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Edible films from seaweed *(Kappaphycus alvarezii)*

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Abstract

A new patent pending process is proposed in this study to produce edible film directly from seaweed *(Kappaphycus alvarezii)*. Seaweed together with other ingredients has been used to produce the film through casting technique. Physical and mechanical tests were performed on the edible films to examine the thickness, colour, transparency, solubility, tensile strength, elongation at break, water permeability rate, oxygen permeability rate and surface morphology. Produced film was transparent, stretchable, sealable and have basic properties as a film for food packaging. This study suggests that the edible film could be used as novel materials in food industry as sachet/pouch/bag for instant coffee, breakfast cereals drinks, seasoning powder, candies etc; as wrapper for seasoning cube and chocolate; as interleaf for frozen foods such as burger patties to avoid the patties from sticking together; and also as material for edible logo in bakeries products. Other than that, the edible film also could be used in pharmaceutical industry as functional strips such as oral freshener strips and drug strips. In cosmetic and toiletries industries, the edible film could be used to produce facial mask and bag for pre-portioned detergent. Compared with edible film developed earlier using alginate and carrageenan, film developed in this research used seaweed directly. The developed film reduced the need to extract the alginate and carrageenan, making material preparation easier and cheaper.

Introduction

Plastic products have become an integral part in our daily life as a basic need. Its broad range of application is in packaging films, wrapping materials, shopping and garbage bags, fluid containers, clothing, toys, household and industrial products, as well as building materials. Once plastic is discarded after its utility is over, it is known as plastic waste. Only small percentages of this plastic waste are recycled and mostly will be ends up in landfills, beaches, rivers and oceans. It is a fact that plastics will never degrade and remains on landscape for several years and subsequently raises environmental issues. Therefore, a small reduction in the amount of plastic materials will reduce environmental concerns. Development of edible and biodegradable material to partially replace petro-based polymers may offer opportunities that would benefit both manufacturers and consumers.

To meet the growing demand of degradable and natural materials, new materials and technologies have been extensively studied including using bio-based polymers. Bioplastic or biopolymer products can be made from raw materials originating from agricultural or marine sources. However, this kind of research is still on the preliminary stage in Malaysia. This paper was focused on the development of edible seaweed based films for application especially in food industry, pharmaceutical industry, cosmetics and toiletries industries.

Seaweed is sustainable natural resources with industrial potential that is not fully utilized. The farming of seaweed has expanded rapidly in Malaysia, especially Sabah, is one of the seaweed producers in the world with total production in year 2010 was 150,000 MT and expected to produce 1,500,000 MT by 2020 (Tan *et al*., 2011). Under the Economic Transformation Programme (ETP), the Federal Government has approved substantial amount of fund to uplift the industry to be one of the economic resources for the country and creating more employment opportunities. Hence, research and development programme was needed to support the industry. Through this project, the development of edible seaweed based film is a promising method for diversifying the usage and adding value to the seaweed.

Seaweed derivatives such as alginate and carrageenan have been widely used to form edible films and their barrier and mechanical properties

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have been studied (Pranoto *et al*., 2005; Maria *et al*., 2007; Maria *et al*., 2008; Maizura *et al*., 2007; Maizura *et al*., 2008; Alicia *et al*., 2009; Mariana *et al*., 2009; Fazilah *et al*., 2011; Song *et al*.; 2011). However, on another aspect, the possibility to form film directly from seaweed has not yet been explored. Since seaweed mainly consists of protein and non-starch polysaccharides (Dawczyski *et al*., 2007), these compounds might possibly provide sufficient properties to form edible film and packages. This film would reduce the need to isolate the alginate and carrageenan, making material preparation easier and cheaper. Therefore, the objectives of this paper are to develop edible film directly from seaweed *(Kappaphycus alvarezii)* and to determine the characteristics of the developed edible film and at the same time to study its potential applications.

Materials and Methods

Sample preparation

Dried seaweed *(Kappaphycus alvarezii)* was obtained from Semporna, Sabah Malaysia. The seaweed was washed under running water to remove debris and salt before being soaked. The seaweed edible film was processed according to the steps outlined in patent pending invention titled: Biodegradable Food Film From Seaweed And Process For Producing The Same (PI2013003883). Films were prepared using solvent casting method by casting film forming solution on the fabricated casting plates and subsequently dried. After drying, the films were peeled off.

Characterization of edible film

Conditioning

Films were conditioned prior subjecting to permeability and mechanical tests according to the Standard Method D618-61 (ASTM, 1993). Films were conditioned at 53% RH and 27 ±2°C by placing them in desiccators over a saturated solution of $Mg(NO_3)_2.6H_2O$ for at least 72 hours. These films were used for water vapour permeability test (WVP), oxygen permeability test (OP), Tensile Strength (TS) and Elongation At Break (EAB). For other tests, films were transferred to plastic bags after peeling and placed in desiccators.

Measurements of thickness

A digital micrometer (Model DM 3025; Digital Micrometers Ltd., Sheffield UK) was used to measure the film thickness to the nearest 0.001 mm. Thickness of each film was measured at room temperature (23°C and 45% RH) and expressed as an average of 10 random measurements.

Measurements of colour

Colour was measured using a Chroma Meter CR10 (Minolta Camera Co. Ltd., Osaka Japan) based on the CIE $L^*a^*b^*$ colour system, where L^* describes lightness (ranging from black to white), a^* and b^* describe the chromatic coordinates (ranging from –a: greenness, –b: blueness, +a: redness, +b: yellowness). Film specimens were placed on the surface of a white standard plate (L^*=92.1, a^*=2.1, b^*=-9.2. A mean value of 10 measurements was reported for each color attribute. Colour differences (ΔE) was calculated by the following equation:

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

Where L^*, a^* and b^* are the colour parameter values of the standard and L, a and b are colour parameter values of the sample.

Measurements of transparency

The light barrier properties of the films were measured by exposing the films to light absorption at wavelength 600 nm. Film transparency was measured according to the method of Bao *et al.* (2009) by placing rectangular film samples into a spectrophotometer test cell directly. This method was modified from the ASTM method D1746, which is the standard test method for transparency of plastic sheeting (ASTM, 2009). Absorbance was recorded using an UV/Vis Spectrophotometer (Optizen 2120UV, Korea). The transparency (T) of films was calculated according to the following equation:

$$T = A_{600}/x$$

where A_{600} is the absorbance at 600 nm and x is the film thickness (mm). According to this equation, a higher value of T would indicate a lower degree of transparency. Tests were run in triplicates for each type of film.

Solubility of edible films

The solubility of the films was determined according to method by Romero-Bastida *et al.* (2005) with minor modification. The film solubility (S%) expresses the percentage of film's dry matter solubilized after immersion of pre-weighted films (2 x 3 cm) in 80 ml of deionized water for 30 minutes at 25°C with constant agitation. The remained pieces of film after immersion filtered using filter paper (Whatman No 1) were dried at 60°C in an oven to constant weight. Film solubility (S%) was calculated from the following equation:

Solubility (%) = (Initial dry weight – Final dry weight) / Initial dry weight x 100

Measurements of mechanical properties

The procedure used to prepare the films for testing of mechanical properties was adapted from ASTM standard test method for tensile properties of thin plastic sheeting (ASTM, 2001). Film samples were cut into 120 mm x 10 mm dumb bell strips and thickness of the individual specimens was determined as a means of five measurements taken over the gage length. It was then used to estimate the cross-sectional area. The tensile properties were determined using a TA-HDi Texture Analyzer (Stable Micro Systems Ltd. Surrey, England) with a load cell of 25 kg and crosshead speed of 1.0 mm/sec. Film samples were clamped between grips, leaving an initial distance between the grips of 75 mm. Tensile Strength (MPa) was calculated by dividing the maximum load (N) necessary to pull the sample film apart by the cross-sectional area (m^2). Percentage of elongation at break was calculated by dividing film elongation at the moment of rupture by the initial grip length of samples multiplied by 100%. A total of 10 samples were tested for each film type.

Measurements of water vapour permeability (WVP)

Water vapor permeability values (WVP) were measured using a modified ASTM method (ASTM, 1989) reported by Gontard *et al.* (1992). The film was sealed in a glass permeation cup containing silica gel (0% RH) with silicone vacuum grease and an “O” ring to hold the film in place. Silica gels were heated at 180°C for at least 3 h prior to use for the determination. The cups were placed in desiccators with distilled water at 30°C. The cups were weighed at intervals of 1 h over a 12 h period and WVP ($g{\bullet}m^{-1}{\bullet}\ s^{-1}{\bullet}Pa^{-1}$) of the film was calculated as follows: WVP=(w• x)/A • t • (P2–P1) (McHugh *et al.,* 1993), where w is the weight gain of the cup (g), x is the film thickness (mm), A is the area of exposed films (m^2), t is the time of gain (s), and (P2–P1) is the vapor pressure differential across the film (Pa).

Measurement of oxygen permeability

The oxygen transmission rates of films were determined using ASTM Standard Method D3985-05 (2010) with some modification. Mocon Oxtrans 2/21 at 0% RH and 23^oC was tested on duplicated samples. The oxygen permeability was calculated by dividing oxygen transmission rate by the oxygen pressure and multiplying by the mean thickness.

Surface morphology

Film samples were examined for surface characteristics using a JEOL JSM-6400 scanning electron microscope (JOEL Ltd., Tokyo, Japan) operated at 15 kV. Five samples were mounted on a bronze stub and sputter-coated (Sputter Coater Bal-tec SCD 005, Liechtenstein) with a layer of gold prior to imaging in order to increase their electrical conductivity. Images were registered at 500 x magnification.

Results and Discussion

Characterization of edible film

The physical and mechanical properties of the edible film are shown in Table 1. The film was easily removed from the fabricated casting plate. The developed film was transparent with the thickness of 0.076 mm. The colour of the film is an important attribute which influences its appearance, marketability and their suitability for various applications. Clear edible films are typically desirable (Sivarooban *et al*., 2008). Visually, the edible film had a slightly yellow appearance with total colour difference (ΔE) value of 4.84.

Transparency is one of the common optical properties of light permeable materials. Spectrophotometer is used to measure the transparency of a material by light-transmittance or absorbance. Development of transparent packaging materials which allow product visibility is a general trend and requirement in packaging films. The edible seaweed film has a transparency of 4.94 indicating that the film was fairly transparent. Data obtained in this study indicated that the transparency of seaweed film was relatively close to synthetic polymer films, polyvinylidene chloride (PVDC) (Yusuke *et al*., 2004) with transparency of 4.58, thus the film are clear enough to be used as see-through packaging material.

Solubility is an important property for biodegradable and edible film applications. Film solubility can be view as a measure of the water resistance and integrity of a film (Rhim *et al*., 2000). Soluble film packaging is convenient to use in ready to eat foods as they dissolved in boiled water or in the consumer’s mouth. 66.08% of the developed edible film was dissolved in the tested conditions. This reading was slightly higher than results for methycellulose films (Turhan and Sahbaz, 2004), methycellulose-chitosan films (Pinotti *et al*., 2007) and rice-chitosan films (Bourtoom and Chinnan,

Table 1. Properties of edible films

Thickness	0.076mm
Total colour difference (ΔE)	4.84
Transparency	4.94
Solubility	66.08%
Tensile strength	6.82 MPa
Elongation at break	17.90%
Water vapour permeability	34.76 g.mm/m^2.day.kPa
Oxygen permeability	18.54 cm^{3}µm/m^{2}daykPa

2008).

Biopolymer materials, such as edible films may be subjected to various kinds of stress during use. Analyzing the mechanical properties of an edible film is relevant in order to predict its behavior when it has been applied to a food product. Tensile strength and elongation at break are very useful parameters for describing the mechanical properties of a film, and are closely related with its internal structure (Mc Hugh and Krochta, 1994). Tensile strength is the maximum tensile stress sustained by the sample during the tension test. If maximum tensile stress occurs at either the yield point or the breaking point, it is designated tensile strength at yield or at break respectively. Elongation at break is an indication of a film's flexibility and stretch ability (extensibility). This is determined as the point when the film break under tensile testing. It is express as the percentage of change of the original length of the specimen between the grips used to stretch the film (Gontard *et al.*, 1992). The studied edible had tensile strength and elongation at break values of 6.82 MPa and 17.90% respectively.

The deterioration of food in packages depends on water transfer between the internal products and the surroundings. The water barrier characteristics of the packages are thus required (Natcharee and Sudip, 2011). In this study, the edible seaweed film had water vapour permeability of 34.76 g.mm/m^2.day.kPa which was much lower than apple puree edible film with reading of 168.96 g.mm/m^2.day.kPa (Maria *et al.*, 2006).

Oxygen is the key factor that might cause oxidation, which initialed several food changes such as odour, colour, flavor and nutrients deterioration (Rungsinee and Natcharee, 2007). Thus, obtaining film with substantial oxygen barrier can help maintaining and extending food shelf life. The seaweed edible film had oxygen permeability rate of 18.54 cm^{3}µm/m^{2}day.kPa which showed excellent barrier properties when comparing with wheat gluten films (24.26-39.67 cm^{3}µm/m^{2}day.kPa) (Patricia and Carlos, 2003) and zein films (13.0-44.9 cm^{3}µm/m^{2}day.kPa) developed by Park and Chinnan (1990).

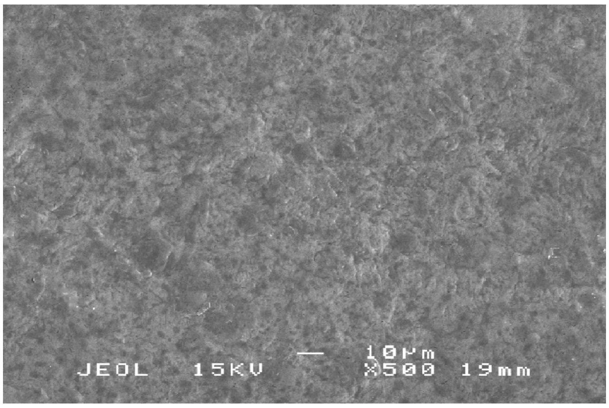

Figure 1. Scanning electron microscopy image (x 500) of seaweed edible film

The edible films should be uniform and free from defects for their functionalities. During the film-forming process, shrinkage of the films due to evaporation of water or rapid drying often causes defects such as cracks or curling in the films (Obara and McGinity, 1995). A scanning electron microscopy (SEM) was used to determine the surface morphology of edible seaweed film. It was observed that the film showed homogeneous, uniform and continuous surface without cracks and porous structure (Figure 1). The homogeneous matrix of film is a good indicator of its structure integrity, and consequently good mechanical properties would be expected (Mali *et al.*, 2002).

Potential areas of applications

The seaweed edible film showed the possible uses in various industries including food industry, pharmaceutical industry, cosmetic and toiletries industries and also agricultural industry.

Food industry

Generally, powdered soup, dried vegetables and other ingredients of precooked noodles and cup noodles, or instant coffee are packaged to maintain their qualities with either aluminum laminated plastic or various plastic films. These packages must be torn to remove the contents prior to cooking or pouring hot water over it. This is not only troublesome, but also caused the packages to end up in the land-fills, where they can be last forever and never degradable. One solution to these problems is to package such products with an edible film. The package can be dissolve by cooking or simply pouring hot water over it, thus making it unnecessary to tear the package and the material can be eaten together with


Sachet — instant drinks
Sachet --- seasoning powder
Sachet — sugar
Wrapper — cheese
Wrapper --- seasoning cube
Pouch — candies
Wrapper — Malay traditional kuih
Interleaf --- frozen foods
Edible logo
Facial mask
Functional strips

Figure 2. Application of edible films

the contents. In addition, this film can also be used as a wrapper for Malay's traditional cakes such as "dodol", "kuih ketayap" and "popiah"; cheese slices and seasoning cube. This film can also be used as interleaf between foods to avoid it from sticking to each other, especially for frozen food such as burger patties and instant "roti canai". The film can be used for printing of edible logo for decoration of cakes and other bakery products (Figure 2).

Pharmaceutical industry

The market for flavored film strips has growth potential, especially in the breath film segment. Incorporating active ingredients opens the door for expansion into new segments such as films with health benefits. Active ingredients can be incorporated directly into the solution prior to the film being cast. These active ingredients become locked into the film matrix and remain stable until consumption. Examples of active ingredients used in film strips include ingredients for oral hygiene, caffeine for alertness, nutrients and botanicals. The film also can be used to produce drugs/medicines strips, a water soluble edible film that can hold active pharmaceutical ingredients. It can be taken without water which is highly recommended for the people who have difficulty in swallowing such as elderly and little children. There is no worry of blocking the airway (Figure 2).

Cosmetic/toiletries industry

The edible film can be used to form facial masks and bags for pre-portioned washing powder where there are no packaging materials need to be dispose as the films will dissolve completely in water (Figure 2).

Agricultural industry

The edible films can be formed into bags for pre-portioned fertilizer mainly targeted for used in home gardening. A small bag of fertilizer can be applied to each pot where the fertilizer will dissolve slowly when in contact with moist soils or during watering.

Conclusion

This study has demonstrated the feasibility of using seaweed directly to produce edible film instead of using extracts of seaweed (agar, carrageenan or alginate). The developed film was transparent, flexible, sealable, dissolvable and had substantial mechanical strength to withstand stress during handling. The film could be used in food industry, pharmaceutical industry, cosmetics and toiletries industries as well as agricultural industry. The development of this edible seaweed based film is a promising method for diversifying and value adding the usage of seaweed and at the same time reducing food packaging waste.

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