

DEVELOPMENT OF VALUE ADDED LOW FAT SPREADS

DR. CHENG LAI HOONG PUSAT PENGAJIAN TEKNOLOGI INDUSTRI (304/PTEKIND/638133)



LAPORAN AKHIR PROJEK PENYELIDIKAN JANGKA PENDEK FINAL REPORT OF SHORT TERM RESEARCH PROJECT Sila kemukakan laporan akhir ini melalui Jawatankuasa Penyelidikan di Pusat Pengajian dan Dekan/Pengarah/Ketua Jabatan kepada Pejabat Pelantar Penyelidikan

1.	Nama Ketua Penyelidik: Dr. Cheng Lai Hoong Name of Research Leader Profesor Madya/ Assoc. Prof. XDr./	Encik/Puan Mr/Mrs/Ms		1.57
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3.	Nama Penyelidik Bersama: Assoc. Prof. Dr. Noor Aziah A Name of Co-Researcher		K	
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ii)	Kualiti output: Quality of outputs			X
iii)	Kualiti impak: Quality of impacts		X	
iv)	Pemindahan teknologi/potensi pengkomersialan: Technology transfer/commercialization potential		X	
v)	Kualiti dan usahasama : Quality and intensity of collaboration		x	
vi)	Penilaian kepentingan secara keseluruhan: Overall assessment of benefits		X	

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ABSTRACT

English:

This work involved the characterization of a low fat spread made of fish gelatin and pectin with or without salt addition and pH adjustment. The samples prepared were characterized by temperature sweep, bulk density determination, texture analysis and morphology evaluation. It was found that a decrease in fish gelatin to pectin ratio resulted in an increase in bulk density, firmness, compressibility, adhesiveness, elasticity and melt ability. In addition, spreads prepared at high acidity gave rise to syneresis, which could be remedied with addition of sodium chloride.

Bahasa Malaysia:

Kajian ini melibatkan pencirian sapuan rendah lemak yang diperbuat daripada gelatin ikan dan pectin dengan penambahan garam dan pengawalan pH. Sampel yang dihasilkan telah dikaji dari segi sapuan suhu, penentuan ketumpatan pukal, analisis tekstur dan penilaian morfologi. Keputusan menunjukkan bahawa penurunan dalam nisbah kandungan gelatin ikan kepada pectin telah meningkatkan ketumpatan pukal, kekerasan, kebolehmampatan, kelekitan, keelastikan dan kebolehleburan sapuan terhasil. Tambahan pula, sapuan yang disediakan dengan kandungan asid tinggi telah menyebabkan berlakunya *syneresis*, yang mana adalah didapati dapat diatasi dengan penambahan natrium klorida.

Comprehensive Technical Report Development of value added low fat spreads

1. Introduction

Nowadays, consumers are more health conscious and have responded to the call for a diet that contains low fat, low sugar, low salt but high fiber. At the same time consumer will never tolerate in terms of palatability. Therefore, this healthy trend has created great challenge to food technologists to tackle the undesirable eating qualities crop from such foods. Among which, low fat products prepared with less than 40% fat content have been an increased market interest and have drawn extensive attention of food technologists.

It is reported that four types of ingredients are required for the production of a low fat spread, namely a viscosifying agent, a gelling agent, a phase separating agent and a synergistic agent. These were reported to be important in playing a role to prevent emulsion breakdown and to give better flavor release in the mouth as well as to inhibit syneresis. This requires the use of protein together with polysaccharide as an emulsifying, thickening and/or gelling agent (Mageen & Jones, 1989; Ross-Murphy, 1992; Chronakis & Kasapis, 1995a, 1995b; Clegg, Moore & Jones, 1996).

In this project, gelatin is used as the protein source. The amphoteric characteristic of gelatin as well as its hydrophobic areas on the peptide chain makes it a strong emulsifying and foaming agent (Galazka, Disckinson & Ledward, 1999; Cole, 2000). Therefore, it has been proven that gelatin has the ability to form a strong adsorbed layer at the interface to prevent droplet coalescence and collapse of air bubbles. However, owing to the relatively high cost of gelatin, alternative ingredient to replace part and parcel of the amount of gelatin use has been sought after. Pectin, an economic thickening and gelling agent that has recently been found to show

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emulsifying properties in an oil in water emulsion (Leroux, Langendorff, Schick, Vaishnav & Mazoyer, 2003) could be a promising replacer for gelatin.

This paper aims at studying the effects of different fish gelatin to pectin ratio and environmental stresses (pH and salts) on the stability of a low fat spread sample. To the best of our knowledge, this has not been studied elsewhere and we believe that the information of the characteristics of such blend system is important to justify its potential use in low fat spread and other food applications.

2. Materials and methods

2.1 Materials

The low fat spread was made of two phases, namely the water phase and the fat phase. The ingredients used for making the water phase were skimmed milk powder, fish gelatin (G7041, Sigma-Aldrich, St Louis, MO), medium rapid set pectin (Sim Company, Penang, Malaysia), sodium chloride or calcium chloride and potassium sorbate. The gelatin was extracted from cold water fish skin with a zero bloom value, and the pectin used was 66-69% esterified. As for the fat phase, commercial margarine fat blend was used. Where necessary, citric acid was used to adjust acidity of the sample.

2.2 Preparation of low fat spread

Fish gelatin and pectin based low fat spread (1 kg / preparation) was prepared following the recipe recommended by Madsen (2000) with some modification. The recipe contained the following ingredients in percentage (w/w): oil blend 30%, water 64.4%, hydrocolloids (fish gelatin plus pectin) 3%, sodium chloride 1.5%, skimmed milk powder 1% and potassium sorbate 0.1%. For studying the effects of salt and

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acidity, calcium chloride was added instead of sodium chloride and citric acid was used to adjust the pH of the water phase, respectively.

Fish gelatin and pectin of different ratio, skimmed milk powder, salt and potassium sorbate were blended together in water at 50 °C and stirred for 20 min. The aqueous phase was then added slowly into the fat phase and blended with a home mixer mixing at low speed for 10 min followed by high speed for 5 min. Samples were kept in sterilized media bottles at room temperature for ~20 hours before analysis. Sample preparation and treatments were carried out in duplicate.

2.3 Temperature sweep

The rheological behavior of the low fat spread samples prepared was characterized using oscillatory temperature sweep. All measurements were carried out with a controlled stress AR1000 Rheometer (TA Instruments, New Castle, DE) equipped with a Peltier Temperature Controller, using parallel plate geometry (40 mm diameter and 0.5 mm gap). Silicone oil was applied to avoid dehydration during heating. Temperature sweeps were run over the range from 5 to 50 °C at a heating rate of 2 °C min⁻¹ with constant strain amplitude of 2% chosen from the linear viscoelastic region of the samples at 1 Hz. The storage modulus (G²) as a function of heating temperature was recorded.

2.4 Bulk density determination

Sample was filled into a square container of known volume. Excess sample was leveled off with a ruler and the content was weighed. Bulk density (g/cm⁻³) was calculated as the ratio of the sample mass to volume of the container. Average of quadruplicate was recorded with two sub-samples from each sample preparation.

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2.5 Texture evaluation

Texture attributes of sample were evaluated at 25-27 °C in terms of firmness, compressibility and adhesiveness using a Texture Analyzer (TA-XT2, UK). The accessory used was Back Extrusion Cell (A/BE) with a 40 mm disc. The sample was filled into an acrylic container (50 mm internal diameter) to a depth of 20 mm. The test speed was fixed at 1.00 mm/s with a test distance of 15 mm and upon compression the disc was returned to the initial starting point. From the resulting force-time curve, firmness (the maximum force), compressibility (the positive area) and adhesiveness (the negative area) were calculated.

2.6 Morphology evaluation

Sample appearance was recorded with a Digital Camera (Olympus Optical Co., Ltd., Japan).

2.7 Statistical analysis

One-way analysis of variance using SPSS 10.0 for windows was performed on all experimental data sets. Post-hoc multiple comparisons were carried out by Duncan analysis to determine significant differences between sample means. All comparisons were made at a 5% level of significance.

3. Results and Discussion

During blending, it was observed that the combination use of fish gelatin and pectin is crucial in producing a low fat spread with only 30% fat content. It is believed that the desirable plasticity and spreadability properties obtained at room temperature

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were inextricably linked to the emulsification and thickening properties of fish gelatin and pectin, respectively. Our observation was in line with those reported by Schultz, Schmidt and Schmandke(1990) and Einhorn-Stoll (1998) who found that pectin helps to stabilize an oil-in-water (O/W) emulsion in the presence of another protein emulsifier.

Figure 1 shows the variations in storage modulus (G') of the samples prepared as a function of heating temperature. It can be seen that G' decreased progressively with increase in heating temperature. This could be attributed to the cumulative effects of fat phase liquefaction together with the dissociation of gelled network formed within the system. The complete melting is seen to occur at 25 $^{\circ}$ C.

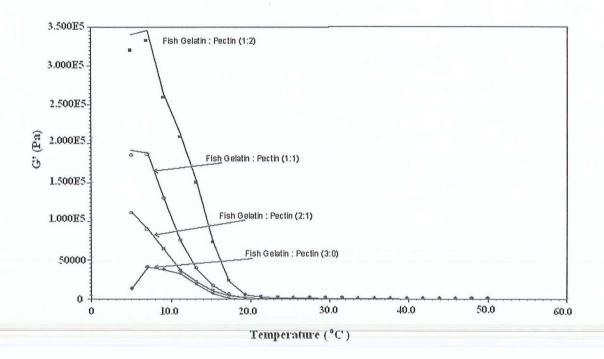


Figure 1: Temperature sweep of low fat spread varied in terms of fish gelatin to pectin ratio.

According to Ronn, Hylgid, Weinberg, Qvist and Laustsen (1998), a steep temperature sweep curve indicates high melt ability and a flat curve suggests low melt ability. As shown in Figure 1, melt ability decreased in the following order: Fish

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gelatin to pectin ratio of 1:2 > 1:1 > 2:1 > 3:0. In addition, the G' value at a specific temperature decreased in the similar order. This is closely related to the number of junction zones formed via the interactions between pectin-pectin, pectin-fish gelatin and fish gelatin-fish gelatin molecules. Nevertheless, pectin-pectin interaction is believed to be the predominant factor in determining the elasticity and sensitivity towards heating due to the fact that pectin and fish gelatin are thermodynamically different polymers (Morris, 1990) and fish gelatin used in this study is a non-gelling agent. This means to say increase pectin content will increase consistency and melt ability of a low fat spread sample.

A fast meltdown property is normally associated with rapid flavour release (Philips & Williams, 2000). From the result depicted in Figure 1, it can be predicted that low fat spread sample made of fish gelatin to pectin ratio of 1:2 could perform better with 'melt in the mouth' characteristic and greater instant in-mouth flavour release effect.

Bulk density is a measure of sample mass divided by its volume. A significant increase in bulk density with substitution of fish gelatin with pectin was evident in Table 1. According to Glicksman (1982), fish gelatin exhibits excellent foaming and emulsifying properties. Thus, higher ratio of fish gelatin to pectin applied in the aqueous phase promotes more air to be trapped during blending.

Table 1 shows some texture attributes of the low fat spread sample prepared. The firmness, compressibility and adhesiveness properties were found to significantly increase with higher substitution of fish gelatin with pectin. Among the parameters reported, compressibility indicates the amount of work required to attain deformation of internal strength of bonds within the system. Thus, it suggests that the presence of pectin enhanced the formation of an extensively associated network as compared to having fish gelatin alone in the system. This is of utmost important in the sense that a

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moderately associated network would probably support the product structure and maintain its consistency throughout storage.

Bulk Density (g/cm ³) ^{*1}	Firmn ess (g) * ²	Compressibility(g.s)* ²	Adhesiveness(g.s)* ²
0.77 ± 0.02^{a}	163.97 ± 5.28^{a}	$1993.17 \pm 53.54^{\circ\circ}$	1301.00 ± 43.89^{a}
0.83 ± 0.01^{bc}	175.90 ± 4.97^{b}	2065.17 ± 45.46 ^a	1282.33 ± 77.64^{a}
0.81 ± 0.01^{b}	$189.37 \pm 8.31^{\circ}$	$2101.50 \pm 1.76.43$ °	$1447.83 \pm 127.69^{\rm b}$
$0.84\pm0.01^{\rm c}$	251.10 ± 8.55^{d}	$2868.33 \pm 83.18^{\rm b}$	$2053.83 \pm 94.28^{\circ}$
	0.83 ± 0.01^{bc} 0.81 ± 0.01^{b}	$(g/cm3)*1 0.77 \pm 0.02a 163.97 \pm 5.28a 0.83 \pm 0.01bc 175.90 \pm 4.97b 0.81 \pm 0.01b 189.37 \pm 8.31c$	$\begin{array}{c} \left(g/cm^3 \right)^{\pm 1} \\ \hline 0.77 \pm 0.02^{a} \\ 163.97 \pm 5.28^{a} \\ 1993.17 \pm 53.54^{a} \\ \hline 0.83 \pm 0.01^{bc} \\ 175.90 \pm 4.97^{b} \\ 2065.17 \pm 45.46^{a} \\ \hline 0.81 \pm 0.01^{b} \\ 189.37 \pm 8.31^{c} \\ 2101.50 \pm 176.43^{a} \\ \end{array}$

Table 1 Bulk density, firmness, compressibility and adhesiveness of low fat spread.

 $*^{1}$ Mean ± standard deviation (n=4). $*^{2}$ Mean ± standard deviation (n=6). Means within a column with the same letter are not significantly different at the 5% probability level.

Figure 2 shows the morphology of low fat spread samples treated with pH adjustment and sodium or calcium salt addition. As clearly shown, spreads prepared at pH 4.5 (samples coded as D, E, and F) seem to be more stable than spreads at pH 3.0 (samples coded as A, B and C). This stability could be due to the fact that pH 4.5 is very close to the isoelectric point of the protein molecules (results not shown). This caused the proteins to have zero net charge along its molecules (Raymundo, Empis & Sousa, 2000; deKruil & Tuinier, 2001) and stabilized. However, at pH 4.5 pectin molecules are slightly charged negative. This resulted in electrostatic repulsion among pectin molecules, that in turn creating a higher solvating or hydrating layers of water molecules around the pectin chains (Glicksman, 1982; Bernal, Smajda, Smith & Stanley, 1987). Subsequently, this helps to prevent water syneresis from taking place.

It is also evident that spread samples adjusted to pH 3 were not stable. Those without addition of salt (sample A) and with addition of calcium chloride (sample C)

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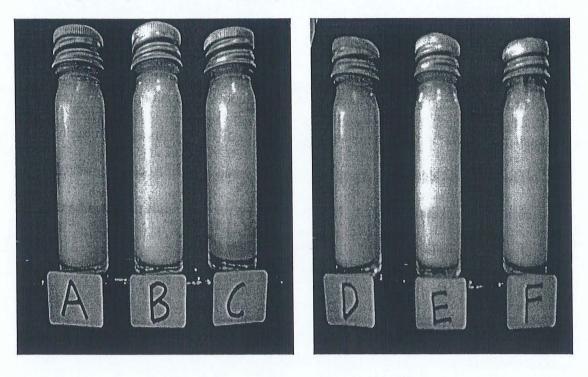


Figure 2: Sample morphology (A) No salt added, adjusted at pH 3; (B) NaCl added, adjusted at pH 3; (C) CaCl₂ added, adjusted at pH 3; (D) No salt added, adjusted at pH 4.5; (E) NaCl added, adjusted at pH 4.5; (F) CaCl₂ added, adjusted at pH 4.5.

experienced severe phase separation. And it is interesting to note that this instability at pH 3 could be overcome with addition of sodium chloride (sample B). At pH 3, aggregates formed due probably to attractive forces between the oppositely charged protein molecules (gelatin and casein) and pectin molecules. The formation of aggregates will exclude water from the matrix resulting in syneresis. So, when sodium ions (Na⁺) was incorporated, it is believed that the shielding or screening effect of sodium monovalent ion on protein and pectin molecules stabilized or neutralized the molecules charges, hence decreasing the ability of the molecules to come into contact to from aggregates (Agboola & Dalgleish, 1995; Ipsen, 1997; Haug, Draget & Smidrod, 2004).

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4. Conclusions

This study showed that low fat spread properties were significantly influenced by the different ratios of fish gelatin and pectin incorporated. Both fish gelatin and pectin complement each other in catering desirable attributes of low fat spread. On the other hand, if high acidity is desired, it is suggested that spread stability can be overcome with the addition of sodium chloride. Nevertheless, further research needs to be done to support these observations.

References

- Agboola, S.O. & Dalgleish, D.G. (1995). Calcium-induced destabilization of oil-inwater emulsions stabilized by caseinate or by β-Lactoglobulin. *Journal of Food Science*, 60(2), 399-404.
- Bernal, V.M., Smajda, C.H., Smith, J.L., & Stanley, D.W. (1987). Interactions in protein/polysaccharide/calcium gels. *Journal of Food Science*, *52*, 1121-1125.
- Chronakis, I.S. & Kasapis, S. (1995a). A rheological study on the application of carbohydrate-protein incompatibility to the development of low fat commercial spreads. *Carbohydrate Polymer, 28,* 367-373.
- Chronakis, I.S. & Kasapis, S. (1995b).Preparation and analysis of water continuous very low fat spreads. *Lebensmittel-Wissenschaft und-Technologie*, 28, 488-494.
- Clegg, s.M., Moore, A.K. & Jones, S.a. (1996). Low-fat margarine spreads as affected by aqueous phase hydrocolloids. *Journal of Food Science*, *61(5)*, 1073-1079.
- Cole, C.G.B. (2000). Gelatin. In F.J. Francis (Ed.). Encyclopedia of Food Science and Technology, 2nd Ed. (pp. 1183-1188). New York: John Wiley & Sons.
- deKruil, G.G. & Tuinier, R. (2001). Polysaccharide protein interactions. *Food Hydrocolloids*, 15, 555-563.

Development of value added low fat spreads

- Einhorn-Stoll, U. (1998). Interactions of whey proteins with different pectins in O/W emulsions. *Nahrung*, *42*, 248-249.
- Glicksman, M. (1982). Functional properties of hydrocolloids. In M. Glicksman (Ed.). Food hydrocolloids (pp. 48-100). Boca Raton, Florida: CRC Press.
- Haug, I.J., Draget, K.I. & Smidrod, O. (2004). Physical and rheological properties of fish gelatin compared to mammalian gelatin. *Food Hydrocolloids*, 18, 203-213.
- Ipsen, R. (1997). Uniaxial compression of gels made from protein and K-Carrageenan. *Journal of Texture Studies, 28,* 405-419.
- Leroux, J., Langendorff, V., Schick, g., Vaishnav, V. & Mazoyer, J. (2003). Emulsion stabilizing properties of pectin. *Food Hydrocolloids*, *17*, 455-462.
- Madsen, F. (2000). Substitution of gelatine in low fat spread. A rheological characterization. In P.A. Williams & G.O. Philips (Eds.), *Gum and the stabilizer for the food industry 10* (pp. 411-420). Cambridge, UK: The Royal Society of Chemistry.
- Mageen, P. & Jones, S.A. (1989). Low fat spreads products. Food Science Technology Today, 3(3), 162-164.
- Morris, V.J. (1990). Starch gelation and retrogradation. *Trends Food Science and Technology*, 7, 2-6.
- Philips, G.O. & Williams, P.A. (2000). *Handbook of Hydrocolloid*. England: Woodhead Publishing Limited.
- Raymundo, A., Empis, J., & Sousa, UI. (2000). Effect of pH and NaCl on rheological and textural properties of Lupin protein emulsion. In P.A. Williams & G.O. Philips (Eds.). *Gum and the stabilizer for the food industry 10* (pp. 350-365). Cambridge, UK: The Royal Society of Chemistry.

Development of value added low fat spreads

- Ronn, B.B., Hylgid, G., Weinberg, L., Qvist, K.B. & Laustsen, A.M. (1998).
 Predicting sensory properties from rheological measurement of low-fat spreads.
 Food Quality and Preference, 9(4), 187-196.
- Ross-Murphy, S.B. (1992). Structure and rheology of gelatin gels. *Recent Progression in Polymer, 33,* 2622-2627.
- Schultz, M., Schmidt, G. & Schmandke, H. (1990). Effects of low methoxy pectin on protein stabilized O/W emulsions. *Die Nahrung*, *34*, 443-447.



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Short Communication

Using fish gelatin and pectin to make a low-fat spread

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Food Technology Division, School of Industrial Technology, Universiti Sains Malaysia, 11800 Penang, Malaysia Received 5 September 2007; accepted 26 October 2007

Abstract

This preliminary work involved the characterization of a low-fat spread made of fish gelatin and pectin with or without salt addition and pH adjustment. The samples prepared were characterized by temperature sweep, bulk density determination, texture analysis and morphology evaluation. It was found that a decrease in fish gelatin to pectin ratio resulted in an increase in bulk density, firmness, compressibility, adhesiveness, elasticity and melt ability. In addition, spreads prepared at high acidity gave rise to syneresis, which could be remedied with addition of sodium chloride.

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Keywords: Low-fat spread; Pectin; Fish gelatin; Bulk density; Temperature sweep

1. Introduction

Nowadays, consumers are more health conscious and have responded to the call for a diet that contains low fat, low sugar, low salt but high fiber. At the same time, consumers will never tolerate in terms of palatability. Therefore, this healthy trend has created great challenge to food technologists to tackle the undesirable eating qualities crop from such foods. Among which, low-fat products prepared with less than 40% fat content have been an increased market interest and have drawn extensive attention of food technologists.

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In this project, gelatin is used as the protein source. The amphoteric characteristic of gelatin as well as its hydrophobic areas on the peptide chain makes it a strong emulsifying and foaming agent (Galazka, Disckinson, & Ledward, 1999; Cole, 2000). Therefore, it has been proven that gelatin has the ability to form a strong adsorbed layer at the interface to prevent droplet coalescence and collapse of air bubbles. However, owing to the relatively high cost of gelatin, alternative ingredient to replace part and parcel of the amount of gelatin use has been sought after. Pectin, an economic thickening and gelling agent that has recently been found to show emulsifying properties in an oil-inwater (O/W) emulsion (Leroux, Langendorff, Schick, Vaishnav, & Mazoyer, 2003) could be a promising replacer for gelatin.

This paper aims at studying the effects of different fish gelatin to pectin ratio and environmental stresses (pH and salts) on the stability of a low-fat spread sample. To the best of our knowledge, this has not been studied elsewhere and we believe that the information of the characteristics of such a blend system is important to justify its potential use in low-fat spread and other food applications.

2. Materials and methods

2.1. Materials

The low-fat spread was made of two phases, namely the water phase and the fat phase. The ingredients used for

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making the water phase were skimmed milk powder, fish gelatin (G7041, Sigma-Aldrich, St. Louis, MO), medium rapid set pectin (Sim Company, Penang, Malaysia), sodium chloride or calcium chloride and potassium sorbate. The gelatin was extracted from cold-water fish skin with a zero bloom value, and the pectin used was 66–69% esterified. As for the fat phase, commercial margarine fat blend was used. Where necessary, citric acid was used to adjust acidity of the sample.

2.2. Preparation of low-fat spread

Fish gelatin- and pectin-based low-fat spread (1 kg/ preparation) was prepared following the recipe recommended by Madsen (2000) with some modification. The recipe contained the following ingredients in percentage (w/w): oil blend 30%, water 64.4%, hydrocolloids (fish gelatin plus pectin) 3%, sodium chloride 1.5%, skimmed milk powder 1% and potassium sorbate 0.1%. For studying the effects of salt and acidity, calcium chloride was added instead of sodium chloride and citric acid was used to adjust the pH of the water phase.

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2.3. Temperature sweep

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2.4. Bulk density determination

Sample was filled into a square container of known volume. Excess sample was leveled off with a ruler and the content was weighed. Bulk density (g/cm^3) was calculated as the ratio of the sample mass to volume of the container. Average of quadruplicate was recorded with two subsamples from each sample preparation.

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Texture attributes of sample were evaluated at 25-27 °C in terms of firmness, compressibility and adhesiveness using a Texture Analyzer (TA-XT2, UK). The accessory used was Back Extrusion Cell (A/BE) with a 40 mm disc. The sample was filled into an acrylic container (50 mm internal diameter) to a depth of 20 mm. The test speed was fixed at 1.00 mm/s with a test distance of 15 mm, and upon compression the disc was returned to the initial starting point. From the resulting force-time curve, firmness (the maximum force), compressibility (the positive area) and adhesiveness (the negative area) were calculated.

2.6. Morphology evaluation

Sample appearance was recorded with a Digital Camera (Olympus Optical Co., Ltd., Japan).

2.7. Statistical analysis

One-way analysis of variance using SPSS 10.0 for windows was performed on all experimental data sets. Post-hoc multiple comparisons were carried out by Duncan analysis to determine significant differences between sample means. All comparisons were made at a 5% level of significance.

3. Results and discussion

During blending, it was observed that use of the combination of fish gelatin and pectin is crucial in producing a low-fat spread with only 30% fat content. It is believed that the desirable plasticity and spreadability properties obtained at room temperature were inextricably linked to the emulsification and thickening properties of fish gelatin and pectin, respectively. Our observation was in line with those reported by Schultz, Schmidt, and Schmandke(1990) and Einhorn-Stoll (1998), who found that pectin helps to stabilize an O/W emulsion in the presence of another protein emulsifier.

Fig. 1 shows the variations in storage modulus (G') of the samples prepared as a function of heating temperature. It can be seen that G' decreased progressively with increase in heating temperature. This could be attributed to the cumulative effects of fat-phase liquefaction together with the dissociation of gelled network formed within the system. Complete melting is seen to occur at 25 °C.

According to Ronn, Hylgid, Weinberg, Qvist, and Laustsen (1998), a steep temperature sweep curve indicates high melt ability and a flat curve suggests low melt ability. As shown in Fig. 1, melt ability decreased in the following order: fish gelatin to pectin ratio of 1:2 > 1:1 > 2:1 > 3:0. In addition, the G' value at a specific temperature decreased in the similar order. This is closely related to the number of junction zones formed via the interactions between pectin-pectin, pectin-fish gelatin and fish gelatin-fish

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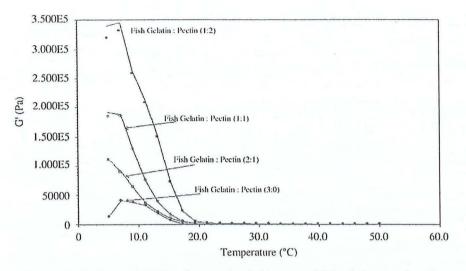


Fig. 1. Temperature sweep of low-fat spread varied in terms of fish gelatin to pectin ratio.

Table 1	
Bulk density, firmness, compressibility and adhesiveness of low-fat sp	oread

Fish gelatin:pectin	Bulk density (g/cm ³)*1	Firmness (g) ^{*2}	Compressibility (gs)*2	Adhesiveness $(gs)^{*2}$
3:0	0.77 ± 0.02^{a}	163.97±5.28 ^a	1993.17±53.54 ^a	1301.00±43.89 ^a
2:1	0.83 ± 0.01^{bc}	175.90 ± 4.97^{b}	2065.17 ± 45.46^{a}	1282.33 ± 77.64^{a}
1:1	0.81 ± 0.01^{b}	$189.37 \pm 8.31^{\circ}$	2101.50 ± 176.43^{a}	1447.83 ± 127.69^{b}
1:2	$0.84 \pm 0.01^{\circ}$	251.10 ± 8.55^{d}	2868.33 ± 83.18^{b}	$2053.83 \pm 94.28^{\circ}$

Means within a column with the same letter are not significantly different at the 5% probability level.

*¹Mean \pm standard deviation (n = 4).

*²Mean \pm standard deviation (n = 6).

gelatin molecules. Nevertheless, pectin-pectin interaction is believed to be the predominant factor in determining the elasticity and sensitivity towards heating due to the fact that pectin and fish gelatin are thermodynamically different polymers (Morris, 1990), and fish gelatin used in this study is a non-gelling agent. This means to say that increase in pectin content will increase consistency and melt ability of a low-fat spread sample.

A fast meltdown property is normally associated with rapid flavor release (Philips & Williams, 2000). From the result depicted in Fig. 1, it can be predicted that low-fat spread sample made of fish gelatin to pectin ratio of 1:2 could perform better with 'melt in the mouth' characteristic and greater instant in-mouth flavor release effect.

Bulk density is a measure of sample mass divided by its volume. A significant increase in bulk density with substitution of fish gelatin with pectin was evident in Table 1. According to Glicksman (1982), fish gelatin exhibits excellent foaming and emulsifying properties. Thus, higher ratio of fish gelatin to pectin applied in the aqueous phase promotes more air to be trapped during blending.

Table 1 shows some texture attributes of the low-fat spread sample prepared. Firmness, compressibility and adhesiveness properties were found to significantly increase with higher substitution of fish gelatin with pectin. Among

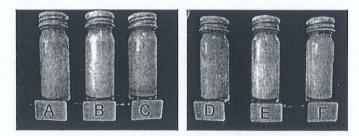


Fig. 2. Sample morphology: (A) no salt added, adjusted at pH 3; (B) NaCl added, adjusted at pH 3; (C) CaCl₂ added, adjusted at pH 3; (D) no salt added, adjusted at pH 4.5; (E) NaCl added, adjusted at pH 4.5; (F) CaCl₂ added, adjusted at pH 4.5.

the parameters reported, compressibility indicates the amount of work required to attain deformation of internal strength of bonds within the system. Thus, it suggests that the presence of pectin enhanced the formation of an extensively associated network as compared with having fish gelatin alone in the system. This is of utmost importance in the sense that a moderately associated network would probably support the product structure and maintain its consistency throughout storage.

Fig. 2 shows the morphology of low-fat spread samples treated with pH adjustment and sodium or calcium salt addition. As clearly shown, spreads prepared at pH 4.5

(samples coded as D, E and F) seem to be more stable than spreads prepared at pH 3.0 (samples coded as A, B and C). This stability could be due to the fact that pH 4.5 is very close to the isoelectric point of the protein molecules (results not shown). This caused the proteins to have zero net charge along its molecules (deKruil & Tuinier, 2001; Raymundo, Empis, & Sousa, 2000) and stabilized. However, at pH 4.5 pectin molecules are slightly negatively charged. This resulted in electrostatic repulsion among pectin molecules, which in turn created a higher solvating or hydrating layers of water molecules around the pectin chains (Bernal, Smajda, Smith, & Stanley, 1987; Glicksman, 1982). Subsequently, this helps to prevent water syneresis from taking place.

It is also evident that spread samples adjusted to pH 3 were not stable. Those without addition of salt (sample A) and with addition of calcium chloride (sample C) experienced severe phase separation. And it is interesting to note that this instability at pH 3 could be overcome with addition of sodium chloride (sample B). At pH 3, aggregates formed probably due to attractive forces between oppositely charged protein molecules (gelatin and casein) and pectin molecules. The formation of aggregates will exclude water from the matrix, resulting in syneresis. So, when sodium ions (Na⁺) was incorporated, it is believed that the shielding or screening effect of sodium monovalent ion on protein and pectin molecules stabilized or neutralized the molecules charges, hence decreasing the ability of the molecules to come into contact to from aggregates (Agboola & Dalgleish, 1995; Haug, Draget, & Smidrod, 2004; Ipsen, 1997).

4. Conclusions

This study showed that low-fat spread properties were significantly influenced by different ratios of fish gelatin and pectin incorporated. Both fish gelatin and pectin complement each other in catering desirable attributes of low-fat spread. On the other hand, if high acidity is desired, it is suggested that spread stability can be overcome with the addition of sodium chloride. Nevertheless, further research needs to be done to support these observations.

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References

- Agboola, S. O., & Dalgleish, D. G. (1995). Calcium-induced destabilization of oil-in-water emulsions stabilized by caseinate or by β -lactoglobulin. *Journal of Food Science*, 60(2), 399–404.
- Bernal, V. M., Smajda, C. H., Smith, J. L., & Stanley, D. W. (1987). Interactions in protein/polysaccharide/calcium gels. *Journal of Food Science*, 52, 1121–1125.
- Chronakis, I. S., & Kasapis, S. (1995a). A rheological study on the application of carbohydrate-protein incompatibility to the development of low fat commercial spreads. *Carbohydrate Polymer*, 28, 367-373.
- Chronakis, I. S., & Kasapis, S. (1995b). Preparation and analysis of water continuous very low fat spreads. Lebensmittel-Wissenschaft und-Technologie, 28, 488-494.
- Clegg, S. M., Moore, A. K., & Jones, S. A. (1996). Low-fat margarine spreads as affected by aqueous phase hydrocolloids. *Journal of Food Science*, 61(5), 1073–1079.
- Cole, C. G. B. (2000). Gelatin. In F. J. Francis (Ed.), *Encyclopedia of food* science and technology (2nd ed, pp. 1183-1188). New York: Wiley.
- deKruil, G. G., & Tuinier, R. (2001). Polysaccharide protein interactions. Food Hydrocolloids, 15, 555–563.
- Einhorn-Stoll, U. (1998). Interactions of whey proteins with different pectins in O/W emulsions. *Nahrung*, 42, 248–249.
- Galazka, V. B., Dickinson, E., & Ledward, D. A. (1999). Emulsifying behavior of globulin Vicia Faba in mixtures with sulphated polysaccharides: Comparison of thermal and high-pressure treatments. *Food Hydrocolloids*, 13, 425–435.
- Glicksman, M. (1982). Functional properties of hydrocolloids. In M. Glicksman (Ed.), Food hydrocolloids (pp. 48–100). Boca Raton, Florida: CRC Press.
- Haug, I. J., Draget, K. I., & Smidrod, O. (2004). Physical and rheological properties of fish gelatin compared to mammalian gelatin. *Food Hydrocolloids*, 18, 203–213.
- Ipsen, R. (1997). Uniaxial compression of gels made from protein and K-Carrageenan. Journal of Texture Studies, 28, 405–419.
- Leroux, J., Langendorff, V., Schick, G., Vaishnav, V., & Mazoyer, J. (2003). Emulsion stabilizing properties of pectin. *Food Hydrocolloids*, 17, 455–462.
- Madsen, F. (2000). Substitution of gelatine in low fat spread. A rheological characterization. In P. A. Williams, & G. O. Philips (Eds.), *Gum and the stabilizer for the food industry*, Vol. 10 (pp. 411-420). Cambridge, UK: The Royal Society of Chemistry.
- Mageen, P., & Jones, S. A. (1989). Low fat spreads products. Food Science Technology Today, 3(3), 162–164.
- Morris, V. J. (1990). Starch gelation and retrogradation. Trends in Food Science and Technology, 7, 2–6.
- Philips, G. O., & Williams, P. A. (2000). Handbook of hydrocolloid. England: Woodhead Publishing Limited.
- Raymundo, A., Empis, J., & Sousa, UI. (2000). Effect of pH and NaCl on rheological and textural properties of Lupin protein emulsion. In P. A.
 Williams, & G. O. Philips (Eds.), *Gum and the stabilizer for the food industry*, Vol. 10 (pp. 350–365). Cambridge, UK: The Royal Society of Chemistry.
- Ronn, B. B., Hylgid, G., Weinberg, L., Qvist, K. B., & Laustsen, A. M. (1998). Predicting sensory properties from rheological measurement of low-fat spreads. *Food Quality and Preference*, 9(4), 187–196.
- Ross-Murphy, S. B. (1992). Structure and rheology of gelatin gels. Recent Progression in Polymer, 33, 2622–2627.
- Schultz, M., Schmidt, G., & Schmandke, H. (1990). Effects of low methoxy pectin on protein stabilized O/W emulsions. *Die Nahrung*, 34, 443–447.