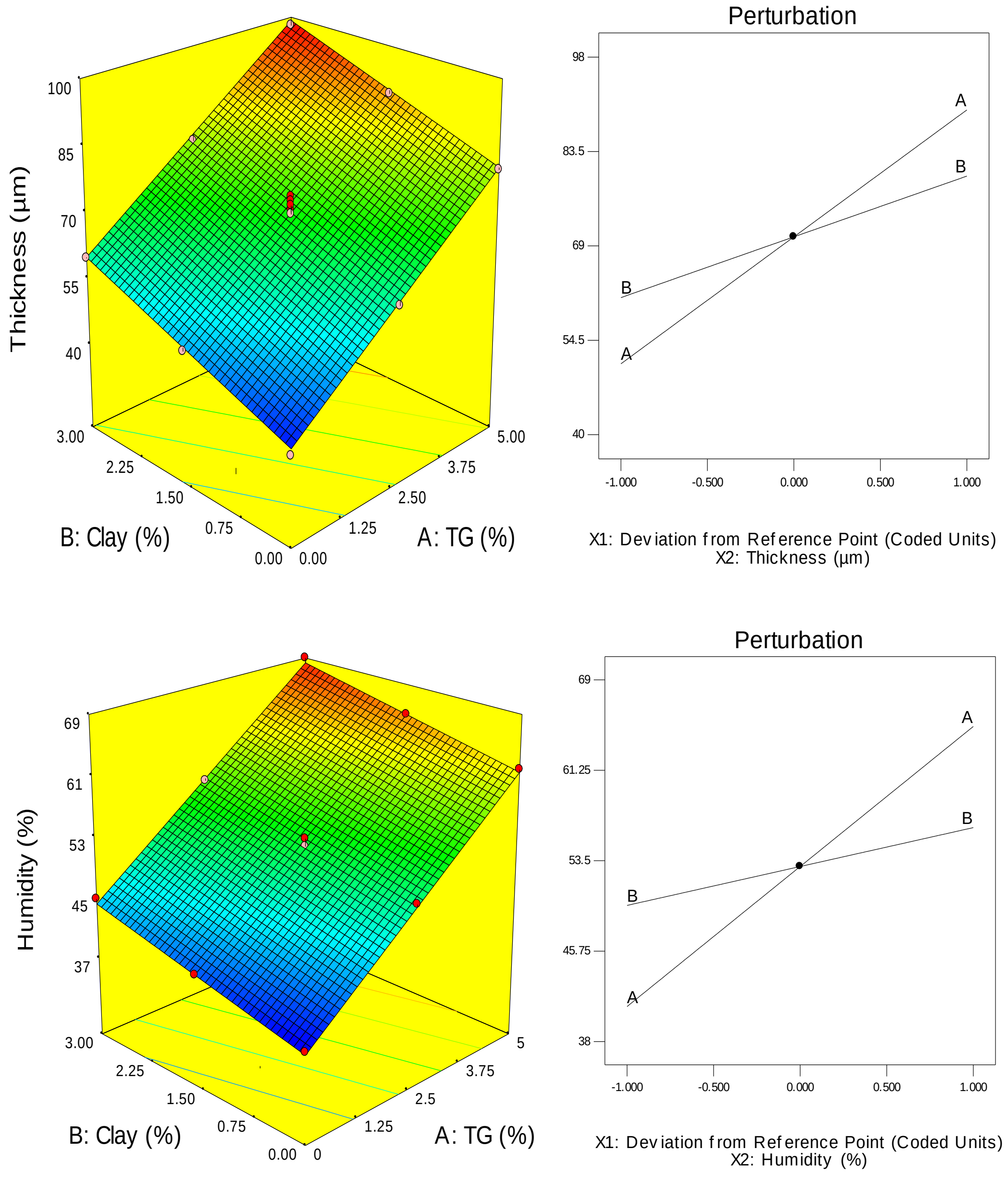


Fig. 1. 3-Dimensional plots and perturbation curves of the effect of TG and Clay on the thickness and humidity content of starch film

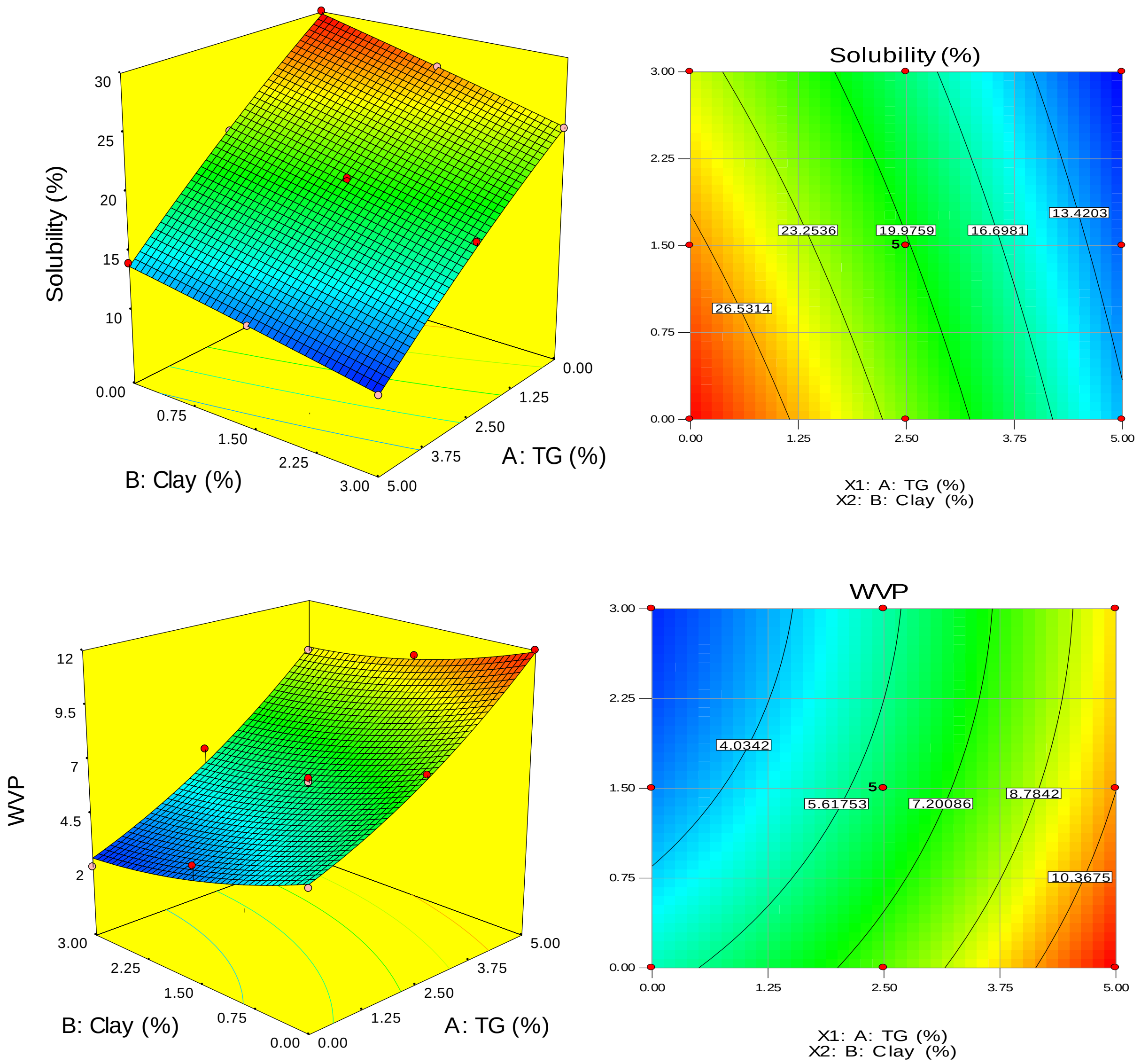


Fig. 2. 3-Dimensional and contour plots of the effect of TG and Clay on the solubility and WVP of starch film

3.3. Mechanical properties

Fig. 3 shows 3-Dimensional plots of the effect of TG and Clay on the TS and EB of starch film. Biodegradable films, although environmentally are interesting and are called environmentally friendly polymers, they are mechanically flawed. These polymers do not have good tensile strength and strain to break.

Therefore, in recent years, many studies have been done on the mechanical properties of biodegradable films. One of the ways to enhance the mechanical properties of films is to compost these materials together and also to composite these films with nanoparticles.

Nanoclay is one of the best nanomaterial to enhance the mechanical properties of biodegradable films. As shown in Fig. 3, tragacanth has dramatically increased the tensile strength of the starch film, so that by adding 5% tragacanth to the starch film, its tensile strength has more than tripled. By placing tragacanth polymers between the starch polymer chains, the van der Waals forces between the polymer chains are likely to be strengthened, which increases the tensile strength of the starch film.

Clay nanoparticles also increase the tensile strength of starch film. In fact, clay nanoparticles are materials that, by creating electrostatic interactions with the polymer chains of starch, increase the cohesion of the polymer network and increase its tensile strength. As for the elasticity of the starch film, as can be seen, Tragacanth has increased the elasticity of the starch film, while the Nanoclay has reduced the tensile strength of the starch film. As we know, tragacanth has the property of absorbing water and can form gels, so by placing tragacanth gel in the structure of the starch film, this gel acts as a plasticizer and increases the elasticity of the film.

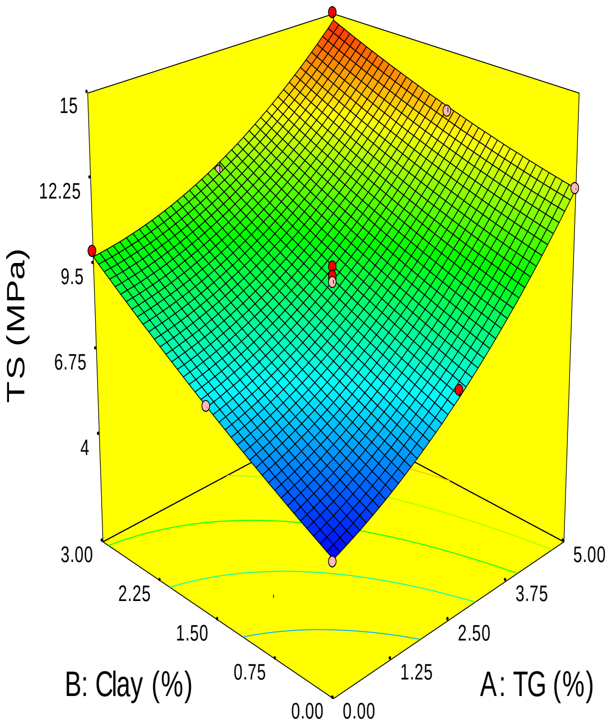
Regarding the effect of clay nanoparticles, it should be noted that these nanoparticles reduce the flexibility of the film and reduce its tensile strength by placing it between polymer chains and increasing the mechanical strength of the film. Wu et al. (2009) examined the effect of agar gel on the mechanical properties of starch film, the results of their research on the tensile strength and elongation of starch film confirm the results of the present study [20]. Lao et al.

(2019) examined the effect of clay nanoparticles on the mechanical properties of biodegradable films, whose their results are consistent with the results of the present study [21].

3.4. Color properties

Fig. 4 shows the contour plots of the effect of TG and Clay on the color properties (L, a and b) of starch film. The color and transparency of the packaging film are one of the most important and influential factors in terms of consumer acceptance. Color is one of the most important characteristics of a food film, which plays an important role in its appearance and marketability. In general, polymer film with high lightness and more similarity to synthetic plastics has high acceptance and application.

The color specifications of the packaging film depend on the type of composition and the process used to make the film. Biodegradable films are usually clear, milky, or colored. Indicator a, indicates the red-green rate of the samples so that the positive numbers represent the redness and the negative numbers indicate the green color of the samples. Similarly, the b-indicator indicates that the color of the samples is yellow-blue, and the negative b indicates the presence of a blue background and a positive b indicates a yellow background in the samples. L indicates the lightness.



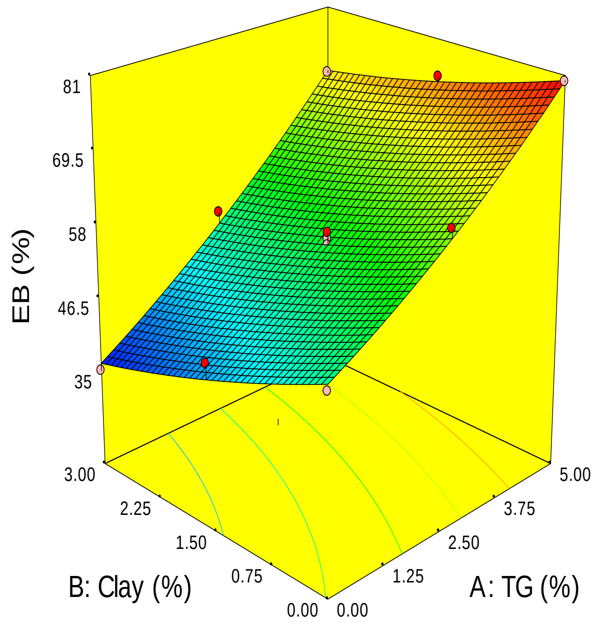
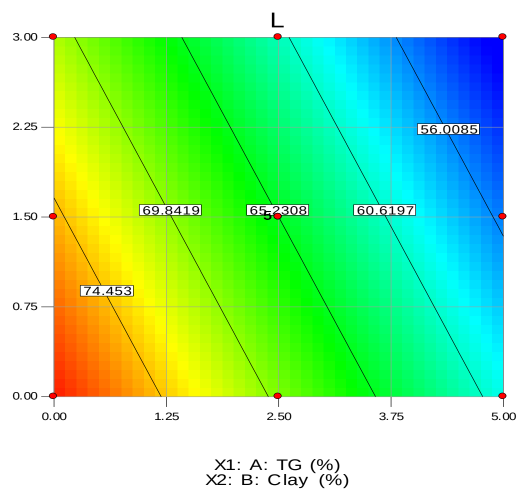


Fig. 3. 3-Dimensional plots of the effect of TG and Clay on the TS and EB of starch film

As can be seen from the results, the addition of tragacanth and Nanoclay to the starch film has reduced the transparency of the film. Tragacanth influence on the film's clarity has been greater than that of the Nanoclay. Due to the fact that starch is a clear film and the color of tragacanth is almost milky or gray, adding tragacanth to the film reduces its transparency. Clay nanoparticles also have a cream-yellow color, so adding clay nanoparticles to the film reduces its transparency and changes factor b to positive numbers, which indicates that by adding these nanoparticles to the film, the color of the film becomes yellow.

Tragacanth and clay nanoparticles have also increased the a-factor (red) of starch film. Kim and Lee (2002) studied the color properties of potato starch and starch-filled linear low-density polyethylene films. Their results confirm the results of the present study [22].



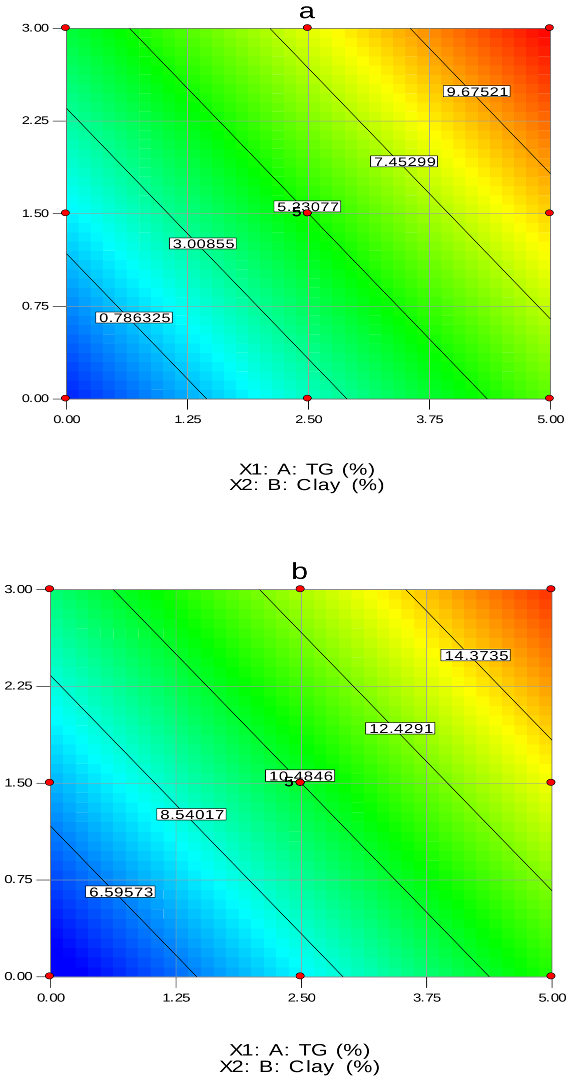


Fig. 4. Contour plots of the effect of TG and Clay on the color properties (L, a and b) of starch film

4. Conclusion

In this study, starch film, starch/tragacanth composite and starch/tragacanth/Nanoclay composite films were prepared. The central compound statistical design was used to investigate the effect of tragacanth concentration at three levels and clay nanoparticle concentration at three levels on the film properties. Physicochemical, mechanical and color characteristics of the prepared films were investigated. Results showed that, the addition of tragacanth and Nanoclay to the starch film increased film thickness. The effect of tragacanth on increasing film thickness was greater than that of Nanoclay. The addition of tragacanth and Nanoclay to the starch film has reduced the transparency of the film. Tragacanth influence on the film's clarity has been greater than that of the Nanoclay. Tragacanth increased the elasticity of the starch film, while the Nanoclay reduced the tensile strength of the starch film. According to the results, tragacanth increased the permeability of starch film to water vapor. Unlike tragacanth, Nanoclay has reduced WVP. Clay nanoparticles are placed in empty spaces between starch polymers and prevent the passage of water vapor molecules, thus reducing WVP. The overall conclusion was that the addition of tragacanth and clay nanoparticles to the starch film improves the mechanical and physicochemical properties of the film and makes it usable for food packaging.

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