

# 1 **A comparison of the sensory and rheological properties of different cellulosic** 2 **fibres for food**

3 Deepa Agarwal, Louise Hewson, Tim J. Foster\*

4 Division of Food Sciences, School of Biosciences, University of Nottingham, Sutton  
5 Bonington Campus, Loughborough, LE12 5RD, UK.

6 \*Corresponding author e-mail: [tim.foster@nottingham.ac.uk](mailto:tim.foster@nottingham.ac.uk)

## 7 **Abstract**

8 The impact of different cellulosic microstructures formed by highly entangled fibre networks  
9 were studied for food applications as dietary fibre. This paper reports the impact of  
10 microstructure on the rheological and sensory behaviour of the aqueous suspensions of  
11 particulate and fibrillated forms of softwood cellulosic fibres, and were compared with citrus  
12 fibre. An aqueous suspension of cellulosic fibres shows stable viscoelastic gel-like behaviour  
13 as a function of frequency. The particulate form of cellulosic fibres showed lowest shear  
14 viscosity as compared to the entangled network system at comparable concentrations. To  
15 provide further insight into the relationship between the structure of cellulosic fibre systems  
16 and perception of salt taste in aqueous suspensions of softwood cellulosic fibres (fibrillated and  
17 particulate form) and citrus fibres with matched shear viscosities were studied. A hypothesis  
18 to explain why softwood cellulosic fibre (CTE) with entangled network structure prolongs the  
19 taste perception is presented.

## 20 **1. Introduction**

21 Polysaccharides are known to be used as functional ingredients in a wide range of commercial  
22 applications such as food, personal care and pharmaceutical products. In the food industry,  
23 polysaccharides are used as thickening, gelling, emulsifying, stabilisation and coating agents  
24 [1]. For these purposes, different natural polysaccharides such as starch, carrageenan, guar  
25 gums and bacterial polysaccharides such as xanthan and bacterial cellulose are used. Typically,  
26 a combination of two or more of these hydrocolloids is used to create a variety of

27 microstructures to formulate stable food applications with specific attributes like acceptable  
28 mouthfeel and flavour perception. The processing conditions such as shearing, heating and  
29 pumping of the polysaccharide mixtures result in microstructures with unique rheological and  
30 sensory properties such as fat mimicking, and texture enhancement [2,3,4]. These  
31 polysaccharides are typically categorised as dietary fibres in the food and pharmaceutical  
32 industry. By definition, according to American Association of Cereal Chemists in 2000, dietary  
33 fibres are referred to as the edible parts of plants or analogous carbohydrates that are resistant  
34 to digestion and absorption in the human small intestine with complete or partial fermentation  
35 in the large intestine [5]. Fibres are often classified as soluble dietary fibre and insoluble dietary  
36 fibre [6]. These dietary fibres may consist of non-digestible carbohydrates, cellulose and lignin  
37 that are an intrinsic part of a plant cell wall [7]. Depending on the source of fibres the amount  
38 of soluble and insoluble components vary, for instance, the dietary fibre from fruits and  
39 vegetables contains considerably higher proportion of soluble fibres, whereas cereal, peel of  
40 fruits or other crops contain more insoluble components such as cellulose and hemicellulose  
41 [8]. Dietary fibres play an important role in human health, as it was reported in past that high  
42 dietary fibre diets are associated with the prevention, reduction and treatment of some diseases,  
43 such as reducing cholesterol and maintain gastrointestinal health [9, 6].

44 The dietary fibre produced from citrus fruit such as CitriFi and Herbacel AQ+ are widely used  
45 in various dairy products as a fat replacer, in low-fat mayonnaise, salad dressing and ice-  
46 creams, providing fibre frameworks to improve water-holding capacity and therefore acting as  
47 a thickening agent. Whereas in bakery products such as biscuits, croissants and muffins *etc.*  
48 these fibres are used as fat and calorie reducing agents without compromising taste, texture and  
49 cost [8, 10]. The rheological parameters such as flow behaviour and viscoelastic behaviour of  
50 the different food applications or model systems can be correlated with the sensory texture  
51 properties and stability of the products [11, 12]. Depending on the source, type and  
52 concentration of fibres used in the product, different rheological and textural properties can be

53 achieved. For instance, the presence of xanthan gum improves the texture and physical shelf-  
54 life of oil-in-water emulsions such as a salad dressing. The citrus fibre in combination with  
55 another stabiliser such as xanthan or LBG improve the physical, chemical and sensory  
56 properties of ice-cream samples [13, 14]. Similarly, a number of other cellulosic fibres are used  
57 in the food industry such as MCC (microcrystalline cellulose), CMC (carboxymethyl cellulose)  
58 and the other chemically derivatised celluloses such as methylcellulose.

59 Rheologically, a number of studies showed that an aqueous suspension of citrus fibres and  
60 cellulosic fibres such as MCC or MFC (microfibrillar celluloses) shows gel-like behaviour  
61 where the storage modulus ( $G'$ ) is higher than the loss modulus ( $G''$ ) over a wide concentration  
62 range. These moduli show little frequency dependence at all concentrations [15, 16, 17, 18,  
63 19]. The higher elastic modulus might be due to long fibrils and fibrillar-aggregates and  
64 entangled microfibrils, forming strong network structures. These aqueous suspensions of  
65 different cellulosic fibres also exhibit shear thinning behaviour [16, 20]. Similar rheological  
66 properties were observed by an aqueous suspension of dietary fibres extracted from tomato  
67 peel/pomace and date [21, 22]. The objective of the current publication is to provide an insight  
68 into the rheological properties of microfibrillar cellulosic fibre extracted from softwood spruce  
69 and understand the fundamental differences between the different cellulosic fibre  
70 microstructures and their functionality in food.

71 In recent years, an increase in demand for the low-salt food products was reported by the food  
72 industry, due to continuous awareness from health professionals. Associations have been made  
73 between a high sodium diet and an increased risk of certain health conditions such as  
74 hypertension and cardiovascular disease [23, 24]. Hence, the World Health Organisation  
75 (WHO) recommendations for a daily salt intake limit of 5g, recognising that many consumers  
76 exceed this limit approx. > 10g [26]. However, salts play many important roles in food products  
77 not just as a tastant enhancing flavour, but also affecting physical properties, shelf-life and

78 finally prevention of fermentation (Lynch *et al.*, 2009). Different salt replacement work has  
79 been presented in the past such as in bread; salt (NaCl) replaced with potassium or magnesium  
80 salts resulted in unpalatable metallic, bitter and off-taste [27]. Rama (2013) showed that the  
81 size of salt crystals influenced the rate of salt perception. It was reported that the larger salts  
82 crystals dissolved relatively slowly as compared to small salt crystals, this prolongs the  
83 duration of the taste perception. Ultimately, the smaller salt crystals meant less salt was  
84 required to achieve a similar level of salty taste [28]. It is well established that as the viscosity  
85 of the hydrocolloid thickened product increases, the flavour perception of the product decreases  
86 especially when the concentration of hydrocolloid exceeds the critical overlap concentration  
87 *i.e.*  $c^*$  [29, 30, 31]. This decrease in flavour perception is due to a reduction in the amount of  
88 tastants reaching the sensing organs [32] due to an increase in viscosity of the system.  
89 Depending on the type of hydrocolloid used in the product a noticeable impact on flavour and  
90 taste perception is observed, for instance, the products thickened with starch showed good taste  
91 and flavour perception as compared to product thickened with xanthan [33, 34].

92 The primary aim of this study then is focused on understanding the impact of highly entangled  
93 networks of cellulosic fibre from various sources and their impact on rheological properties of  
94 the suspension. It is hoped that this understanding will shed light on the potential application  
95 of cellulosic fibres extracted from softwood (spruce) in food applications. A detailed study of  
96 microstructure was performed by using light microscopy and correlated with water retention  
97 capacity and rheological behaviour of the suspensions. The second objective of the work was  
98 to test the impact of the highly entangled network of cellulosic fibres on overall taste (sensory)  
99 perception from a basic food model system composed of cellulosic fibres, water and salt. A  
100 detailed study of rheological behaviour and the sensory perception of the cellulosic fibres,  
101 when correlated with light microscopy, as presented here, will enable important structural  
102 features of these cellulosic materials to be identified which are of relevance to the food and  
103 personal care industries alike. The hypothesis underpinning this research is that the highly

104 entangled network microstructure of cellulosic fibres are responsible for higher water retention  
105 capacity which also reflects significantly on higher rheological properties and lowers the taste  
106 (sensory) perception.

## 107 **2. Materials and methods**

### 108 *2.1. Materials*

109 For this study different food grade, cellulosic fibres: citrus fibres CF100 and CFAQ+ were  
110 provided by Cybercolloids, Ltd (Ireland) and CTE (Flakes and Powder form, and are composed  
111 on softwood spruce cellulose (CTE) and carboxymethyl cellulose (CMC)) was provided by  
112 Borregaard AS (Norway). Reverse osmosis (RO) water was used for all sample preparation.  
113 Sensory data was collected using FIZZ 2.0 software (Biosystems, Couternon, France).

### 114 *2.2. Sample preparation*

115 All cellulosic fibres were dispersed in RO-water by using a high shear Ultra-turrax  
116 homogeniser at 18000rpm for 4 minutes at different concentrations (between 0.1% - 2.5%  
117 w/w). All the samples were left to hydrate overnight on roller bed (60rpm speed) at ambient  
118 temperature before analysis. The concentration of all the sample was checked by using OHAUS  
119 MB25 moisture analyser (OHAUS, US). All samples were freshly prepared in two batches and  
120 analyses were made in duplicate. For sensory analysis different cellulosic fibres (*i.e.* CTE  
121 (flakes), CTE (Powder), CF100 and CFAQ+) were dispersed in 0.2% NaCl stock solutions at  
122 different concentrations at comparable viscosities *i.e.* high (0.2Pas) and low (0.01Pas) at 50s<sup>-1</sup>  
123 shear rate (summarised in Table 1) and also at constant concentration (1.5% w/w). All samples  
124 were mixed by using a high shear mixer (Silverson, UK) at 5000rpm for 5mins. All samples  
125 were stored at 4°C overnight and stirred well before serving to panellists. For sensory analysis,  
126 all ingredients are commercially available and commonly used in a variety of food products.  
127 Prior to sensory evaluation, all panellists were informed of the ingredients and any possible  
128 allergens highlighted in accordance with local Sensory Centre procedures.

129 **Table 1:** *Different concentrations (Concn %) of cellulosic fibres and corresponding shear*  
 130 *viscosity (at 50s<sup>-1</sup>) used for sensory analysis.*

Sample	High Viscosity		Low viscosity	
	Concn (%)	Shear Viscosity (Pas)	Concn (%)	Shear Viscosity (Pas)
CTE (F)	1	6.56	0.2	0.194
CTE (P)	1.5	5.59	0.5	0.0918
CFAQ+	1	5.35	0.8	0.0812
CF100	2	7.45	0.5	0.0918

131

### 132 2.3. Rheological Analysis

133 The rheological measurements were carried out on a stress-controlled Rheometer (Physica  
 134 MCR 301, Anton Paar, Austria) with a serrated parallel plate (50mm diameter with a gap of  
 135 1mm) at 20±1°C, controlled by a Peltier system. Small oscillation amplitude sweeps were  
 136 generated by log ramping strain 0.01-100% at a constant frequency of 1Hz. Frequency sweeps  
 137 were performed over the frequency range 0.1-15Hz at a constant strain of 0.2% which lay  
 138 within the linear viscoelastic region. Rotational measurements were performed by increasing  
 139 the shear rate from 0.01-1000 1/s log. Data presented is an average of four replicates.

### 140 2.4. Sensory evaluation

141 Panellists (n= 74, aged 20–40, mixed male and female volunteers) were recruited from the  
 142 University of Nottingham staff and students. The four samples (CTE (flakes), CTE (Powder),  
 143 CF100 and CFAQ+) were compared for saltiness using a round robin of paired comparison  
 144 (PC) tests (BS EN ISO 5495:2007), such that each sample was evaluated against every other  
 145 sample within the set of four, ensuring a total of 6 paired comparison tests. Three separate  
 146 sessions were performed to examine the saltiness perception at a low viscosity (0.1Pas,  
 147 Panellist: 74), high viscosity (6Pas, Panellist: 74) and at matched fibre concentration (1.5%  
 148 w/w, Panellist: 60). The sample size was 10ml throughout and samples were served at room  
 149 temperature (20±1°C). For each test the panellist had to take the whole sample in their mouth,

150 allow the sample to coat the roof of their mouth, hold in the mouth for a minimum of 5 seconds  
151 before swallowing, and then cleanse their palate with unsalted crackers (99% Fat Free,  
152 Rakusen's, Leeds, UK) and mineral water (Evian, France) before tasting the next sample.  
153 Panellists were instructed to determine which of the 2 samples was highest in 'saltiness'. Rest  
154 breaks were given between every 3 paired comparison tests. The test was used in forced-choice  
155 mode, so panellists were required to give an answer even if the perceived difference was  
156 negligible. Panellists were asked to provide additional comments regarding any other  
157 differences between the samples. All tests were carried out at the University of Nottingham's  
158 Sensory Science Centre, within individual sensory booths under controlled temperature and  
159 humidity. Testing was performed under red light in order to minimise any small differences in  
160 sample colour not relevant to the test. All the experiments were performed in compliance with  
161 UK legislation (ISO standards), and in accordance with the institutional framework and  
162 practices established by the University of Nottingham Ethics Committee. All participants  
163 received written information about the study before giving their informed consent.

#### 164 *2.5. Microscopic analysis*

165 Light microscopy of all aqueous suspensions of samples was performed by using Olympus  
166 BX5 bright field light microscopy at 20X magnification, scale bar 200µms, all fibres were dyed  
167 using Congo red dye (Sigma-Aldrich, UK).

#### 168 *2.6. Water Retention Values (WRV):*

169 Approximately 0.1g (A) of powder was added to 100g water and mixed with an Ultra-turrax  
170 for 4mins at 18000rpm. The mixture was placed in a centrifuge tube and allowed to rest for  
171 2hrs followed by centrifugation (Beckman Centrifuge machine, Model: J2-21) for 30mins at  
172 2141g. The top water layer was removed and the bottom layer weighed (B). This was done in  
173 duplicate, and WRV was calculated by using Equation 1 (Eq.1).

174 Calculation:  $WRV (\%) = (\text{Bottom layer (B)-starting material (A)})/\text{starting material (A)}$  Eq. 1

## 175 2.7. Data Analysis

176 Sensory data were collected by using FIZZ 2.0 sensory software (Biosystems, Couternon,  
177 France) and statistical analysis was performed by using Friedmans approach (significant level  
178  $\alpha=0.05$ ). Rheology and WRV data were analysed by using ANOVA. The main purpose of the  
179 ANOVA test is to identify and quantify the factors which are responsible for the variability of  
180 the response.

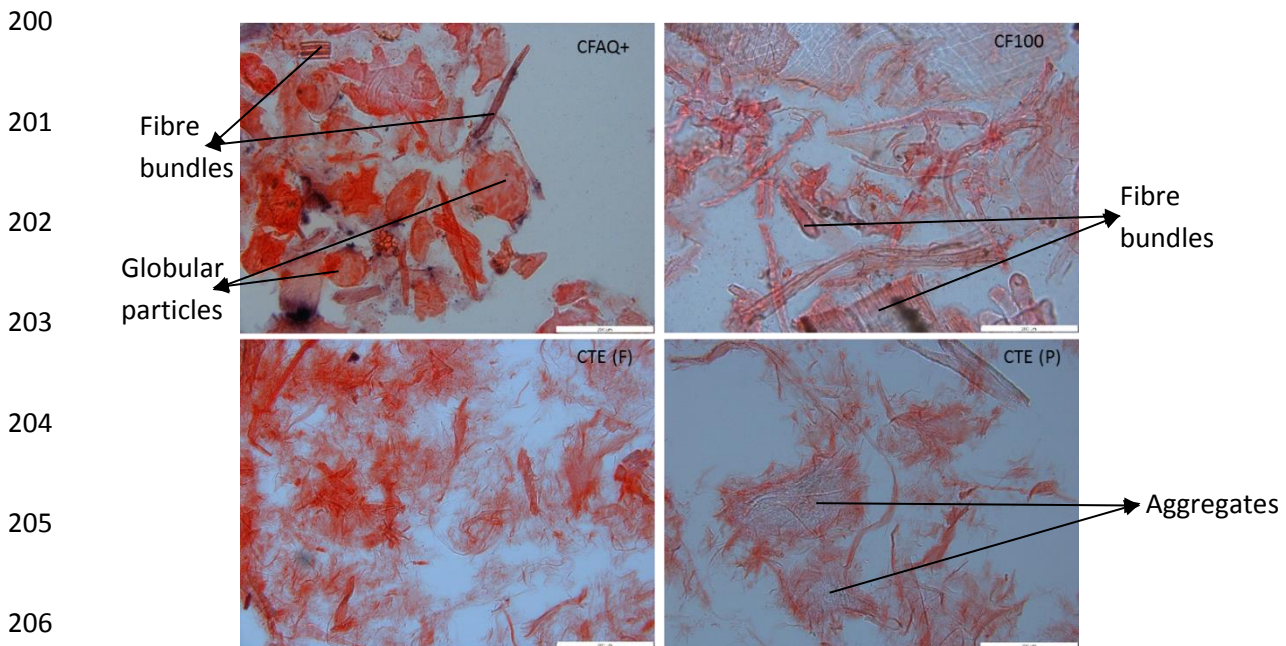
## 181 3. Results and Discussion

### 182 3.1. Microstructure of cellulosic fibres:

183 Light microscopy images of different cellulosic dietary fibres at 1.5% w/w concentration are  
184 presented in Figure 1. A noticeable difference in the microstructure was observed while  
185 comparing CTE(F) or CTE(P) with the citrus fibres CF100 and CFAQ+. The aqueous  
186 suspension of CTE(F) showed a dense entangled fibre network whereas larger aggregates and  
187 fibre bundles were observed with CTE(P). CTE(P) samples were produced by further milling  
188 process of CTE(F) product. During the milling process, the system exhibits slight moisture  
189 loss, hence fibres form strong intermolecular interactions (common phenomena known for  
190 cellulosic fibres upon drying or moisture loss with an increase in temperature), which explains  
191 the noticeably higher amount of fibre aggregates upon hydration. The aqueous suspension of  
192 citrus fibres *i.e.* CF100 and CFAQ+ showed multiple components (both soluble and insoluble)  
193 in the system such as short fibre bundles of fibre, globular structures which are believed to be  
194 pectin and other cell wall material (similar microstructures were observed by Córdoba *et al.*,  
195 2010 with lemon fibres). Larger cellulosic fibre bundles and noticeably less interconnected  
196 fibre-network were observed in the case of both CFAQ+ and CF100 (Figure 1). These highly  
197 entangled fibre network microstructures are responsible for the noticeable difference in both



198 water retention value (also known as water retention capacity) and rheological properties of the  
199 suspensions, discussed below.

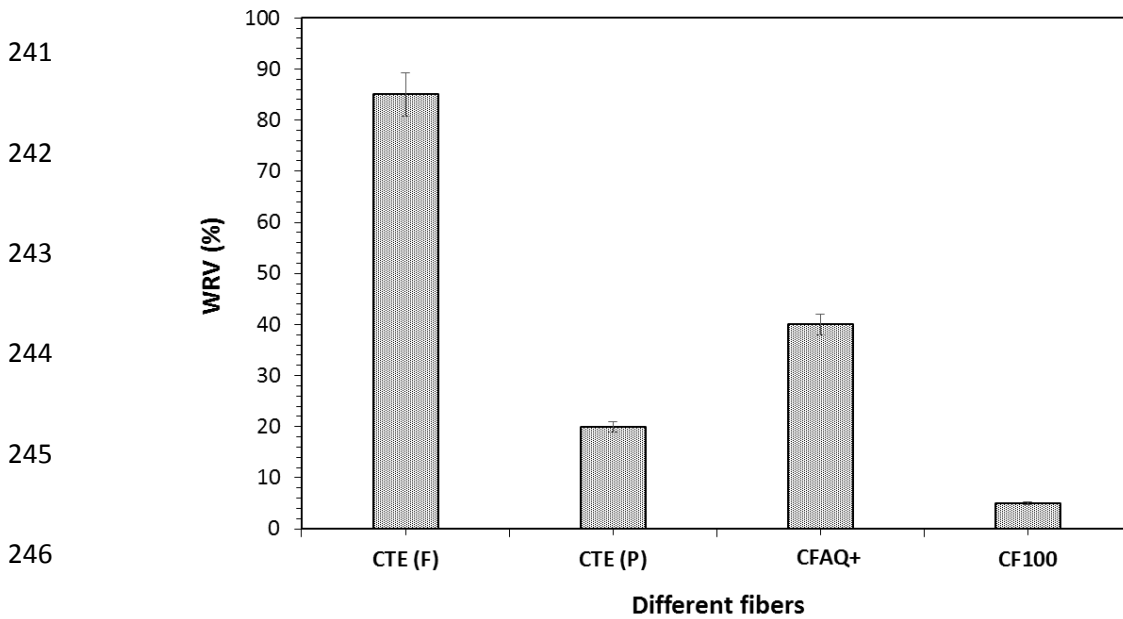


207 **Figure 1:** *Light microscopy images of 1.5% aqueous suspension of different cellulosic fibres*  
208 *stained with Congo red dye, scale bar: 200µms.*

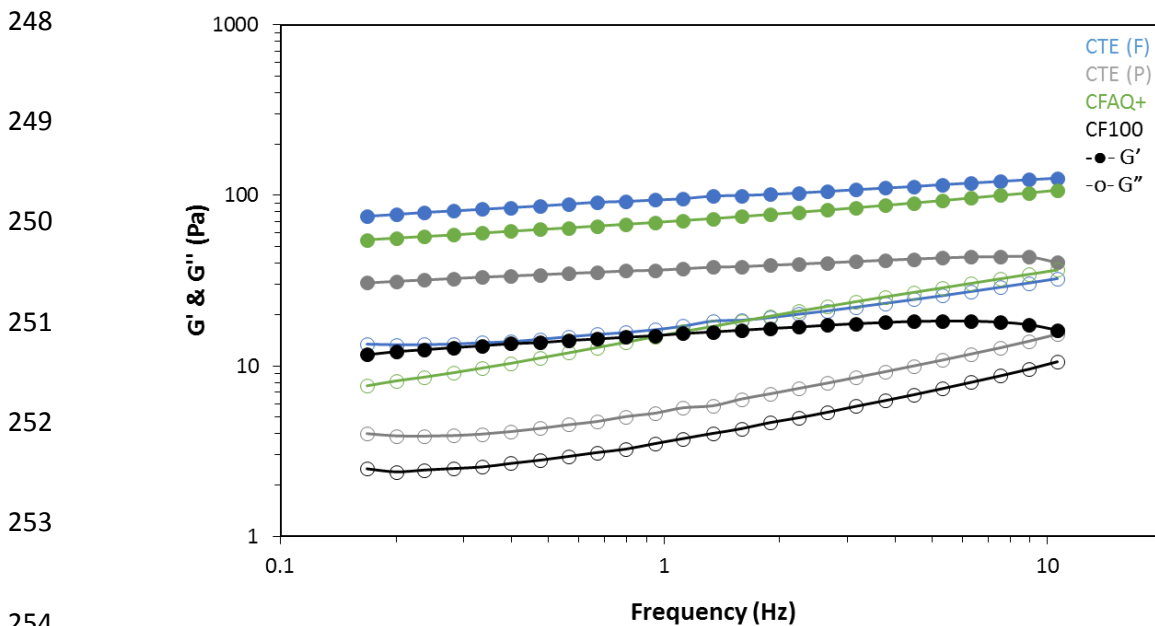
209 Water retention value (WRV) is an important property of dietary fibres from both a  
210 physiological and technological point of view. It helps in understanding the behaviour of  
211 dietary fibres in food applications or during gut transit. WRV of the different cellulosic fibres  
212 are presented in Figure 2, a significant difference ( $p$ -value $<0.05$ ) in WRV was observed when  
213 comparing the softwood cellulosic fibre (CTE(F) and CTE(P)) and citrus fibres (CF100 and  
214 CFAQ+). This significant difference in the WRV can be explained by the different  
215 microstructures, inherent formulation (soluble and insoluble components present in the system)  
216 and different processing. It is well established that the processes such as drying, grinding,  
217 heating or extrusion process modifies the physical properties of the fibre matrix and hydration  
218 properties [35, 36]. Sangnark & Noomhorm (2003) and Elleuch (2011) reported that the  
219 grinding can damage the regions of potential water retention capacity and, therefore, decrease  
220 the capacity to hold water [37, 38]. This explains why lower amounts of water were retained

221 in the CTE(P) fibre-network (milled product) whereas higher amounts retained in CTE(F)  
222 network structures. A slight loss of moisture during the process might have introduced different  
223 intermolecular interactions, resulting in larger amount of aggregates (evident in Figure 1), these  
224 aggregates are difficult to hydrate hence reducing the water retention capacity. Whereas lower  
225 WRV of citrus fibres suspensions can be explained by larger fibre bundles, a noticeably less  
226 interconnected fibre network and the presence of other soluble and insoluble components  
227 present in the case of CFAQ+ and CF100 (Figure 2 and Figure 1; similar behaviour with other  
228 citrus fibres was reported by Grigelmo-Miguel *et al.*, 1999 [39]). Interestingly, the WRV of  
229 CFAQ+ was higher than CF100 this can be explained by two factors *i.e.* (1) inherent differences  
230 in the soluble and insoluble content in the formulation, and (2) entangled network structure  
231 formed during the processing of these fibres. The difference due to the formulation correlate  
232 well with WRV reported with orange dietary fibres [39], lime peel [40], mango dietary fibre  
233 [41], peach dietary fibre [42] and carrot dietary fibre [43]. Also, it is evident from the  
234 microstructure of citrus fibres in Figure 1, that the CFAQ+ has slightly smaller fibre size and  
235 is much more entangled than CF100, hence affecting the water retention capacity of the fibres.  
236 It is well established that the hydration and water retention capacity of dietary fibres are very  
237 important factors in the food industry as these factors can influence the ingredients  
238 functionality, shelf life and product yield [44, 45]. The high WRV of CTE(F) suspension

239 suggested that the material could be used as a functional ingredient in food applications just  
 240 like the industry established citrus fibres.



247 **Figure 2:** Water retention values (WRV %) of different cellulosic fibres.

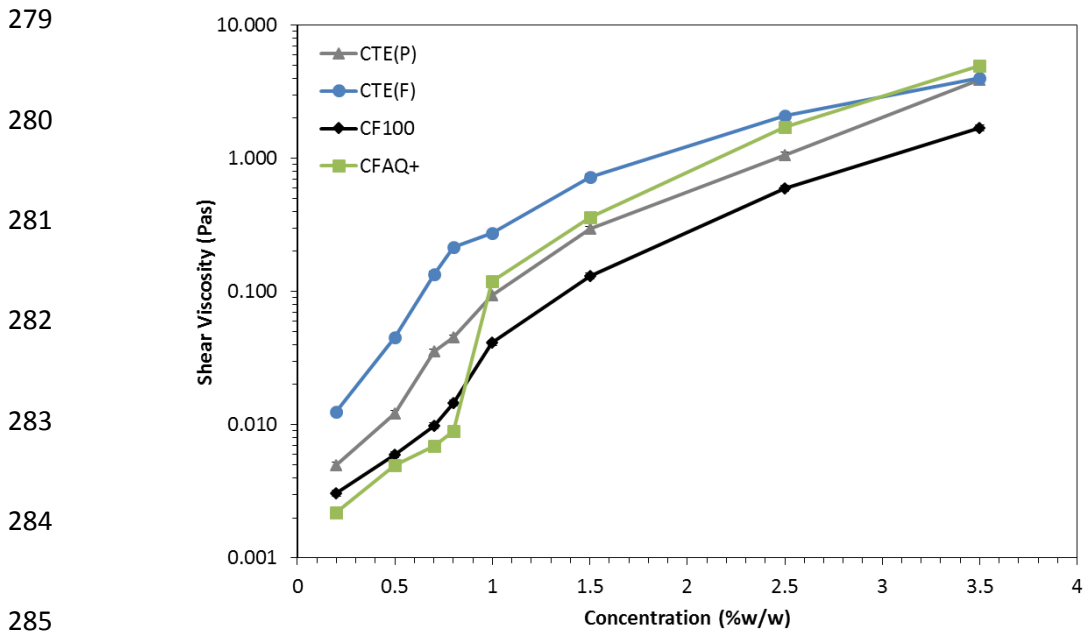


255 **Figure 3:** Dynamic mechanical spectra of 1.5% w/w aqueous suspension of CTE(F), CTE(P)  
 256 and citrus fibres i.e. CFAQ+, CF100, where storage modulus (solid symbols) and loss modulus  
 257 values (unfilled symbols) are represented as a function of frequency measured at 20±1°C.

258 **3.2. Rheological properties of cellulosic suspensions**

259 **3.2.1. Viscoelastic behaviour:**

260 Storage modulus ( $G'$ ) and loss modulus ( $G''$ ) as a function of the frequency of an aqueous  
261 suspension of CTE (F and P) and citrus fibres *i.e.* CFAQ+ and CF100 are presented in Figure  
262 3. All suspensions showed viscoelastic gel-like behaviour, where storage modulus was higher  
263 than loss modulus with little dependency on frequency. Similar behaviour was observed with  
264 lemon fibres by Cordabo *et al.*, 2010 [17] and softwood cellulosic fibres by Tatsumi *et al.*,  
265 2007 [19]. Slight dependency of  $G'$  &  $G''$  on the frequency indicates that the network structure  
266 formed by cellulosic fibres (independent of source) is in an active mode of forming  
267 entanglements to form a stable network of fibres, producing a suspension with gel-like  
268 properties. Chen (2013) suggested that high frequency increases the mobility of microfibers in  
269 aqueous suspension, this increased mobility of the microfibers results in increases the  
270 entanglement and formation of densely ordered network structure which reflects on viscoelastic  
271 behaviour [46] . At a comparable concentration of 1.5% w/w, the elastic moduli of CTE(F) was  
272 highest, where  $CTE(F) > CFAQ+ > CTE(P) > CF100$ , following the same trend for WRV (Figure  
273 2), and visually explained when considering the highly entangled network of the CTE(F)  
274 aqueous suspension (Figure 1). The aqueous suspension of CF100 showed the lowest moduli,  
275 WRV values and have relatively large and discrete fibre particulates in the matrix, which  
276 explains the weak viscoelastic behaviour of the suspension. Whereas, CTE(F) flakes show  
277 higher moduli when compared to CTE(P) powder form, and can be explained by the retention  
278 of a more fibrillated structure (Figure 1) resulting in higher water retention values (Figure 2).



286 **Figure 4:** Concentration dependence of shear viscosity (Pas) recorded at shear rate  $50s^{-1}$  for  
 287 four different cellulosic fibres, where (○) CTE(F), (Δ) CTE(P), (◇) CF100, and (□) CFAQ+.

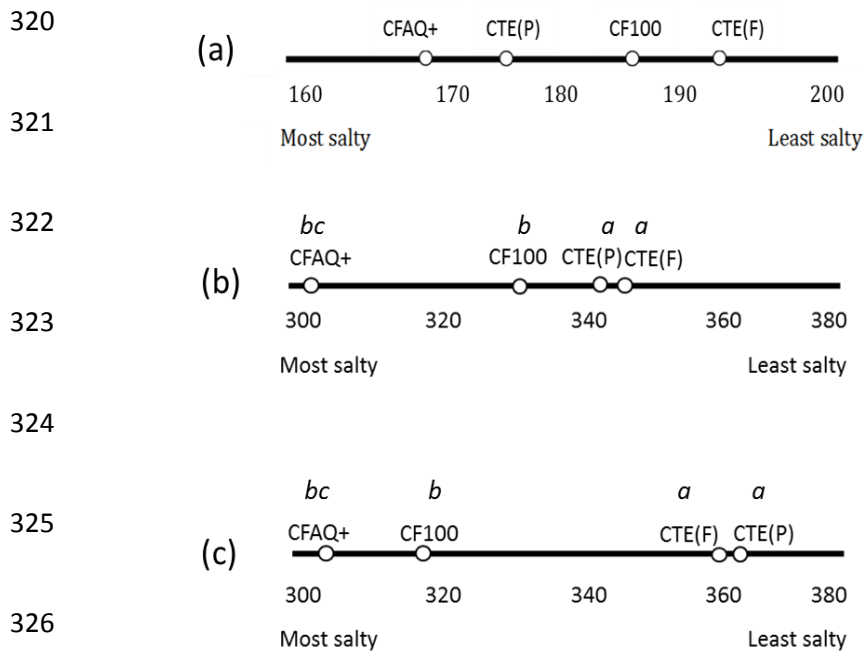
288 **3.2.2. Concentration dependence of shear viscosities:**

289 Shear viscosity recorded at a shear rate of  $50s^{-1}$  as a function of concentration is presented in  
 290 Figure 4, and again shows the trend  $CTE(F) > CFAQ+ > CTE(P) > CF100$ , indicating that the  
 291 shear viscosity is dependent on the source, processing and microstructure of cellulosic fibres.  
 292 At the highest concentration studied (3.5% w/w) the CFAQ+ showed a higher viscosity than  
 293 CTE(F), indicating that when the dispersions become highly packed the insoluble particles of  
 294 CFAQ+ become dominant in the measured viscosity outcome, and the entangled nature of the  
 295 CTE(F) is less effective at providing a measured viscosity. From a colloidal point of view, this  
 296 then may be considered as an effect of ‘hard’ versus ‘soft’ and deformable particles. However,  
 297 for some food applications such as ice-cream, mayonnaise, salad dressings *etc.*, a maximum  
 298 concentration of 0.8% w/w is recommended for citrus fibres considering the sensory perception  
 299 without any off-flavour, body and texture defects [13]. Considering the maximum  
 300 concentration 0.8% w/w for certain application, CTE(F) showed higher shear viscosity as  
 301 compared to other cellulosic fibres (Figure 4). These results indicate that to achieve specific

302 target viscosity (in the range of normal liquid-like foods) a lower concentration of CTE(F) is  
303 required as compared to other fibres(Figure 4 and Table 1). Such results of being able to match  
304 the viscosity of the different fibres in model systems can be considered for the purpose of  
305 investigating whether the inherent properties of the materials themselves can have an impact  
306 on sensory characteristics of texture and taste perception analysis.

### 307 *3.3. Sensory perception*

308 Figure 5 summarises the results from the sensory panel at the same concentration and matching  
309 viscosities (low and high viscosity) of four cellulosic fibres. In figure 5a, no significant  
310 differences (p-value > 0.05) was found in saltiness perception of the four product at the same  
311 concentration (1.5%w/w). This can be attributed to the fact that at this concentration, where  
312 differences in moduli and WRV were seen, all measured viscosities, at  $50\text{s}^{-1}$ , were  $>500\text{mPas}$ .  
313 This is significantly higher than the viscosity known to be important for decreasing the taste  
314 perception in entangled polymeric systems [29, 30], above critical concentration ( $c^*$ ), and  
315 therefore at these higher viscosities, the effect of the fibre type is not apparent. Figuerola  
316 (2005) showed that texture was strongly dependent on the particle size in the case of citrus  
317 fibres [47]. Due to a noticeable difference in the microstructure of all four fibres at the same  
318 concentration, as expected, panellists reported CTE(F) suspensions were much thicker as  
319 compared to other cellulosic fibres suspensions (results not shown).



**Figure 5:** Rank sum scores of each sample (CFAQ+, CF100 and CTE(F) and CTE(P)) for saltiness perception, where a decreasing numerical value corresponds to an increase in the attribute. (a) At constant concentration i.e. 1.5% w/w, (b) At matched high viscosity (0.2Pas), and (c) At matched low viscosity (0.01Pas) salt suspensions [a, b, c represents the statistical significance, where the same letter indicates no significant difference, different letters indicate a significant difference with  $p$ -value  $< 0.05$ . Note: \*bc indicates that no significant difference between CF100 and CFAQ+, but there is a significant difference ( $p$ -value  $< 0.001$ ) between CFAQ+ and CTE fibres.

It was evident from the rheological analysis in Figure 4, that an aqueous suspension of CTE(F) shows higher shear viscosities and this difference in shear viscosity explains the difference in thickness perception noted by the panellists during sensory analysis of 1.5% w/w suspension of different fibres. In the comments section, most of the panellists found a strong off-taste (described as ‘citrus/lemon taste’) with CF100 and little off-taste with CFAQ+ suspensions. Whereas the absence of such off-taste was reported by the panellists (evident with no comments from the panels and even some panel mentioned no-off-taste when comparing with CF100 and CFAQ+) in the case of CTE(F) and CTE(P) suspensions.

343 In order to remove the impact of the difference in viscosities at the same concentration,  
344 different suspensions were formulated with matched viscosities (low and high) but different  
345 fibre concentrations. Using the paired comparison test, it was found that at high viscosity, a  
346 significant reduction ( $p$ -value  $< 0.05$ ) in saltiness perception was observed with CTE(F) and  
347 CTE(P) suspensions as compared to CF100 and CFAQ+ (Figure 5b). No significant difference  
348 in terms of saltiness perception was observed between CTE(F) and CTE(P) as  $p$ -value  $> 0.05$ .  
349 Similar taste perceptions were observed with the suspensions at lower viscosities of different  
350 cellulosic fibres (Figure 5c). The granular suspension of CF100 and CFAQ+ is believed to be  
351 the cause of their higher saltiness perception, in line with similar behaviour found in particulate  
352 suspensions such as starch and xanthan, in that if the granular structure was maintained during  
353 processing, the system does not reduce the taste perception at high concentration [33, 34, 48].

354 A significant difference in saltiness perception and thickness between CTE products and citrus  
355 fibres (CFAQ+ and CF100) can be explained by the dense network structure afforded by the  
356 fibrillated cellulose – now acting more like a polymeric solution, resulting in reduced taste  
357 perception. While the differences in taste perception were significant between the CTE samples  
358 and the citrus samples, for both high and low viscosity, the positioning on the rank sum scoring  
359 for the higher viscosity systems was narrower. This then also indicates that all systems at the  
360 higher viscosities tend to behave as a concentrated dispersion, exemplified at the higher  
361 viscosities seen for the 1.5%w/w samples, where there was no difference seen between  
362 samples. In summary, it was evident from the sensory analysis, that the CTE samples with  
363 highly entangled network structure lowers the taste perception as compared to particulate  
364 suspensions such as CF100 and CFAQ+.

#### 365 **4. Conclusions**

366 The influence of a highly entangled fibre network of cellulosic fibres on the rheological  
367 properties of a suspension is consistent with water retention values of these fibres and a key



368 factor which may responsible for lower taste perception. Rheological measurements show that  
369 all cellulosic suspensions showed viscoelastic gel-like behaviour, due to highly dense fibrillar  
370 and particulate networks affording high water retention capacity. The difference in  
371 microstructures and inherent composition of different cellulosic fibres are responsible for  
372 difference in sensory (tastant) perception. Aqueous salt suspension at matched viscosities of  
373 softwood cellulosic fibre samples showed lower saltiness perception as compared to citrus  
374 fibres. It appears that the particulate structure releases the tastant more effectively and faster as  
375 compared to highly fibrillated and networked systems. The results presented in this paper  
376 clearly highlights that the choice of dietary fibre needs to be made carefully when considering  
377 the application in food products. A highly entangled network microstructure of cellulosic  
378 fibres, responsible for higher water retention capacity greater rheological properties may be  
379 beneficial for certain structural and nutritional aspects of food products, but if taste release is  
380 of importance, for increase sensory perception, then a fibrillated fibre would not be preferred  
381 over a more particulate material.

### 382 **Conflict of Interest**

383 There are no conflicts of interest to declare.

### 384 **Acknowledgement**

385 This work was supported by the Oslofjordfond, Norway grant scholarship (2012 - 2015).  
386 Special thanks to the Sensory Science Centre (the University of Nottingham) and Wenting Yin  
387 for help in running sensory sessions and all participants for their contribution.

### 388 **References**

389 [1] T.J. Foster. Technofunctionality of hydrocolloids and their impact on food structure. Gums  
390 and stabilisers for the food industry 15, Williams, P.A., and Phillips, G.O. (Eds). RSC:  
391 Cambridge, 2010, 103-112.

- 392 [2] V. Tolstoguzov. Some thermodynamic considerations in food formulation. Food  
393 Hydrocolloids. 2003, 17:1, 1-23.
- 394 [3] J. L. Kokini, J. B. Kadane and E. L. Cussler. Liquid texture perceived in mouth. Journal of  
395 Texture Studies. 1977, 8:2, 195-218.
- 396 [4] C. M. Christensen. Oral perception of solution viscosity. Journal of Texture Studies. 1979,  
397 10:2, 153-164.
- 398 [5] B.C. Tunland, D. Meyer. Non-digestible oligo- and polysaccharides (dietary fibre): their  
399 physiology and role in human health and food. Comprehensive Reviews in Food Science and  
400 Food Safety. 2002, 1, 73-92.
- 401 [6] S. Gorinstein, Z. Zachwieja, M. Folta, H. Barton, J. Piotrowicz, M. Zember, M. Weisz, S.  
402 Trakhtenberg, O. Martin-Belloso. Comparative content of dietary fibre, total phenolic, and  
403 minerals in persimmons and apples. Journal of Agricultural and Food Chemistry. 2001. 49,  
404 952–957.
- 405 [7] J. Slavin Impact of the proposed definition of dietary fibre on nutrient databases. Journal of  
406 Food Composition and Analysis. 2003, 16, 287-291.
- 407 [8] Herbafood. (2002). Herbacel AQ Plus. Apple and citrus fibre. Available from  
408 [www.herbafood.de/eaqplus.pdf](http://www.herbafood.de/eaqplus.pdf).
- 409 [9] J. W. Anderson, B. M. Smith, N. S. Guftanson. Health benefit and practical aspects of high-  
410 fibre diets. American Journal of Clinical Nutrition. 1994, 59, 1242–1247.
- 411 [10] Fiberstar. (2005). The product portfolio of Fiberstar Inc., US. Available at  
412 <http://fiberstar.net/>
- 413 [11] M.A. Hill, J.R. Mitchell, P.A. Sherman. The relationship between the rheological and  
414 sensory properties of a lemon pie filling. Journal of Texture Studies. 1995, 26, 457-470.
- 415 [12] K. Maruyama, T. Sakashita, Y. Hagura, K. Suzuki. The relationship between rheology,  
416 particle size and texture of mayonnaise. Food Science and Technology Research. 2007, 13:1,  
417 1-6.
- 418 [13] M. Dervisoglu, F. Yazici. Note. The Effect of citrus fibre on the physical, chemical and  
419 sensory properties of ice-cream. Food Science Technology International. 2006, 12:2,159-164.

- 420 [14] H-U. Endress, J. Fischer. Fibres and fibre blends for individual needs: a physiological and  
421 technological approach. *Advanced Dietary fibre technology*. 2001, 26, 283-297.
- 422 [15] M. Pääkkö, M. Ankerfors, H. Kosonen, A. Nykänen, S. Ahola, M. Österberg, J.  
423 Ruokolainen, J. Laine, P.T. Larsson, O. Ikkala, T. Lindström. Enzymatic Hydrolysis Combined  
424 with Mechanical Shearing and High-Pressure Homogenization for Nano-scale Cellulose Fibrils  
425 and Strong Gels. *Biomacromolecules*. 2007, 8, 1934-1941.
- 426 [16] G. Agoda-Tandjawa, S. Durand, S. Berot, C. Blassel, C. Gaillard, C. Garnier, L.J  
427 Doublier.. Rheological characterization of microfibrillated cellulose suspensions after freezing.  
428 *Carbohydrate Polymers*. 2010, 80, 677-686.
- 429 [17] A. Cordoba, M.D.M. Camacho, N.M. Navarrete. Rheological behaviour of an insoluble  
430 lemon fibre as affected by stirring, temperature, time and storage. *Food and Bioprocess  
431 Technology*. 2010, 5-3, 1083-1092.
- 432 [18] D. Tatsumi, S. Ishioka, T. Matsumoto. Effect of fibre concentration and axial ratio on the  
433 rheological properties of cellulose fibre suspensions. *Journal of the Society of Rheology Japan*.  
434 2002, 30:1, 27–32.
- 435 [19] D. Tatsumi. Rheology of cellulose fibre disperse systems and cellulose solutions. *Nihon  
436 Reoroji Gakkaishi*. 2007, 35:5, 251–256.
- 437 [20] E. Cepeda, & I. Collado. Rheology of tomato and wheat dietary fibres in water and in  
438 suspensions of pimento puree. *Journal of Food Engineering*. 2014, 134, 67-73.
- 439 [21] A. Torbica, M. Belovic, J. Mastilovic, Z. Kevresan, M. Pestoric, D. Skrobot, T.D.  
440 Hadnadev. Nutritional, rheological, and sensory evaluation of tomato ketchup with increased  
441 content of natural fibres made from fresh tomato pomace, *Food and Bioproducts Processing*.  
442 2016, 98, 299-309.
- 443 [22] J. Ahmed, A.S. Almusallam, F. Al-Salman, M.H. Abdul Rahman, E. Al-Salem.  
444 Rheological properties of water insoluble date fiber incorporated wheat flour dough. *LWT -  
445 Food Science and Technology*. 2013, 51:2, 409-416.
- 446 [23] N. R. Cook, J. A. Cutler, E. Obarzanek, J. E. Buring, K. M. Rexrode, S. K. Kumanyika,  
447 L. J. Appel, P. K. Whelton. Long term effects of dietary sodium reduction on cardiovascular  
448 disease outcomes: Observational follow-up of the trials of hypertension prevention (TOHP).  
449 *British Medical Journal*. 2007, 334, 885–888.

- 450 [24] F. M. Sacks, L. P. Svetkey, W.M. Vollmer, L. J. Appel, G. A. Bray, D. Harsha, E.  
451 Obarzanek, P. R. Conlin, E. R. Miller, D.G. Simons-Morton, N. Karanja, P. H. Lin, M. Aickin,  
452 M. M. Most-Windhauser, T. J. Moore, M.A. Proschan, J. A. Cutler. Effects on blood pressure  
453 of reduced dietary sodium and the dietary approaches to stop hypertension (DASH) diet. The  
454 New England Journal of Medicine. 2001, 344, 3–10.
- 455 [25] X. Tian, I.D. Fisk. Salt release from potato crisp. Food and Function. 2012, 3, 376-380.
- 456 [26] E.J. Lynch, F.D. Bello, E.H. Sheehan, K.D. Cashman, E.K. Arendt. Fundamental studies  
457 on the reduction of salt on dough and bread characteristic. Food Research International. 2009,  
458 42, 885-891.
- 459 [27] A. Sullo, R.L. Watson, I.T. Norton. The design of colloidal foods for healthier diets. Gums  
460 and Stabilisers for the food industry: Changing the face of food manufacture: the role of  
461 hydrocolloids -17. Royal society of chemistry, Cambridge, UK. 2014, 289-299.
- 462 [28] R. Rama, N. Chiu, M.C.D. Silva, L. Hewson, J. Hort, I.D. Fisk. The impact of salt crystal  
463 size on the in-mouth delivery of sodium and saltiness perception from snack foods. Journal of  
464 Texture Studies. 2013, 44, 338-345.
- 465 [29] Z. V. Baines, & E. R. Morris. Flavour/taste perception in thickened systems: the effect of  
466 guar gum above and below  $c^*$ . Food Hydrocolloids. 1987, 1:3, 197-205.
- 467 [30] D. J. Cook, T. A. Hollowood, R. S. T. Linforth, A. J. Taylor. Oral shear stress predicts  
468 flavour perception in viscous solutions. Chemical Senses. 2003, 28:1, 11-23.
- 469 [31] R.J. Hill. Elastic modulus of microfibrillar cellulose gels. Biomacromolecules. 2008, 9,  
470 2963-2966.
- 471 [32] T. A. Hollowood, R. S. T. Linforth, A. J. Taylor. The effect of viscosity on the perception  
472 of flavour. Chemical Senses. 2002, 27:7, 583-591.
- 473 [33] A. L. Ferry, J. Hort, J. R. Mitchell, D. J. Cook, S. Lagarrigue, B. V. Pamies. Viscosity and  
474 flavour perception: why is starch different from hydrocolloids? Food Hydrocolloids. 2006,  
475 20:6, 855-862.
- 476 [34] R. Abson, S.R. Gaddipati, J. Hort, J.R. Mitchell, B. Wolf, S.E. Hill, A comparison of the  
477 sensory and rheology properties of molecular and particulate forms of xanthan gum. Food  
478 Hydrocolloids. 2014, 53, 85-90.

- 479 [35] J.F. Thibault, M. Lahaye, F. Guillon. Physiochemical properties of food plant cell walls.  
480 In: T. F. Schweizer, C. A. Edwards (eds) Dietary fibre, a component of food. Nutritional  
481 function in health and disease. Springer-verlag, Berlin. 1992, 21-56.
- 482 [36] A. L. Nelson. Properties of high-fibre ingredients. *Cereal Foods World*. 2001, 46, 93–97.
- 483 [37] A. Sangnark, A. Noomhorm. Effect of particle sizes on functional properties of dietary  
484 fibre prepared from sugarcane bagasse. *Food Chemistry*. 2003, 80, 221–229.
- 485 [38] M. Elleuch, D. Bedigian, O. Roiseux, S. Besbes, C. Blecker, H. Attia. Dietary fibre and  
486 fibre-rich by-products of food processing: Characterisation, technological functionality and  
487 commercial applications: A review. *Food Chemistry*. 2011, 124, 411–421.
- 488 [39] N. Grigelmo-Miguel, O. Martina-Belloso. Characterization of dietary fibre from orange  
489 juice extraction. *Food Research International*. 1999a, 131, 355–361.
- 490 [40] J. Ubando, A. Navarro, M. A. Valdivia. Mexican lime peel: Comparative study on contents  
491 of dietary fibre and associated antioxidant activity. *Food Chemistry*. 2005, 89, 57–61.
- 492 [41] N. Vergara-Valencia, E. Granados-Pereza, E. Agama-Acevedo, J. Tovar, J. Rualesc, L.  
493 A. Bello-Pereza. Fibre concentrate from mango fruit: Characterization, associated antioxidant  
494 capacity and application as a bakery product ingredient. *Lebensmittel-Wissenschaft und*  
495 *Technologie*. 2007, 40, 722–729.
- 496 [42] N. Grigelmo-Miguel, O. Martina-Belloso. Influence of fruit dietary fiber addition on  
497 physical and sensorial properties of strawberry jams. *Journal of Food Engineering*. 1999b, 41,  
498 13–21.
- 499 [43] V. S. Eim, S. Simal, C. Rosselló, A. Femenia. Effects of addition of carrot dietary fibre on  
500 the ripening process of a dry fermented sausage (sobrassada). *Meat Science*. 2008, 80, 173–  
501 182.
- 502 [44] C. Collar, E. Santos, C. M. Rosell. Assessment of the rheological profile of fibre-enriched  
503 bread doughs by response surface methodology. *Journal of Food Engineering*. 2007, 78, 820–  
504 826.
- 505 [45] C. M. Rosell, E. Santos, C. Collar. Mixing properties of fibre enriched wheat bread  
506 doughs: A response surface methodology study. *European Food Research and Technology*.  
507 2006, 223, 333–340.

- 508 [46] P. Chen, H. Yu, Y. Liu, W. Chen, X. Wang, M. Ouyang. Concentration effects on the  
509 isolation and dynamic rheological behaviour of cellulose nanofibres via ultrasonic processing.  
510 Cellulose. 2013, 20, 149–157.
- 511 [47] F. Fernando, M. LuzHurtado, A. M. Estévez, I. Chiffelle, F. Asenjo. Fibre concentrates  
512 from apple pomace and citrus peel as potential fibre sources for food enrichment. Food  
513 Chemistry. 2005, 91:3, 395-401.
- 514 [48] E.R. Morris. Rheological and organoleptic properties of food hydrocolloids K. Nishinari,  
515 E. Doi (Eds.), Food hydrocolloids, Plenum Press, New York. 1993, 201-210.