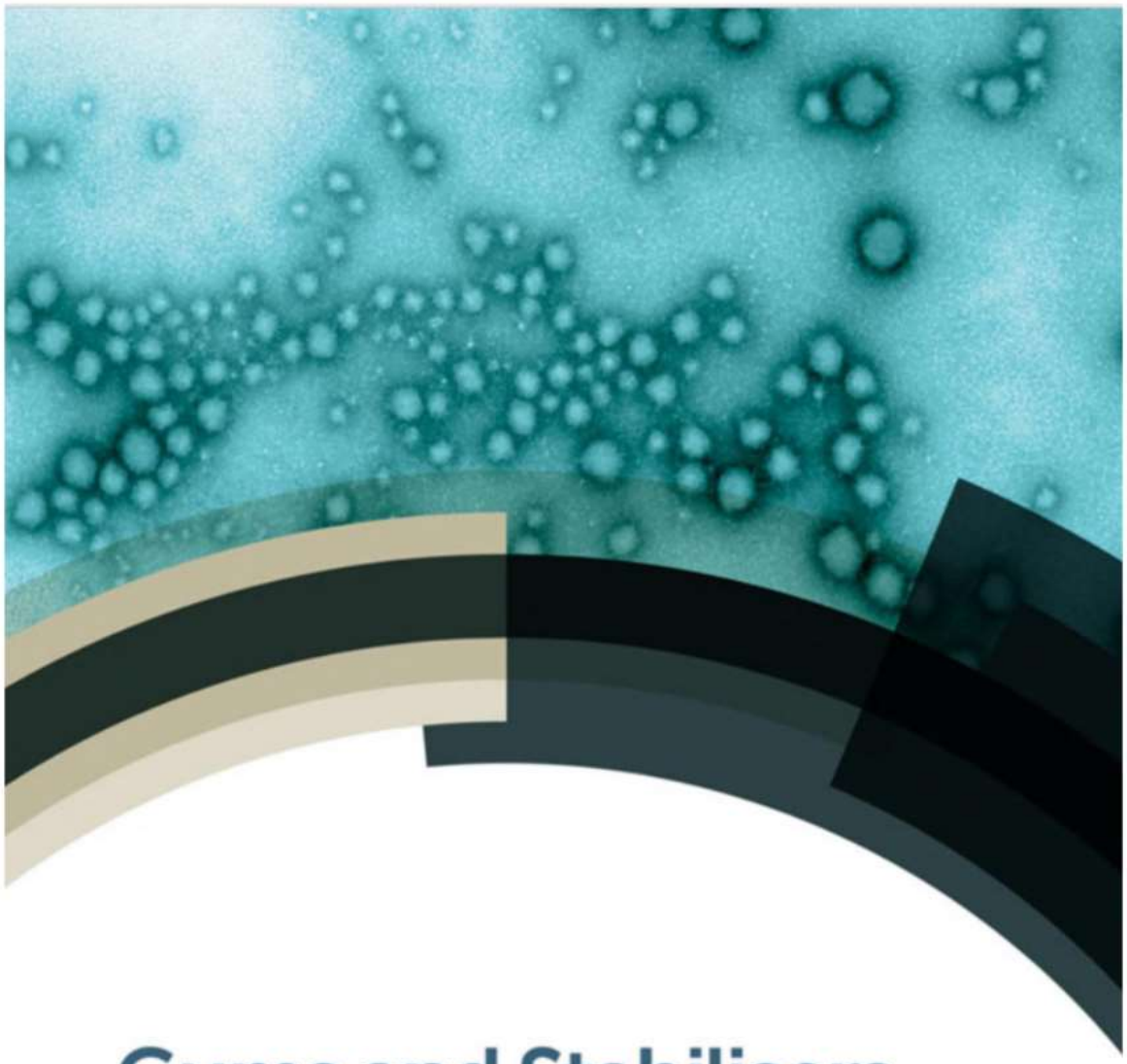

Gums and Stabilisers for the Food Industry 18

Hydrocolloid Functionality for Affordable and Sustainable Global Food Solutions

Edited by Peter A. Williams and Glyn O. Phillips

ROYAL SOCIETY OF CHEMISTRY

PREFACE

It is now approaching 40 years since the first Gums and Stabilisers Conference was held in July 1981. Robert Harrison of Dari Tech Atlanta, the President, opening the first meeting, said that *"one pound of stabiliser is more important for product performance and eating quality of frozen dessert than is 20 pounds of any other ingredient."* The state of the technology at that time was more akin to cooking than the application of functional science.

How has the subject changed since then? A scrutiny of the Proceedings over the subsequent years illustrates how food hydrocolloid science has grown in complexity, understanding, and while introducing new hydrocolloids slowly, is still relying on the main stabilisers of 40 years ago. Xanthan is good example and it is unlikely that the necessary resources will be provided in the current financial industrial climate to produce fundamentally new emulsifiers and texture controllers.

The Proceedings of this Volume 18 provides an excellent state of the art (or science?) that underpins the industrial products of today. In the absence of fundamentally new ingredients more resourcefulness is required to achieve improved product fabrication. And the search for new products continues, but only as a research activity.

The sections of the current volume are:

- CHEMICAL AND PHYSICOCHEMICAL CHARACTERISATION
- EMULSIONS, FOAMS AND FILMS
- ENCAPSULATION AND CONTROLLED RELEASE
- HEALTH ASPECTS
- PRODUCT FORMULATION

The sections of the original volumes were:

- ANALYSIS, STRUCTURE AND PROPERTIES
- GELATION AND RHEOLOGICAL PROPERTIES
- EMULSION STABILISATION
- CURRENT DEVELOPMENTS

There is a familiarity in the language but the present volume illustrates the greater material and technological diversity associated with current research. We now have microstructure and nanostructure processing and the search for new materials such as pectins from pumpkin and collagen from quail feet.

But further exploiting the blends of iota and kappa carrageenan continues.

The search for new emulsification materials and technologies is extended to using ultrasonics, okra pectin, hydrophobically modified inulins and barley-hull hemicelluloses.

But the use of citrus pectins in oil in water emulsions continues.

Controlled release systems have certainly moved on and now have applications to reduce toxic influences and protection against oxidation. However, it is in the health applications area that the contrast is most marked. The benefits of dietary fibre, the lowering of blood glucose, controlling immunomodulatory activity, engineering digestion are new interesting areas.

But the further exploitation of gum arabic continues.

The technology squeeze is extended also to find improved formulations such as beefing up soluble fibre with UHT beverage, adding gelling maltodextrin, cellulose ethers, protein based aggregates and casein micro-particles.

But the search for stability continues.

Continuing too was the great friendly interaction which is a feature of these meetings. The Ruthin Castle Banquet, the Rhos Male Voice Choir concert and chats around the bar made for a happy occasion which by popular acclaim will be repeated and perhaps in another European location.

Mention should be made also of the award of the Food Hydrocolloids Trust Medal to our colleague Claus Rolin. His inspirational presentation showed the benefits which pectin research receives from its industrial usage.

Thanks also to those members of the Organising Committee who worked together to plan and arrange details of the meeting. However, the main thanks must be given to Professor Peter A. Williams who holds all arrangements together in association with our tireless friend of so many years Haydn Hughes.

Thank you all for coming and do keep in touch.

Glyn O. Phillips
Chairman, Organising Committee

Acknowledgements

The Food Hydrocolloids Trust is indebted to members of the conference organising committee for their efforts in arranging this conference which was held in June 2015 at Glyndwr University, Wrexham, UK

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Part 3

Encapsulation and Controlled Release

RELEASE MECHANISM OF ESSENTIAL FATTY ACIDS IN POLYSACCHARIDE MATRICES UNDERGOING GLASS TRANSITION

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ABSTRACT

Identification of theoretical mechanisms governing molecular diffusion of essential fatty acids (oleic and α-linolenic acid) in high solid matrices was carried out on two polysaccharide matrices of high-methoxy pectin and κ-carrageenan in the presence of co-solute glucose syrup and polydextrose, respectively. Physicochemical analysis of this system utilised modulated DSC, dynamic oscillation in shear, ESEM, FTIR and WAX diffraction. The carbohydrate matrices were conditioned through an extensive temperature range to induce changes in molecular morphology and identify the network glass transition temperature. Thermally induced variation in phase morphology was employed to rationalise transportation patterns of the bioactive compound within the high-solid preparation. Thus, experimental observations using UV-vis spectroscopy modelled diffusion kinetics to document the mobility arresting effect of the vitrifying matrix on the micro-constituent. Within the glass transition region, results argue that free volume theory is the molecular process governing structural relaxation. Further, Less Fickian diffusion follows well the rate of molecular transport of the fatty acids as a function of time and temperature of observation in the condensed matrices.

1 INTRODUCTION

Oxidation of essential fatty acids leads to undesirable changes in sensory perception and nutritional profile. Thus, protective and controlled delivery of essential fatty acids in processed foods attempts to significantly improve preservation status. Strategy for lipid preservation has been focused on controlling water and oxygen transports in biopolymer shells[1-3], however, limited information is available on the lipid diffusion in high solid system (>70% total solid).

The novelty behind the current approach elucidates the importance of glass transition temperature to control physicochemical, biological and enzymatic reactions[4]. Structural consistency during glassy state of biopolymers is taken as a limitation parameter on molecular rearrangements thus allows entrapment of small and large molecules[3,7]. Transformation of structural properties within the glass-to-rubber transformation of polymeric biomaterials was suggested to allow a controlled release of bioactive substances, i.e. caffeine and vitamin C[5-6]. The current investigation aims to extend fundamental understanding on the physics and kinetic rates of molecular mobility of essential fatty acids in condensed carbohydrate matrices for further potential applications in added value food and pharmaceutical products.

2 MATERIALS AND METHODS

2.1 Materials

Experiments were conducted in two systems, the first system consisted of 3% high methoxyl pectin and 81% glucose syrup with 1% oleic acid; the second system was 2% κ-carrageenan and 82% polydextrose with 1% α-linolenic acid.

High-methoxy pectin from citrus peel was purchased from Sigma Aldrich Co (Sydney, Australia) with a degree of methyl esterification (DE) of about 65%. Glucose syrup, as the co-solute, was a product of Edlyn Foods Pty Ltd (Victoria, Australia). The total level of solids was 81% with 40-45% of glucose residues present as reducing end groups (dextrose equivalent, DE is about 42). 1-Oleoyl-rac-glycerol, as a source of oleic acid (OA) in its glyceride form, was obtained from Sigma Aldrich Co (Sydney, Australia). It contained 40% monoglyceride and 60% di- and triglyceride mixture (TLC).

κ-Carrageenan was purchased from Sigma Aldrich Co (Sydney, Australia). The polysaccharide is extracted from *Euchema cottonii* type III and used as the basic material for further purification prior to our experimentation[8].
Polydextrose, as the co-solute, was Sta-Lite III powder, supplied by Tate & Lyle ANZ, Pvt. Limited (Decatur, IL). α-Linolenic acid is obtained from Sigma Aldrich Co (Sydney, Australia) with 70% purity with the remaining being 20% linoleic acid and 10% oleic acid.

2.2 Methods

2.2.1 Sample preparation. Polysaccharide solutions (high-methoxy pectin and purified κ-carrageenan) were dispersed in appropriate amounts in Milli-Q water with constant stirring on a magnetic plate at 90°C to form a clear solution within 10-20 min. Temperature was reduced to about 80°C upon mixing of co-solutes but further reduced to 50 and 40 °C, respectively, prior to oleic and linoleic acid, addition. To promote gelation, 2 M HCl was added dropwise to meet pH 3 to high-methoxy pectin/glucose syrup system and 50 mM KCl to κ-carrageenan/polydextrose system.

2.2.2 Rheology measurements. The Advanced Rheometer Generation 2 equipped with magnetic thrust bearing technology (TA Instruments, New Castle, DE) was used. Samples were loaded onto the preheated Peltier plate at 90°C with a 5 mm parallel plate measuring geometry and edges were covered with silicone oil (BDH, 50 cS) to minimize moisture loss. Cooling (followed by heating runs) was performed at 1°C/min to -50°C in controlled strain of 0.01% and constant oscillatory frequency of 1 rad/s (normal force was maintained at 0.05 ± 0.01 N).

2.2.3 Dynamic diffusion of fatty acid in biopolymeric matrices. Two grams of sample containing polysaccharide matrices with fatty acid and a separate 2 ml of ethanol or dichloromethane (CH_2Cl_2), served as the solvent, were kept in experimental temperatures within the range of -30 to 22°C. As the commencement of the experiments, solvents were transferred to the top of matrices forming two immiscible phases. Time zero set upon addition of solvents and samples taken at succeeding intervals of time for 6 hours for the first experiments and 3.5 hours for the second.

Release kinetics was determined by estimating the amount of fatty acid liberated at each time interval using the Sulfo-Phospho-Vanillin (SPV) method and Lambda 35 UV-vis spectrophotometry (Perkin Elmer, Singapore). Aliquots (100 µl) of fatty acid were analysed based on the formation a pink adduct as a result of the reaction between phosphorous vanillin and carbonium ions from the acid digested fatty acid. Total lipid was analysed a set wavelength (λ_{max} = 525 nm). Analysis was carried out in triplicate and average values are reported.

3 RESULTS

2.1 Thermomechanical characterisation

Thermomechanical analysis features prominently in the characterisation of phase and state transitions of biopolymers as a function of time and temperature [9]. Figure 1 shows the thermal history and the manifestation of shear moduli for carbohydrate/lipid system in the temperature range from 90 to -30 °C experiencing rubbery to glass transformations.

As seen in Figures 1-2, storage modulus (*G'*) is well above the loss modulus (*G''*) for both systems suggesting a rubbery plateau in the high temperature range (50-90 °C) following the dramatic increment at 50 °C as the system enters the glass transition region. A further feature of this system is the increase in values of *G'* and sudden drop of *G''*, which demarcates the glassy state of the materials from -15 °C. Vitrification of biopolymer composites indicates glassy state of supercooled structures recorded as the viscous element decreases significantly.

alongside the viscoelastic relaxation of the condensed matrix. The oleic acid mobility produces a good quality linear relationship that allows utilisation of the modified Arrhenius equation to estimate the energy of activation (E_a = 24 kJ/mol). This is contrasted with the corresponding value for the high-methoxy pectin/ glucose syrup system within the glassy state yielding an E_a value of 251 kJ/mol. Similarly, E_a = 20 kJ/mol was recorded for α-linolenic acid and 233 kJ/mol for κ-carrageenan/ polydextrose. Clearly, the energy barrier for structural relaxation of the carbohydrate molecules is much higher than for the diffusional mobility of the fatty acid.

Next, looking deeper into the mobility of the fatty acid by considering a Power Law equation that was first developed from Fick's Law to monitor the sorption/desorption of water molecules in breakfast cereals[13]:

$$\frac{M_t}{M_\infty} = kt^n \qquad (5)$$

where, n is the kinetic diffusion exponent, M_t/M_∞ is the extent of fatty acids release at experimental and equilibrium time, k is a constant characteristic of the bioactive compound-polymer system and t is given in seconds.

Plotting the natural logarithms of M_t/M_∞ *versus* time from data in the first sixty minutes of experimental data in Figure 3, produces the required values of n and k for the various experimental temperatures range. The kinetic diffusion exponents (n) were calculated in this investigations to be from 0.20 to 0.34 for the high-methoxy pectin/ glucose syrup matrix and from 0.26 to 0.35 for κ-carrageenan/polydextrose system. Based on the Fick's Law, the ideal transport mechanism has an n-value equal to 0.5, whereas the rapid Case II transport is characterised by an n-value of 1.0. This makes the range of n-values between 0.5 and 1.0 a Non-Fickian or anomalous diffusion[14]. The low diffusion exponent of these investigations, i.e. Less Fickian with values below 0.5, reflects a low mobility due to the dense state of the vitrified polymeric matrix and the amphiphilic nature of α-linolenic acid that may interact in part with κ-carrageenan and/or polydextrose whilst oleic acid may undergo hydrophobic association with high-methoxy pectin/glucose syrup matrix.

Fick's second law can be further utilised to estimate the diffusion coefficient, D_{eff}, of a small molecule within solid-like macromolecular systems[14]. Simplification of Siepmann & Peppas (2011) equation for samples of slab shape where diffusion occurs through the slab's surface without any edge effects produces the following model[15-16]:

$$\frac{M_\infty - M_t}{M_\infty - M_i} = 4\left(\frac{D_{eff}\ t}{\pi L^2}\right)^{1/2} \qquad (6)$$

where, M_i, M_t and M_∞ denote the absolute amounts of the diffusant compound released at times zero, during experimentation and infinity/equilibrium, respectively, and L is the thickness of the slab.

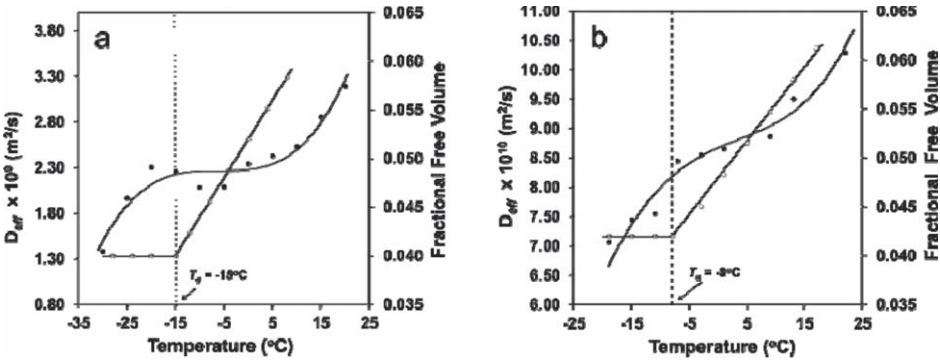

Figure 4 *Fractional free volume (○, trace on the right y-axis) of (a) 2% κ-carrageenan with 82% polydextrose and (b) 3 % high-methoxy pectin with 81% glucose syrup and relative diffusion coefficient of the fatty acids (oleic and linolenic acid, respectively) from these carbohydrate matrices within 60 min of observation (●, trace on the left y-axis).*

Figure 4 reproduces values of the diffusion coefficient over the temperature range that leads to carbohydrate vitrification. This is further compared with the fractional free volume of the matrix producing a discontinuity at the mechanical glass transition temperature. Clearly, vacant spaces amongst the adjacent macromolecules of carbohydrate matrices govern the nature and mobility rate of fatty acid in the vitrified matrix. Phenomenological estimations of the diffusion coefficient follow an approach that has proved to be a mainstay of utility in recording and then modeling experimental data to unveil the kinetics of drug delivery[15]. Furthermore, interest in this work lies in proposing a relationship between free volume of the polymeric matrix and diffusion kinetics of the bioactive compound in the vitrified mixture.

3 CONCLUSIONS

Diffusion processes in manufactured food products are largely dominated by small molecules including oxygen, water and bioactive compounds. The present study examines the transportation mechanism of fatty acids within a carbohydrate matrix that undergoes a thermally induced glass transition. Informed manipulation of thermomechanical parameters and spectroscopic data documented the physics and kinetic rates of the release mechanism. The carbohydrate matrices are of an amorphous nature and exhibit a mechanical glass transition temperature that dictates the diffusional mobility of fatty acid. This is at its lowest within the glassy state where the free volume of the polymeric sample has collapsed to insignificant levels. The kinetic diffusion exponent and diffusion coefficient obtained from Fick's second law document a

controlled release process that is affected by the macromolecular system and the amphipathic nature of the micro-constituents.

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