GRADUATE AND POSTDOCTORAL STUDIES

McGILL UNIVERSITY

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FOR THE DEGREE OF
DOCTOR OF PHILOSOPHY

OF

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BIORESOURCE ENGINEERING

Extraction of Hydrocolloids from Vegetable Seeds and Identification of their Physicochemical Properties Compared with Selected Commercialized Gums

July 9th, 2018
1:15 pm
Raymond Building, Room R3-011
McGill University, Macdonald Campus

COMMITTEE:
Dr. Pierre Dutilleul (Pro-Dean) (Department of Plant Science)
Dr. G.S.V. Raghavan (Departmental Chair or deputy) (Department of Bioresource Engineering)
Dr. V. Orsat (Supervisor) (Department of Bioresource Engineering)
Dr. M. Lefsrud (Internal Examiner) (Department of Bioresource Engineering)
Dr. M.J. Dumont (Internal Member) (Department of Bioresource Engineering)
Dr. H. Ramaswamy (External Member) (Department of Food Science and Agricultural Chemistry)

Dr. Josephine Nalbantoglu, Dean of Graduate and Postdoctoral Studies
Members of the Faculty and Graduate Students
are invited to attend

ABSTRACT

Hydrocolloids are carbohydrates, or protein-polysaccharide biopolymers with great affinity to make hydrogen bonding with aqueous systems. Due to the diversity of functionalities they can provide (thickening, emulsification, gel formation, mouth-feel improvements, microencapsulation, etc.), they are employed in multi-faceted applications in various fields including the food industry. These hydrophilic biopolymers can be extracted from a diversity of sources including plants, seaweeds, animals or microorganisms. Due to the increasing market demand for hydrocolloids, annual plants show a high potential to answer this demand as a natural sustainable source of hydrocolloids.

The first chapter is dedicated to provide a summary of the literature about hydrocolloids in general including the already commercialized hydrocolloids extractable from a variety of sources. It also explored a little bit more around plant-sourced hydrocolloids and focused on some specified hydrocolloids extracted from the seeds (or seed husks) of annual plants. The cell walls of plants (also plant seeds) consist of remarkable sources of carbohydrates which are actually considered as plants' energy reservoirs. Reasonable amount of hydrocolloids can be extracted from these reservoirs, the chemistry of which may be different from source to source. Galactomannans, glucomannans, arabinoxylans, etc., can be extracted from these sources, each with varying functional properties. This interesting diversity, can perfectly respond to the existing and growing need for having diversity in functional ingredients besides in addition to being sustainable. Therefore, within the following chapters, the extraction of hydrocolloids was studied from some specific annual plant sources, namely *Ocimum basilicum* L. or basil seed, *Trigonella foenum-graecum* or fenugreek seed, and *Plantago ovata* Forsk or psyllium seed husk.

Hence, in the second chapter, the screening of the microwave-assisted hydrocolloid extraction from *Ocimum basilicum* L. (basil seed) was performed and compared with conventional heating extraction (CHE). A full factorial randomized design was selected for the experiments in which the yield of extraction was selected as the experiment response. pH, temperature, seed to water ratio, and ±

extraction time were selected as the extraction variables (based on the preliminary results), the effect of each, as well as their combinatory effects, were screened and evaluated on the experiment response (yield %). The physicochemical properties including chemical composition analysis, rheology, and Fourier transform infrared (FTIR) spectroscopy of the extracts were analyzed and compared between the two extraction methods. The extracted basil seed hydrocolloid contained 83.43% carbohydrate and 1.18% protein residues and it possessed high apparent viscosity ($\eta > 200$ mPa.s) with storage moduli (G') > loss moduli (G"), i.e. weak gel properties. The microwave-assisted extraction (MAE) of the hydrocolloid extracted from *Ocimum basilicum* L. (a glucomannan) resulted in higher yield of extraction (10.46 vs 6.17%) as well as improved physicochemical properties when compared with the conventional heating extraction (e.g. $G', G''_{MAE} > G', G''_{CHE}$).

In a third study, the optimization of the microwave-assisted hydrocolloid extraction from *Trigonella foenum-graecum* (fenugreek) seed was performed and compared with conventional heating extraction. Central composite, randomized fractional factorial design was selected for the experiments and the single effect, as well the combinatory effects of pH, temperature, and the extraction time (experiment variables), were evaluated on the experiment responses (the extraction yield % and the apparent viscosity, mPa.s). The extraction results as well as the physicochemical analysis revealed that MAE led to a higher extraction yield (24.29 vs 18.94%) and enhanced physicochemical properties compared with the conventional heating extraction (e.g. $G', G''_{MAE} >> G', G''_{CHE}$). Fenugreek seed hydrocolloid (FSH) contained ~85.5% carbohydrate and 0.86% protein, a statistically higher viscosity for MAE compared with CHE (~73.5 > ~17.5 mPa.s) with G" > G' for all the extracts.

In a fourth study, the microwave-assisted hydrocolloid extraction from *Plantago ovata* Forsk (Psyllium) seed husk was optimized and compared with conventional heating extraction. A semi-factorial central composite design was chosen for the experiments. The effect of variables (pH, temperature, and time) was evaluated on the

experiments independent responses (the yield of extraction %, the apparent viscosity, and tan δ). The physicochemical analysis results of psyllium seed husk hydrocolloid (PSH) showed a very high carbohydrate content (88.58%), with a low protein content of 0.87%. MAE of PSH resulted in higher yield of extraction when compared with the CHE (63.43 vs 49.44%). A statistically significant higher extraction yield (mean value) was obtained by MAE compared with CHE (~127 > ~40 mPa.s). The $G'_{MAE} > G'_{CHE}$; whereas the $G''_{CHE} > G''_{MAE}$. Also in most of the experimental conditions, $\tan \delta_{MAE} < \tan \delta_{CHE}$.

In a fifth study, the validation of the predicted responses at the defined screened or optimized conditions was performed for *Ocimum basilicum* L. (basil) seed, *Trigonella foenum-graecum* (fenugreek) seed, and *Plantago ovata* Forsk (psyllium) seed husk hydrocolloids and each for both MAE and CHE methods. The optimized extraction results and the physicochemical properties were compared for the three extracted hydrocolloids (BSH, FSH, and PSH). The optimized conditions differed for MAE and CHE for each of the hydrocolloids. The obtained extraction yields and apparent viscosities at the optimized conditions were higher or comparable with the predicted values. The Fourier transform infrared (FTIR) spectroscopy results showed highly similar absorbance peaks within the 4500 – 500 cm^{-1} of the analyzed region; however each of the hydrocolloids exhibited their very own specific absorbance peaks especially within the polysaccharides' fingerprint region (1200 – 800 cm^{-1}). In BSH and FSH, at ~870 – 800 cm^{-1}, the absorbance peaks were assigned to the stretching vibrations of glucopyranosyl, galactopyranosyl, and manopyranosyl groups of glycosidic bonds and in PSH, the peaks absorbed at ~1065 and ~1020 cm^{-1} were related to C-O-H groups of glycosidic linkages. In terms of molecular weight, PSH > BSH > FSH was observed. The rheological properties of the three hydrocolloids were also compared with selected commercialized gums including xanthan, guar, carboxymethylcellulose, locust bean gum, commercialized fenugreek seed gum, gum Arabic, and zedu gum.

In a sixth study, the three MAE hydrocolloids (BSH, FSH, and PSH) were applied as emulsion stabilizers and their emulsion

stabilizing capacity was evaluated and compared with defined well-known commercialized emulsifiers (gum Arabic and zedu gum). Gum Arabic exhibited a shear-thickening flow behavior; whereas all the other emulsions displayed the characteristics of shear-thickening fluids. Their hydrodynamic diameter (d, μm), diffusion coefficient (D, cm^2/s), polydispersity index (PDI), as well as viscosity (η, mPa.s) and emulsion stability (ES%) of the emulsions were compared. The η was proportional to "d", PDI, and ES%; whereas emulsions with higher η possessed smaller "D" values. It was observed that emulsions with smaller "d" possessed smaller PDI values as well. However, small PDI values were not necessarily resulting in higher ES%. The ES% of the three extracted hydrocolloids (BSH > PSH > FSH) was significantly higher compared with gum Arabic and zedu gum. The microstructure of the emulsions displayed the finest and the most homogenous particle size distribution for the gum Arabic emulsion. Signs of flocculation, however, were observed for all the emulsions except for the BSH-containing emulsion which showed to be the most stable of all.

CURRICULUM VITAE
UNIVERSITY EDUCATION

2015 – 2018	**PhD in Bioresource Engineering** McGill University, Canada
2012-2014	**M.Sc. in Food Science & Agricultural Chemistry** McGill University, Canada
1998-2002	**Bachelor of food industry engineering** Azad University, Iran

EMPLOYMENT

Mar. 2018 – Present	**Science-oriented Area Manager** Colin Ingredients / Diafood, France
2013	**Food product developer and Marketing** Maple Leaf / Canada Bread, Canada
2009 – 2010	**Field support manager, domain food** KERRY Ingredients & Flavours, Iran
2006 – 2009	**Technical representative, domain food** DERA Food Technology N.V., Iran & Belgium
2004 – 2006	**Food expert** Representation office of SENSIENT, Iran
2002 – 2004	**Technical support supervisor** Representation office of CARA GUM Intl., Iran

AWARDS

Walter M Stewart Scholarship, October 2015

PUBLICATIONS

1. Keisandokht, S., Haddad, N., Gariépy, Y., & Orsat, V. (2018). Screening the microwave-assisted extraction of hydrocolloids from *Ocimum basilicum* L. seeds as a novel extraction technique compared with conventional heating-stirring extraction. Food Hydrocolloids, 74, 11-22.

II. Submitted journal papers

2. Keisandokht, S., Sodano, T., Orsat, V., & Ngadi, M. (2018). Optimization of the extraction conditions and investigation of the physicochemical properties of *Trigonella foenum-graecum* seed hydrocolloids from conventional heating-stirring extraction compared with microwave-assisted extraction (submitted to the journal of "Industrial Crops and Products, Elsevier").

III. Journal papers in preparation for submission

3. Keisandokht, S., Sodano, T., Orsat, V., & Ngadi, M. (2018). Optimization of the extraction conditions and physicochemical analysis of the gel-forming *Plantago ovata* Forsk (Psyllium) seed husk hydrocolloids comparing conventional heating-stirring extraction with microwave-assisted extraction.

4. Keisandokht, S., Orsat, V., & Karboune, S. (2018). Microwave-assisted extraction of *Ocimum basilicum* L. (basil) seed, *Trigonella foenum-graecum* (fenugreek) seed, and *Plantago ovata* Forsk (Psyllium) seed husk hydrocolloids compared with conventional heating extractions at their optimum conditions and comparison of some physicochemical properties (with selected commercialised gums).

5. Keisandokht, S., Orsat, V., & Karboune, S. (2018). Application of single and binary mixtures of selected extracted novel seed hydrocolloids in stabilization of oil in water emulsions compared with selected common commercialized emulsifying agents.