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Effect of pH and addition of corn oil on the properties of gelatin-based biopolymer films

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ABSTRACT

Response surface methodology (RSM) was used to investigate pH and corn oil (CO) addition on gelatin film properties. Test films were evaluated for thickness (T), tensile strength (TS), puncture strength (PT), percentage elongation at break point (E), water vapour permeability (WVP) and oxygen permeability (OP). T, TS and PT increased linearly ($P < 0.01$) with increasing CO content, but were not significantly affected by pH. pH had both linear and quadratic effects on E values ($P < 0.01$), while CO content had a lesser effect. WVP decreased as the pH of the film-forming solutions was adjusted away from pH 7.0 or CO addition level increased above 27.25%. OP was affected in both a quadratic and linear manner by pH adjustment and CO addition, respectively. Optimal film-forming conditions were pH (10.54) and CO level (55.18%, w/w), as predicted by response surface methodology. Scanning electron microscopy (SEM) was performed to investigate the microstructures of gelatin films, and confocal scanning laser microscopy (CSLM) was used to investigate fat distributions and protein phase in gelatin films.

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1. Introduction

The development of biopolymer films has increased the amount of research on edible packaging, since they consist of natural and biodegradable substances which may lead to a replacement for plastics (Gennadios and Weller, 1990; Krochta and de Mulder-Johnston, 1997). Gelatin is a natural polymer obtained from animal by-products through acid or alkaline hydrolysis. It is a high molecular weight polypeptide composed of amino acids: mainly glycine (27%), hydroxyproline and proline (25%) (Mark, 1985). Gelatin has been widely employed in the manufacture of foodstuffs (Moley, 1984; Slade and Levine, 1987; Shewfelt, 1987), sausage casings (Johnston-Banks, 1990), pharmaceuticals (Digenis et al., 1994; Johnson, 1965; Torres, 1994), photographic and other specialized industrial product applications (Jolley, 1970; Slade and Levine, 1987; Nishio and Hayashi, 1985). Due to its abundance, biodegradability, low cost and its potential to form films for use in edible/biodegradable food packaging applications, an interest in gelatin as a primary biopolymer film component has recently emerged (Menegalli et al., 1999; Bigi et al., 2002). Edible gelatin films prepared from bovine and porcine skins have been shown to have high puncture strength, low puncture deformation and high water vapour permeability (Sobral et al., 2001; Wang et al., 2007). However, some researchers have shown that gelatin film properties can be altered by modifying the gelatin structure (Carvalho and Grosso, 2004; Sarmento et al., 2000).

Conversely, films manufactured from lipids alone have suitable water vapour barrier properties but poor mechanical properties (Guilbert, 1986; Kamper and Fennema, 1984a,b). The production of composite films incorporating both gelatin and lipid components may enhance the suitability of gelatin-based edible films for greater commercial application. Furthermore, a variable such as pH of film-forming solutions may also prove to be an important factor influencing functional properties of biopolymer films (Anker et al., 2000; Banker, 1966; Banerjee and Chen, 1995; Gennadios et al., 1993a). Therefore, a gelatin-based film with optimised pH and lipid concentration could produce a film that is suitable for food packaging applications. However, from extensive review of the scientific literature, no reports could be found documenting the effects of manipulating both pH and oil content in gelatin-based biopolymer solutions for the purpose of film manufacture.

The objective of the present study was to determine the effects of pH and CO addition on mechanical and permeability properties of gelatin films using response surface methodology (RSM) in an attempt to optimise film formulation. RSM is a statistical model frequently used for the optimisation of complex systems and uses quantitative data from an appropriate experimental design to determine and simultaneously solve multivariate problems (Madamba, 2002).

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2. Materials and methods

2.1. Ingredients for film formation and reactants

The following ingredients were used for film formation: Porcine skin gelatin (Bloom 180), average molecular weight 1×10^5 Da, hydrophile–lipophile balance (HLB) value approximately 17 (Redbook Ingredient Services Ltd., Dublin, Ireland); pure corn oil HLB value 9 (Best Foods UK Ltd., Surrey, UK) and glycerol (100% purity, Cahill May Roberts Ltd., Dublin 20, Ireland). For pH adjustment, NaOH was obtained in pellet form from Lab Pak Ltd., Filongley, UK and lactic acid from Alkem Chemicals Ltd., Cork, Ireland.

2.2. Preparation of film solutions for film formation

Gelatin (20 g) powder was solubilised in 480 ml of distilled water at room temperature (23 °C). Addition of glycerol to solutions was set at a glycerol:powder ratio of 1:2 (w:w). CO was added into the above solutions and pH adjusted using lactic acid or 1 M NaOH according to Table 1 before heating the dispersions. All dispersions were stirred continuously using a magnetic stirrer hotplate for 2 h, until powders were completely dissolved. dispersions were homogenised at 480 bar (first stage at 30 bar, second stage at 450 bar) using an APV homogeniser 2000 series (APV, Alberslund, Denmark) three passes after heated to 80 °C. All dispersions were homogenous with no visible phase separation. Films were cast by pouring dispersions onto level Teflon-coated Perspex plates and dried for 24 h at 50 ± 5% relative humidity and 23 ± 2 °C. Formed films were subsequently peeled from the casting plates and held under the same conditions for a further 12 h period prior to testing.

2.3. Film thickness

Film thickness (T) was measured using a 0–25 mm micrometer screw gauge (Mitutoyo Corporation, Kawasaki, Kanagawa, Japan) with overall thickness being expressed as an average (n = 15) taken randomly from each film. Film thickness was used in calculating the tensile strength (TS), water vapour pressure (WVP) and oxygen permeability (OP) values of test films.

2.4. Film opacity

The opacity of gelatin films was tested using a spectrophotometer (DU 640, Bachman, USA), set at a wavelength of 500 nm. Five film specimens were taken from each film sample and cut into rectangular pieces 45 × 10 mm, then placed on the inner side of a transparent plastic 10 mm cuvette and the absorbance measured. The opacities of films were calculated by the following equation according to the method described by Gontard and Guilbert (1994):

$$\text{Opacity} = \text{Absorbance at } 500\text{ nm} \times \text{film thickness}$$

2.5. Film tensile strength, elongation and puncture strength

Mechanical properties of films were evaluated according to the ASTM-D882 standard test methodology (ASTM, 1985) using an Mecmesin Force and Torque Test software (Imperial 2500 Instruments, Mecmesin Ltd., Slinfold, West Sussex, England). Test film samples were cut into strips (100 × 25.4 mm) and analysed for tensile strength (TS), elongation at break point (E) and puncture strength (PT).

2.6. Film water vapour permeability

Circular water vapour permeability cups made from Perspex were manufactured to the specifications reported by McHugh et al. (1993). Distilled water (6 ml) was placed in each test cup and a film sample was mounted across the cup opening. The cups were stored under controlled temperature and humidity (50 ± 3% RH, 23 ± 2 °C). A constant air velocity of 152 m/min was maintained over the cups to ensure uniform air movement across the WVP test cells. The weight loss of the cups was monitored over a 24 h period with weights recorded at 2 h intervals. Water vapour permeability was calculated according to a modification of the ASTM E-96 standard method (ASTM, 1990) for determining WVP of synthetic packaging materials.

2.7. Film oxygen permeability

Oxygen permeability was measured under controlled condition (50 ± 3% relative humidity, 23 ± 2 °C) according to the method developed by Papkovsky et al. (2000). The films were mounted between the upper lid and rubber ring with silicon lubricant and fixed to the lower cup by screws, with an oxygen sensor housed inside. Nitrogen gas was blown into the chamber through one pipe, while exhausting air through another, until the nitrogen reading became stable within the chamber. Both pipes were then shut. The sensor measured the increase in oxygen content over time. The original data was calculated using the following equation:

$$\text{OP} = [S/(60 \cdot \beta)] \cdot (V/20.5) \cdot (273/298) \cdot [(T \cdot 1000)/A]/101.625 \times 10^9/24$$

where OP = oxygen permeability (cm^3 μm/m^2 d kPa), S = slope indicating the transmission rate of oxygen, β = permeability coefficient (4.776), a constant value in this study as all treatments were conducted at the same conditions (50 ± 3% relative humidity,

Table 1
Experimental design levels and data for gelatin films

Runs	Coded variable level		pH	CO% (w/w)	T (μm)	TS (MPa)	E (%)	PT (N)	WVP (g mm/kPa d m^2)	OP (cm^3 μm/m^2 d kPa)
1	−1.4142	0	3.46	27.25	50	7.45	265	19.73	56.80	28.60
2	0	–	7.00	0.00	42	6.05	193	13.24	55.92	32.75
3	1	1	9.50	47.00	55	15.30	264	28.49	53.36	34.00
4	1.4142	0	10.54	27.25	49	9.20	351	15.66	56.64	26.80
5	1	−1	9.50	7.50	46	7.79	211	18.29	60.75	27.75
6	−1	−1	4.50	7.50	46	7.28	197	16.46	62.94	28.00
7	−1	1	4.50	47.00	50	15.02	153	28.33	60.23	31.33
8	0	1.4142	7.00	55.18	66	12.60	139	26.86	50.55	35.51
9	0	0	7.00	27.25	52	7.28	215	17.44	70.30	33.80
10	0	0	7.00	27.25	52	7.73	220	17.77	72.70	33.33

T: thickness of gelatin films; TS: tensile strength of gelatin films; E: percentage elongation at break point of gelatin films; PT: puncture strength of gelatin films; WVP: water vapour permeability of gelatin films; and OP: oxygen permeability of gelatin films.

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