Strategies for Evaluation of Edible Coating Formulations - Applications on Food Quality and Safety

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Abstract. Edible coatings can be used to improve shelf-life and food quality by providing effective and selective barriers to moisture transfer, oxygen uptake, better visual aspect, and reduction of microbiological contamination. Wettability defines the ability of a coating to adhere to a food’s surface and is determined through the values of the spreading coefficient. Other factors, such as gas barrier properties (permeabilities), opacity and mechanical properties must also be considered in order to ensure the adequate behavior of the coatings aiming at the improvement of food shelf-life and quality. The effectiveness of edible coatings depends, in a first stage, on the control of the wettability of the coating in order to ensure a uniformly coated surface, and in a second stage on other factors (water vapour permeability; oxygen and carbon dioxide permeability; opacity; mechanical properties) that can also affect the effectiveness of the coating and that strongly depend on the food surface that will be coated. A methodology for evaluating edible coating formulations has been developed by our group which is applicable to coatings for fruits, vegetables, cheese and fish (Cerqueira et al., 2009, 2010; Lima et al., 2010; Martins et al., 2010; Souza et al., 2010). In all cases different formulations of edible coatings (polysaccharides, proteins, plasticizers, surfactants and lipids) were evaluated through the measurement of the wettability on food surface. Then the coating compositions with the best values of wettability were selected and the barrier, colour and mechanical properties were evaluated in order to select a unique coating composition. The results have shown that the final composition allows in all cases a good coating performance with a good coating adhesion, confirmed by shelf-life evaluating of food.

This methodology allows testing a great number of formulations and its application has generated an important amount of data on the use of edible coatings on several foods, particularly in order to extend their shelf-life and quality.

Keywords. Edible coatings, edible films, wettability, shelf-life, food quality, food safety.
Introduction

Natural polymers can be an alternative source for packaging development due to their edibility and biodegradability (Siracusa, Rocculi, Romani & Rosa, 2008). Edible coatings and films appear as alternative for synthetic plastic for food applications. Their use based on natural polymers and food grade additives has been constantly increasing in the food industry. The coatings/films can be produced with a great variety of products such as polysaccharides, proteins and lipids, with the addition of plasticizers and surfactants. The two terms, coatings/films, are used in food packaging, sometimes without any distinction. However, the “film” is a thin layer (e.g. formed by casting of the biopolymer solution) prepared separately from the food that is later applied to it, while the coating can be formed by direct application of a suspension or an emulsion on the surface of the food, leading to the subsequent formation of a film (Krochta, 2002). The functionality and performance of edible films mainly depend on their barrier, mechanical and colour properties, which in turn depend on film composition and its formation process. In the case of the edible coatings, the method of application on the product, and the capacity of the coating to adhere to the surface are the most important parameters. Food products are usually coated by dipping or spraying, forming a thin film on the food surface that acts as a semi-permeable membrane, which can control the moisture loss or/and suppress the gas transfer (Lin & Zhao, 2007).

Edible coatings application on food products is evaluated through quality parameters of the coated products that are measured as indicators (e.g. water loss, respiration rate and colour). This evaluation provides information on the functionality of the coatings in the food product (Lin & Zhao, 2007). Due to the difficulties in evaluating the barrier and mechanical properties of the coatings once formed on the food product, such evaluation is often made on the corresponding film. To do that, films are cast and their properties are measured in order to mimic as closely as possible their properties when applied on the food product. In the last years a great number of works appear with the application of edible coatings on food products. Only in a few cases the choice for a given edible coating formulation has been justified.

Materials used in edible coatings

A great diversity of materials is used to produce edible coatings and films, but most of them can be included in one of three categories: polysaccharides, proteins and lipids. The great diversity of structural features of polysaccharides have origin from differences in the monosaccharide composition, linkage types and patterns, chain shapes, and degree of polymerization, influencing their physical properties. Polysaccharides, which are commercially available for use in food and nonfood industries as stabilizers, thickening and gelling agents, crystalization inhibitors, and encapsulating agents, etc (Stephen & Churms, 2006). The main polysaccharides used in edible coatings/films are chitosan, starch, alginate, carragennan, modified cellulose, pectin, pullulan, chitosan, gellan gum, xanthan gum, etc (Han & Gennadios, 2005). Proteins cover a broad range of polymeric compounds that provide structure and biological activity. They are heteropolymers comprised of more than 20 different amino acids, and have specific sequences and structures (Nelson & Cox, 2000).

Protein film-forming materials derived from animal sources include collagen, gelatin, fish myofibrillar protein, keratin, egg white protein, casein, and whey protein (Han et al., 2005). Due their apolar nature and hydrophobic behaviour, lipids are of great importance since they can be used as a barrier against moisture migration, thus covering the drawback of polysaccharides and proteins in terms of their great water sensitivity. Some of the films based on lipids present
water permeability values close to synthetic plastic films, but they are in most cases very brittle when used alone, and they should be blended e.g. with polysaccharides or proteins in order to improve their mechanical properties (Morillon, Debeaufort, Blond, Martine & Voilley, 2002).

Coatings based on polysaccharides and proteins require in the most cases the presence of a plasticizer and/or a surfactant. Films without plasticizer present a brittle and stiff structure due to the extensive interactions between polymer molecules. Most plasticizers are very hydrophilic and hygroscopic and they can attract water molecules; furthermore, plasticizers disrupt molecular hydrogen bonds, increasing the distance between polymer molecules, and reduce the proportion of crystalline to amorphous regions (Rivero, García & Pinnoti, 2010). Surfactants are amphoteric substances due to their simultaneous hydrophilic and hydrophobic properties. They are conventionally added to enhance the stability of the emulsion in the formulae of emulsified films. They can be incorporated into coatings formulations to reduce the surface tension of the solution, improving its wettability (Choi, Park, Ahn, Lee & Lee, 2002; Ribeiro, Vicente, Teixeira & Miranda, 2007).

**Edible coatings properties**

When referring to a coating’s performance, wettability can be used as a measurement of its effectiveness to coat a food surface. Undoubtedly, the success of edible coatings depends very strongly on the control of the wettability of the coating solutions. They must wet and spread on the surface of the food product, and upon drying they must form a film that has the adequate properties and durability. The coating process involves not only wetting of the food product by the coating solution, but also some degree of penetration of the solution into the product (Lin et al., 2007). The theoretical background of the wettability measurement was discussed e.g. by Choi et al. (2002) and Ribeiro et al. (2007) (Choi et al., 2002; Ribeiro et al., 2007). Briefly, the wettability of a solid by a liquid is determined by the balance between adhesive forces of the liquid on the solid and cohesive forces of the liquid, where adhesive forces cause the liquid to spread over the solid surface while cohesive forces cause it to shrink. This balance is expressed in the form of a spreading coefficient ($W_s$). In practical terms, the closer the $W_s$ values are to zero, the better a surface will be coated.

Also the surface energy or surface tension of the food product is a controlling factor in the process that involves wetting and coating of surfaces (Karbowiak, Debeaufort & Voilley, 2006). The determination of the surface tension usually involves the measurement of the contact angles that several standard liquids make with that surface. The surface energy of the solid surface is then related to the surface tensions of the liquids and the corresponding contact angles. This method invokes an estimation of the critical surface tension of the surface of the solids studied, by extrapolation from the Zisman plot (Zisman, 1964). The characterization of food surface properties (such as the surface tension and critical surface tension) is recognized as the key to understand mechanisms of wetting and spreading (Karbowiak et al., 2006). Food surfaces are very heterogeneous; the same fruit can have different surfaces characteristics. The food surface tension of several fruits, fish and cheeses has been characterized in the last years, and it has been shown that depending of the kind of food there will be different values of that parameter. Table 1 summarizes some of the values of critical surface tension and surface tension (including its polar and dispersive components) from different food products analyzed by our group.
Table 1. Values of critical tension, surface tension (including its dispersive and polar components) of food products.

<table>
<thead>
<tr>
<th>Food</th>
<th>Critical tension (mN/m)</th>
<th>Surface tension (mN/m)</th>
<th>Dispersive component (mN/m)</th>
<th>Polar component (mN/m)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strawberry</td>
<td>18.8</td>
<td>28.94</td>
<td>22.99</td>
<td>5.95</td>
<td>(Ribeiro et al., 2007)</td>
</tr>
<tr>
<td>Mango</td>
<td>19.5</td>
<td>26.48</td>
<td>24.77</td>
<td>1.71</td>
<td>(Lima et al., 2010)</td>
</tr>
<tr>
<td>Apple</td>
<td>25.4</td>
<td>27.81</td>
<td>27.13</td>
<td>0.68</td>
<td>(Lima et al., 2010)</td>
</tr>
<tr>
<td>Fish (Salmon salar)</td>
<td>30.13</td>
<td>60.64</td>
<td>18.18</td>
<td>42.46</td>
<td>(Souza et al., 2010)</td>
</tr>
<tr>
<td>Semi-hard Cheese</td>
<td>18.33</td>
<td>37.79</td>
<td>29.93</td>
<td>7.87</td>
<td>(Cerqueira et al., 2009a)</td>
</tr>
</tbody>
</table>

Applications

In order to select edible coatings to be applied on food products our group has chosen a two-step criterion: in a first stage, to evaluate wettability values of the coating formulations (the coatings with the best values are selected); in a second stage, to determine the values of other relevant properties (e.g. high or low water vapour, oxygen or carbon dioxide permeabilities, good mechanical resistance, etc.) depending on the kind of food and on the desired effects. This allows refining the selection made in stage one and ending eventually with the best formulation for a given application. In 2007, Ribeiro et al. tested the ability of polysaccharide coatings of starch, carrageenan and chitosan to extend the shelf-life of strawberry fruit (*Fragaria ananassa*). The coatings and strawberries were characterized in terms of their physical properties (wettability and then oxygen permeability) in order to optimize coating composition. In both cases, to enhance the wettability of the coating solutions, Tween 80 was added, reducing the superficial tension of the liquid and thus increasing the spreading coefficient ($W_s$). The effects of application of these coatings to fresh strawberries were assessed by determining colour change, firmness, weight loss, soluble solids and microbiological growth. The results show that the edible coating leads to the improvement of strawberries firmness loss, to the decrease of mass loss and rate of microbial growth.

In 2009b, Cerqueira et al., studied the utilization of coatings composed of galactomannans from two different sources (*Caesalpinia pulcherrima* and *Adenanthera pavonina*) and glycerol for application on five tropical fruits: acerola (*Malpighia emarginata*), cajá (*Spondias lutea*), mango (*Mangifera indica*), pitanga (*Eugenia uniflora*) and seriguela (*Spondias purpurea*). The wettability was determined using different aqueous galactomannan solutions (0.5 %, 1.0 % and 1.5 %) with glycerol (1.0 %, 1.5 % and 2.0 %). For the solutions having the best (closest to zero) wettability values, films were cast and water vapour permeability, oxygen permeability, carbon dioxide permeability, tensile strength and elongation-at-break were determined. Taking into account the surface and permeability properties of the obtained films, four compositions were selected as the best coatings to apply on the studied fruits. Also Lima et al. (2010) studied the application of coatings based on mixtures of polysaccharide (galactomannan from *C. pulcherrima* and *A. pavonina*), collagen and glycerol on mangoes and apples. The same methodology was used in order to choose one coating formulation for application on the mango and apple. Finally, the coatings were applied on fruits in order to determine their influence in gas transfer rates. Coated mangoes and apples present less $O_2$ consumption and less $CO_2$ production than uncoated fruits.
In 2009, Cerqueira et al. studied the application of edible coatings from three different polysaccharide sources in a semi-hard cheese. Chitosan, galactomannan from Gleditsia triacanthos, and agar from Glacilaria birdiae were tested as coatings, with different polysaccharide, plasticizer and corn oil concentrations. The surface properties of the cheese and the wetting capacity of the coatings on the cheese were determined. The three best solutions for each polysaccharide were chosen, further films were cast, and permeability to water vapour, oxygen, and carbon dioxide was determined, along with opacity. The edible coating of G. triacanthos was chosen and was applied on cheese. The O₂ consumption and CO₂ production rates of the cheese with and without coating were evaluated, showing a decrease of the respiration rates when the coating was applied. The uncoated cheese had an extensive mould growth at the surface when compared with the coated cheese. In 2010, Fajardo et al. studied the effects of the application of chitosan coating (selected in the previous work) containing natamycin on the physicochemical and microbial properties of semi-hard cheese. Microbiological analyses showed that natamycin coated samples presented a decrease on moulds/yeasts compared to control after 27 days of storage. Edible coatings of galactomannans from G. triacanthos incorporating nisin were evaluated in the shelf-life extension of Ricotta cheese against Listeria monocytogenes (Martins et al., 2010). Nine different formulations were evaluated and based on wettability only one formulation was chosen, being their transport, mechanical and color properties determined. Three different treatments were tested in cheese samples inoculated with L. monocytogenes: samples without coating, samples with coating without nisin, and samples with coating containing nisin. Results showed that the cheese coated with nisin-added galactomannan film was the treatment presenting the best results in terms of microbial growth delay. Recently, Souza et al. (2010), using the same methodology, evaluated the effect of chitosan coating on shelf life extension of salmon (Salmo salar) fillets. The selected coating formulation, based on wettability, was chosen to be applied on fish fillets. The results showed that chitosan-based coatings were effective in extending for an additional 3 days the shelf life of the salmon. These results demonstrate that chitosan-based coatings may be an alternative for extending the shelf life of salmon fillets during storage at 0 °C.

Conclusions

The methodology developed by our group has been applied to fruits, vegetables, cheese and fish with good results. Improvements in the amount of knowledge on edible films and coatings, acquired through research and product development work, as well as advances in material science and processing technology have occurred. Results suggested that the selected coating formulations can reduce gas transfer rates in fruits and cheese, and can be therefore important tools to extend food products shelf life. A good choice of the coating formulation is essential for the durability and maintenance of the coating on the food products. The determination of wettability is therefore fundamental for the correct application of edible coatings. Further work has to be performed to understand how different factors as such temperature and the application method can be important in the coating performance.

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