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Edible iron yam and maize starch convenient food flavoring packaging films with lemon essential oil as plasticization

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Abstract

Lemon essential oil was used as plasticizers to prepare iron yam/maize starch edible films. Changes of physical, microcosmic and antimicrobial properties of films were studied. Results showed that the addition of lemon essential oil led to the decrease of moisture content, transparency, whiteness index, water vapor permeability, solubility and tensile strength and the increase of b^*, elongation and haze values. With the increase of lemon essential oil content, the antibacterial activity increased, and the surface and internal microstructure of the film became more and more heterogeneous. Compared with the control group, the tensile strength, water content and WVP of IYM/2LO films decreased by 21.9%, 23.5%, and 23.6%, respectively. When surfactant was added, the antimicrobial activity, solubility, haze, surface coarseness and porosity increased significantly, and the tensile strength decreased from 11.8MPa (IYM/1LO) to 7.60MPa (IYM/1LO/0.1S) obviously. The iron yam/maize starch edible films with good physical and antibacterial properties can be considered as convenient food flavoring packaging materials.

Keywords: iron yam/maize starch film; lemon essential oil; plasticization; edible flavoring packaging materials.

Practical Application: Provide a convenient food flavoring packaging material without chemical synthetic plasticizer.

1 Introduction

Nowadays, convenient food such as instant noodles has become very popular due to its ease of preparation, reasonable price, and delicious taste. Instant noodle flavoring mix has generally been provided in a separately packaged plastic film pouches designed for single usage (Aguirre-Joya et al., 2018). However, there are some shortcomings because of the using plastic film pouches for condiments including: (1) plastic products are hard to be degradation, causing great white pollution to our environment; (2) leftover condiments may remain in the pouch after application to food; (3) there is a potential threat to people's health due to the transfer of additives originally incorporated in the plastic films into the condiments.

Edible packaging materials produced from agro-resources may provide an alternative to non-degradable polymers (Bui et al., 2016; Podshivalov et al., 2017; Wang et al., 2017). Among various available biopolymers such as polysaccharides, lipids and proteins, starch is one of the most commonly used natural material, since it is widely available, inexpensive, and relatively easy to handle (Kim et al., 2015; Moreno et al., 2015). However, plain starch films without other compounds are readily broken into fragments because of the poor mechanical strength when they are dried in ambient conditions. The addition of plasticizers to pure starch-based materials is essential to overcome film brittleness caused by high intermolecular forces, to enhance film-forming characteristic, workability and serviceability of the coatings. Plasticizer plays a significant role in the starch film formation affecting the film structure and, accordingly, all the physical properties of films (Mali et al., 2005). The basic rationale of the plasticization is that plasticizers can attract the water molecules, reduce the intermolecular interactions between the starch molecules, and increase the flexibility of films (Cao et al., 2018). At present, the most commonly plasticizers used in starch-based films are polyols, such as sorbitol (Cao et al., 2018), glycerol (Chevalier et al., 2018), ethylene glycol (Illiger et al., 2009), maltitol and xylitol (Saberi et al., 2017) which are often used to modify the mechanical properties of the films. Besides that, the monosaccharides such as glucose, mannose, galactose, and fructose were also used as plasticizers to investigate the effects on physical and mechanical properties of starch-based films (Cao et al., 2018).

However, it has been found that the use of synthetic plasticizers has potentially adverse effects on human health (Bui et al., 2016). Therefore, the use of natural materials instead of chemical synthetic additives is preferred. Essential oils are aromatic oily liquids extracted from plant materials and commonly studied as additives in edible/biodegradable films due to their antimicrobial (Zhang et al., 2015; Roselló et al., 2015; Aldana et al., 2015; Jouki et al., 2014) and antioxidant (Dashipour et al., 2015; Alves-Silva et al., 2013; Ruiz-Navajas et al., 2013; Bonilla et al., 2013) capacity. Moreover, they have proved to have some impact on mechanical properties of edible films (Altiok et al., 2010; Pires et al., 2011; Tongnuanchan et al., 2012; Shojaee-Aliabadi et al., 2014a). In this sense, essential oil can

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be potentially used as plasticizers to improve the mechanical properties of edible films.

Iron yam is a dual-use food announced by the Ministry of health in China. Starch is the main nutritional component of iron yam. The paste viscosity of iron yam starch is stable, and its viscosity changes little at high temperature. One of the main characteristics of maize starch is high amylose content, which is favorable for film formation. Up to our knowledge, there is not enough number of published articles dealing with the plasticizing activity or mechanism of lemon essential oil in iron yam/ maize starch films. The main objective of this work was the development and the characterization of iron yam/ maize starch films using lemon essential oil as plasticizers for the purpose of producing edible films with better properties. The influences of different concentrations of lemon essential oil on the films' mechanical properties, permeability to water vapor, color, solubility, antimicrobial activity and microstructure were evaluated. In addition, the effects of surfactant on these properties of iron yam/ maize starch film were also studied.

2 Materials and methods

2.1 Materials

Iron yam starch was provided by Hangzhou Mei Yan Trade Co., Ltd. (Hangzhou, China). Maize starch was provided by Shanghai Luyuan Starch Co., Ltd. (Shanghai, China). Sodium alginate, carboxy methyl cellulose sodium, span 80 were provided by Luoyang Haohua Chemical Reagent Co., Ltd. (Luoyang, China). Lemon essential oil (LO) was provided by Shanghai Tiamay Aromatic Plant Science and Technology Co., Ltd. (Shanghai, China). Tryptone soy broth (TSB) and luria–bertani broth (LB) were supplied by Shanghai Huzhen Industrial Co., Ltd. (Shanghai, China). Nutrient agar was provided by Shanghai Haoyang biological technology Co., Ltd. (Shanghai, China). Staphylococcus aureus ATCC6538 and Escherichia coli CMCC44102 were provided by Henan University of Technology (Zhengzhou, China).

2.2 Film preparation

Preparation of iron yam/maize starch edible films form solutions

36 g iron yam starch and 24g maize starch were dissolved in 1000 mL distilled water to prepare iron yam/ maize (IYM) starch film-forming solutions (6%, w/v). The solution was treated at 100 °C in water bath and stirred for 15 min under the condition of 700 rpm, so that the starch was completely dissolved and gelatinized. Then added 14g carboxy methyl cellulose sodium, 14g sodium alginate to the solution and stirred for 1h. The LO was first dissolved in 0.1% (v/v film-forming solutions) surfactants (Span 80) and then added to the film-forming solution, and the final concentration is 0.5%, 1%, 1.5% and 2% (v/v), respectively. All solutions were homogenised at 10000 rpm for 4 min and the air bubbles were removed by vacuum. Sample codes: IYM/nLO or IYM/nLO/S. n represents the ratio of lemon oil to starch, S represents Span 80.

Preparation of composite films

200 mL film formation solution was casted on a leveled glass plate (25 × 25 cm) and dried at 25 ± 1 °C for at least 16 h. Then the dry films were carefully peeled and stored at 25 ± 1°C and 53 ± 1% RH in desiccators containing saturated magnesium chloride for 7 days.

2.3 Thickness

Thickness of each film was measured in five places using a thickness gauge (GM280F, Shenzhen Huaqing Instrument Co., Ltd., Shenzhen, China) with an accuracy of 0.01 mm. One of the measuring places was the center of the film, the remaining four measuring places were selected in four sides' outer edge, and average values were used. Eight replications of each formulation have been made.

2.4 Mechanical properties

A TA-XT2i Texture Analyzer (Stable Microsystems Ltd., UK) was used to measure the tensile properties according to the method described by Song et al. (2018). The edible films were cut into 20 mm × 80 mm rectangles and fixed between the tensile grips. Initial grip separation was 60 mm and crosshead speed was 1.0 mms^{-1}. Tensile strength and elongation at break were used to evaluate the mechanical properties. Each sample was repeated 8 times.

2.5 Color

Color changes were measured by a color difference meter (WSC-S, Shanghai Precision & Scientific Instrument Co., Ltd., Shanghai, China). The CIE Lab scale was used to record color measurements. The color was presented in terms of L^*, a^* and b^* values. Whiteness index (WI) was calculated using the following Equation 1 (Atarés & Chiralt, 2016).

$$WI = 100 - [(100 - L^*)^2 + a^{*2} + b^{*2}]^{0.5} \quad (1)$$

where L^* was 0 for black and 100 for white, a^* indicated red (+) to green (-) and b^* indicated yellow (+) to blue (-).

2.6 Water content

Water content (WC) was measured before and after dried in an oven at 105 °C for 24 h. WC was calculated as the following Equation 2:

$$WC = (M_0 - M)/M \quad (2)$$

where M_0-the initial mass (g), M-the bone-dry mass (g). WC was expressed as g H_2O/g dry solids. Measurements were repeated 3 times.

2.7 Film solubility

Film solubility was measured according to the method described by Kim et al. (2015). Films were cut into 20 × 20 mm pieces and dried in a vacuum oven (Shanghai Yiheng Technology

Co., Ltd., Shanghai, China) for 24 h at 100 °C to get the initial dry mass. The films were then placed in a beaker with 30 mL of distilled water. The beakers were sealed with fresh-keeping film and stored at 25 ± 1 °C for 24 h. Discarded the residual water from the beakers and gently rinsed the edible film pieces with distilled water. The dry mass was determined by drying the residual film pieces at 100 °C in a vacuum oven. The loss of total soluble matter was calculated from the initial and final dry weight of films. Measurements were taken 3 times for each treatment.

2.8 Water vapor permeability

Water vapor permeability (WVP) was measured gravimetrically according to the modified method described by Song et al. (2018). The thickness of each film was measured by a micrometer (0.02 mm accuracy) at five randomly selected points. The films were sealed onto permeation cells (1384.74 mm × 25 mm) filled with granular (Φ < 2 mm) anhydrous calcium chloride. The permeation cells were then placed in desiccators filled with saturated sodium chloride solutions at three relative humidities (RH) gradients (0, 75%, and 100%) at 25 °C, and were weighed until weight changes were close to 0.001 g. WVP was calculated use the following Equation 3:

$$WVP = \frac{mL}{At\Delta P} \quad (3)$$

where: *m*-weight (g), *L*-thickness (m), *A*-permeation area (m^2), *t*-time (s), ΔP-water vapor pressure difference (Pa). Each test was made 5 times.

2.9 Transmittance and haze

Films' transmittance and haze were measured by a transmittance haze meter (WGT-S, Shanghai Yanhe Scientific Instrument Co., Ltd., Shanghai, China). Films were cut into 40×40mm strips and mounted between the magnetic clamp. Then film specimen was flattened and against the integrating sphere. Measurements were repeated 6 times.

2.10 Antimicrobial activity

Films' antimicrobial properties with *E. coli* and *S. aureus* as model bacteria were determined by the agar diffusion method described by Acevedo-Fani et al. (2015). A loop of *E. coli* was inoculated into 25 mL LB and a loop of *S. aureus* was inoculated into 25 mL TSB, respectively, in 50 mL flasks, which were then incubated in a constant temperature vibrator (Changzhou Putian Instrument Manufacturing Co., Ltd, Jiangsu, China) with 200rpm at 37 °C for 24 h. In order to display the antibacterial properties differences more clearly, appropriate amounts of suspensions were again transferred to nutrient broths and incubated at 37 °C to the exponential phase of growth, forming bacterial cell suspensions for following tests. The concentrations of the *S. aureus* and *E. coli* suspensions were 6×10^7 CFU mL^{-1} and 4×10^7 CFU mL^{-1}, respectively.

Films were cut into 6 mm diameter discs and then placed on nutrient agar in petri dishes filled with 20 mL bacterial cell suspensions. The petri dishes were examined for zone of inhibition after 24 h incubation at 37 °C. Films with no LO and span 80 were treated by using the same protocol and were used as control. All tests were repeated 3 times.

2.11 Microstructure determination

A JSM-7401F field emission scanning electron microscope (JEOL Ltd., Akishima, Japan) operating at 20 kV was used to analysis the microstructure of the films. Films were previously conditioned desiccators (0% RH) filled with P_2O_5 at 25 °C for 2 weeks. Films were cut into 6 × 1 mm pieces and then fractured in liquid nitrogen. Samples were mounted on metal grids using double-sided adhesive tape and coated with gold under vacuum.

2.12 Statistical analysis

Analysis of variance was performed by ANOVA procedures of the SPSS software (version 13.0, Statistical Package for the Social Sciences Inc., Chicago, USA). LSD test was used to determine the difference of means, and P<0.05 was considered to be statistically significant.

3 Results and discussion

3.1 Thickness, color and mechanical properties

As shown in Table 1, the average thickness of the prepared films was 75.86 μm, and no remarkable difference (P<0.05) was observed among all the films. This result indicated that the agglomerations of small particles of essential oil in the film had little effect on the thickness variation of the prepared film. This results were in agreement with other studies reported previously by Altiok et al. (2010) and Hoque et al. (2011). Different conclusion was also reported, where the thickness of films increased significantly with the addition of Zataria multiflora Boiss essential oil (Atarés & Chiralt, 2016).

Table 1. Thickness and color of Iron yam / Maize composite films[a].

Films	Thickness (μm)	L*	a*	b*	WI
Control	73.33 ± 1.36[a]	59.98 ± 0.52[a]	-1.73 ± 0.49[a]	1.72 ± 0.54[c]	59.94 ± 0.21[a]
IYM/0.5LO	74.49 ± 1.25[a]	59.46 ± 0.31[ab]	-1.98 ± 0.42[ab]	2.49 ± 0.10[b]	59.39 ± 0.35[b]
IYM/1LO	75.69 ± 1.12[a]	59.45 ± 0.38[ab]	-2.06 ± 0.68[ab]	2.50 ± 0.15[b]	59.37 ± 0.13[b]
IYM/1.5LO	76.53 ± 1.28[a]	59.37 ± 0.13[ab]	-2.19 ± 0.82[ab]	2.57 ± 0.14[b]	59.28 ± 0.31[bc]
IYM/2LO	76.88 ± 1.31[a]	59.22 ± 0.36[b]	-2.54 ± 0.31[ab]	2.65 ± 0.11[b]	59.13 ± 0.52[bc]
IYM/1LO/0.1S	78.21 ± 1.31[a]	59.29 ± 0.21[c]	-2.98 ± 0.67[b]	3.12 ± 0.26[a]	59.17 ± 0.37[c]

[a]Data were shown in mean ± standard deviation. Different superscript letters in the same column indicated significant differences (P<0.05).

The color of the edible film is an important indicator of the acceptability of the consumer. The properties of the additives influence the optical properties of edible film (Song et al., 2018). From Table 1 we can see that compared with the control, the lightness of the edible film decreased significantly when the amount of essential oil was 2%. When the content of essential oil was 1%, the addition of surfactant significantly reduced the lightness of the edible film.

As shown in Table 1, the b* value of the control film was 1.72. When the lemon oil was added to the composite film, the yellow color of the film increased significantly as evidenced by the significantly ($P<0.05$) increased b* value compared with the control films. The experimental results of WI also proved the conclusion, as evidenced by the significantly ($P<0.05$) decreased WI values. The main reasons were that the natural yellow color of lemon essential oil, and the concentration of essential oil incorporated in the iron yam/maize starch matrix could induce the light scattering on films' surface. This results are in agreement with another reported previously (Song et al., 2018). However, there were no obvious difference in the b* values among edible films with different essential oil content. From Table 1 we can also see that the b* of the composite film with emulsifier (IYM/1LO/0.1S) was higher, because the emulsifier reduced the tension of the oil-water interface and emulsified more essential oils in the composite film.

As shown in Table 2, compared with the control group, the tensile strength of edible films decreased with the increase of essential oil content, while the elongation at break increased with the increase of essential oil content. The decrease of tensile strength may be attributed to the addition of essential oil which developed heterogeneous thin film structures with discontinuities. As the essential oil is liquid at room temperature, it will exist in the form of easily deformed oil droplets in the oil film, thus increasing the extensibility of the film (Fabra et al., 2008). From Table 2 we also see that the addition of surfactant reduced the tensile strength and increased the elongation at break of IYM/LO films. Tensile strength decreased from 11.8MPa (IYM/1LO film) to 7.60MPa (IYM/1LO/0.1S film), and elongation at break increased from 31.86% to 38.25%. The possible reason was the hydrophobicity surfactant influenced the intermolecular hydrogen bonding within starch–starch or starch–water.

Table 2. The mechanical properties of Iron yam / Maize composite films[a].

Films	Tensile strength (MPa)	Elongation at break (%)
Control	11.80 ± 1.16[a]	31.86 ± 0.84[c]
IYM/0.5LO	10.16 ± 1.40[b]	34.99 ± 0.96[b]
IYM/1LO	9.84 ± 1.20[b]	35.54 ± 0.83[b]
IYM/1.5LO	9.68 ± 0.79[b]	35.85 ± 0.58[b]
IYM/2LO	9.21 ± 0.70[b]	36.27 ± 0.87[b]
IYM/1LO/0.1S	7.60 ± 1.08[c]	38.25 ± 1.18[a]

[a]Data were shown in mean ± standard deviation. Different superscript letters in the same column indicated significant differences ($P<0.05$).

3.2 Water content

As shown in Table 3, the water content of film decreased with the increase of essential oil content compared with the control films. The decrease of water content was mainly due to the interaction between starch and essential oil which limiting polysaccharide–water interactions. Similar results have been reported by Dashipour et al. (2015) and Hafsa et al. (2016). From Table 3 we can also see that the incorporation of surfactant into the composite films affected its water content. When the surfactant span 80 existed, the water content of the films decreased significantly ($P<0.05$) because of the lower HLB value. Villalobos et al. (2006) also found that hydrophobic surfactant such as span could decrease water vapor adsorption of hydroxypropyl methylcellulose films and give rise to greater reduction in water content.

3.3 Film solubility

As can be seen from Table 3, compared with the control films, the solubility of the edible film decreased with the addition of lemon essential oil. The solubility values of control, IYM/0.5LO, IYM/1LO, IYM/1.5LO and IYM/2LO films were 53.16%, 52.25%, 52.01%, 51.10 and 50.21%, respectively. The main reason for the decrease of water solubility was the decrease of the hydrophilicity of the film, as well as the interaction between the components of the essential oil and the hydroxyl of the edible film, which reduced the effectiveness of the interaction between the hydroxyl and water molecules, and made the edible film's water resistance stronger. From Table 3 we can also see that IYM/1LO/0.1S film showed remarkable ($P<0.05$) higher solubility in water compared with IYM/1LO film. The interaction between starch and oil was weakened, resulting in the migration of lemon essential oil to the surface of the film. The increase of irregular surface structure and surface roughness increased the contact area of the film and water, resulting in an increase in water solubility. Previous studies have also got similar results (Peng et al., 2013; Zhong & Li, 2011).

Table 3. Physical properties of corn and wheat starch films[a].

Films	Water content (%)	Solubility (%)	WVP ($gm^{-1}s^{-1}Pa^{-1} \times 10^{-11}$)	Transmittance (%)	Haze (%)
Control	18.83 ± 0.41[a]	53.16 ± 1.13[b]	4.95 ± 0.07[a]	90.07 ± 0.12[a]	28.00 ± 0.77[d]
IYM/0.5LO	15.50 ± 0.38[b]	52.25 ± 1.05[c]	4.93 ± 0.07[a]	89.24 ± 0.11[b]	44.00 ± 0.83[c]
IYM/1LO	15.35 ± 0.40[b]	52.01 ± 1.09[c]	4.75 ± 0.05[a]	88.45 ± 0.14[c]	44.20 ± 0.74[c]
IYM/1.5LO	14.88 ± 0.31[b]	51.10 ± 1.08[d]	4.25 ± 0.06[b]	88.31 ± 0.17[c]	45.40 ± 1.18[c]
IYM/2LO	14.40 ± 0.29[b]	50.21 ± 1.07[d]	3.78 ± 0.08[c]	88.18 ± 0.16[c]	54.34 ± 1.40[b]
IYM/1LO/0.1S	11.08 ± 0.30[c]	55.23 ± 1.11[a]	3.57 ± 0.08[c]	87.17 ± 0.15[d]	68.10 ± 1.58[a]

[a]Data were shown in mean ± standard deviation. Different superscript letters in the same column indicated significant differences ($P<0.05$).

3.4 Water Vapor Permeability (WVP)

The WVP of edible film is one of the most important factors in the quality of food packaging materials. The edible film should be able to avoid, or at least reduce the water transfer between the environment and the food. From Table 3 we can see that the WVP of control film was 4.95×10^{-11} $gm^{-1}s^{-1}Pa^{-1}$. The WVP of the edible films decreased with the concentration of lemon essential oil from 0.5% to 2%. Compared with the control, the WVP values of IYM/1.5LO and IYM/2LO were significantly reduced by 14.1% and 23.6% respectively. The results could be attributed to reduced bonding ability to water and the enhancement of tortuosity factor of the vapor diffusion-path through film as result of lipid globules presence (Hashemi et al., 2017). Similar conclusion was given by Chen & Liu (2016). But other authors (Dashipour et al., 2015; Jouki et al., 2014) found that the addition of essential oil increased the WVP of edible films. As shown in Table 3, the WVP value of IYM/1LO/0.1S film decreased significantly from 4.75×10^{-11} $gm^{-1}s^{-1}Pa^{-1}$ to 3.57×10^{-11} $gm^{-1}s^{-1}Pa^{-1}$ compared with IYM/1LO film. From Table 3, we can also see that the WVP value of the edible film decreased with the addition of the surfactant. The WVP was $4.75 \times 10^{-11}gm^{-1}s^{-1}Pa^{-1}$ for the IYM/1LO film, which decreased markedly to $3.57 \times 10^{-11}gm^{-1}s^{-1}Pa^{-1}$ for the film that containing 0.1% span 80 (IYM/1LO/0.1S). The results of different studies on the effect of surfactant on the edible films' WVP were not the same (Chen et al., 2009; Peng et al., 2013), which indicated that the influence of surfactant on the MVP of edible films could be related to the kinds and concentrations of surfactants, and the properties of edible film materials.

3.5 Transmittance and haze

The transmittance of the film directly reflects the visual effect of the inner packaging, and the degree of haze can reflect the opacity of the film. As shown in Table 3, as compared with the control films, with the addition of lemon essential oil, the transmittance of the edible film gradually decreased, and the values of the haze increased gradually. This conclusion is similar to the results that have been published previously (Choi et al., 2016; Dashipour et al., 2015). The cause of this phenomenon might be due to the presence of oil droplets in the network of edible films, resulting in an increase in light scattering. The light scattering depended mainly on the content and particle size of the dispersed phase. In general, the more droplets, the greater the intensity of light scattering, the lower the transmittance (Shojaee-Aliabadi et al., 2014b). It can be seen from Table 3, the existence of surfactant affected the edible films' transmittance and haze. The transmittance values of IYM/1LO/0.1S film were lower than ($P < 0.05$) that of IYM/1LO film. On the other hand, the addition of span 80 significantly ($P<0.05$) increased the haze values compared with IYM/1LO film. The reason may be that the essential oil was easy to migrate to the surface of the film. The transmittance and haze of the edible film were caused by the irregular characteristics of the reflected surface. In the process of film forming, the degree of dispersion, the type of additives and the compatibility of each component would affect the irregular characteristics of the films' surface (Song et al., 2018). The results of the SEM experiment in this study confirmed the conclusion.

3.6 Antimicrobial activity

Figure 1 shows the antibacterial properties of the compound film solution with essential oil. It can be seen that the sensitivity of Gram-positive bacteria to edible film was significantly higher than that of Gram negative bacteria. This may be caused by the relatively impermeable outer membrane around the Gram-negative bacteria (Biddeci et al., 2016). This result was in line with other previously reported papers, in which the edible film's antibacterial activity was affected by the clove bud oil (Lee et al., 2015) and

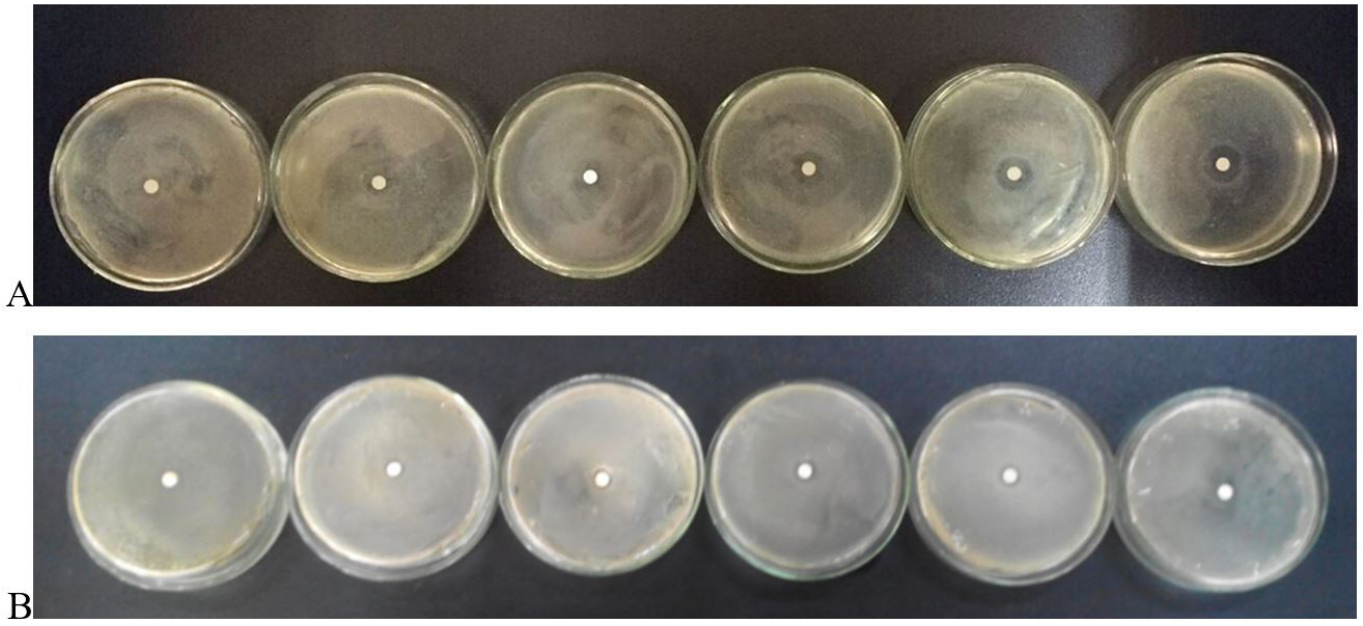

Figure 1. Images of zones of inhibition against (A) *S. aureus*; (B) *E. coli* (from left to right: Control, IYM/0.5LO, IYM/1LO, IYM/1LO/0.1S, IYM/1.5LO, IYM/2LO).

the lemon oil (Fisher & Phillips, 2008). From Figure 1 we can see that the control film did not show the inhibitory halo around the membrane, and there was no obvious bacteriostasis effect. In the presence of lemon essential oil without adding surfactant, IYM/2LO had the largest inhibitory area, indicating that it had the best antibacterial effect. Essential oil affected the microbial cells that attack the phospholipid bimolecular layer of the cell membrane, destroyed the enzyme system, and affected the genetic material of the bacteria (Atarés & Chiralt, 2016; Chen & Liu, 2016). Figure 1 also shows that the addition of 0.1% surfactant obviously increased ($P<0.05$) the antimicrobial activity compared with the IYM/1LO film. The possible reason is that the cell membrane's normal function is affected by the permeability of the surfactant alkyl in the cell membrane.

3.7 Microstructure

The SEM microstructure of the surface and cross section of the composite film are shown in Figure 2. On the premise of not adding essential oil and surfactant, the film's surface

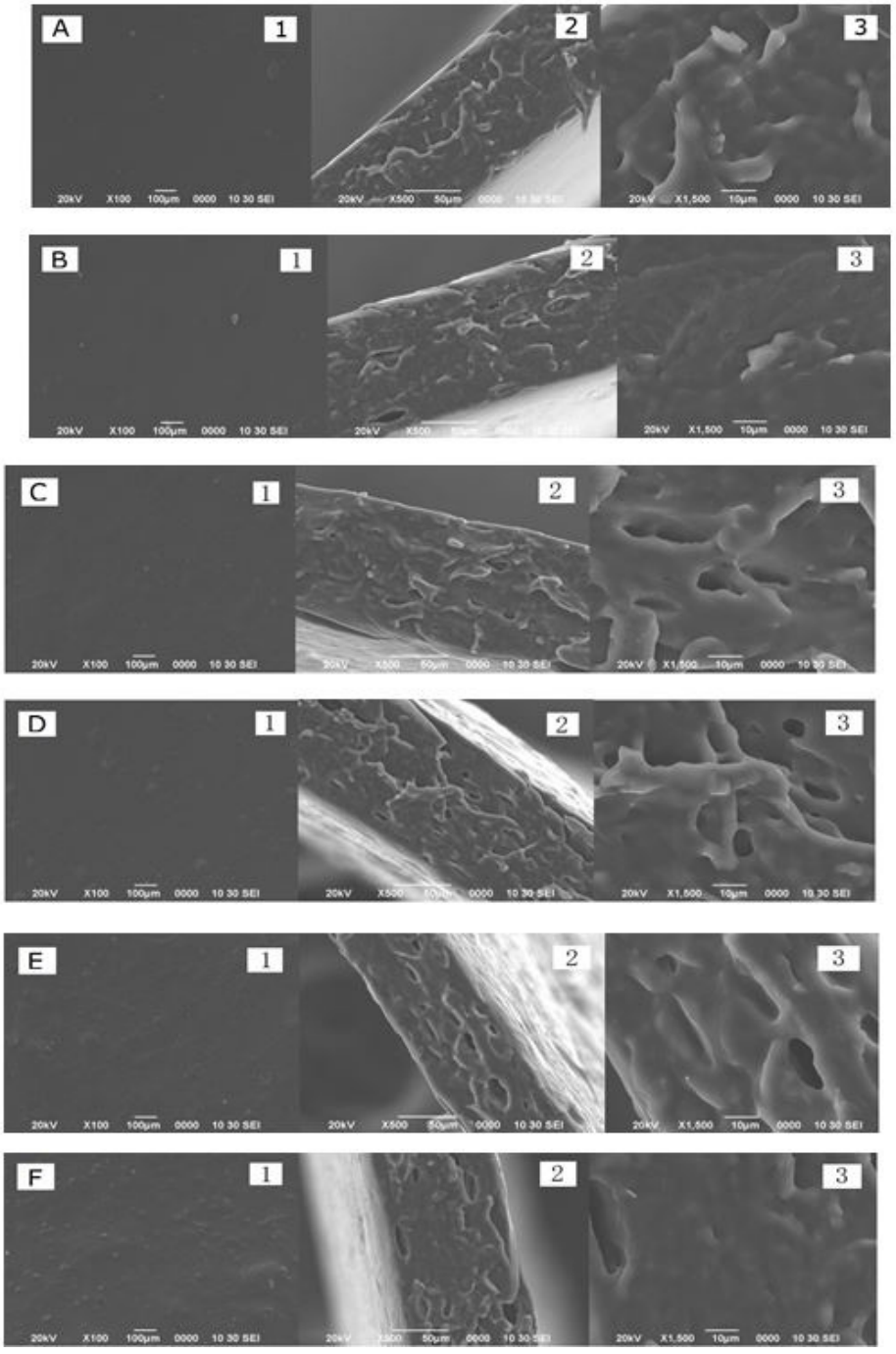

Figure 2. SEM micrographs of the surface (left column, 100×) and cross section (middle column, 500×, and right column, 1500×) of iron yam/maize films. (A) control; (B) IYM/0.5LO; (C) IYM/1LO; (D) IYM/1.5LO; (E) IYM/2.0LO; (F) IYM/1LO/0.1S).

of the control group was smooth and uniform (Figure 2A1). Shojaee-Aliabadi et al. (2014b) studied the microstructure of the pure carrageenan film, and the results were similar to ours. However, Acosta et al. (2016) found that the surface of pure starch gelatin film showed heterogeneous structure, probably due to the not completely miscibility of starch and gelatin as well as the polymer phase separation. As shown in Figure 2B-F, the addition of essential oil made the surface of edible film appear irregular structure, and the roughness of the surface increased with the increase of essential oil content. Similar conclusions were also given in previous papers, which may be due to coalescence, flocculation and wrinkling during the edible film's drying process. Some of the oil droplets migrated to the surface of the film and evaporated (Acevedo-Fani et al., 2015; Choi et al., 2016; Hafsa et al., 2016).

From Figure 2, we can see that although the control film showed smooth and uniform surface structure, there appeared a fibrillar network structure in the cross section (Figure 2A2,3). The main reason is probably the incomplete dissolution/gelatinization of starch granules. Similar conclusions have been drawn from published papers (Bonilla et al., 2013; Shi et al., 2013).

The addition of essential oil led to microporous holes structure in the cross section of the edible film, and the number and size of the micropores increased with the increase of the content of the essential oil (Figure 2B2,3; C2,3; D2,3; E2,3; F2,3). The presence of many holes correspond to the position of the oil droplets. As previously reported, because of high vacuum, these substances may be partially evaporated during the scanning electron microscope analysis (Biddeci et al., 2016; Fisher & Phillips, 2008). After adding the lion essential oil and the tea tree essential oil to the chitosan film and the hydroxypropyl methylcellulose film, the similar phenomenons were found by Peng & Li (2014) and Song et al. (2018).

Figure 2 also shows that compared with the surface microstructure of IYM/1LO film (Figure 2C1), the addition of surfactants led to more granular protuberance on the surface of IYM/1LO/0.1S film (Figure 2F1), which resulted in the increase of surface roughness. The reason may be that the surfactant span 80 migrated to the surface of the film during the drying process of the film. In addition, the microstructure of the cross section of the film (Figure 2F2,3) also changed obviously because of the addition of surfactant. The number of micropores increased, but the size of the micropores decreased. It may be because the interaction between the surfactant and the macromolecule changed the distribution of the surfactant and led to the changes of the microstructure.

4 Conclusions

Under the condition of no essential oil, the iron yam/maize starch films were basically transparent, smooth and homogeneous appearance without concave convex matrix. In the presence of essential oil, compared with the control film, the thickness was not obviously changed, however, the water vapor permeability, water content, solubility and tensile strength were reduced. With the increase of the concentration of essential oil, the whiteness value decreased, the b* value increased, the antimicrobial properties increased, and the microstructure became more heterogeneous. The existence of surfactant significantly changed the mechanical properties, bacteriostatic, transparency, solubility and microstructure of edible film. The findings suggested that iron yam/maize starch films used lemon essential oil as plasticization can be considered as a new inner package material for instant food.

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