MASSEY UNIVERSITY

Behaviour of Emulsion Gels in the Human Mouth and Simulated Gastrointestinal Tract

A thesis presented in partial fulfillment of the requirements

for the degree of

Doctor of Philosophy in Food Technology

at Massey University, Manawatu, New Zealand.

Qing Guo

2015

Riddet Institute
FOOD | INNOVATION | HEALTH

Abstract

Food structure greatly impacts the digestion process of food in the human body. Food structure design is a potential strategy to modulate food digestion and develop foods with controlled nutrient digestion and release. This research aimed to understand the dynamic processes of digestion of whey protein emulsion gels from the mouth to the intestine. Therefore, a series of heat-set whey protein emulsion gels, the structure of which was designed by varying NaCl concentration (10, 25, 100 and 200 mM) and oil droplet size (1, 6 and 12 μm), were formed. Mechanical properties in linear viscoelastic region, large deformation and fracture region and microstructure of gels were evaluated. *In vivo* oral processing and *in vitro* oral-to-gastrointestinal digestion of whey protein emulsion gels with different structures were investigated with a focus on the effect of gel structure on the gel disintegration and lipid digestion.

Results showed that the gel strength increased with increasing NaCl concentration. At the micro-scale, the gel structure became from homogenous to porous with increasing NaCl concentration from 10 to 200 mM. The fragmentation degree of whey protein emulsion gels in the mouth showed a positive linear relationship with the gel strength (i.e. a higher gel strength, the greater gel fragmentation degree). During oral processing, the small oil droplets (~ 0.45 μm) incorporated in the protein matrix were stable without oil droplet release. During gastric digestion, the bolus of the gel containing 10 mM NaCl (soft gel) disintegrated much faster than that of the gel containing 200 mM NaCl (hard gel) in the human gastric simulator (HGS). The disintegration of the soft gel in the HGS was caused by both the abrasion and fragmentation while the abrasion was the predominant mechanism of the disintegration of the hard gel. The larger particle size of the soft gel bolus slowed down the emptying of gels from the HGS. With continued digestion, the emptying of both gels from the

HGS was accelerated by gel disintegration. The gel structure greatly influenced the gel disintegration at the micro-scale. The soft gel particles were gradually disrupted into individual oil droplets, with the protein matrix dissolving after gastric digestion for 4 hours while the hard gel particles still retained the oil droplets inside the protein matrix. The colloidal structure of emptied gastric digesta, which generated from original gel structure, still significantly impacted the digestion of whey protein emulsion gels in an *in vitro* intestinal model. In general, the colloidal structure of the emptied gastric digesta of the hard gel hindered the breakdown of gel particles and hydrolysis of oil droplets more effectively than that of the soft gel. The remaining structure within the hard gel particles limited the free motion of oil droplets, which led to a lower degree of coalescence and breakup of oil droplets. Interestingly, coalescence appeared to occur between neighboring oil droplets inside the emptied gastric digesta of the hard gel during intestinal digestion.

The structure of the gels containing 100 mM NaCl became from aggregated particle to emulsion-filled with increasing oil droplet size from 1 to 12 μm. The gel strength also decreased with the increase of droplet size. For the gels containing large oil droplets (6 and 12 μm), oil droplets were released from the protein matrix along with some coalescence during oral processing. During gastric digestion, a higher degree of coalescence of oil droplets occurred and coalesced oil droplets creamed to form a top oil layer. This slowed down the emptying of gels from the HGS. For the gels containing 1 μm oil droplets, most oil droplets still retained in the protein matrix during oral and gastric digestion with minimal instability of oil droplets. In addition, increasing interactions between oil droplets and protein matrix by decreasing oil droplet size hindered the protein hydrolysis.

Overall, this research provides an understanding of the way in which food

disintegrates in the human body and highlights the role of food structure on the digestion of food in the human body. These findings could assist in designing the functional new foods that deliver health benefits (e.g. lipid regulation, encapsulation and release of nutrients) and improving human health related to food digestion (e.g. dysphagia, dyspepsia).

Acknowledgement

When I look back the journey, I remember all the friends and family who have helped and supported me along this long but fulfilling road. It is a rewarding and exciting experience. I have felt their love, generosity, support and friendship in my heart.

First and foremost, I wish to express my sincere gratitude to my chief supervisor (Dr Aiqian Ye) and co-supervisors (Prof Harjinder Singh, Prof Douglas Dalgleish and Dr Mita Lad), for their continuous support, encouragement, insightful comments, hard questions and immense knowledge. Their guidance supported me throughout my study. I really appreciate that they have given me enormous freedom to do research independently. Their deep scientific insights often enlightened me when I was confronted with difficulties. Their dedication, enthusiasm, patentice, motivation and intellectual capacity and critical way of thinking and analyzing in academic areas have been a learning experience. I could not have asked for better role models. I could not be prouder of my academic roots and the dreams which they have given to me.

I wish to thank the Riddet Institute for providing me the financial assistance during my study and funding my project as well as providing travel grants to attend the international conferences. I would like to thank the Massey University Doctoral Completion Bursary for providing me financial assistance in the final stage of my PhD study.

I would like to thank Dr Maria Ferrua for her help in preparing the publication regarding to the gel disintegration in the model stomach. I am thankful to Prof John Bronlund for his constructive suggestions to my oral study. I would like to thank Dr Jianyu Chen for the training and guidance of the use of the confocal laser scanning microscopy. I am thankful to Dr Derek Haisman for demonstrating the human gastric

simulator. I would like to thank Prof Matt Golding to be my examiner of the PhD confirmation as well as his valuable advice and critical comments to my project. I am thankful to Prof Paul Moughan for his encouragement and motivation of my PhD journey. I would like to thank Dr Sophie Gallier, Dr Ashling Ellis, Dr Jaspreet Singh and Dr Simon Loveday for their academic discussions about my project.

I would like to thank Ms Janiene Gilliland, Mr. Chris Hall (Riddet Institute), Ms. Michelle Tamehana, Mr Warwick Johnson, Mr Steve Glasgow and Mr Gary Radford for being excellent lab managers, providing the lab induction, training of instruments, ordering of chemicals, technical help, safety advice and scientific advice. I would like to thank Dr Sylvia Chung and Mr Eli Gray-Stuart for their constructive advice for the application of human ethic approval for my oral study. I would like to thank Dr Peter Zhu, Ms Nok Sawatdeenaruenat, Ms Maggie Zou, Mr Trent Olson and Mr Jack Cui for their help in the lab. I am grateful to Ms Terri Palmer, Ms Ansley Te Hiwi, Ms Felicia Stibbards and Ms Yvonne Parkes for their administrative help and assistance. I would like to thank Mr Matt Levin and Mr Peter Jeffery for their timely help and services in information systems. I am also grateful to Mr Mark Ward, Ms Paula McCool, Ms Willi Twilight and Mr John Henley-King. I am grateful to all the human subjects who attended my oral study.

I would like to thank all the staff and research fellows at the Riddet Institute and the Institute of Food Nutrition and Human Health for their help in my PhD study. My special thanks go to my dear office mates: Dr Anant Dave, Mr Vikas Mittal, Mr Dave Poddar, Dr Jiahong Su, Ms Natascha Stroebinger, Ms Lakshmi Dave, Mr Shakti Singh and Mr Prateek Sharma for their company, support, encouragement, and academic discussion. I would like to thank all my friends in New Zealand and China for their encouragement and support.

And last, but not least, to my wife (Dongfang) and my beloved parents, who share my passion, depression, joy, pain, failure and success during this journey, thank you for your support, encouragement, listening and consoling. I cannot complete this manuscript without you.

Table of Contents

List of Figures

List of Tables

List of Symbols

G'	Shear storage modulus
$D_{4,3}$	Average volume-weighted diameter
$D_{3,2}$	Average surface-weighted diameter
pI	Isoelectric point

List of Abbreviations

α-La	α-lactalbumin
β-Lg	β-lactoglobulin
WPI	Whey protein isolate
Ig	Immunoglobulin
IgG	Immunoglobulin G
IF	Lactoferrin
SA	Serum albumin
BSA	Bovine serum albumin
TAG	Triglyceride
SDS	Sodium dodecyl sulfate
SGF	Simulated gastric fluid
SIF	Simulated intestinal fluid
HGS	Human gastric simulator

List of Peer-reviewed Publications

Guo, Q., Ye, A., Lad, M., Dalgleish, D., & Singh, H. (2013). The breakdown properties of heat-set whey protein emulsion gels in the human mouth. *Food Hydrocolloids*, 33(2), 215-224.

Guo, Q., Ye, A., Lad, M., Dalgleish, D., & Singh, H. (2014). Effect of gel structure on the gastric digestion of whey protein emulsion gels. *Soft matter*, 10(8), 1214-1223.

Guo, Q., Ye, A., Lad, M., Dalgleish, D., & Singh, H. (2014). Behaviour of whey protein emulsion gel during oral and gastric digestion: effect of droplet size. *Soft matter*, 10(23), 4173-4183.

Guo, Q., Ye, A., Lad, M., Ferrua, M., Dalgleish, D., & Singh, H. (2015). Disintegration Kinetics of Food Gels During Gastric Digestion and its Role on Gastric Emptying: an *in vitro* Analysis. *Food and Function*, 6(3), 756-764.

Guo, Q., Ye, A., Lad, M., Dalgleish, D., & Singh, H. (2015). Impact of colloidal structure of gastric digesta on in vitro intestinal digestion of whey protein emulsion gel (In preparation).