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3D Printing to Modulate the Texture of Starch-based Food

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Abstract

Food 3D Printing is a novel additive manufacturing process that allows fabricating three-dimensional food products with customized shape, structure or composition. This technology was used in this study to create semi-solid bite-size food made of pre-gelatinized starch with engineered size, number of pores, and pore size. Mechanical, and geometrical properties of the prints were quantified by uniaxial compression tests and X-Ray tomography. Finally, an in-vitro set-up was used to investigate the combined effect of food hydration and uniaxial compression, in conditions inspired by tongue-palate compression.

The hydration of the starch prints leads to a drastic change in mechanical properties. Most 3D printed food designs yield and collapse under a uniaxial compression of 10 kPa, applied to mimic the squeezing of the food between the palate and the tongue. After breakage, starch dispersion increases rapidly due to the higher surface area in contact with the liquid, and the yielding point depends on the internal structure and height. Structures with a higher initial surface area in contact with the liquid have a lower yielding point, probably due to the faster hydration.

These results help understanding the dynamics of semi-solid food deconstruction during oral processing and the approach developed could provide useful insights when developing food products for specific consumer needs.

24 *Keywords:* Food 3D Printing, Starch, Food Oral Processing, Food Design, Texture

25 **1. Introduction**

26 Food 3D Printing (3DP) is a novel fabrication technology where edible structures are
27 manufactured with a controlled shape, size, internal structure, and texture (Prakash et al., 2019).
28 3DP foods are usually soft pastes made of gels and thickeners, often used to treat patients with
29 dysphagia (Hori et al., 2015; Marconati et al., 2019; Marconati and Ramaioli, 2020). One of the
30 drawbacks reported from patients having this disease is the loss of appetite due to unappealing
31 food texture. 3DP allows to easily modify the texture of food products and can be used as a tool
32 to manufacture more appealing food products (Godoi et al., 2016).

33 Numerous food formulations were 3D printed: starch-based products (Liu et al., 2020;
34 Huang et al., 2019; Chen et al., 2019; Liu et al., 2018; Masbernat et al., 2021), eggs' white and
35 yolk (Anukiruthika et al., 2020), edible gels (Liu et al., 2019; Schutyser et al., 2018; Yang et al.,
36 2018), chocolate (Rando and Ramaioli, 2021; Mantihal et al., 2019, 2017; Lanaro et al., 2017),
37 pectin based products (Vancauwenberghe et al., 2018), healthy snacks for children (Derossi et
38 al., 2018) and many others. Often authors vary the texture of the food (Guénard-Lampron et al.,
39 2021), changing the infill parameter; criterion defined in the printer software, called Slicer,
40 which expresses the degree of internal structures of the desired product shape. Even though the
41 infill parameter allows to easily change the food texture, it depends on the Slicer that can be
42 different for each printer, giving poor control of the internal structure during the design phase.

43 Mantihal et al. (2019) and Liu et al. (2020) studied the texture attributes of 3DP foods
44 having a different internal structure, after manufacturing. Results showed that structures with
45 higher infill have a higher hardness and lower facturability. Due to the layer-by-layer
46 manufacturing method, the printed structures with 100% infill showed poorer mechanical
47 properties compared to prints prepared with the casting method. Huang et al. (2019) studied the

48 effect of printing parameters such as nozzle size and layer thickness on the texture of food prints
49 made of brown rice pastes. In particular, when structures with the same shape and size are
50 manufactured with bigger nozzle and layer thickness, the number of layers extruded is reduced
51 leading to a change in the mechanical behavior. The hardness of the prints was found lower at a
52 lower nozzle diameter. Furthermore, the degree of filling of a certain structure varies due to
53 larger layers deposited in the inner part of the prints. These studies limited the investigation to
54 determine the food texture attributes after manufacturing and did not consider the conditions of
55 the oral phase such as the effect of food hydration, and temperature, during compression.

56 Mechanical compression tests are often used in the food industry to characterize the
57 behavior of food products during the first bite. However, they can be inadequate to explain the
58 food behavior during oral processing, which is a complex interplay of mechanical and physico-
59 chemical phenomena. The food structure breakdown depends on the type of food (liquid, solid or
60 semi-solid) and on the human subject (gender, age, habits) (Campbell et al., 2017). Solid and
61 semi-solid food products, after they are introduced into the mouth, are subject to a gradual
62 deconstruction of the products and are continuously hydrated from saliva, providing lubrication
63 until they are transformed to a bolus ready to swallow. The time needed to prepare the bolus
64 depends on the degree of structure and on its initial water content. Also, the body temperature is
65 approximately 37 °C and it can enhance the dissolution and dispersion of certain food
66 components.. Soft solid foods are often squeezed between the tongue and the hard palate (Chen,
67 2009). The typical pressure measured during in-vivo studies was found of the order of 10 kPa
68 (Hori et al., 2015).

69 The hydrolysis and dispersion of starch contained in bread products due to the action of
70 pH and α -amylase during the oral and digestion phases were studied by Freitas et al. (2018) The

71 authors used an experimental apparatus allowing to accurately reproduce the condition of the
72 oral and digestion phases. In the former step (set to 2.5 min) the total starch hydrolyzed varies
73 between 2 and 20%, depending on the type of product. There are few studies reporting the
74 dissolution of 3D printed tablets having different shapes, inner structures, and compositions,
75 aiming to determine the drug release kinetics. Goyanes et al. (2015b, 2014, 2015a) varied the
76 infill parameter during the manufacturing of tablets and studied the dissolution kinetics, finding
77 that increasing the degree of filling of tablets increases the amount of drugs released. Differently
78 from foods, the characteristic dissolution time occurring during digestion and gastric phases are
79 significantly higher varying from minutes to hours. Moreover, tablets are generally directly
80 swallowed, whereas food products are deconstructed in the mouth, drastically increasing the
81 surface area in contact with the saliva and improving dispersion.

82 To our best knowledge, the effect of hydration was never considered during the
83 mechanical characterization of food 3D printed products.

84 Based on these premises, an in-vitro apparatus was developed to study the combined
85 mechanical and dispersion behavior of the 3D printed structures. Experimental parameters were
86 inspired by food oral processing conditions. Food 3DP was used to manufacture starch structures
87 having controlled size, porosity, and pore size, which varied systematically during the *in-vitro*
88 experiments. The results of this study can be helpful for understanding the effect of food
89 hydration on mechanical properties during food oral processing.

90 **2. Material & Methods**

91 *2.1. Materials*

92 Starch 1500 (Colorcon, UK) was used as a printing material. It is a partially
93 pregelatinized maize starch in form of powder with a composition of 73% amylopectin and 27%

94 amylose. It has a gelatinization level of 20%, a mean particle size of 65 μm , and a moisture
95 content of 7.2%.

96 Starch was mixed with deionized water (20 ± 1 °C) to obtain a 30% w/w suspension. That
97 was mixed using a magnetic stirrer (IKA RCT basic) at 850 rpm for 1 min. Afterwards, a
98 stainless-steel spatula was used for 2 more minutes to homogenize the mixture and reduce lumps.
99 The suspensions were used the same day to avoid any moisture evaporation.

100 *2.2. Rheological Analysis*

101 The rheological behavior was characterized using a PhysicaMCR-301 rheometer (Anton
102 Paar GmbH, Germany) equipped with a parallel plate geometry ($d = 25\text{mm}$; $\text{gap} = 1\text{mm}$) at 20
103 and 50 °C. A frequency sweep was performed between 100 and 0.1 rad/s at 1% strain. Moreover,
104 an amplitude sweep was performed at a constant angular frequency of 10 rad/s and varying the
105 strain from 0.1 to 100%. The stress at which the complex modulus (G^*) deviates from the Linear
106 Viscoelastic Region (LVR) was considered as the yield stress. The measurements were
107 performed in triplicates.

108 *2.3. Structure Design and Extrusion 3D Printing*

109 Cubic food structures were designed to study systematically the effect of size, porosity,
110 and pore size. Three different groups of designs were considered for this study. The first group
111 (G1) comprises structures characterized by the same internal pattern and number of holes, but
112 sizes varying in the range of 10 to 25 mm. The second group (G2) includes structures having the
113 same size (20 mm) but different internal patterns and number of holes. The structures from the
114 third group (G3) had the same external width as those in group 2, but were printed using fewer
115 layers and therefore had a different height. The geometrical characteristics are summarized in
116 Table 1 and complementary measurements are shown in Table A.6. Each structure was labeled

117 based on the length of its side (L) and the number of pores (P) (i.e. L20P4 is a print having a side
118 of 20 mm and 4 pores).

119

120 An extrusion-based Food 3D Printer (Foodini, Spain) was used to print 3D structures
121 made of pre-gelatinized starch. 3D structures were designed using the Foodini JavaScript Editor,
122 which allows defining the coordinates of the 3D structures with a Java programming language. A
123 code giving information on the specific path followed by the extruder was developed for each
124 structure. The printing paths of the first two layers are shown in Figure A.10. Before printing,
125 starch suspensions were loaded into the temperature-controlled printer capsule. The printing
126 material was kept inside the capsule for 30 min at constant temperature. The uniformity was
127 verified with a thermocouple.

128 The printing material was extruded from a 0.8 mm nozzle at $T_n = 46 \pm 2$ °C in an ambient
129 temperature of 20 °C. The printer includes a cartridge that moves the syringe at a set printing
130 velocity $V_p = 0.3$ mm/s with a volumetric flow rate of $Q = 0.8$ mm³/s along the X and Y axes. All
131 structures were printed continuously: the extruder moves while starch is deposited without
132 interruption.

133 2.5. Geometrical Characterization

134 After printing, images of the top (x-y plane) and lateral (z-x plane) views of the prints
135 were taken using a camera (BASLER, Germany), with a 75 μm resolution. Moreover,
136 measurements of the prints length, width, height, and pore size were taken manually using a
137 caliper.

138 A Desktom 130 X-ray micro-computed tomography (μ-CT RX Solutions, Chavanod,
139 France) was used to measure the 3D microstructure of the printed structures. The X-ray source

140 settings were: voltage of 50 kV, current source of 160 μ A, and a power of 8 W. Slices were
141 acquired with an image resolution of 20.03 μ m, while the prints were rotated 180° at a step of
142 0.3°.

143 The reconstructed images were analyzed using the Simpleware SCANIP software
144 (Synopsys, Mountain View, CA, USA), to determine the structure porosity and surface area.
145 Images were treated with median filtering and greyscale thresholding. The porosity (Φ) was
146 defined as the ratio between the void volume and the total volume of the structure's external
147 envelope (Eq. 1).

148 (1)

$$\Phi = \frac{V_{voids}}{V_{tot}} = \frac{V_{tot} - V_{starch}}{V_{tot}}$$

149 Where V_{tot} , V_{starch} , V_{voids} are respectively the total volume of an enclosing cube, the
150 volume of printed starch and the voids' volume. V_{voids} takes into account only the open pores and
151 was obtained from the difference between V_{tot} and the volume of starch. Furthermore, the surface
152 area (A_s) is defined as the total interfacial area between starch and air. The surface area, from the
153 caliper and the Image-J measurements, was calculated as $A_s = 2 \cdot A_c + 4 \cdot A_{s,lat} + no.pores \cdot 4 \cdot$
154 $L_{por} \cdot h_s$; where A_c , represents the cross-section area of the top view, $A_{s,lat}$, L_{por} and h_s are
155 respectively the lateral surface area of the prints, the length of one pore and the height of the
156 structure.

157 2.6. Uniaxial Compression

158 The mechanical properties of 3D printed structures were characterized using a Texture
159 Analyzer TA.HD.Plus (Stable Micro System, UK) equipped with a 5 Kg cell and a cylindrical
160 probe (with a flat surface and a diameter of 40 mm). The tests were performed in a force-

161 controlled mode at room temperature (25 ± 1 °C). The probe moves down at a pre-test speed of 1
162 mm/s until the trigger force of 0.05 N is reached; then, the probe moves down compressing the
163 prints at a test speed of 2 mm/s until the imposed maximum force is reached; finally, the probe
164 moves back to the initial position at a post-test speed of 2 mm/s. The maximum forces (F_{max})
165 were set in order to achieve a maximum stress of around 10 KPa, typically observed during in-
166 vivo tongue-palate measurements (Chen, 2009). Moreover, to measure the yielding point of all
167 the prints in wet and dry conditions, other tests were performed with maximum stress (σ_{max}) of
168 50 KPa.

169 The cross-section area (A_c) was measured from the images of the top for each print,
170 using the ImageJ software. Each measurement was repeated three times. Since each structure has
171 a different A_c , different maximum forces were computed and applied during the compression
172 tests, based on the target maximum pressure, as $F_{max} = \sigma_{max} \cdot A_c$. A summary of different forces
173 applied to each structure is reported in Table 1.

174 *2.7. Water Hydration and Compression Tests*

175 A novel setup was developed to investigate the coupled effect of food hydration and
176 repeated compressions, inspired by food oral processing conditions. A sketch of the dispersion
177 apparatus is shown in Figure 1. A heated jacketed glass vessel of 250 mL contained a cylindrical
178 customized stand which allows supporting the printed structures while they were compressed.
179 The conductivity was measured with an InPro 7108-25-VP Conductivity Sensor (METTLER-
180 TOLEDO, France). The water was continuously stirred at 350 rpm to achieve a uniform
181 concentration of the starch dispersed in the water. The customized stand has an inner diameter of
182 2 cm and an external diameter of 4 cm, where 1 mm holes were drilled to improve the mixing.
183 The conductivity probe was calibrated to verify a linear relation between the starch concentration

184 and water conductivity, using partially pre-gelatinized maize starch (Starch 1500) in a range of
185 starch concentrations between 0 and 0.06 m_s/m_{s0} . The measured conductivity due to starch
186 dispersion was converted into mass fraction, using the calibration curve, reported in Figure A.11,
187 and normalized by the maximum mass fraction of starch dispersed.

188 During the experiments, the jacketed vessel was filled with 150 ml of deionized water
189 and connected to a water bath to control the temperature (37 ± 0.5 °C). The printed structures
190 were partially immersed in deionized water. The water level reached 2 mm height measured
191 from the center of the stand. The compression protocol begins 30s after the prints were partially
192 immersed in the water. We consider this as the initial time of the experiments ($t = 0s$). The
193 compression protocols follow the same parameters explained in section 2.6. Finally, videos were
194 recorded during the experiments using a camera (BASLER, Germany), with a 75 μm resolution
195 and at a frame rate of 25 Hz.

196 *2.8. Simulated Salivary Fluid (SSF) Hydration and Compression Tests*

197 For the purpose of simulating more closely food oral processing, two structures (L20P4,
198 L20P49) were compressed as described in Section 2.7 but using Simulated Salivary Fluid (SSF)
199 to imitate human saliva. These two structures were selected to consider different porosity levels,
200 close to upper and lower limits of the range studied. Prior to commencing the study, the SSF was
201 prepared using the method detailed by the InfoGest Consensus Method (Brodkorb et al., 2019).
202 The salivary amylase solution was adjusted to have a final activity of 75 IU/ml by using SSF and
203 *Bacillus sp.* salivary α -amylase in the form of a lyophilized powder (A6380, Sigma-Aldrich).
204 Every trial contained 148.6 mL of SSF, 0.4 mL of $CaCl_2$ 0.3M and 1 mL of salivary amylase
205 solution to achieve a final volume of 150 mL.

206 **3. Results and Discussion**

207 3.1. Rheological properties

208 The rheological properties of the printing material directly affect different steps of the 3D
209 printing process: flow in the nozzle, stability after deposition, and layer sintering (Chaunier et al.,
210 2021, 2019; Rando, 2021; Rando and Ramaioli, 2022). In this study, the rheological properties
211 relative to starch paste owing through the nozzle and structure stability were evaluated at their
212 respective temperatures.

213 Figure 2 shows the results of starch viscosity and storage (G') and loss (G'') moduli carried
214 out at 20 and 50 °C, to understand the rheological behavior of the printing material in the nozzle
215 and after deposition; whereas, the yield stress (τ_0) was estimated at 20 °C (Figure 2).

216 The viscosity drops from 90000 to 90 Pa · s when the angular velocity was increased
217 from 0.1 to 100 rad/s, showing a shear-thinning behavior, which is beneficial during extrusion
218 3D printing since in the printer nozzle shear stress is higher. The maximum shear rate at the wall
219 ($\dot{\gamma}_w$) was estimated considering the Rabinowitsch-Mooney equation for a Power Law fluid
220 (Rando, 2021). Equal to 17.32s^{-1} , confirming that the rheological tests were carried out in the
221 relevant range.

222 At all shear rates, the storage modulus is higher compared to the loss modulus, showing a
223 gel-like behavior of the starch suspension. Interestingly, there is no difference in viscosity
224 between 20 and 50 °C. A higher nozzle temperature could improve the extrusion of starch
225 mixtures. However, increasing the temperature above 46 °C leads to drying of the starch solution
226 and clogging of the nozzle.

227 The yield stress, as shown in Figure 2, was obtained considering the stress at which the
228 complex modulus (G^*) deviates from the Linear Viscoelastic Region (LVR) and is found equal
229 to 182 ± 2 Pa. Similar results were found by Azam et al. (2018) who studied the rheological

230 properties of orange leather by mixing wheat starch and water at 30% w/w and finding yield
231 stress of 139 Pa. Also Chen et al., 2019 estimated values of yield stress between 32 to 455 Pa in
232 corn, rice, and potato starch suspensions with concentrations from 15 to 25% w/w at a
233 temperature between 70 and 85 °C.

234 The maximum theoretical stable height (H_y) can be estimated from the yield stress as
235 $H_y = \frac{\tau_0}{\rho g}$ where ρ and g are respectively the starch density and the gravitational acceleration
236 (Rando and Ramaioli, 2021). A yield stress of 182 ± 2 Pa would allow printing simple quasi-2D
237 wall structures with a maximum theoretical height of approximately 18 mm. However, stable
238 prints were manufactured at a height of 25 mm. The presence of an internal structure could be
239 beneficial to improve the distribution of the stresses and the stability of the printed structure.
240 Drying can also increase progressively the yield stress after printing, explaining the stability
241 observed experimentally.

242 *3.2. Mass and Geometrical Properties of the Prints*

243 The mass of the prints was measured after printing for each structure. The results are shown in
244 Table 2. As expected, the mass of the prints increases when increasing the degree of filling and
245 the size.

246 The dimensions of all printed geometries (length, width, height and pore size) were measured
247 after printing using a caliper (Table A.6 and Table A.7) showing that the length and width are
248 approximately 4-10 % higher compared to the size defined in the design phase; whereas the
249 height of the prints is consistent with the original target. A comparison of the geometrical
250 properties measured after deposition with image analysis and the X-ray CT (XCT) are reported
251 in Table 3 and in Figure 3. Experimental measurements were taken i) with a caliper after
252 printing, ii) from images taken after printing using Image-J, and iii) with XCT. From the top

253 view images of the prints, shown in the Table 3, it is possible to see that there is an accumulation
254 of material on one edge for all structures. This accumulation is constant throughout the edge
255 vertical section and it is probably caused by the continuous printing sequence. The extruder stops
256 and starts extruding on the same x-y coordinates. Whenever the extrusion starts for each layer a
257 small excess of starch it is deposited on the initial x-y coordinates. This creates the accumulation
258 of starch on one edge. Changing the printing sequence aiming to begin the starch extrusion at
259 different x-y coordinates in each adjacent layer could improve the quality of the prints.

260 The porosity of the structures measured with different techniques is compared Figure 3(a).
261 Structures with a lower number of pores show a higher porosity, due to the bigger pores. Also
262 increasing the size of the printed structures increases the porosity, owing to the rather constant
263 thickness of the walls separating the pores. XCT and caliper results show good agreement.
264 However, the porosity measured with the caliper and Image-J could be slightly over-estimated
265 since only lateral measurements were taken and it was assumed that the pore cross-section is
266 constant throughout the height. Whereas experimental results suggest that the pore cross-section
267 is larger at the base and decreases within the height. In addition, some caliper and Image-J
268 inconsistency may be due to a non-uniformity of the pore cross section, for some structures in
269 groups 2 and 3.

270 A comparison between the surface areas is reported in Figure 3(b). The surface area
271 increases when the size of the structure increases (G1). Moreover, XCT suggest that the surface
272 area increases also when the number of pores increases (G2). However, too many pores result in
273 a reducing surface in contact with air (G3). XCT measurements suggest higher surface areas than
274 other techniques, because XCT considers correctly the roughness of the structure due to the
275 layer-by-layer building approach. Conversely, caliper and Image-J measurements were taken

276 assuming that the cross-section is constant. Therefore structures are assumed to be completely
277 smooth and the computed surface areas are underestimated compared to the final prints. XCT is
278 therefore expected to be more accurate.

279 Finally, Figure 3(c) shows the specific surface area calculated as the ratio between the
280 surface area and the mass of each structure. In general, prints with higher mass have a lower
281 specific surface area, but the differences are not as big as the absolute differences in surface area
282 discussed above. The structures L20P81 and L20P100 show even lower specific areas due to the
283 tiny and partially closed pores.

284 3.3. Deconstruction under Compression and Hydration

285 3D printed starch structures manufactured with different sizes and porosity were tested
286 with the *in-vitro* dispersion apparatus, providing compression and hydration conditions inspired
287 by oral processing. In this section, structures from G2 were compressed at maximum stress of 10
288 kPa, sufficient to cause their yielding. Conversely, structures belonging to G3 did not yield at 10
289 kPa. For this reason maximum stress of 50 kPa was used, as indicated in Table 4.

290 This higher stress guaranteed yielding and a controlled dispersion of the starch. It should
291 be noted, however, that all structures in group G3 yielded below 20kPa. For the sake of clarity,
292 the results of G1 are not shown in this section due to different ending times; as the height
293 increases the displacement of the Texture Analyzer probe increases.

294 Figure 4 reports a typical graph showing the coupled effect of starch dispersion and
295 mechanical response over time, during the deconstruction experiments. Images of the test taken
296 at different times are shown above the graph.

297 On the left axis of Figure 4, is reported the normal stress applied on the starch structure;
298 whereas the right axis shows the increase in starch mass dispersed in water (normalized by the

299 total starch mass) under the combined effect of hydration. During the first compression, there is
300 yielding at 9.8 kPa of the print ($t = 2.1\text{s}$) which causes the structure to fracture. After yielding (t
301 $= 8.8\text{s}$) the force increases again until the maximum set point of 10 kPa is reached. At this point,
302 the print is completely deconstructed and the amount of starch dispersed increases. Finally, the
303 print is compressed four more times, while the amount of starch dispersed into the liquid kept
304 increasing until the end of the experiments. Multiple compressions enhance the breakage into
305 smaller pieces and the starch dispersion. The amount of starch dispersed in the 90 s is around
306 35% of the maximum amount of starch. The yielding occurs during the first 10 s, whereas the
307 timescale of the starch dispersion is significantly longer. The increase of starch dispersed in
308 water is almost linear, albeit some sudden increases in dispersion rate seem to occur after the
309 second and third compression. This is probably due to the short duration of the experiments,
310 whereas for longer times an exponential curve is expected, as found by Gao et al. (2021).

311 Figure 5 presents the results obtained with Group 2 and 3: (a) the mass of starch dispersed
312 over time, (b) the mass dispersed (m_s) normalized by the total starch mass ($m_{s,0}$), (c) stress-time
313 compression plots under hydration (first cycle) of group 2 and (d) stress-time compression plots
314 under hydration (first cycle) of group 3.

315 The structures from G2 show similar yielding times (structures with the same size) and
316 the normalized dispersion rates follow all the same master curve. However, there is a slight
317 difference at structure L20P4, this may be due to the lower surface area in contact with the water
318 initially as shown in Figure A.12. As the number of pores increase the structure easily hydrates
319 internally such as L20P9 and L20P16 which could explain their faster rate of dispersion and the
320 higher percentage of normalized starch at the end of the experiments (Table 4).

321 Structures from G2 disperse more rapidly when structures have more pores, (P16>P4),
322 probably due to the higher mass. The dissolution tests of 3DP pharmaceutical tablets carried out
323 by Goyanes et al., (2014) showed that structures with higher infill (internal structure) dissolve
324 faster.

325 Structures from G3 (Figure 5) show a different behavior than G2. In G3 the structures
326 with higher mass (L20P81 and L20P100) dispersed slower, this is probably due to their lower
327 porosity. However, when normalized starch is compared (Figure 5 (b)) the structure L20P25,
328 dispersed at a faster rate probably due to its higher specific surface area favouring its hydration.
329 Also, the structure L20P25 yield at lower stress which helps to a faster breakage and dispersion.
330 After breakage, the surface area of the starch in contact with water increases due to comminution
331 and partial dispersion of starch. Modelling both these behaviours considering a breakage
332 function (Chen, 2009) and a dispersion model (Gao et al., 2021) could be an interesting
333 extension of this study.

334 Overall, the structures showed that the amount of starch dispersed in water starts
335 increasing after breakage. Furthermore, the increase dispersion rate is visible upon each
336 compression. The dispersion results from L20P49 were excluded due to an artefact in the
337 conductivity probe, its behavior under hydrated compression is presented in Figure 5 (d).

338 Table 4 shows the characteristic dispersion times t_{20} , when 20% of starch is dispersed,
339 and the fraction of starch mass dispersed at the end of the experiments. Comparing the dispersion
340 time t_{20} it is possible to see that structures with more pores have a slightly faster dispersion rate,
341 except for L20P81 and L20P100, for which XCT showed a low porosity and surface areas,
342 suggesting many closed pores.

343 Prints having the same size are characterized by almost the same t_{end} , it is possible to see
344 that the amount of dispersed starch increases when the number of pores is higher, probably due
345 to the higher amount of material needed to manufacture the prints. On the other hand, structures
346 with lower porosity and higher manufacturing material showed less percentage of starch
347 dispersed.

348 The amount of starch dispersed at the end of the experiments varies between 29.6% and
349 51.2%, depending on the structures. Freitas et al. (2018) considered the oral and gastric phase of
350 bread digestion and reported a lower starch hydrolyzed (approx. 5%) during the oral phase ($t =$
351 2.5 min) in absence of salivary amylase, while the addition of amylase increased the amount of
352 starch hydrolyzed to 18.5%. The different product structure, composition and protocol prevent a
353 direct comparison with the results obtained in this study.

354 As explained in Section 2.8, two structures (L20P4 and L20P49) were evaluated using a
355 Simulated Salivary Fluid (SSF) to better reproduce oral processing conditions. Figure 6 shows
356 that no significant effect of α -amylase was observed, except a slight decrease in the young
357 modulus of the material (initial slope). Further work should be done to develop and adapt the
358 methodology in this study to compare 3DP structures under deconstruction using liquids with
359 closer characteristics to human saliva.

360 *3.4. Mechanical Properties of the 3D Printed Structure*

361 The mechanical properties of the prints were characterized by uniaxial compression using
362 a texture analyzer (TA). The yield point was measured by identifying the local maximum in the
363 stress-strain curves (σ_y) at which structures begin to yield (Figure 7).

364 The Young modulus (E) was estimated from the linear slope of the stress-strain curve as:

$$E = \frac{\Delta F}{A_c \Delta \varepsilon}$$

366 Where F and ε are respectively the force and strain measured from the TA; whereas A_c is
367 the initial cross-section area measured after printing using ImageJ. The same protocol was used
368 by Vancauwenberghe et al., 2018 to characterize the mechanical properties of honeycomb and
369 cube structures made of pectin. Data were fitted considering a range of strain $\Delta \varepsilon = [0.1 - 0.2]$,
370 from the stress-strain curves reported in Figure 7 in dry conditions. In each figure, the structures
371 were grouped aiming to compare only prints with the same number of pores or length size.

372 All plots show a similar trend: the stress gradually increases when the strain increases
373 until a maximum is reached at strains between 0.2 and 0.3 where the structures yield and break.
374 Finally, the stress keeps increasing until the maximum stress imposed by the TA protocol is
375 reached between $\varepsilon = 0.6$ and 0.8. At this point, the prints are completely deconstructed under the
376 imposed force.

377 Figure 8 shows the stress-time curves measured from the compression in dry conditions.
378 All plots showed a first peak, which represents the yielding point and varies between 12 and 18
379 kPa, depending on the structures. After yielding, the prints are continuously compressed until the
380 maximum force imposed from the TA is reached. Structures from Group 1 which present
381 difference in size, show that prints with higher heights have lower yielding points. The tallest
382 print L25P4 showed less repeatability. On the contrary, increasing the pore size in Group 2 leads
383 to lower yielding points. In fact, structures with bigger pores have a higher void fraction. Similar
384 behavior was reported by Huang et al. (2019), who found lower hardness in 3DP foods made of a
385 rice paste, having a lower degree of filling. Conversely for Group 3 no significant difference was

386 observed when comparing dry yielding points. Probably due to their similar values of surface
387 area with respect to G2.

388 All yielding properties and Young modulus were evaluated during the first compression
389 cycle. A comparison between results in dry and wet conditions is reported in Table 5. In dry
390 conditions, all structures yield at stresses higher than 10 kPa, varying between 12.2 and 18.3 kPa,
391 depending on the structure's size and porosity. On the contrary, when the prints are in contact
392 with water structures yield at significantly lower stresses varying between 8.3 and 16.6 kPa. The
393 higher surface area in contact with the liquid could explain the lower yielding points.

394 Figure 9 shows that increasing the size of the structures (G1) reduces the yield point (σ_y)
395 under dry and wet conditions. For the second group (G2), the σ_y and E increase while increasing
396 the number of pores under dry conditions. These two effects could be due to the change in
397 porosity in both groups. Contrarily, for group 3 (G3) no significant effect was observed in the
398 values of σ_y and E under dry conditions regardless the change of porosity. Similar values of
399 surface area within the group could explain this behavior.

400 For G2 and G3 a significant difference was observed when yielding under wet conditions.
401 This behavior could be the higher surface area in contact with the liquid. This might explain the
402 higher yielding points in wet conditions of structures L20P81 and L20P100. When increasing the
403 number of pores there is no hydration in the internal structure due to the absence of voids.

404 Most importantly, the young modulus of structures in wet conditions was always lower
405 compared to the values estimated in dry conditions. The addition of water, makes structures less
406 elastic enhancing the deconstruction in mouth. In fact, in Figure 9 the values of σ_y were found
407 significantly higher in dry conditions for all structures except for L20P81. For values of E (Figure
408 9(c)) structures from G1 had no significant difference between dry and wet except for L20P4.

409 Figure 9 (a) and (b) show a comparison between the yielding points in wet and dry
410 conditions. The structures fracture always at lower stress when are in contact with the water, no
411 significant differences were seen in structures L20P81 and L20P100. In dry condition, structures
412 with higher porosities have a lower yielding points. The structure L20P100 was found to have a
413 slightly lower yielding point than expected.

414 Figure 9 (d) shows a comparison between the Young Modulus (E) of all structures against
415 the initial surface area in contact with the liquid. The initial contact area between the starch and
416 the liquid was calculated as: $A_{s,s-H2O} = \frac{A_{s,lat} \cdot h_{H2O}}{h_S} + A_c = \frac{(A_s - 2A_c) \cdot h_{H2O}}{h_S} + A_c$ where h_{H2O} is the
417 height of the water in contact with the starch structures equal to 2 mm. Structures with a higher
418 surface area in contact with water seem to be less elastic. The higher surface area for moisture
419 uptake could result in a faster mass transfer of water at the print base, facilitating the
420 deconstruction.

421 **4. Conclusion and Perspectives**

422 In this study, Food 3D printing was used to manufacture starch-based structures having
423 controlled size, porosity and pore size. A starch paste with $m_s = 30\%$ w/w showed good
424 extrudability at a $T_n = 46$ °C and stability after deposition at room temperature due to its shear-
425 thinning behaviour and high yield stress. Moreover, the presence of an internal design improves
426 stability of the bite sized prints. In addition, a simple in-vitro apparatus was developed, inspired
427 by the combined effect of tongue-palate compression and hydration to study the semi-solid food
428 deconstruction.

429 The geometrical and mechanical properties of the 3D bite-sized structures were
430 quantified. The uniaxial compression in wet conditions showed significantly lower yield stress

431 than in dry conditions. Dry structures with higher porosity and height showed lower yielding
432 points. After wetting, structures with higher surface area in contact with the liquid fracture at
433 lower stress, probably due to the faster mass transfer of water at the base of the structure. Finally,
434 after breakage of the food structure, the starch dispersion rate increases proportionally to the
435 mass of starch present in the system for all structures, except the structures with the lowest
436 porosity, showing slower dynamics, probably induced by the lower specific surface area.

437 Whilst this study did not aim to simulate food oral processing, it did contribute to
438 understanding the deconstruction behavior of semi-solid food during an innovative approach
439 inspired by food oral hydration. This study was limited by the lack of samples studied under
440 simulated salivary fluid. Nonetheless, future work should focus on understanding the effect of
441 the geometrical properties of the prints in the dispersion-deconstruction experiments. The in-
442 vitro apparatus can be further improved to reproduce better oral conditions.

443 **Appendix A. Additional Data**

444 *Appendix A.1. Printing Path from Foodini JavaScript*

445 Figure A.10 reported the printing path of the first and second layers used to design the
446 starch prints. The path was repeated several times gradually increasing the z coordinate allowing
447 to manufacture of 3D structures.

448 *Appendix A.2. Structure Dimensions and Mass*

449 The dimensions of the prints measured with the caliper after manufacturing are summarized in
450 Table A.6 and Table A.7.

451 *Appendix A.3. Conductivity Probe Calibration*

452 The conductivity probe was calibrated to ensure a linear relationship between the
453 amounts of starch dispersed and water conductivity. Partially pre-gelatinized maize starch in a

454 range of starch concentrations between 0 and 0.06 m_s/m_{s0} was dispersed in water while the
455 conductivity was recorded. The parameters of the calibration curve were obtained by linear
456 fitting, finding a slope of 34.793 and an intercept of 0.532. Data shows good agreement showing
457 an $R^2 = 0.996$. A comparison between the experimental results and the fitting is reported in
458 Figure A.11.

459 *Appendix A.4. Surface area in contact with the liquid under hydration*

460 The initial contact area between the starch and the liquid is presented for each structure in Figure
461 A.12.

462 **Acknowledgement**

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464 or not-for-profit sectors.

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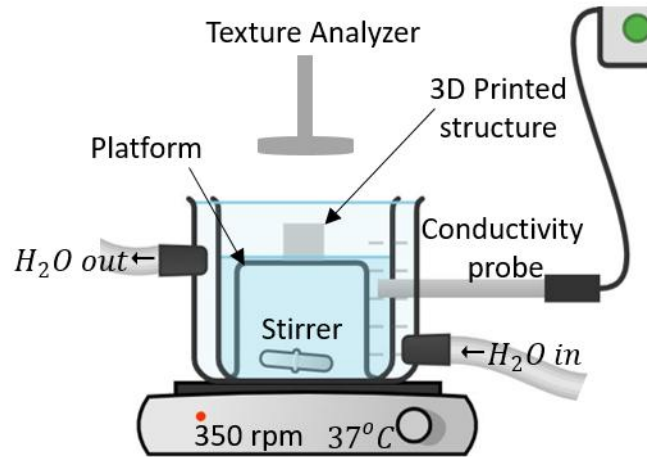
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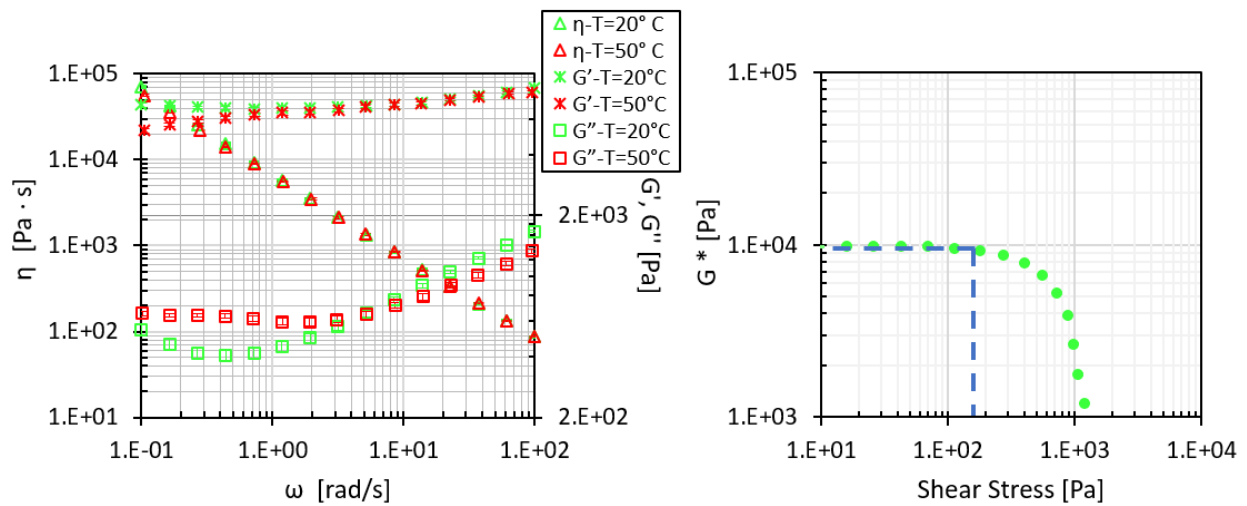
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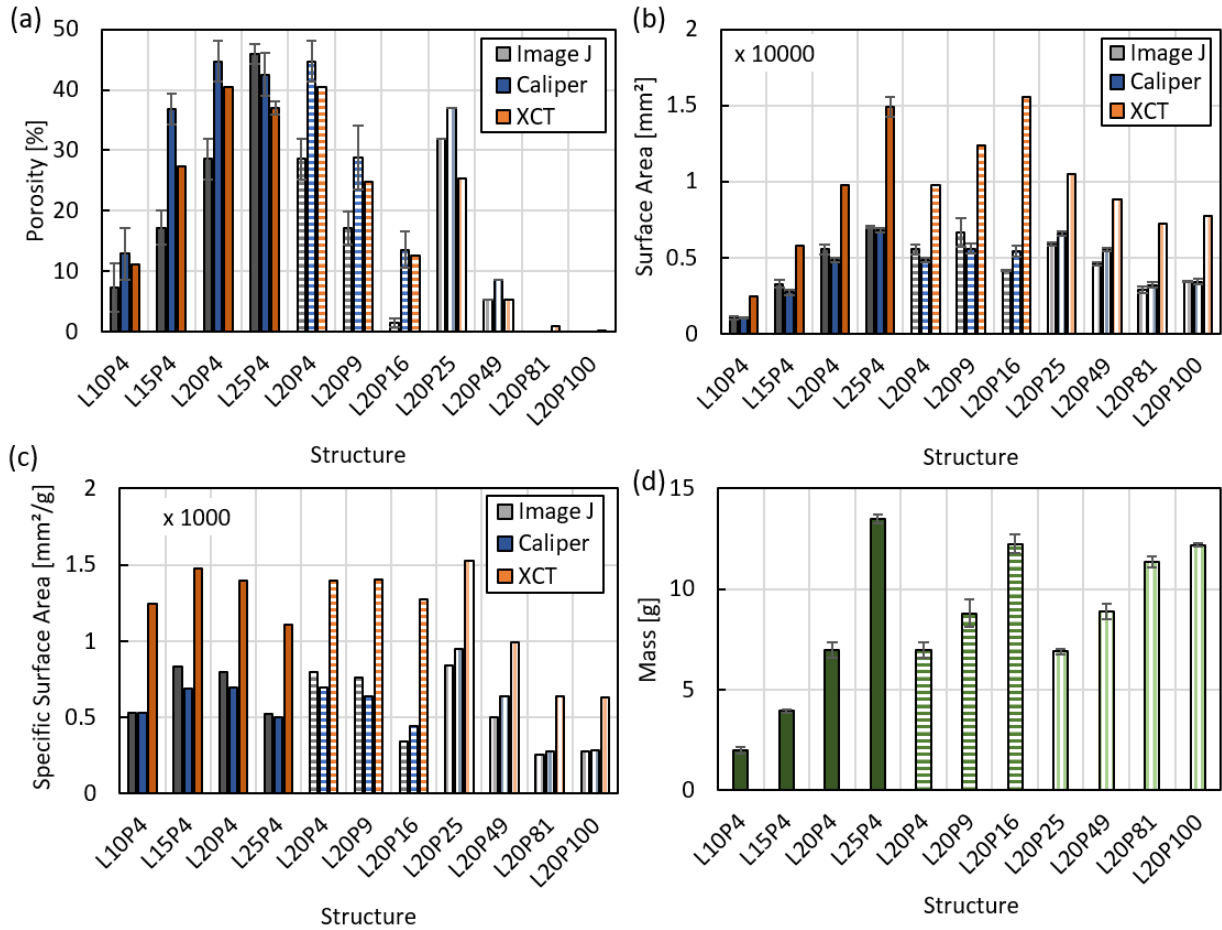
Figure 1. Sketch of the dispersion cell setup.



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579 Figure 2. Rheological properties of a starch suspension at $m_s = 30\%$, measured by frequency sweep and amplitude sweep tests:

580 (left) Complex viscosity (η^*), storage modulus (G') and loss modulus (G''), (right) Yield stress τ_0 estimation at 20°C .



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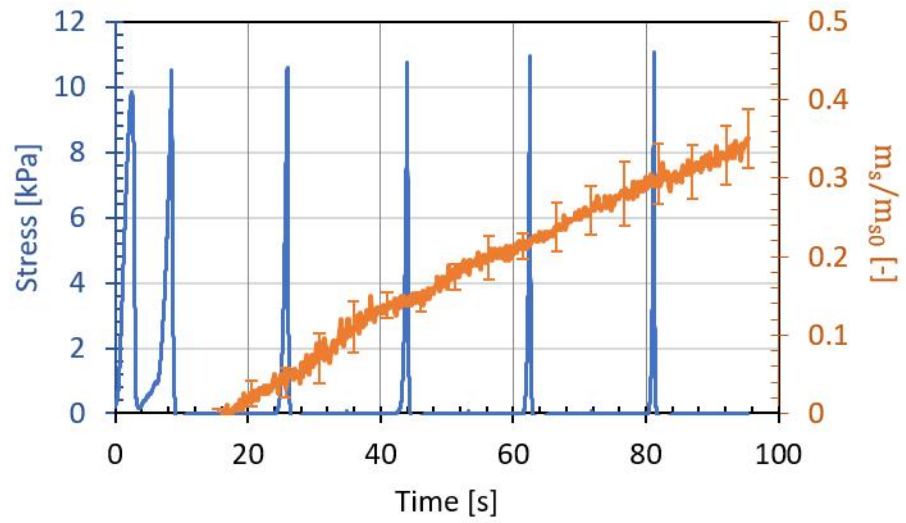
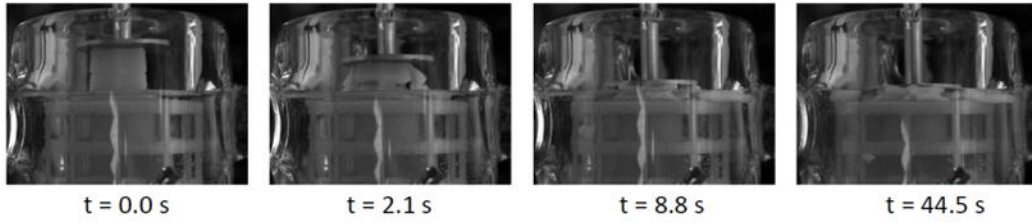
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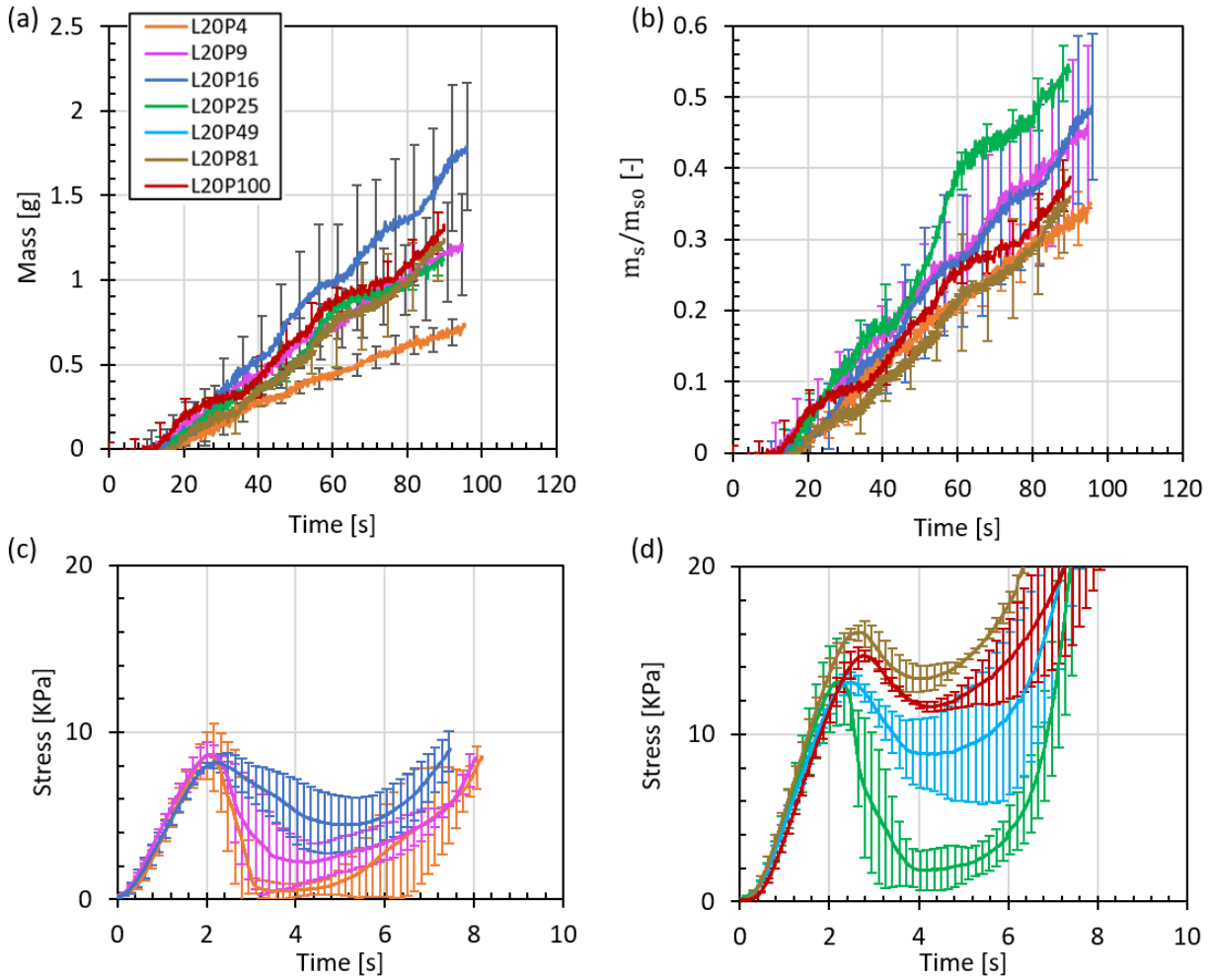
Figure 3. Comparison between measured and the design geometrical parameters for different starch structures: Image-J (gray), Caliper (dark blue), XCT (orange). (a) Porosity, (b) Surface area, (c) Specific surface area, (d) Mass. Each structure was labeled based on the length of its side (L) and the number of pores (P) (i.e. L20P4 is a print having a side of 20 mm and 4 pores).



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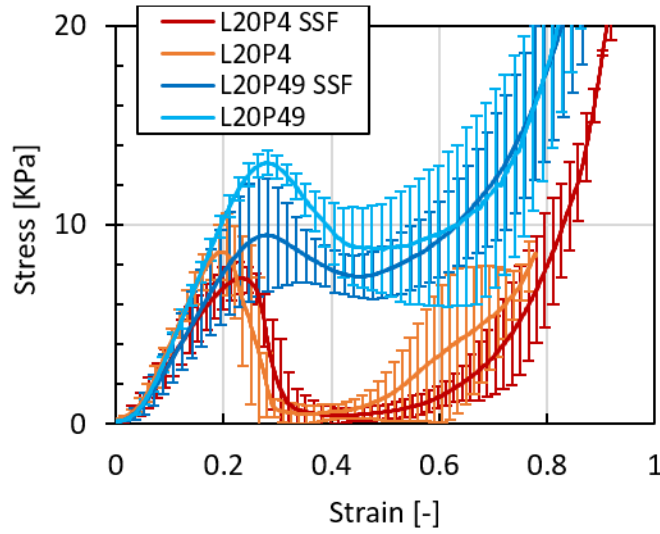
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Figure 4. A typical, dispersion-compression plot of a starch 3D printed structure



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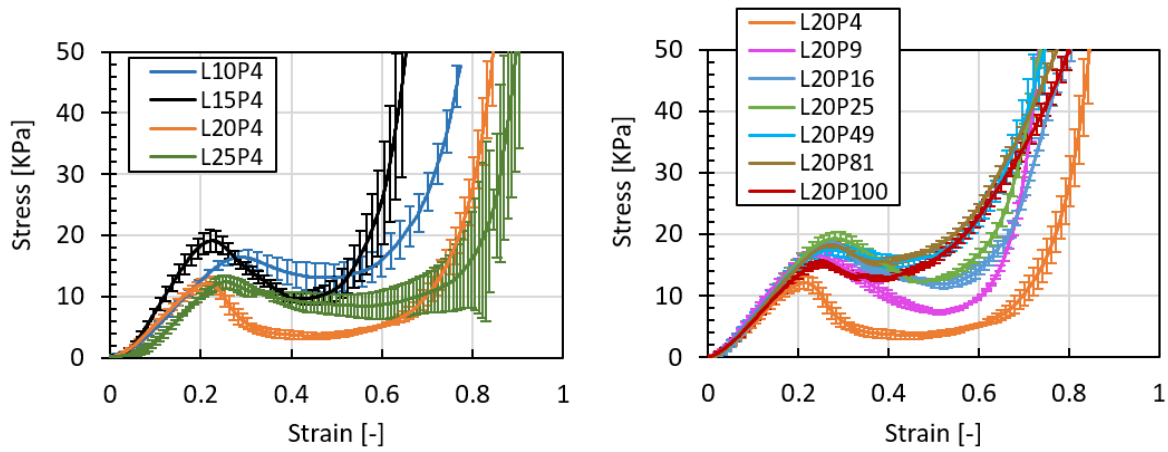
590 *Figure 5. Group 2 under maximum stress of 10 kPa (L20P4, L20P9 and L20P16) and Group 3 under maximum stress of 50 kPa*
 591 *(L20P25, L20P49, L20P81 and L20P100). (a) Starch mass dispersion of the Group 2 and 3 under hydration, (b) Normalized*
 592 *dispersion of starch from Group 2 and 3 under hydration, (c) Stress-time plots for structures of Group 2 under hydration, (d)*
 593 *Stress-time plots for structures of Group 3 under hydration.*



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Figure 6. Stress-Strain plot under hydration with water and SSF for structures L20P4 and L20P49



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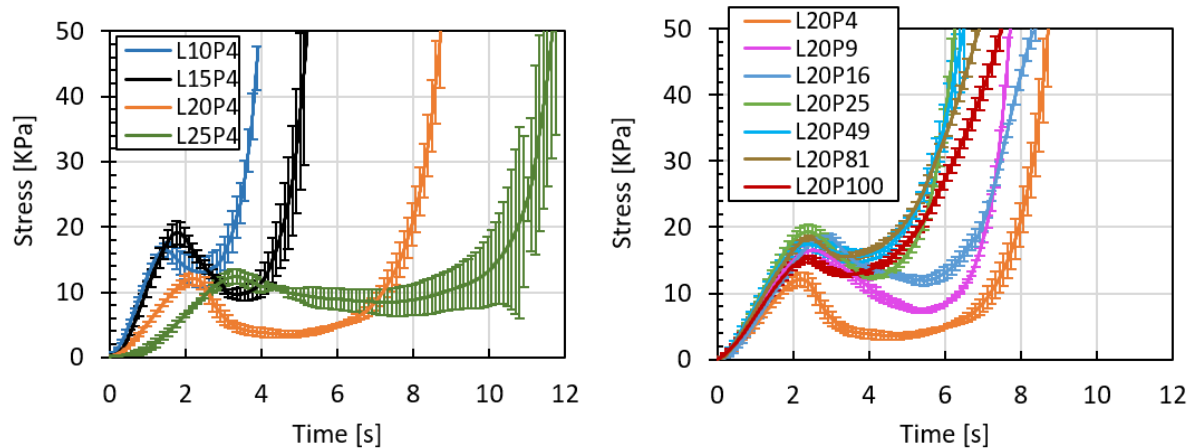
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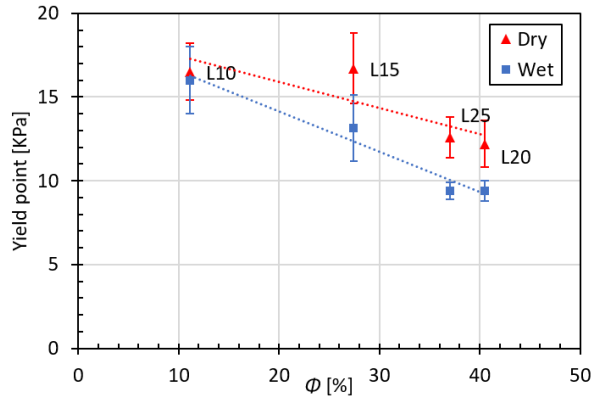
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Figure 7. Uniaxial compression in dry conditions of 3DP starch structures respectively: Group 1 (L10P4, L15P4, L20P4, L25P4), Group 2 (L20P4, L20P9, L20P16) and Group 3 (L20P25, L20P49, L20P81, L20P100). (left) Stress-strain plots for structures of Group 1, (right) Stress-strain plots of structures of group 2 and 3.

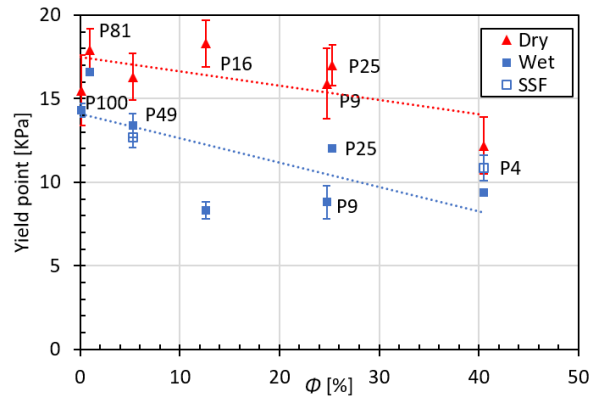
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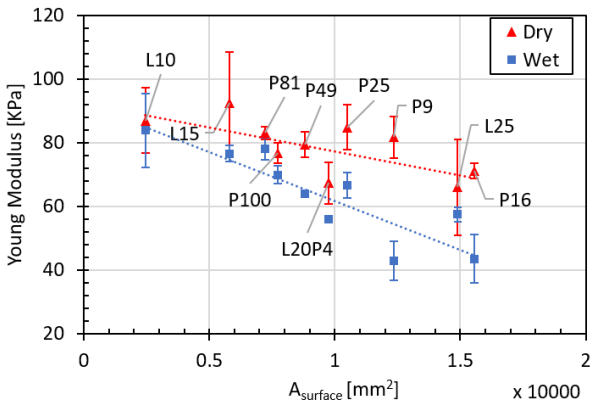
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 602 *Figure 8. Mechanical behavior under uniaxial compression in dry conditions of the prints respectively: Group 1 (L10P4, L15P4,*
 603 *L20P4, L25P4), Group 2 (L20P4, L20P9, L20P16) and Group 3 (L20P25, L20P49, L20P81, L20P100). (Left) Stress-time plots*
 604 *for structures of Group 1, (right) Stress-time plots for structures of Group 2 and 3.*



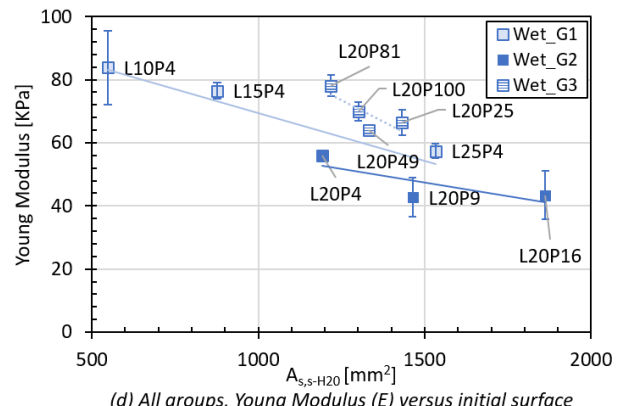
(a) Group 1 Yield point (σ_y) versus Porosity (Φ)



(b) Group 2 & 3 Yield point (σ_y) versus Porosity (Φ)



(c) All groups, Young Modulus (E) versus surface area (A_s)



(d) All groups, Young Modulus (E) versus initial surface area in contact with the liquid

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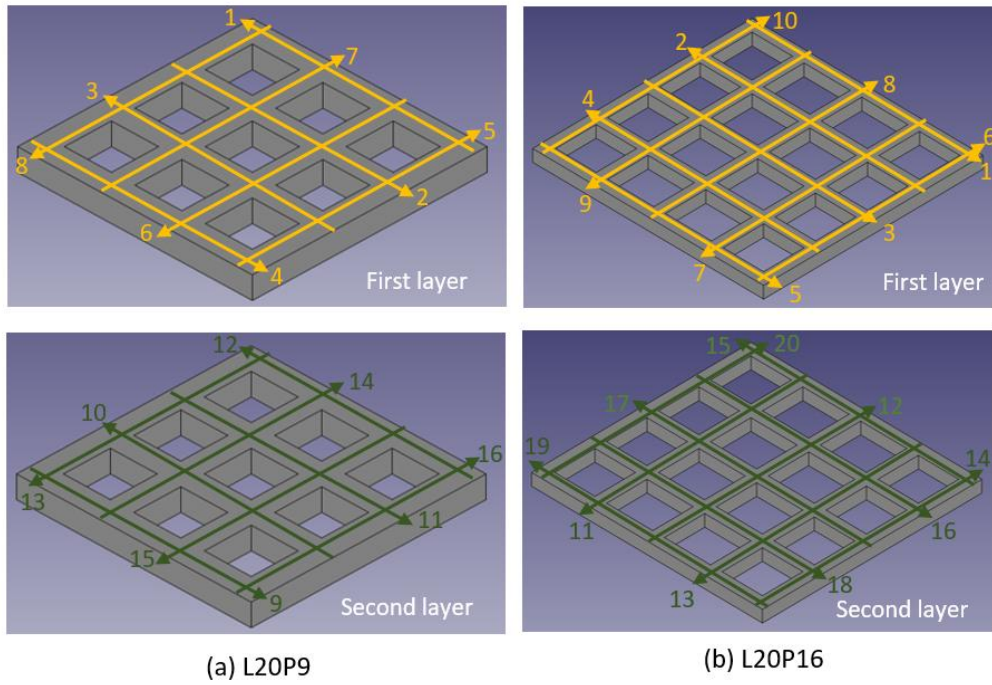
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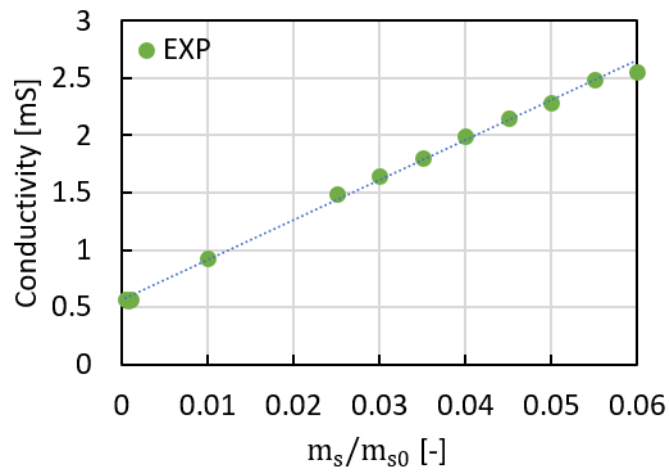
Figure 9. Comparison between yielding points in dry, water hydration conditions. The normal compression in wet conditions lowered the yielding properties of the prints. (a) Group 1 yield point (σ_y) versus Porosity (Φ), (b) Group 2 & 3 Yield point (σ_y) versus Porosity (Φ), (c) All groups, Young Modulus (E) versus surface area (A_s), (d) All groups, Young Modulus (E) versus initial surface area in contact with the liquid



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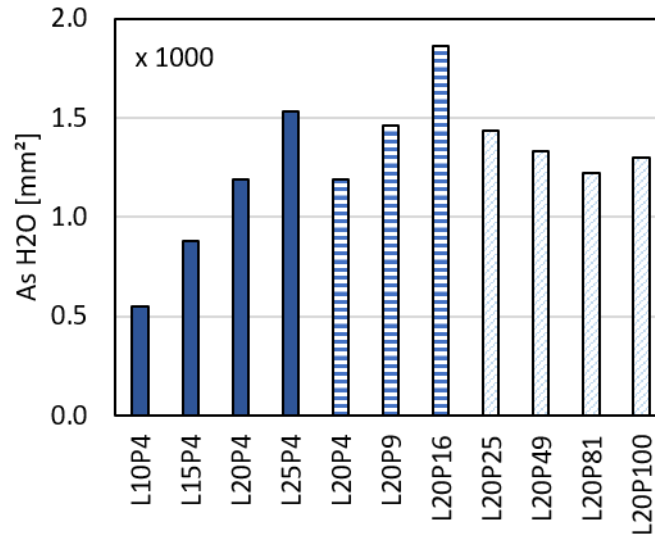
Figure A.10. Printing sequences used to print respectively the structures L20P9 and L20P16.



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Figure A.11. Calibration curve of the conductivity probe using pre-gelatinized starch Colorcon 1500



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Figure A.12. Initial contact area between the starch and the liquid

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Table 1. Summary of printed cubic structures, classified by the number of pores, size, and the number of layers. The measured

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Cross Section (A_c) and Target Maximum Forces (F_{max}) computed for the compression protocol to achieve a uniaxial

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compression at 10 KPa and 50 KPa are listed in the last two columns.

Structure Name		No. of pores	Size (mm)	No. of layers	$A_c [mm^2]$	$F_{max} [N]$	
						$\sigma_{max} = 10 \text{ KPa}$	$\sigma_{max} = 50 \text{ KPa}$
G1	L10P4	4	10	25	134.8 ± 0.7	1.3	6.5
	L15P4		15	40	199.8 ± 5.1	2.0	10.0
	L20P4		20	51	288.8 ± 15.9	2.9	14.5
	L25P4		25	64	400.2 ± 38.8	4.0	20.0
G2	L20P4	4	20	51	288.8 ± 15.9	2.9	14.5
	L20P9	9			356.6 ± 18.2	3.5	17.8
	L20P16	16			437.9 ± 29.5	4.4	21.9
G3	L20P25	25	20	23	289.5 ± 7.5	2.9	14.5
	L20P49	49			433.8 ± 15.2	4.3	21.5
	L20P81	81			553 ± 7.3	5.5	27.6
	L20P100	100			590.1 ± 17.4	5.9	29.5


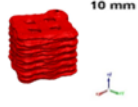
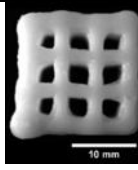
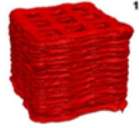


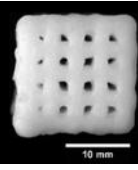
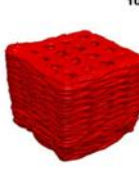
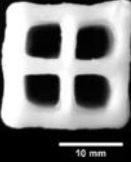

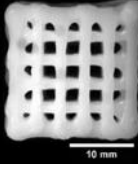

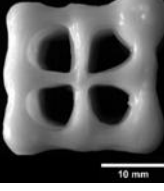

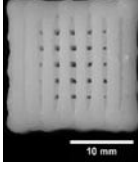
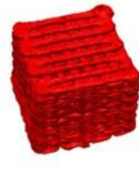
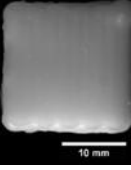

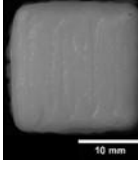

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Table 2. Mass of the structures measured after printing

Structure Name		Mass [g]
G1	L10P4	1.97 ± 0.15
	L15P4	3.98 ± 0.09
	L20P4	6.98 ± 0.39
	L25P4	13.48 ± 0.23
G2	L20P4	6.98 ± 0.39
	L20P9	8.79 ± 0.68
	L20P16	12.25 ± 0.47
G3	L20P25	6.89 ± 0.14
	L20P49	8.88 ± 0.37
	L20P81	11.35 ± 0.28
	L20P100	12.20 ± 0.08

623 Table 3. Comparison of porosity and surface area of printed structures estimated using a Caliper and measured using XCT

Name	Top View Camera	XCT	Name	Top View Camera	XCT
L10P4			L20P9		
Φ [%]	12.95	11.13	Φ [%]	28.79	24.75
A_s [mm^2]	1048.04	2454.10	A_s [mm^2]	5593.96	12350.47
V_s [mm^3]	1218.25	1564.01	V_s [mm^3]	7540.16	7450.00
L15P4			L20P16		
Φ [%]	36.82	27.44	Φ [%]	13.56	12.62
A_s [mm^2]	2712.76	5802.52	A_s [mm^2]	5411.96	15557.86
V_s [mm^3]	2792.41	3634.89	V_s [mm^3]	8612.64	9891.18
L20P4			L20P25		
Φ [%]	44.73	40.49	Φ [%]	36.93	25.32
A_s [mm^2]	4848.11	9757.74	A_s [mm^2]	6578.59	10550.00
V_s [mm^3]	5735.00	6126.18	V_s [mm^3]	7978.37	6595.00
L25P4			L20P49		
Φ [%]	42.52	37.03	Φ [%]	8.67	5.27
A_s [mm^2]	6807.00	14900.00	A_s [mm^2]	5527.04	8805.00
V_s [mm^3]	8872.67	11566.67	V_s [mm^3]	8285.88	7290.00
L20P81			L20P100		
Φ [%]	0	1.00	Φ [%]	0	0.11
A_s [mm^2]	3211.62	7215.00	A_s [mm^2]	3427.18	7725.00

$V_s [mm^3]$	9935.67	8195.00	$V_s [mm^3]$	10670.99	9005.00
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625 *Table 4. Characteristic dispersion times t_{20} when 20% of starch is dispersed and fraction of starch dispersed at the end of*626 *deconstruction and hydration experiments.*

Maximum uniaxial stress [σ_{max}]	Structure name		t_{20} [s]	m_s/m_{s0} (@ t_{end}) [%]
10 kPa	G2	L20P4	58.2	31.9 ± 0.0
		L20P9	46	43.9 ± 0.1
		L20P16	47	45.0 ± 0.1
50 kPa	G3	L20P25	44.4	53.7 ± 0.0
		L20P81	57.5	36.0 ± 0.1
		L20P100	52.7	38.3 ± 0.0

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628 *Table 5. Summary of Young Modulus (E) and Yield Point (σ_y), measured in dry and wet conditions.*

Structure Name		Dry		Wet	
		σ_y [KPa]	E [Pa]	σ_y [KPa]	E [Pa]
G1	L10P4	16.5 ± 1.7	86.9 ± 10.3	16.0 ± 2.0	83.8 ± 11.7
	L15P4	16.7 ± 2.1	92.5 ± 15.9	13.1 ± 2.0	76.5 ± 2.5
	L20P4	12.2 ± 1.4	67.3 ± 6.6	9.4 ± 0.6	55.9 ± 1.0
	L25P4	12.6 ± 1.2	66.0 ± 15.1	9.4 ± 0.5	57.4 ± 2.3
G2	L20P4	12.2 ± 1.4	67.3 ± 6.6	9.4 ± 0.6	55.9 ± 1.0
	L20P9	15.9 ± 1.3	81.7 ± 6.5	8.8 ± 1.0	42.8 ± 6.1
	L20P16	18.3 ± 2.1	71.1 ± 2.3	8.3 ± 0.5	43.4 ± 7.6
G3	L20P25	17.0 ± 1.1	84.8 ± 7.1	12.0 ± 0.2	66.5 ± 4.0
	L20P49	16.3 ± 1.0	79.4 ± 4.1	13.4 ± 0.7	64.0 ± 1.0
	L20P81	17.9 ± 0.6	83.0 ± 2.1	16.6 ± 0.2	78.1 ± 3.4
	L20P100	15.5 ± 0.5	76.7 ± 3.3	14.3 ± 0.4	69.9 ± 2.9

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630 *Table A.6. Summary of the dimensions measured with the caliper after printing*

Structure Name		Length [mm]	Width [mm]	Height [mm]
G1	L10P4	10.46 ± 0.44	11.48 ± 0.71	10.58 ± 0.13
	L15P4	16.74 ± 0.45	16.50 ± 0.38	15.97 ± 0.52
	L20P4	22.49 ± 0.43	22.63 ± 0.12	20.34 ± 0.42
	L25P4	26.72 ± 0.48	26.30 ± 0.23	25.47 ± 0.42
G2	L20P4	22.49 ± 0.43	22.63 ± 0.12	20.34 ± 0.42
	L20P9	22.41 ± 0.15	22.47 ± 0.09	21.02 ± 0.03
	L20916	22.00 ± 0.45	22.01 ± 0.68	20.57 ± 0.39
G3	L20P25	21.45 ± 0.19	21.42 ± 0.22	17.36 ± 0.29
	L20P49	21.72 ± 0.28	21.64 ± 0.39	17.62 ± 0.10

	L20P81	23.59 ± 0.18	23.46 ± 0.18	17.95 ± 0.11
	L20P100	24.15 ± 0.45	24.14 ± 0.43	18.29 ± 0.24

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Table A.7. Summary of the dimensions measured with the caliper after printing (cont.)

Structure Name		Pore Length [mm]	Pore Width [mm]
G1	L10P4	1.96 ± 0.07	2.11 ± 0.33
	L15P4	5.02 ± 0.22	5.05 ± 0.26
	L20P4	7.54 ± 0.30	7.54 ± 0.12
	L25P4	8.62 ± 0.42	8.66 ± 0.15
G2	L20P4	7.54 ± 0.30	7.54 ± 0.12
	L20P9	4.06 ± 0.37	3.95 ± 0.48
	L20916	2.1 ± 0.23	1.94 ± 0.31
G3	L20P25	2.61 ± 0.03	2.61 ± 0.03
	L20P49	0.97 ± 0.08	0.94 ± 0.02
	L20P81	-	-
	L20P100	-	-

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