UNIVERSITI PUTRA MALAYSIA

INFLUENCE OF pH AND DIFFERENT EMULSION COMPONENTS ON STABILITY AND PHYSICOCHEMICAL PROPERTIES OF CANOLA OIL-IN-WATER EMULSION

NEGAR MOHAMMADIAN RASNANI

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MASTER OF SCIENCE
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By

NEGAR MOHAMMADIAN RASNANI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

January 2010

This thesis is especially dedicated to my beloved father and mother that always giving me their unlimited support

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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January 2010

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Faculty : Food Science and Technology

The main objective of the present study was to investigate the effect of pH (5-7), processing conditions (i.e. pressure, cycle and temperature) and main emulsion components (namely, propylene glycol, caseinate sodium, pectin, sodium chloride and canola oil) on the physicochemical properties of sodium caseinate-pectin stabilized emulsions. In this work, the physicochemical emulsion properties assessed by measuring the zeta-potential, average droplet size, apparent viscosity, turbidity, and creaming stability were considered as response variables. The results indicated that the pH and temperature had significant ($p < 0.1$) effects on all the response variables studied. The highest creaming stability was observed at pH 5, which was attributed to the formation of multilayer in the interfacial area. It was found that the average droplet size increased when pH was increased from 5 to 7. The prepared

emulsions showed shear-thinning behaviour at different pH values (5-7). The highest emulsion viscosity was also observed at pH 5; while it decreased when pH was increased from 5.5 to 7.

The influence of processing conditions (i.e. pressure, cycle of high pressure homogenizer and temperature of environment to solubilize the pectin) on the physicochemical properties of sodium caseinate-pectin stabilized emulsions were investigated. The results indicated that the independent variables had the most and least significant ($p < 0.05$) effect on average droplet size and stability, respectively. The effect of cycle of the homogenizer was significant ($p < 0.05$) in all response variables. The nonlinear regression equations were significantly ($p < 0.05$) fitted for predicting the changes in all the response variables with relatively high coefficient of determination ($R^2 > 0.825$). It was concluded that desirable physicochemical properties can be achieved when the pressure, cycle, and temperature of the pectin solution were adjusted to 13 MPa, 3, and 80 ° C, respectively.

It was observed that the physicochemical properties of emulsions were significantly ($p < 0.1$) influenced by the concentration of pectin. It could be explained by the fact that the presence of carboxyl groups (–COOH) in the molecular structure of pectin provided the proper surface activity in the interfacial area. The pectin molecules adsorbed to the droplet surfaces and increased the repulsion forces between the emulsion onto droplets, thereby preventing extensive droplet flocculation. The zeta potential remained negative in all emulsion samples, possibly because of negatively charged (-COO^-) groups presence in the molecular structure of pectin. The second order regression equations were significantly ($p < 0.1$) fitted for predicting the changes in all the response variables with relatively high coefficient of determination ($R^2 > 0.7$). The results indicated that the emulsion containing 0.9% (w/w) propylene glycol, 1.0% (w/w) sodium caseinate, 3.0% (w/w) pectin, 0.2% (w/w) sodium chloride and 15.0% (w/w) canola oil provided the optimum emulsion formulation with desirable physicochemical properties. The adequacy of response surface equations was confirmed by indicating no significant ($p > 0.1$) difference between the experimental and predicted values.

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

KESAN pH DAN KOMPONEN EMULSI YANG BERLAINAN KEATAS KESTABILAN DAN SIFAT FIZIKOKIMIA EMULSI MINYAK KANOLA - DALAM-AIR

Oleh

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Objektif utama kajian ini dijalankan adalah untuk menyiasat kesan pH (5-7), keadaan pemprosesan (tekanan, bilangan kitaran dan suhu) dan komponen utama emulsi (propilena glikol, natrium kaseinat, pektin, NaCl dan minyak kanola) terhadap sifat-sifat fizikokimia emulsi yang distabil menggunakan gabungan natrium kaseinat-pektin. Dalam kajian ini, sifat-sifat fizikokimia emulsi seperti potensi-zeta, purata saiz partikel, kelikatan, kekeruhan dan kestabilan pengkriman akan dinilai dan dijadikan sebagai pemboleh-ubah respons. Hasil kajian menunjukkan pH dan suhu mempunyai kesan yang signifikan ($p < 0.05$) terhadap semua pemboleh-ubah respons yang dikaji. Kestabilan pengkriman yang paling stabil dapat diperlihatkan pada pH 5, dikaitkan kepada pembentukan berbilang lapis di zon antara muka. Keputusan juga menunjukkan purata saiz partikel meningkat apabila pH berubah dari 5 ke 7. Emulsi tersebut juga menunjukkan sifat pencairan ricihan di pH yang berlainan (pH 5-7).

Kelikatan emulsi adalah paling tinggi di pH 5, manakala kelikatannya menurun apabila pH meningkat dari pH 5.5 ke 7.

Pengaruh keadaan pemprosesan (tekanan, bilangan kitaran penghomogen tekanan tinggi dan suhu untuk melarutkan pektin) ke atas sifat-sifat fizikokimia emulsi yang distabil menggunakan gabungan natrium kaseinat-pektin juga dikaji. Hasil kajian menunjukkan pemboleh-ubah tersebut mempunyai kesan signifikan yang paling tinggi dan terkecil ($p < 0.05$) masing-masing ke atas purata saiz partikel dan kestabilan. Faktor bilangan kitaran penghomogen adalah signifikan ($p < 0.05$) ke atas semua pemboleh-ubah respons. Persamaan regresi tak linear yang signifikan ($p < 0.05$) bagi meramalkan perubahan semua pemboleh-ubah respons mempunyai pekali kolerasi relatif yang tinggi ($R^2 > 0.825$). Jadi, dapat disimpulkan bahawa, sifat-sifat fizikokimia yang dikehendaki dapat dicapai apabila tekanan, bilangan kitaran penghomogen dan suhu larutan adalah masing-masing 13 MPa, 3 dan 80 ° C.

Keputusan menunjukkan kepekatan pektin mempunyai kesan yang signifikan ($p < 0.05$) ke atas sifat-sifat fizikokimia emulsi. Ini dapat dijelaskan dengan kehadiran kumpulan berfungsi karboksil (-COOH) di dalam struktur molekul pectin yang memberikan fungsi surfaktan di zon antara muka. Molekul pektin akan menjerap ke permukaan titisan dan meningkatkan daya penolakan antara titisan emulsi bagi mengelakkan flokulasi titisan. Oleh kerana kumpulan berfungsi karboksil ($-COO^-$) di dalam struktur pektin, semua sampel emulsi menunjukkan nilai potensi-zeta yang negatif. Persamaan regresi tertib kedua yang signifikan ($p < 0.1$) bagi meramalkan perubahan semua pemboleh-ubah respons juga mempunyai pekali kolerasi relatif yang tinggi ($R^2 > 0.7$). Keputusan menunjukkan emulsi dengan 0.9% (w/w) propilena glikol, 1.0% (w/w) natrium kaseinat, 3.0% (w/w) pectin, 0.2% (w/w) NaCl dan 15.0% (w/w) minyak kanola memberikan formulasi emulsi yang paling optimum dengan sifat-sifat fizikokimia yang dikehendaki. Kepadaan persamaan permukaan gerak balas disahkan dengan perbezaan yang tidak bererti ($p > 0.1$) antara nilai eksperimen dan ramalan.

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I certify that an Examination Committee has met on **………………………** to conduct the final examination of **Negar Mohammadian Rasnani** on her Master thesis entitled "**Influence of pH and Different Emulsion Components on the Stability and Physicochemical Properties of Canola Oil-In-Water Emulsion**" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the (Name of relevant degree).

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institutions.

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LIST OF ABBREVIATIONS

PG	Propylene glycol
NaCl	Sodium Chloride
CAS	Sodium caseinate
HMP	High methoxy pectin
LMP	Low methoxy pectin
RSM	Response Surface Methodology

CHAPTER I

INTRODUCTION

Emulsion-based food products are common everyday products, which are produced worldwide. Emulsions form the basis of many food products including milk, flavored milks, creams, salad dressings, dips, coffee whitener, ice cream, soups, sauces, mayonnaise, butter ,margarine, fruit beverages and whipped cream (Friberg and Larsson, 1997 ; Krog *et al.*, 1993 ; Dickinson and Stainsby, 1982; Dickinson, 1992).This kind of food products exist partly or wholly as emulsions and display a wide diversity of physicochemical and organoleptic characteristics, such as appearance, rheological behaviors, taste, aroma and shelf life, which is the result of the different type of ingredients and processing conditions utilized to produce each type of product.

Emulsions are colloidal dispersions (a colloid or colloidal dispersion is a type of homogeneous mixture and comprises a collection of small particles, droplets, or bubbles of one phase, having at least one dimension between 1 and 1000 nm, and dispersed in a second phase), consist of mixture of two unblendable liquids (usually oil and water). One of the liquids (the dispersed phase) is dispersed as small spherical droplets in the other (continuous phase). In most foods the diameter of the droplets is usually between 0.1 and 100 µm (Dickinson and Stainsby, 1982).

Emulsions are classified into two groups based on the distribution of the oil and aqueous phases: oil-in-water (o/w) emulsions that consist of oil droplets dispersed in an aqueous phase (e.g., mayonnaise, milk, cream etc.), and water-in-oil (w/o) emulsions which consist of water droplets dispersed in an oil phase (e.g. margarine, butter and spreads).

All oil-in-water emulsions have a basic compositional structure with almost the same components. First, there is the water that makes up about (70-83%) of the oil-in-water emulsions. The water used is separately pretreated to remove impurities, microorganisms, colloidal and suspended matter and other undesirable attributes, such as off-tastes, odors, turbidity, alkalinity and hardness. The second major component is the oil phase including the key elements responsible for sensory properties and textural stability of the oil-in-water emulsions. The other ingredients common to almost all the oil-in-water emulsions are stabilizers. Stabilizers have been used in food products for more than half a century. However, the functionality of stabilizers in new dairy products, have been attracting the interest of food manufacturers in more recent years (Lal *et al.*, 2006). Production of dairy products that have desirable quality attributes (appearance, texture and flavor) over a sufficient shelf life is what dairy manufacturers are seeking for. In order to achieve this objective, stabilizers have been utilized to enhance the kinetic stability of food emulsions (Dickinson, 1992). As emulsions are thermodynamically unstable systems, a stabilizer is any ingredient that can be used to enhance the stability of an emulsion for a reasonable period of time (a few days, weeks, months, or years) and may be either an emulsifier or a thickening agent. The most common emulsifiers used in food industry are amphiphilic proteins, small molecule surfactants and phospholipids. Thickening agents are ingredients which are used to increase the viscosity of the continuous phase of emulsion and they enhance emulsion stability by retarding the movement of the droplets. The most common thickening agents used in the food industry are polysaccharides.

Most food emulsions are much more complex than the simple three –component (oil, water and stabilizer). The aqueous phase may contain a variety of water-soluble ingredients, including: salts, acids, bases, surfactants, proteins, carbohydrates, hydrocolloids, colors, preservatives and sweeteners. The oil phase usually contains a complex mixtures of lipid-soluble components such as: Triacylglycerols, diacylglycerols, free fatty acids, sterols, flavor oils, weighting agents, antioxidants and cloudifying agents.

Emulsion-based food products are subjected to significant changes in their overall properties during production, storage and handling. The first reason for this phenomenon is the presence of different ingredients (e.g. salts, acids), wich may create various types of structural entities in the different phases of oil-in-water emulsions such as fat crystals, ice crystals, protein aggregates, air bubbles, liquid