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PHYSICAL PROPERTIES OF SODIUM CASEINATE EDIBLE FILMS AND ITS APPLICATION AS COATING ON BREAD BUNS

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By

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PHYSICAL PROPERTIES OF SODIUM CASEINATE EDIBLE FILMS AND ITS APPLICATION AS COATING ON BREAD BUNS

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Chairman: Professor Russly A. Rahman, PhD
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Breads and buns are known to have relatively short shelf life, and staling process is the major reason for such deterioration. Packaging, which serves as a protective layer to food, is able to retard the staling process. Non-biodegradable petroleum based synthetic packaging is among the commonly used materials for food packaging due to its good mechanical, moisture, gas and aroma barrier properties. However, the rise of non-biodegradable packaging waste in an alarming rate has led to a paradigm shift from synthetic packaging to biodegradable packaging. Therefore, various natural resources have been used for edible films and coatings development. The main purpose of the study was to establish an effective edible packaging system developed from sodium caseinate to prolong the shelf life of buns. Edible films consisted of various level of sodium caseinate (6%, 7% and 8% (w/w)), sorbitol (1:0.25, 1:0.5, and 1:0.75 (sodium caseinate: sorbitol)) and palm olein (1:0.0, 1:0.5, and 1:0.75 (sodium caseinate: palm olein)) were prepared in emulsion form and were casted on disposable petri dishes and dried in the oven for 24 hours. Dried films were kept under controlled environment for at least 48 hours before various tests. Barrier properties of films were tested according to ASTM Standard Method D3985 and ASTM Standard Method E96. For mechanical properties, it was tested based on the ASTM Standard Method D882, and film opacity was measured according to the BSI standard. Emulsion with optimum criteria was selected and applied on buns as coating. Uncoated, coated and cling film wrapped buns quality and shelf life were tested and compared. Results showed that, concentration of sodium caseinate, sorbitol and palm oil had significantly affected ($p<0.05$) the physical properties of the edible films. However, the concentration of sodium caseinate did not significantly affect ($p>0.05$) tensile strength of the film and concentration of palm oil did not significantly affect ($p>0.05$) elongation properties of the film. Overall, sodium caseinate edible films showed better oxygen barrier property but weaker water barrier property than cling film. However, as the content of lipid increased, there was a decrease in the water vapour permeability. For mechanical properties, cling film generally showed better tensile strength and elongation, but some of the edible films showed comparable stiffness (Young's modulus) with cling film. Besides that, edible films without lipid showed comparable transparency with the cling film. As the protein and lipid content

increased, the films showed higher opacity. However, the increased in plasticizer content, resulted in less opaque film. As for the application of coating on buns, coated buns and cling film wrapped buns had longer shelf life than uncoated buns. However, coated buns shelf life was one day shorter than cling film wrapped buns. For texture, although cling film wrapped buns and coated buns did not show better overall quality, however, due to the excellent gas barrier properties, it prolonged the buns shelf life. In conclusion, although application of coating prolonged the buns shelf life, however coated buns may become unacceptable due to the deterioration in eating quality. Therefore, modifications need to be done to improve the physical properties of the edible film.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

SIFAT- SIFAT FIZIKAL FILEM NATRIUM KASEINAT BOLEH DIMAKAN DAN APPLIKASINYA SEBAGAI SALUTAN ROTI BAN

Oleh

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Roti dan ban diketahui mempunyai tempoh simpanan yang singkat, dan proses *staling* merupakan punca utama degradasi roti dan ban. Pembungkusan makanan memainkan peranan sebagai lapisan perlindungan bagi makanan, dan dipercayai dapat memperlahankan proses *staling*. Pembungkusan sintetik petroleum antara bahan pembungkusan yang sering digunakan dalam industri pembungkusan makanan. Ini kerana bungkusan berlandaskan petroleum mempunyai ciri-ciri mekanikal, rintangan kelembapan, gas, dan aroma yang baik. Namun begitu, pembungkusan sintetik petroleum merupakan bahan pembungkusan yang tidak dapat dibiodegradasikan. Penambahan sisa buangan pembungkusan tidak terbiodegradasi dalam kadar yang merisaukan telah membawa kepada anjakan paradigma dari penggunaan bungkusan sintetik ke bungkusan mesra alam. Oleh yang demikian, pelbagai sumber semula jadi telah digunakan dalam pembangunan filem-filem dan salutan boleh dimakan. Tujuan utama kajian ini adalah untuk mewujudkan satu sistem pembungkusan yang efektif dengan menggunakan natrium kaseinat untuk memanjangkan hayat penyimpanan roti ban. Filem-filem boleh dimakan yang diperbuat daripada kombinasi natrium kaseinat (6%, 7% dan 8% (w/w), sorbitol (1:0.25, 1:0.5 dan 1:0.75 (natrium kaseinat: sorbitol)), dan olein sawit (1:0.0, 1:0.5 dan 1:0.75 (natrium kaseinat: olein sawit)) pada pelbagai tahap disediakan dalam bentuk emulsi. Emulsi kemudian dituangkan ke dalam piring petri dan dikeringkan dalam ketuhar selama 24 jam. Filem yang telah dikeringkan disimpan dalam persekitaran terkawal untuk sekurang-kurangnya 48 jam sebelum digunakan untuk analisis seterusnya. Ciri-ciri rintangan filem telah diuji menggunakan *ASTM Standard Method D3985* dan *ASTM Standard Method E96*. Bagi ciri-ciri mekanikal, filem-filem telah diuji menurut *ASTM Standard Method D882*, manakala kelegapan filem telah diuji mengikuti standard *BSI*. Formulasi yang mempunyai kriteria optimum dipilih dan disadurkan di atas ban sebagai salutan. Ujikaji dan perbandingan dibuat untuk membandingkan kualiti dan hayat simpanan di antara roti ban tanpa salutan, roti ban yang bersalutan emulsi, dan roti ban yang dibungkus dengan filem berpaut. Keputusan menunjukkan bahawa, kandungan natrium kaseinat, sorbitol dan olein sawit memberi kesan signifikan ($p<0.05$) kepada sifat-sifat fizikal filem-filem boleh dimakan. Namun begitu, kandungan minyak kelapa sawit tidak memberi sebarang kesan signifikan ($p>0.05$) kepada pemanjangan filem

boleh dimakan. Secara keseluruhannya, filem boleh dimakan natrium kaseinat menunjukkan sifat rintangan oksigen yang baik, tetapi rintangan kelembapan yang lemah. Namun begitu, kadar telapan air berkurang apabila kandungan minyak bertambah. Bagi ciri-ciri mekanikal, filem berpaut mempunyai ketegangan dan pemanjangan yang lebih baik secara keseluruhannya. Namun, ada juga filem boleh dimakan yang menunjukkan kekakuan yang serasi dengan filem berpaut. Secara keseluruhan, filem boleh dimakan tanpa minyak menunjukkan kelegapan yang serasi dengan filem berpaut. Namun demikian, apabila kandungan protein dan minyak bertambah, filem yang terbentuk menjadi semakin legap, tetapi kelegapan filem berkurangan apabila kandungan *plasticizer* bertambah. Dalam aspek aplikasi sebagai salutan ban, didapati bahawa ban yang bersalutan dan ban yang dibalut dengan filem lekap dapat melanjutkan hayat simpanan berbanding dengan ban yang tidak bersalutan. Namun begitu, ban bersalutan mempunyai hayat simpanan satu hari lebih pendek daripada ban dibalut dengan filem lekap. Walaupun ban bersalutan dan ban dibalut dengan filem lekap tidak menunjukkan kualiti yang lebih baik secara keseluruhan, namun ciri-ciri rintangan gas yang baik filem yang boleh dimakan dapat membantu dalam memanjangkan hayat simpanan roti. Kesimpulannya, walaupun aplikasi salutan dapat memanjangkan hayat simpanan roti, namun kemerosotan kualiti makan roti ban tidak diterima oleh kalangan pengguna dan mengakibatkan pembaziran makanan. Oleh demikian, modifikasi diperlukan untuk mempertingkatkan kriteria filem.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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This is to confirm that:

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CHAPTER 1

INTRODUCTION

Packaging has been used as a tool to provide moisture barrier, gases barrier and mechanical protection to food. It also serves as a communication channel to provide product information to consumer. Various materials such as plastics, paper, metal and glass have been used in food packaging production (Lee, Yam, & Piergiovanni, 2008). Petroleum based synthetic polymer films are one of the commonly used packaging materials due to abundant availability at low cost, and good mechanical and barrier properties. Gourmelon (2015) mentioned that, plastic production has been growing and it is used to replace materials such as glass and metals. As most of the food packaging are meant for one-time usage, they will be removed and discarded once the food is consumed. Hence, the increase usage of plastics packaging in food industry has been associated as part of the reasons that contributed to drastic increase in non-degradable waste, which resulted in environment pollution. Therefore, the increase in environment consciousness among the consumers have led to a paradigm shift from non-biodegradable packaging material to biodegradable and edible packaging.

Edible packaging is made from natural resources such as plant and animal proteins, polysaccharides, gums, plasticizers, lipid and resins (Khwaldia, Perez, Banon, Desobry, & Hardy, 2004; Krochta, 2002; Robertson, 2006). These packaging are found to have similar functions as synthetic packaging. They provide moisture, oxygen and aroma barrier for various foods, yet they are biodegradable. This has become a solution for the waste disposal issues that are found with the synthetic packaging. Edible films and coating is no longer a new technique to prolong food shelf life, it has been implemented since many years ago in food industry. One of the examples is the application of Carnauba wax on fruits to preserve the freshness. Beeswax and whey protein isolates are among the popular ingredients that have been used in the previous studies (Shellhammer & Krochta, 1997; Pérez-Gago & Krochta, 2002). This is due to the good moisture barrier properties of the films and the property of the whey protein isolate to form edible film. Besides that, there were also studies carried out by using essential oil, such as oregano, rosemary oil and garlic essential oil to form edible films (Seydim & Sarikus, 2006) to enhance the antimicrobial properties of the edible films. However, these materials are expensive for mass production and may not be cost effective for the manufacturers. According to Malaysia Palm Oil Council (2012), Malaysia is one of the world major palm oil producer and exporter. Currently, Malaysia hold the account of 39% of world palm oil production and 44% of world exports. Even though there is abundance of palm olein available in Malaysia, and obtainable at lower price, however there was lack of studies of edible films and coating using palm olein as lipid in the system. Therefore, in this study modification of the sodium caseinate edible system was done using palm olein, so that edible films and coating systems can be implement in the food industry in order to reduce food packaging material waste.

Among the natural ingredients used for edible films and coating development, proteins and polysaccharides are the major components for film and coating formation. However, compared to polysaccharides, particularly milk proteins possess more interesting

mechanical and barrier properties and they are highly nutritious, and highly soluble in water. Furthermore, milk proteins can act as effective emulsifier in film formation (Khwaldia et al., 2004).

Although milk proteins are known as good film formers with excellent oxygen, lipid and aroma barrier at low relative humidity, but the hydrophilic nature of proteins structure results in poor moisture barrier characteristics. Therefore, lipid has been added to enhance the moisture barrier property (Kokoszka, Debeaufort, Lenart, & Voilley, 2010). Furthermore, the addition of lipid also provides gloss and enhance the visual appearance of the food products (McHugh, 2000). Although adding lipid can enhance the oxygen and moisture barrier of films, it increases the films brittleness as well. Besides that, milk protein edible films also possess brittle and stiff nature. Hence, plasticizer plays an important role in regulating the structure and the flexibility of the films (Chick & Ustunol, 1998; Chen, 2002). The structure of the film is disrupted by the plasticizers due to its molecule sizes are small enough to interrupt more hydrogen bonds, resulting in lower tensile strength of films (Chick & Ustunol, 1998; Chen, 2002). However, the composition of proteins, plasticizer and lipids are closely related, therefore, proportion of each of the ingredients should be properly adjusted to produce an optimum film and coating system with optimum barrier properties and mechanical properties.

Consumer acceptability and recognition towards bread is determined by the fresh flavour of the bread (Heenan, Hamid, Dufour, & Delahunty, 2009). Freshly baked breads and buns are usually associated with key sensory attributes such as crispy crust, soft crumb, and appealing aroma. However, as soon as the baked products are out from oven, some physical and chemical changes take place and subsequently result in sensory changes. This deterioration process is known as staling process. Chin, Abdullah and Yusof (2011) stated that, staling process is the major factor that contributes to the quality deterioration in baked goods other than the microbial growth. The sensory changes that take place upon staling process include increase in crumb firmness, reduce in crust crispiness, increase in crumbliness, loss of fragrance or aroma, and assume a stale flavour (Schiraldi & Fessas, 2001; Cauvain, 1998). These changes led to the unacceptable sensory characteristics by consumers, and subsequently create unnecessary food waste.

The quality deterioration of baked goods was mainly due to the storage condition, temperature, and packaging (Cauvain, 1998). These factors not only affect the rate of staling process but the shelf life as well. Shelf life of food products is referring to the duration, at which the food products retain an acceptable level eating quality from the safety and organoleptic point of view (Galić, Ćurić, & Gabrić, 2009). It is closely related to the formulation, processing methods, packaging, and storage condition. Other than that, moisture content, pH, addition of microbial preservatives and antioxidant of the bread also affect the shelf life of bread (Galić et al., 2009).

Breads are commonly wrapped by using polyethylene plastics, which are non-biodegradable. Although these materials can be recycled, however they are usually being disposed instead. The current practice in local bakeries is that most of the freshly baked goods are kept on the shelf until they are purchased by consumers. As the bakery products are exposed to oxygen and without proper packaging or coating, it results in

accelerating staling process and microbial growth. Packaging which provides barriers to moisture, oxygen, lipid, and aroma, is able to protect food from microbial contamination, quality deterioration, and provides mechanical protection to food during the handling process. Furthermore, it acts as the communication channel to provide important information such as nutritional information, shelf life of the food products and ingredients to the consumers (Han, 2005; Krochta, 2002; Kelsey, 1985). Some studies have been carried out to develop effective edible film and coating systems to provide alternative solutions to prolong and protect the food shelf life (Doulia, Katsinis, & Mougin, 2000; Galić et al., 2009). However, most of those studies were regarding the application of coating on meat, fruits, and vegetables. Besides that, most of these studies regarding edible films and coatings were focused on the mechanical properties and barrier properties of various types of films and coatings. There are still lack of studies regarding the application of films and coatings on bakery products particularly on breads and buns. The general objective of this study is to apply the best coating formulation for shelf life extension of bread bun.

The specific objectives of this thesis are:

1- To develop edible films and coating using different combination of concentration levels of sodium caseinate, sorbitol and palm olein;
2- To determine mechanical, barrier and optical properties of sodium caseinate edible films;
3- To evaluate the quality of bread buns coated with the developed film.

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