

1                   **Assessment on bulk solids best practice techniques for flow**  
2                   **characterization and storage/handling equipment design for biomass**  
3                   **materials of different classes**

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11                  **Abstract**

12                  This paper shows the results of a collaboration project in which four different laboratories have carried out  
13                  complementary characterizations of samples of the same set of lignocellulosic biomass samples with the  
14                  purpose to better understand material properties and to highlight possible critical features of different  
15                  biomass characterization procedures. Three different types of materials were used as biomass models: 1)  
16                  scots pine wood chips, as an example of coarse and flaky with some elastic properties; 2) chopped straw of  
17                  reed canary grass as nesting biomass having chops (flaky, long and nesting fibers) 3) Scots pine wood  
18                  powder as (elastic and cohesive). Particle size and shape analyses were carried out with calipers, with 2D  
19                  image analysis, with 3D image analysis (ScanChip) and through sieving. Applications and validity limits of  
20                  each of these techniques were evaluated and discussed. Flow function and internal friction was determined  
21                  by the use of a Schulze ring shear tester; a Brookfield powder flow tester and a large ring shear tester and  
22                  no major large differences in results were found between them. The Schulze ring shear tester; a Brookfield  
23                  powder flow tester; a large Jenike shear tester and a Casagrande shear box. Results, in this case showed

24 bigger differences. Tensile strengths were used for wall friction measurements. A higher wall friction  
25 coefficient was obtained with the larger shear cell; as well as the Schulze and Brookfield tester. clear  
26 Additionally, tensile strength of biomass materials were also measured by the use of a novel measurement  
27 technique. Arching tests were carried out in a pilot scale plane silo with variable hopper geometry and  
28 results were compared with those predicted by applying the Jenike procedure and with those predicted  
29 when assuming tensile strength as the controlling material property. Finally, safety of handling and storage  
30 was assessed by carrying out explosion tests on dusts from Scots pine and reed canary grass.

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32 KEYWORDS: lignocellulosic biomass, particle size, particle shape, flowability, arching, explosion

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34 RESEARCH HIGHLIGHTS:

- 35 • Suitable test methods were classified for three biomass classes
- 36 • Different particle sizing methods give a wide range of results
- 37 • Shear tests do not discriminate flowability differences between the biomass classes
- 38 • Arching behavior depends more on tensile strength than on compressive strength
- 39 • Scots pine and reed canary grass dusts are flammable

40

## 41 **1 Introduction**

42 The interest in solid biomass has been increasing over the last decades for their potential as renewable  
43 energy sources and as raw material for biorefineries, producing a great variety of added-value products in  
44 well integrated production chains [1-3]. Industrial use of lignocellulosic biomass implies an increase in  
45 demand for robust and reliable solid bulk handling. In general, plants involving use of solids are  
46 characterized by significantly longer start up times, larger start up costs and by plant through-puts which  
47 may be significantly reduced with respect to their design value [4]. Furthermore, feedstock from

48 lignocellulosic biomass as raw materials, by-products, residuals, and waste is highly versatile. There are  
49 differences between plant species but also in structural elements within individual plants (e.g. phloem,  
50 xylem, bark, leaves/needles, roots, fruits etc.) adding more variation to physical and chemical properties,  
51 besides that of moisture content and potential contaminants. Moreover, mechanical and thermal  
52 pretreatments before feeding to conversion plants can significantly change the properties of biomass  
53 solids. In particular, milling [5-7], densification [8, 9] and torrefaction [10, 11,] affect particle size and shape  
54 distribution, bulk density and energy density. With specific reference to solids made by particulate  
55 biomasses, handling and feeding present further difficulties due to peculiar properties both at particle level  
56 and at bulk level that make these materials even more unpredictable than other granular materials  
57 traditionally processed by industry, especially in terms of flow reliability and control [12-14]. Therefore  
58 robust and reliable characterization methods for particulate biomasses are urgently needed, but also as  
59 reference for calibration models enabling fast non-destructive methods suitable for on-line analysis of rapid  
60 material streams, e.g. spectrometry [15-17].

61 Flow problems can be correctly addressed through knowledge of flow properties of bulk solids and by  
62 availability of reliable design methods for industrial silos. However, standard characterization methods  
63 used for flow properties of common granular solids are not always suitable for biomass materials. A  
64 significant problem is that conventional shear testers have been mainly developed for measuring frictional  
65 and cohesive properties of bulk solids with quite regular particle shapes and with particle top sizes below a  
66 few millimeters [18-20]. Few biomass materials have both particle size and shape distributions that meet  
67 these requirements. Thus for large biomass particles one possibility is to use a larger scale shear tester [21].  
68 However, for the biomass materials formed of elongated particles or fibers, like straws and grasses, there  
69 are question over the validity of shear testing as a characterization technique. These materials are highly  
70 compressible and comprise particles that are severely entangled hindering shear zone formation and the  
71 attainment of a steady state flow condition [22-24]. Moreover, preliminary studies indicate that tensile  
72 strength could be a more relevant flow property than compressive strength due to mechanical interlocking  
73 caused by entanglements between fibrous solids [25]. These observations indicate that there is a need to

74 more deeply assess the validity of elasto-plastic constitutive models and the choice of suitable testers for  
75 characterization of biomass particulate solids flow properties.

76 In addition to this, common design methods of storage units to ensure flow, based on Jenike analysis [26],  
77 have not been proved yet to work for biomass bulk solids. Tendencies of arch or bridge formation at the  
78 hopper outlet has been investigated in several studies. Flat bottomed containers with an opening slot were  
79 used to experimentally derive the critical outlet size for arching as a function of particle size distribution,  
80 particle shape and moisture content of the biomass sample [27-30], of air promotion flow [31] or of the  
81 milling procedure [32]. A similar apparatus based on the same method was also recently proposed as a  
82 reference method for European standardization [33]. However, the applicability of these results on real  
83 scale silos and on conical or wedge-shaped hoppers was not proved. With this respect, a clue on the effect  
84 of bulk material consolidation, dependent on storage unit size, is the observed influence of material bed  
85 height on the critical opening size [27, 30]. The role of consolidation has been confirmed by arching tests on  
86 biomass beds compacted by an external load [34, 35]. Barletta and Poletto carried out a direct assessment  
87 of the Jenike design method performing experiments on a wedge shaped hopper [36, 37]. The results of  
88 these studies indicate that the design procedure is adequate for the tested wood powder samples. Further  
89 investigations are needed to complete the assessment. However, the effect of material consolidation state,  
90 related to bed height and container diameter, on arching propensity was not fully addressed.

91 Furthermore, safety issues related to dust generation during [38] biomass handling need to be addressed.  
92 Fires (due to self-heating during storage) and dust explosions are two important issues in biomass bulk  
93 handling, because they may result in worker injuries, loss of lives, considerable economic losses and  
94 environmental damage. A dust explosion is the result of a quick combustion of fine particles dispersed in  
95 air. In presence of an ignition source they react with oxygen, generating an exothermic chain reaction. If  
96 these reactions occur inside a vessel the system pressure increases rapidly [39, 40]. Under these  
97 circumstances, venting devices are designed to release pressures, and indeed, as protection measures  
98 usually are the only options. Explosion pressures can reach 7-10 bar in a closed vessel with no protective  
99 system. Considering that walls and roofs of typical biomass containers are not designed to bear such

100 pressures, explosions may lead to serious structural damages, including complete silo destruction. The  
101 most significant parameters characterizing the violence of an explosion are the maximum pressure reached  
102 ( $P_{max}$ ) and the maximum rate of pressure rise ( $K_{st}$ ). However, there is a lack of data concerning these  
103 parameters for biomass materials. Still, the majority of reported industrial explosions had their origin in  
104 organic (carbon) dusts, and compositional similarities with biomass materials suggests that there may be  
105 hidden dangers of explosivity among these materials.

106 This paper shows the results of a collaboration project (Bio4Flow) in which four different laboratories have  
107 carried out complementary characterization of three sets of biomass samples with the purpose to better  
108 understand the material properties and to highlight possible critical features of biomass characterization  
109 procedures. The laboratories involved are:

- 110 - the Biofuel Technology Centre at the Swedish University of Agricultural Sciences, Sweden (SLU BTC);
- 111 - the Wolfson Centre for Bulk Solids Handling Technology at the University of Greenwich, United  
112 Kingdom (UG Wolfson);
- 113 - the Powder Technology Group of the University of Salerno, Italy (US PTG);
- 114 - the BIPREE Research Group of the Technical University of Madrid, Spain (UPM BIPREE).

## 115 **2 Materials and Methods**

### 116 **2.1 Materials**

117 Three material assortments were chosen to represent common types of biomass bulk solids: wood chips,  
118 wood powder, and straw chops. Scots pine (*Pinus sylvestris* L.) wood chips (Figure 1a) with particle sizes of  
119 approximately <math>25 \times 15 \times 5</math> mm and a moisture content of 10-15% (wet basis) were collected at a sawmill  
120 (Sävar såg, Sävar, Sweden). Wood powder (Figure 1b) was produced from the wood chips assortment by  
121 hammer milling (Vertica Hammer Mill DFZK-1, Bühler AG, Uzwil, Switzerland) using a screen size of 4 mm.  
122 Straw chops with a moisture content 10-15% (wet basis) was produced from reed canary grass (RCG)

123 (*Phalaris arundinacea* L.) and shredded in a single shaft shredder (Lindner Micromat 2000, Lindner-  
124 Recyclingtech GmbH, Spittal, Austria) with 40 mm screen size (Figure 1c).

125 Different methods were used to characterize these biomass materials. The methods are presented in the  
126 following sections. Table 1 provides a summary of the methods adopted.

## 127 **2.2 Particle size distribution measurement methods**

128 Particle size distributions and shapes were measured with the following techniques (also listed in Table 2):

### 129 **Sieve analysis**

130 Particle size distributions of wood chips and straw chops were determined with an oscillating screen  
131 method according to the European standard for determination of particle size distribution of solid biofuels  
132 with particle sizes >3.15 mm [41]. Wood powder particle size distribution was determined with a vibrating  
133 screen method according to the European standard for determination of particle size distribution of solid  
134 biofuels with particle sizes <3.15 mm [42]. Sieve analyses were assumed to provide a measure of the  
135 particle width.

### 136 **2D image analysis**

137 The particle size distribution via 2D image analysis [43] was carried out only on samples of the biomass  
138 types with larger particles, i.e. wood chips and RCG straw. This procedure, in fact, includes hand  
139 preparation of samples which was not affordable with wood powder particles.

140 The first operation was the powder sampling. A sample of about 100 g of the powder was scooped from  
141 three positions in the bag. The sample was gently mixed and divided into four wood chips and sixteen RCG  
142 portions. In both cases, each single portion was weighed and sieved on a standard ASTM (American Society  
143 for Testing and Materials) screen with 2 mm aperture size. Obtained samples were spread over a black A4  
144 paper sheet over the plane of a photographic bench. A caliper ruler was placed at one side of the sheet, in  
145 order to calibrate images. The photographic bench was equipped with 4 lamps (each power 250 W)  
146 connected to a DC supply to avoid light fluctuations. Digital pictures were taken with a Nikon D100 (Nikon  
147 Co, Tokio, Japan), equipped with standard Nikkor 50 mm focal length lenses. Digitized images were

148 analyzed with the help of Image-Pro Plus Software of the Media Cybernetics, Inc (Rockville, MD USA). After  
149 calibrating the image with the ruler and selecting an area of interest which excluded image borders, the  
150 built in procedure “count/size” was run. Selected particle measurements were:

- 151 • Particle area projected area,  $A$  [ $\text{mm}^2$ ]
- 152 • Maximum diameter [ $\text{mm}^2$ ], i.e. the longest line joining two points of the object outline and passing  
153 through the centroid
- 154 • Minimum diameter [ $\text{mm}^2$ ], i.e. the shortest line joining two points of the object outline and passing  
155 through the centroid
- 156 • Major axis [ $\text{mm}^2$ ], i.e. the length of the major axis of an ellipse with the same moments of order 1  
157 and 2 order as the particle image
- 158 • Minor axis [ $\text{mm}^2$ ], i.e. the length of the minor axis of an ellipse with the same moments of order 1  
159 and 2 order as the particle image
- 160 • Particle roundness [-], defined as  $P^2/(4\pi A)$ , where  $P$  is the perimeter [ $\text{mm}$ ] of the projected particle  
161 image, i.e. the ratio of the area of a circle having the perimeter of the projected particle over the  
162 particle projected area

163 From these measurements other quantities were obtained, namely:

- 164 • Particle equivalent diameter [ $\text{mm}$ ], i.e. the diameter diameter of the circle having the same  
165 projected area as the particle
- 166 • Particle elongation ratio [-], i.e. the ratio of the maximum axis over the minimum axis.
- 167 • Size ratio [-], i.e. the ratio of the maximum diameter over the minimum diameter.

168 Tabular outputs of the “count/size” Image-Pro Plus procedure were exported to a spreadsheet file to  
169 collect the results of more images. To analyze about 60 g of wood chips, 8 images were used for a total  
170 count of about 1100 particles. To analyze about 4.2 g of RCG, 7 images were used for a total count of about  
171 8800 particles.

172 For each material, all data collected on the Excel file was ordered and grouped in order to obtain  
173 experimental cumulative distributions.

174 ***3D image analysis***

175 Length, width and thickness of single particles were scanned with resolution of 0.22×0.22×0.17 mm using a  
176 ScanChip analysis system for optical measurement (Andritz, Iggesund Tools, Iggesund, Sweden). This device  
177 uses vibratory conveyors to separate particles from each other before measuring particle dimensions at a  
178 calibrated speed on a transport band by a laser-supported camera and laser triangulation. The system was  
179 in this study set to measure particles with thickness >1 mm.

180 ***Caliper***

181 A pair of digital Vernier calipers was used to manually measure the approximate length, breadth and  
182 thickness of a cuboid around the particle (to the nearest 0.5 mm) of small samples of each biomass type.  
183 Note that due to the elasticity and fragility of the fibers the micrometer was lined up to the respective  
184 dimension by eye, rather than by gently tightening the jaws, as it is usually done for solid objects. The  
185 samples were taken from the bulk and cut at above 2 mm. Fines were not measured due to the difficulties  
186 involved in manual handling.

187 ***2.3 Flow properties test methods***

188 ***2.3.1 Internal flow properties***

189 Internal flow properties of materials were tested with different equipment and methods. These are  
190 described in the following:

191 ***Schulze ring shear tester - RST***

192 Yield loci measurements for straw chops and wood powder particles were made with a Schulze [44] ring  
193 shear tester RST-01.01 (Dr. Dietmar Schulze Schüttgutmesstechnik, Wolfenbüttel, Germany) according to  
194 the ASTM standard [45]. Materials were sheared at four different consolidating stresses (ranging from 4.6  
195 to 20.9 kPa). It was not considered meaningful to take measurements for wood chips due to too large  
196 particle sizes.



197 ***Brookfield Powder Flow Tester - PFT***

198 The Brookfield powder flow tester (PFT) is an automated annular shear tester as described in [46]. The  
199 main specification is as follows; the powder is stored in an annular trough of inner and outer diameters 100  
200 and 150 mm, respective, and depth 19 mm. The trough is enclosed by an annular lid of the closed pocket  
201 design after Walker [47] where the 18 lid pockets are formed by a radius cavity with equally spaced vertical  
202 vanes.

203 The sample is consolidated and failed by applying a torsional load by rotating the trough while a controlled  
204 axial is applied through the lid. The rotational speed of the trough is one revolution per hour, the normal  
205 stress range of the machine is 0.3 to 4.8 kPa. The sampling frequencies for the axial and torsional loads is  
206 50Hz.

207 The shear test algorithm followed is essentially similar to the one described in ASTM 6128 for the Jenike  
208 shear tester. The key difference is the inclusion of shear stress peak at the consolidation normal stress in  
209 the failure locus construction.

210 ***Large Annular Shear Tester - LAST***

211 The large annular shear tester (LAST) at Wolfson Centre is a large manually operated annular shear tester.  
212 Outer and inner diameters of the trough are 1 m and 0.75m, respectively. The trough depth is 0.15 m. The  
213 annular lid is of the open pocket design (Schulze [44]) where the underside of the lid is flat, with pockets  
214 formed by 18 evenly spaced vertical vanes with sides that are open.

215 The 52 kg lid is suspended from its centre on a counter balance beam. The desired consolidation load is  
216 achieved by applying dead weights to the lid or counter balance. To shear the sample, the trough is slowly  
217 rotated at (1 rev/hr) while the rotation of the lid is prevented by a torque arm connected to load cells by a  
218 pair of tie bars. The normal stress range is low due to the large normal loads required on the large area of  
219 the lid.

220 The test algorithm followed manually is the same as that described above for the PFT.

## 221 ***Tensile tester -TT***

222 The tensile tester (TT) is at present a prototype for proving the principal and still requires significant  
223 refinement. The tester comprises a pair of identical rectangular cells with a vertical split down the centre.  
224 The cells are clamped together and the extreme shape biomass evenly filled into the cell and leveled. A pair  
225 of independent lids and dead weights is applied to the top free surface of the biomass in the two cell halves  
226 to generate the required normal stress. To enable tensile failure of the sample, one cell half is fixed to the  
227 frame of the machine while the other half is suspended from the frame via four wires in the form of a  
228 parallelogram linkage. On the end of this moving cell, half a horizontal cord passes over a pulley to a weight  
229 hanger. With a given normal load acting on the sample, increasing horizontal tensile loads can be applied  
230 by increasing dead weight on the pulley until failure and separation occur. Only the peak load is measured.  
231 As the cell is on a parallelogram linkage the vertical component of the force must be subtracted to  
232 determine actual horizontal force at failure. The height of the sample at failure is measured to determine  
233 bulk density and the area of the tensile failure plane. The data is presented as a tensile strength function  
234 i.e. tensile strength as a function of uniaxial consolidation stress. The consolidation stress range of the  
235 equipment is approximately from 0.2 to 3kPa.

## 236 **2.3.2 Wall friction properties**

237 Wall friction properties of materials were tested with different equipment and methods. Besides the  
238 already mentioned Schulze ring shear tester these are described in the following:

### 239 ***Large Jenike wall friction - LWFT***

240 The large wall friction tester (LWFT) is a linear device with a 270 mm diameter shear ring (20mm depth)  
241 that rest on a wall sample of 300 x 500mm dimension. The biomass sample is enclosed with a compression  
242 lid. The wall normal stress range of the tester is 0.5 to 12kPa, while the wall shear stress range is 0 to 6kPa.  
243 Shearing is facilitated by pulling the sample along the wall. This motion is produced by a linear slide (driven  
244 by an electric pistol drill) travelling at a constant speed of 0.6mm/s.

## 245 ***Casagrande shear box – CG WFT***

246 The device used was a circular shear tester, 10 cm in diameter and 3 cm deep. Its design (Figure 2) was  
247 based on the Casagrande shear box (CG WFT) which allows relative displacement of parts of the box to be  
248 controlled [48, 49]. Direct shear tests were used to determine the particle-to-wall friction coefficient. The  
249 cell of the direct shear apparatus was modified by inserting a steel mold as shearing surface (Figure 2).

## 250 ***2.4 Arching test methods***

251 Discharge experiments were carried out in a plane silo (Figure 3), with a total volume of about 0.3 m<sup>3</sup>,  
252 formed by a parallel-piped bin and a wedge-shaped hopper in which it is possible to independently change  
253 both hopper steepness and width of the outlet slot. Transparent glass front and rear walls of the silo allow  
254 visual inspection of the flowing solids inventory. All other silo walls are made of stainless steel. The adopted  
255 experimental procedure includes: i) adjustment of hopper steepness and outlet slot width; ii) loading of  
256 biomass from the silo top while the opening of the hopper is closed by a slab, held by a hydraulic piston; iii)  
257 biomass levelling with a rake; iv) very slow lowering of the closing slab by operating the hydraulic piston  
258 while monitoring the flow regime (flow or arching) by photo or video recording. For each value of hopper  
259 angle,  $\alpha$ , within the hopper mass flow range, experiments were repeated with different outlet openings to  
260 find the maximum opening size,  $D_c$ , giving rise to stable arch formation.

## 261 ***2.5 Explosion test methods***

262 Values defining the maximum explosion pressure ( $P_{\max}$ ) and the characteristic constant ( $K_{st}$ ) for explosion  
263 class have been determined in this research work according to the current normative for explosibility  
264 characterization (UNE-EN 14034-1:2005+A1:2011). The maximum pressure  $P_{\max}$  is the difference between  
265 pressure at time of ignition (normal pressure) and pressure at the highest point in the pressure-time  
266 record. The test device used was a Kühner 20-l sphere. Three series of tests were carried out, and results  
267 showed a deviation of less than 10% from the average, which is considered acceptable. The maximum  
268 explosion pressure rise maximum explosion pressure rise  $(dP/dt)_{\max}$  is defined as the maximum slope of the  
269 tangent to the pressure vs. time curve at each nominal fuel concentration. The characteristic constant ( $K_{st}$ )

270 is obtained from the product  $(dP/dt)_{\max}$  multiplied by the cubic root of the explosion enclosure volume. The  
271 explosion class is defined as a function of the  $K_{st}$  values, as follows: St0 (non-explosible) for  $K_{st}=0$  mbar/s;  
272 St1 (weak) for  $K_{st}=1-200$  mbar/s; St2 (strong) for  $K_{st}=201-300$  mbar/s; St3 (very strong) for  $K_{st}=300$  mbar/s.

## 273 **3 Results**

### 274 **3.1 Particle size distributions**

275 Comparisons between results for different particle size measurement methods are shown in Figure 4.  
276 Caliper measurements and 2D analysis are very work intensive. Hence, sample sizes were not as large as for  
277 sieving or 3D image analysis. Representative sampling is crucial for comparisons, but is very difficult to  
278 ensure. Results are all expressed in terms of weight fractions. Particles passing the smallest sieve aperture  
279 size of 3 mm were assumed to have an aperture size of 1.5 mm. Length distribution of wood chips (Figure  
280 4a) and RCG (Figure 4b) shows a good agreement between techniques except for 2D image analysis for  
281 RCG. Width distribution of wood chips (Figure 4c) and RCG (Figure 4d) shows that 3D and caliper as well as  
282 2D and sieving generally match in couples. The above discrepancy may be due to that 2D and sieving  
283 covered the full size range, whereas manual calipers and the ScanChip instrument used for 3D analysis  
284 (which was set to detect particles larger than 1 mm in thickness) could not take into account finer particles.  
285 Measurements of the particle thickness could only be performed with caliper and 3D image analysis (Figure  
286 4e for wood chips and Figure 4f for RCG). However, it must be noted that is somewhat skewed only  
287 particles with a thickness  $>1$  mm were analyzed in the 3D data.

288 2D analysis provided some information on particle shape. Figure 5a presents the cumulative elongation  
289 distribution with reference to the particle projected area. RCG shows much more elongated particles than  
290 wood chips. In order to understand if shape is related to particle size Figure 5b reports the average  
291 equivalent particle diameter in terms of projected area, for the different elongation classes used for the  
292 cumulative distribution. is concluded For wood chips, elongations aresmaller particles are more elongated  
293 than large particles. This is probably due to fragmentation of larger particles which preferentially are  
294 separated in the fiber direction. On the contrary, larger size RCG particles are more elongated than smaller

295 ones. This is not surprising since RCG particles are obtained through chopping at length larger than the  
296 straw width. Furthermore, RCG particle fibers are very frail and breakage can occur also perpendicularly to  
297 the fiber direction.

298 Wood powder particles were too small to be measured with 3D Scanchip, 2D image analysis, or with a  
299 caliper. Therefore only sieving measurement is available for this powder and reported in Figure 6. In  
300 particular, laser diffraction was applied only to the cut obtained after sieving by a 1400  $\mu\text{m}$  sieve. The  
301 cumulative particle size distribution reported was calculated taking into account the volumetric fraction  
302 under and over 1400  $\mu\text{m}$ .

303

### 304 ***3.2 Flow properties***

305 The flow properties were measured using a range of shear testers, namely the Brookfield PFT, Schulze RST,  
306 the Casagrande shear box (CG WFT), the Wolfson Centre large annular shear tester (LAST) and large wall  
307 friction tester (LWFT) as well as the tensile tester (TT). The full size RCG was tested in the large cells while  
308 fines were tested in the standard testers. For wood powder, the full size range was tested on all machines,  
309 while only the large cells were capable of testing the wood chips.

310 Extreme shape materials do not develop a shear plane (coincident with the underside of the lid) when  
311 exposed to extended torsional displacement in the cell. However, due to the irregular particle shape and  
312 ability to interlock, extended shear also causes the material to be redistributed in the lid pocket rather than  
313 simply shear (see Figure 7). Material at the back of the pocket does not move, and thus, as the lid rotates,  
314 the length of the powder sample contained in the pocket gets short and increases in height causing a lifting  
315 of the lid and formation of a void at the back of the pocket. Thus, the assumption of stresses being  
316 uniformly applied over the cross sectional area of the cell are no longer valid and actual normal stress is  
317 significantly higher than inferred by the load over the cell area. This is similar to the behaviour seen with  
318 cohesive powders if a large number of vanes are removed from the cell, i.e. suggesting that for high  
319 internal friction materials like fibrous biomass a shearing lid with more vanes is necessary to grip the

320 powder and cause shear. Or simply, this is a sign that these materials do not shear and therefore an  
321 alternative approach is required to characterise their strength. This behaviour is evidenced by the shear  
322 stress vs shear strain traces which shows an under-consolidated response to torsional displacement with  
323 the shear stress rising slowly and never reaching consolidation due to the relative movement of the  
324 material in the shear cell. Figure 8 shows an example of this behaviour compared with that of sand, a  
325 conventional particulate material.

326 Inspection of the biomass flow functions of wood chips (Figure 9), RCG (Figure 10) and wood powder  
327 (Figure 11) shows that all would be classed as easy flowing/cohesive materials, with fair agreement  
328 between different shear testers. Manual handling suggests that the flaky wood chips are elastic/free-  
329 flowing, the fibrous RCG exhibits nesting behavior, and the wood powder is an elastic/cohesive material.  
330 The problem is the relevance of shear testing to fibrous/flaky particulates that do not shear, as  
331 demonstrated by internal frictions in the Figures 9 to 11, showing large variations in angle  $\approx 10^\circ$  for the  
332 fibrous RCG and flaky woodchips. The tensile tester (Figure 12) detected significant differences, with wood  
333 powder having no measureable strength whereas the fibrous and elongated RCG showed the greatest  
334 tensile strength. Wall friction data for all three materials (Figure 13) show that the LWFT, with a larger, and  
335 thus, more representative surface area than the other methods, showed significantly higher friction  
336 coefficients than the Brookfield PFT, Schulze RST, and Casagrande shear box for the three materials tested.

### 337 ***3.3 Arching tests***

338 In arching tests, hopper inclination,  $\alpha$ , was chosen to be steep enough to ensure mass flow conditions  
339 during material discharge. For each hopper inclination, the minimum outlet size preventing formation of a  
340 stable arch of bulk material at the outlet was experimentally determined. Experimental data for the three  
341 materials (reported in Figure 14 as hollow symbols) show critical outlet sizes between 0.02 and 0.28 m.  
342 Critical outlet sizes were generally increasing in this order: wood powder, RCG, wood chips. This ranking  
343 does not correspond to the flowability classification obtained from shear test derived flow functions.

344 Results confirm that propensity of different types of biomass cannot be explained solely by unconfined  
 345 yield strength characteristics. Critical outlet size values,  $D_c$ , were also calculated according to the hopper  
 346 design procedure due to Jenike [50], as reported also by Cannavacciuolo *et al.* [22]. Following this  
 347 procedure, when the arch is on the verge of collapsing, its weight is just balanced by the vertical  
 348 component of the maximum normal stress close to the walls. Jenike and Leser [51] derived inequality  
 349 (Eq. 1) from the force balance on the arch and by assuming that the arch is unstable if material resistance is  
 350 lower than the abutment stress:

$$351 \quad f_c < \frac{\rho_b g D}{H(\alpha)} \quad (1)$$

352 where  $f_c$  is the unconfined yield strength of the powder in use,  $D$  is the effective outlet size,  $\rho_b$  is the  
 353 powder bulk density,  $g$  is the acceleration due to gravity,  $H(\alpha)$  is a function which takes into account effects  
 354 of variation of thickness of the arch with the silo geometry and the hopper half-angle  $\alpha$ . Jenike and Leser  
 355 [51] reported a graphical solution of  $H(\alpha)$  that is well approximated by the following equation [52]:

$$356 \quad \frac{1}{H(\alpha)} = \left( \frac{65}{130 + \alpha} \right)^i \left( \frac{200}{200 + \alpha} \right)^{1-i} \quad (2)$$

357 where silo geometry is accounted for by the exponent  $i$ ,  $i=0$  for wedge hoppers and  $i=1$  for conical hoppers.  
 358 In mass flow silos, consolidation stress at outlet,  $\sigma_1$ , depends on distance from virtual hopper vertex.  
 359 According to Jenike [50], it is possible to show that:

$$360 \quad \sigma_1 = \rho_b g D \frac{(1 + \sin \phi_e) s(m, \alpha, \phi_e, \phi_w)}{2 \sin \alpha} \quad (3)$$

361 where  $s$  is a complicated function depending on hopper geometry (wedge or conical), on its half angle,  $\alpha$ ,  
 362 on the tensional state ( $m=1$  for active state,  $m=-1$  for passive state), on powder effective angle of internal  
 363 friction,  $\phi_e$ , and on powder wall friction,  $\phi_w$ . Combining Equations (1) and (3) it is possible to obtain the free  
 364 flow criterion to be applied on the plane  $f_c - \sigma_1$ :

$$365 \quad f_c < \frac{\sigma_1}{ff} \quad (4)$$

366 where  $ff$  is the flow factor

$$367 \quad ff = H(\alpha) \frac{(1 + \sin \phi_e) s(m, \alpha, \phi_e, \phi_w)}{2 \sin \alpha} \quad (5)$$

368 Diagrams reporting flow factors for conical and wedge hopper are given by Jenike [26] for different values  
369 of  $\alpha$ ,  $\phi_e$  and  $\phi_w$ . Flow factors estimates can also be obtained by using the mathematical procedure proposed  
370 by Arnold *et al.* [53]. The flow factor line, determined by the LHS term of Equation (5), generally cuts in two  
371 parts the powder flow function  $FF$ , that is the experimental material constitutive equation in which  
372 unconfined yield stress  $f_c$  is given as a function of consolidation stress  $\sigma_1$ :

$$373 \quad f_c = FF(\sigma_1) \quad (6)$$

374 The intersection between the flow function and the line representing the flow factor provides the critical  
375 unconfined yield strength of the material,  $f_c^*$ . The smallest outlet size,  $D_c$ , providing arch free flow is given  
376 by:

$$377 \quad D_c = \frac{f_c^* \cdot H(\alpha)}{\rho_b g} \quad (7)$$

378 In fact, in agreement with Eq. (1),  $D$  values larger than  $D_c$  provide arch free flow of powders. According to  
379 the design theory presented above, flow properties reported in the previous section were used to evaluate  
380 design values reported in Table 3. Values of  $H$  functions and flow factors were evaluated according to  
381 Arnold and Mc Lean [52]. The intersection of flow factor,  $ff$ , and flow functions lines ( $FF$ ), for each material  
382 and shear testing method, determined critical values of the unconfined yield strength  $f_c^*$  which, in turn,  
383 were used in Eq. (7) to determine theoretical values of  $D_c$ . Design values of  $D_c$  are compared with  
384 experimental data in Figure 14. Comparison shows that design values largely overestimate critical sizes for  
385 all biomass materials. Flow properties data obtained by different shear testing techniques significantly  
386 affected the design values of  $D_c$ . However, the discrepancy between experimental values and design values  
387 does not seem to be fully explained by this uncertainty. Additional reasons might be related to the main  
388 assumptions of the Jenike analysis (Coulomb solid, radial stress field, balance between material  
389 compressive strength and stresses internal to the arch) whose validity for biomass materials need a deeper



390 assessment. For fibrous biomass materials it can be argued that arch stability is related to material tensile  
391 strength,  $\sigma_t$ , rather than material unconfined compressive yield strength. In this case, dimensional analysis  
392 suggests that an order of magnitude evaluation of  $D_c$  can be expressed as:

$$393 \quad D_c \cong \frac{\sigma_t}{\rho_b g} \quad (8)$$

394 For RCG at low hopper angles and for wood chips within the entire hopper angle range, values of  $D_c$   
395 calculated according to Eq. (8), reported in Figure 14, show better agreement with experimental data.

### 396 **3.4 Explosion tests**

397 Dust was separated from the original sample of wood powder and of RCG by sieving. The Scots pine dust  
398 has the 80% of the volumetric particle size distribution between 47 and 378  $\mu\text{m}$  and a moisture content of  
399 8.4% by weight. The RCG dust has the 80% of the volumetric particle size distribution between 19 and  
400 279  $\mu\text{m}$  and a moisture content of 8.1% by weight. Dust explosion classes according to the  $K_{st}$ -value reveal  
401 that handling of Scots pine and RCG presents a dust explosion hazard like other organic materials, i.e. coals.  
402 For the sake of comparison, lignite was selected as a reference material [40] (Table 4). Although RCG and  
403 wood powder from scots pine fell into the lowest explosive (the studied biomass are classified as St1), this  
404 does not necessarily indicate a lower level of hazard. Some of the most devastating dust in the process  
405 industry have occurred with dusts in the lower ranges of the St 1 class [54], )

## 406 **4 Discussion**

407 Table 5 summarizes characterisation methods appropriate for each of the three biomass materials  
408 considered. An X indicates the possibility to use that method on a specified material. For size  
409 measurements a sequence of characters is used including L, W and T indicating the possibility to measure  
410 particle length, width and thickness, if appropriate. The three different material tested present different  
411 characteristics which make different measurement procedures more or less appropriate. These will be  
412 discussed in the following for each of the materials. Firstly, for the tested materials they all produce fines  
413 which has to be properly accounted for to correctly evaluate the explosion risks.

#### 414 **4.1 Wood chips**

415 Wood chips appear as a flaky free flowing material characterized by large particles, although this material  
416 can bring a certain portion of fines. Particle size distribution can be measured in a meaningful way with all  
417 techniques proposed, except laser diffraction. 3D image analysis is particularly interesting, due to the  
418 completeness of information provided and for the rapid measurement procedure. Some care is however  
419 required to account for correct contribution especially with reference to width and thickness distributions.  
420 Regarding to width, sieving is the fastest and most accurate procedure. However, this technique cannot  
421 provide any information regarding other shape characteristics. Shape distributions obtained with 2D  
422 analysis clearly indicate the tendency of this material to break along the fiber length.

423 Since wood chips are characterized by relatively coarse particles, not all powder flow testers can be used to  
424 carry out powder flow measurements. It is verified that shearing of this material in testers may not occur  
425 according to generally assumed shearing features, such as the formation of a defined shear plane of known  
426 surface, and that this uncertainty might impair the final results. However, given these limits, internal flow  
427 properties obtained with the large shear tester and the ring shear tester are almost equivalent. This was  
428 not the case. For wall friction measurements, a large shear tester provides higher values compared to  
429 standard size equipment. In this case, standard size testers may provide lower wall friction angles which  
430 could lead to underdesign storage unit dimensions.

431 The critical hopper outlet size for wood chips predicted with the Jenike [50] procedure, using data both  
432 from conventional and large testers, are in all cases extremely conservative. Instead, as indicated in Figure  
433 14 a, arch stability in the silo flow experiments is better described by the material tensile strength.

#### 434 **4.2 Reed canary grass (RCG)**

435 RCG appears as a flaky nesting material characterized by large and very flat and frail particles which  
436 produce a significant amounts of fines. Particle size distribution can be measured with all the techniques  
437 proposed, except laser diffraction. Also here, 3D image analysis is particularly appropriate for the  
438 completeness of the information provided and for the rapid measurement procedure but care is required

439 to account for the correct contribution of fines. With reference to particle width, sieving is the fastest and  
440 most accurate procedure. However, this technique cannot provide any information of other shape  
441 characteristics. Further, sieving of straw materials is challenging due to particle nesting and, when treated  
442 for longer time periods, brittle materials may break into pieces. Shape distributions obtained with the 2D  
443 analysis clearly indicate a tendency for this material to break across the length of the fibers. Therefore,  
444 fines show a less elongated shape.

445 With RCG, all powder flow testers can be utilized. However, in the same way as for wood chips, flaky and  
446 long particles may not produce the generally assumed features of formation of a defined shear plane with  
447 known surface area. Shear tests and wall shear tests showed the same trends of similarities and  
448 dissimilarities as for wood chips.

449 Also for RCG, the critical hopper outlet size estimated with the Jenike [50] was found to be extremely  
450 conservative. In particular, arch stability and silo flow was found to depend more on tensile strength  
451 properties than shear test data at low hopper angles. Arch stability depending on unconfined yield strength  
452 estimated from RST provided a better agreement with experimental data at higher hopper angles.

453 .

#### 454 ***4.3 Wood powder***

455 Wood powder appears as an elastic cohesive material characterized by fine needle shaped particles.  
456 Particle size distribution can be measured with techniques able to measure large amounts of small  
457 particles, including laser diffraction. Among the methods tested, only sieving and laser diffraction were  
458 suitable. Unfortunately both these techniques can not provide any information on the particle shape and its  
459 distribution.

460 All powder flow testers are suited to carry out wood powder flow measurements. As for wood chips and  
461 RCG, the same pattern of result consistency between different shear testers was found. Also with wood  
462 powder, the large shear cell gave higher wall friction values than small shear cells did.

463 Arching behavior of wood powder, predicted with the Jenike [50] procedure with data from conventional  
464 and large testers, was extremely conservative. However, since wood powder shows no measurable tensile  
465 strength, the critical silo outlet size cannot be predicted by measurement of this property.

466

## 467 **5 Conclusions**

468 Particle size measurements using caliper, 2D image analysis, 3D image analysis, and sieving, gave a wide  
469 range of results. Caliper as well as 2D and 3D image analysis are suitable only for materials made of  
470 relatively large particles but are powerful instruments for providing significant particle shape information  
471 than sieving.

472 The different shear tester measurements for determination of flow function and internal friction could not  
473 discriminate between the three biomass materials. Larger differences in results from different testers were  
474 found for wall friction clear compared to flow function. Arching tests revealed that the critical outlet size  
475 was over-predicted by applying the Jenike [50] procedure with unconfined yield strength data. The tensile  
476 tester measured significant strength differences between the materials that were consistent with  
477 experimental arching behavior.

478 Scots pine and RCG dust are classified as St1, ; being flammable and, when mixed with air, being able to  
479 form an explosive atmosphere.

480 The different test methods were evaluated with respect to their ability of relevant characterization of each  
481 of the three representative biomass materials tested (Table 5).

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606 **Table captions**

607 Table 1. Tests performed by the Bio4Flow research partners.

608 Table 2. Particle size measurement techniques.

609 Table 3. Main outlet design values

610 Table 4. Explosion parameters of the analysed samples.

611 Table 5. Summary of applied characterisation methods. An X indicates the possibility to use the method on  
612 the line with the material in the column. For size measurements, L, W and T indicate the possibility to  
613 measure the particle length, width and thickness.

614 **Figure captions**

615 Figure 1. Biomass materials used in the experiments: a) Scots pine (*Pinus sylvestris*) wood chips; b) Scots  
616 pine (*Pinus sylvestris*) wood powder; c) reed canary grass (*Phalaris arundinacea*) straw chops.

617 Figure 2. Sketch of the Casagrande direct shear tester prepared for: a) internal friction and b) wall friction.

618 Figure 3. Experimental plane silo with variable shape a) sketch; b) full size view; c) material leveling before  
619 experiments; d) and e) silo opening; f) stable arch; g) material collected in the discharge basin.

620 Figure 4. Particle size distributions in cumulative weight fraction for particle length (a and b), particle width  
621 (c and d), and particle thickness (e and f), of wood chips (a, c and e) and reed canary grass (b, d and f)  
622 measured with various techniques: —, 2D image analysis; ·····, 3D ScanChip analysis; ---, single particle  
623 caliper measurements; - · - · -, sieving.

624 Figure 5. Particle shape distributions for: a) the elongation in cumulative area % and b) equivalent area  
625 diameter for different elongation classes. — and ●, wood chips; — — —, and ▲, reed canary grass.

626 Figure 6. Particle size distributions obtained by sieving for wood powder.

627 Figure 7. Photograph of the large annular shear cell showing the formation of voids at the front of the lid  
628 pockets for the wood chips

629 Figure 8. Torsional and axial load traces from the PFT for a) wood powder and b) sand.

630 Figure 9. Bulk flow properties measured for wood chips using a range of shear testers.

631 Figure 10. Bulk flow properties measured for reed canary grass using a range of shear testers.

632 Figure 11. Bulk flow properties measured for wood powder using a range of shear testers.

633 Figure 12. Tensile Strength Functions measured for the three biomass materials in The Wolfson Centre  
634 Tensile Tester.

635 Figure 13. Wall friction functions measured in a range of different shear testers for a) reed canary grass  
636 RCG, b) wood powder and c) wood chips.

637 Figure 14. Critical hopper outlet size for arching: a) wood chips; b) reed canary grass; c) wood powder;  
638 ●, experiments; ○, theory with RST data; □, theory with PFT data; ◇ theory with LAST data; ▽, theory  
639 with tensile test data.

Table 1. Tests performed by the Bio4Flow research partners.

Characterisation method	SLU BTC	UG Wolf	US PTG	UPM BIPREE
<i>Particle size distribution</i>				
Sieve analysis	X			
2D image analysis			X	
3D image analysis - Scanchip	X			
Caliper		X		
<i>Powder flow properties</i>				
Shear tests powder flow tester		X	X	
Shear tests - Schulze shear tester	X			
Shear tests - large annular shear cell		X		
Tensile tests		X		
Wall friction-powder flow tester		X	X	
Wall friction-large Jenike shear cell		X		
Wall friction-Casagrande shear box				X
Arching test in a model silo			X	
<i>Safety properties</i>				
Explosion test				X

Table 2. Particle size measurement techniques.

Technique	Method(s)	Materials analyzed	Sample size
Caliper	Manual measurements	wood chips	320 pieces
		RCG	100 pieces
2D image analysis	Image analysis software	wood chips	1100 pieces
		RCG	8800 pieces
3D image analysis	ScanChip	wood chips	16000 pieces
		RCG	12000 pieces
Sieving	EN 15149-1:2010	wood chips	2 kg
	EN 15149-2:2010	RCG	1 kg
		wood powder	50 g

Table 3. Main silo outlet design values

Material	$\alpha$ [°]	$H$ [-]	$ff$ [-]	$f_c^*$ or $\sigma_t^*$ [Pa]	$D_c$ (design) [m]
wood chips LAST data	40	1.20	1.50	528	0.43
	38	1.19	1.48	526	0.43
	35	1.18	1.45	523	0.42
	33	1.17	1.40	519	0.41
	30	1.15	1.35	515	0.40
	28	1.14	1.32	512	0.40
	25	1.13	1.28	509	0.39
	20	1.10	1.24	506	0.38
wood chips Tensile test data	40		1.50	96	0.066
	38	-	1.48	96	0.065
	35	-	1.45	96	0.065
	33	-	1.40	95	0.065
	30	-	1.35	94	0.064
	28	-	1.32	94	0.064
	25	-	1.28	94	0.064
reed canary grass RST data	40	1.20	1.38	126	0.171
	35	1.18	1.32	124	0.165
	30	1.15	1.29	123	0.160
	25	1.13	1.28	123	0.156
	20	1.10	1.29	123	0.153
reed canary grass PFT data	40	1.20	1.59	255	0.35
	35	1.18	1.57	254	0.34
	30	1.15	1.58	254	0.33
	25	1.13	1.61	257	0.33
	20	1.10	1.65	263	0.33
reed canary grass LAST data	40	1.20	1.47	337	0.46
	35	1.18	1.36	334	0.44
	30	1.15	1.29	332	0.43
	25	1.13	1.25	331	0.42
	20	1.10	1.23	331	0.41
reed canary grass Tensile test data	40	-	1.47	52	0.059
	35	-	1.36	65	0.074
	30	-	1.29	60	0.068

	25	-	1.25	56	0.064
	20	-	1.23	53	0.061
wood powder RST data	38	1.19	1.23	285	0.15
	35	1.18	1.21	285	0.15
	33	1.17	1.21	284	0.14
	30	1.15	1.21	284	0.14
	28	1.14	1.22	285	0.14
	25	1.13	1.23	286	0.14
	23	1.12	1.25	287	0.14
	20	1.10	1.28	289	0.14
wood powder PFT data	38	1.19	1.30	519	0.27
	35	1.18	1.28	516	0.26
	33	1.17	1.27	514	0.26
	30	1.15	1.27	514	0.26
	28	1.14	1.27	514	0.25
	25	1.13	1.29	516	0.25
	23	1.12	1.30	518	0.25
wood powder LAST data	38	-	1.76	453	0.23
	35	-	1.64	439	0.22
	33	-	1.58	431	0.22
	30	-	1.49	423	0.21
	28	-	1.44	418	0.21
	25	-	1.38	412	0.20
	23	-	1.35	409	0.20



Table 4. Explosion parameters of the analysed samples.

Dust sample	Pmax (bar.g)	(dP/dt)max (bar/s)	Duration of the combustion (ms)	Kst (bar.m/s)	Explosion class
Scots pine	7,4	553	41	150	St1
reed canary grass	7,2	579	32	157	St1
lignite	7,3-10	--	--	32-176	St1

Table 5. Summary of applied characterisation methods. An X indicates the possibility to use the method on the line with the material in the column. For size measurements, L, W and T indicate the possibility to measure the particle length, width and thickness.

Characterisation method	wood chips	RCG	wood powder
<i>Particle size distribution</i>			
Sieve analysis	W	W	W
2D image analysis	LW	LW	
3D image analysis - Scanchip	LWT	LWT	
Caliper	LWT	LWT	
<i>Powder flow properties</i>			
Shear tests - powder flow tester		X	X
Shear tests - Schulze shear tester	X	X	X
Shear tests - large annular shear cell	X	X	X
Shear tests - Casagrande		X	X
Tensile tests	X	X	X
Wall friction - powder flow tester		X	X
Wall friction - large Jenike shear cell	X	X	X
Wall friction-Casagrande	X	X	X
Arching test in a model silo	X	X	X
<i>Safety properties</i>			
Explosion test		X	X

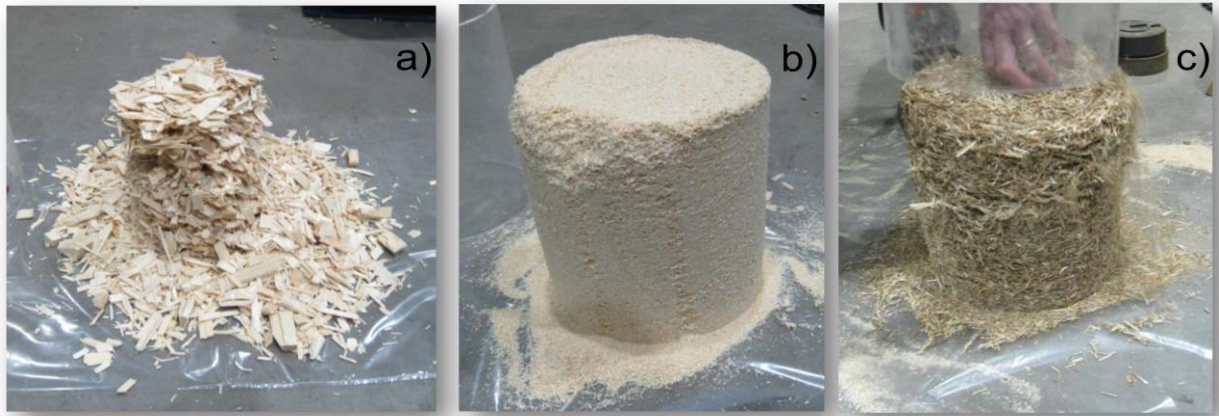


Figure 1. Biomass materials used in the experiments: a) Scots pine (*Pinus sylvestris*) wood chips; b) Scots pine (*Pinus sylvestris*) wood powder; c) reed canary grass (*Phalaris arundinacea*) straw chops.

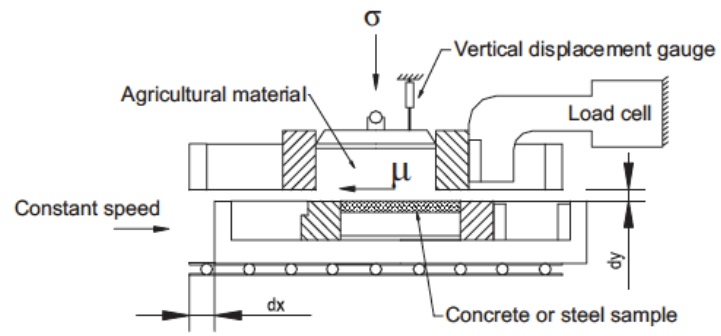


Figure 2. Sketch of the Casagrande direct shear tester prepared for wall friction test.



Figure 3. Experimental plane silo with variable shape a) sketch; b) full size view; c) material leveling before experiments; d) and e) silo opening; f) stable arch; g) material collected in the discharge basin.

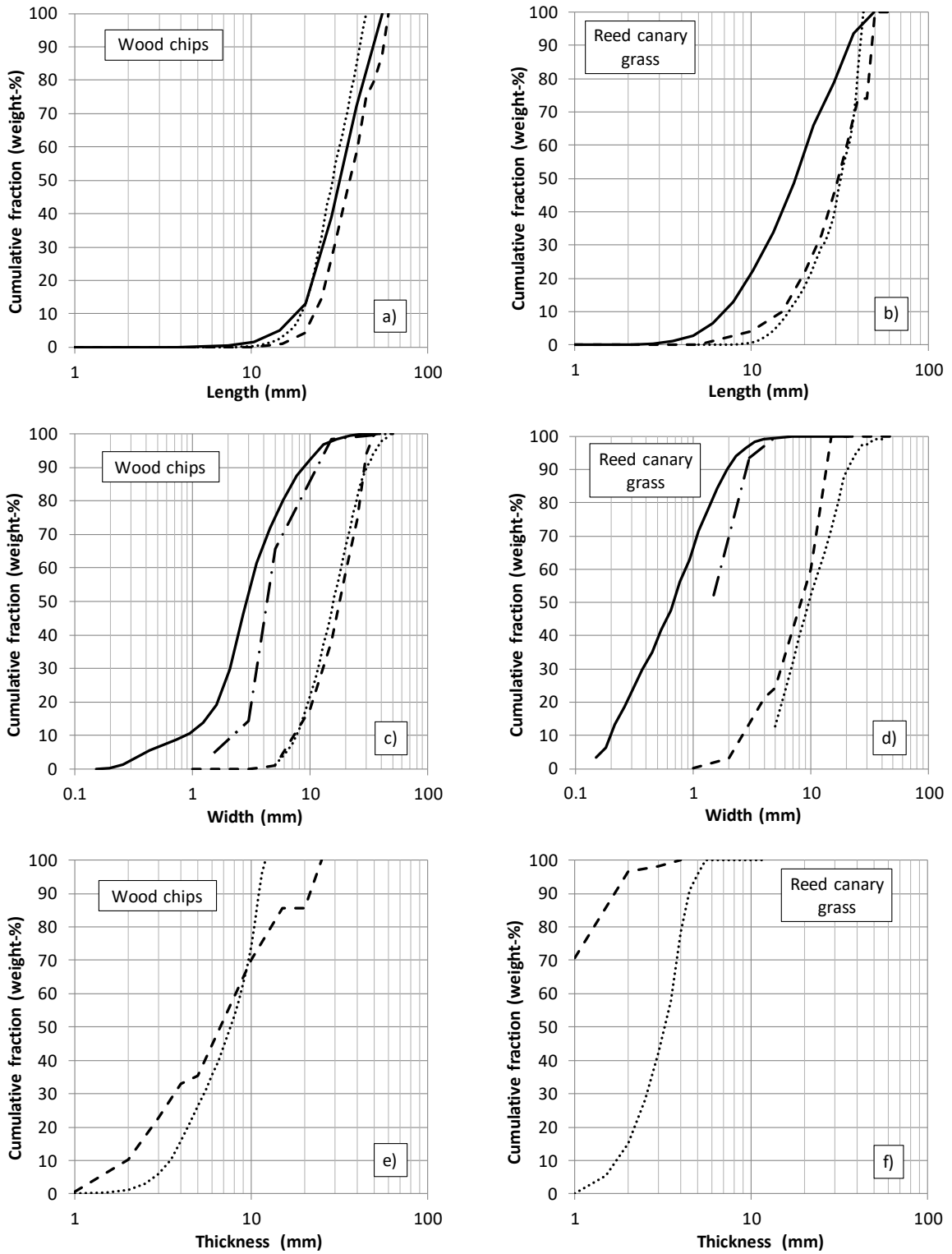


Figure 4. Particle size distributions in cumulative weight fraction for particle length (a and b), particle width (c and d), and particle thickness (e and f), of wood chips (a, c and e) and reed canary grass (b, d and f) measured with various techniques: —, 2D image analysis; ....., 3D ScanChip analysis; ---, single particle caliper measurements; - · - · -, sieving.

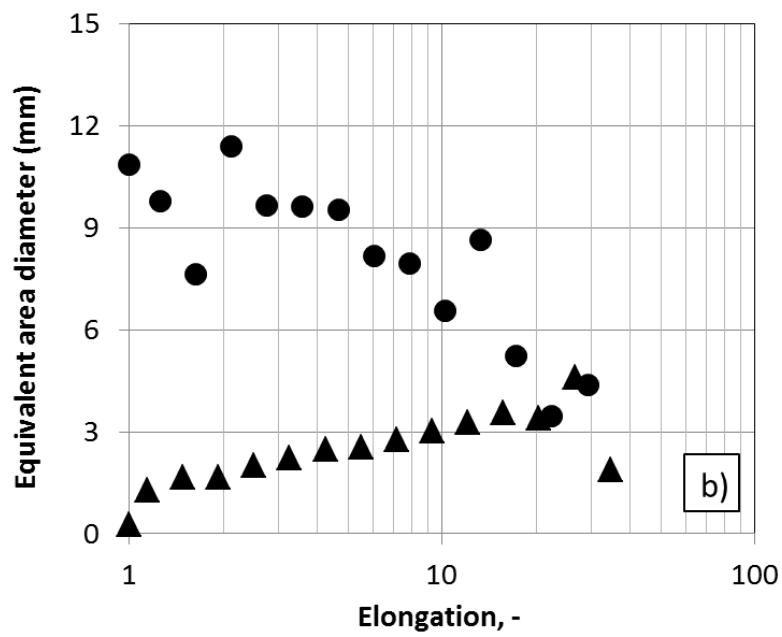
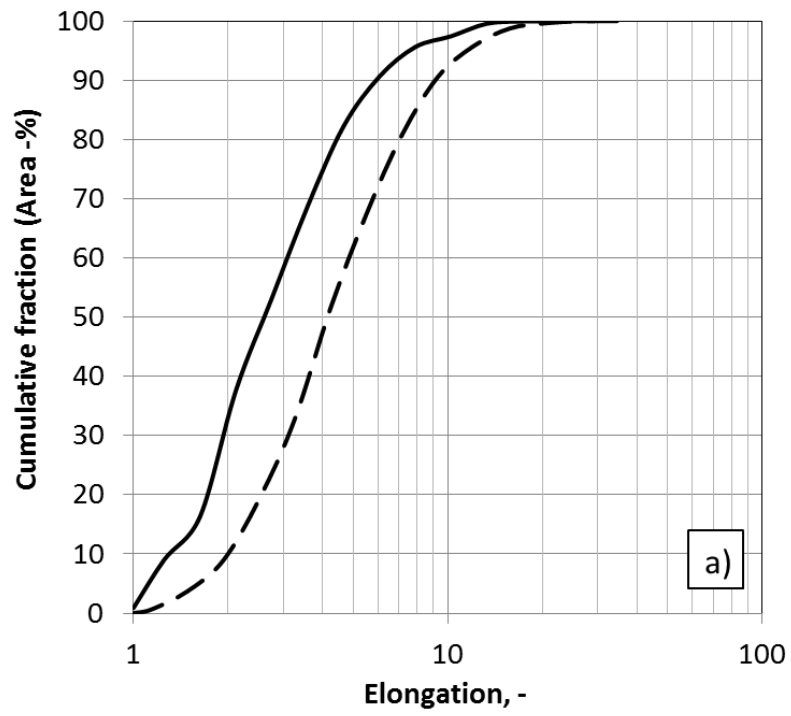


Figure 5. Particle shape distributions for: a) the elongation in cumulative area % and b) equivalent area diameter for different elongation classes. — and ●, wood chips; ---, and ▲, reed canary grass.

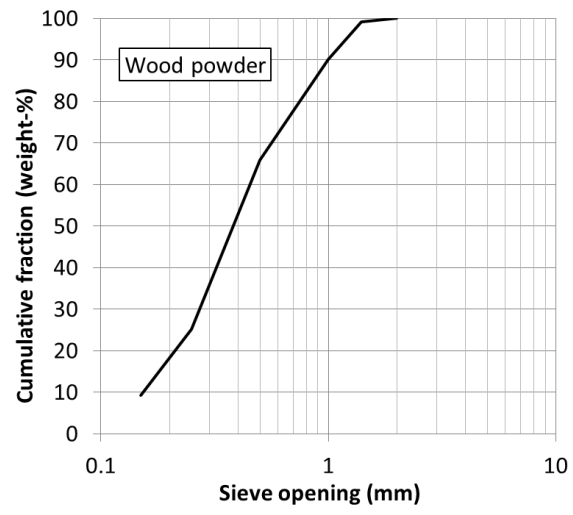


Figure 6. Particle size distribution obtained by sieving for wood powder.



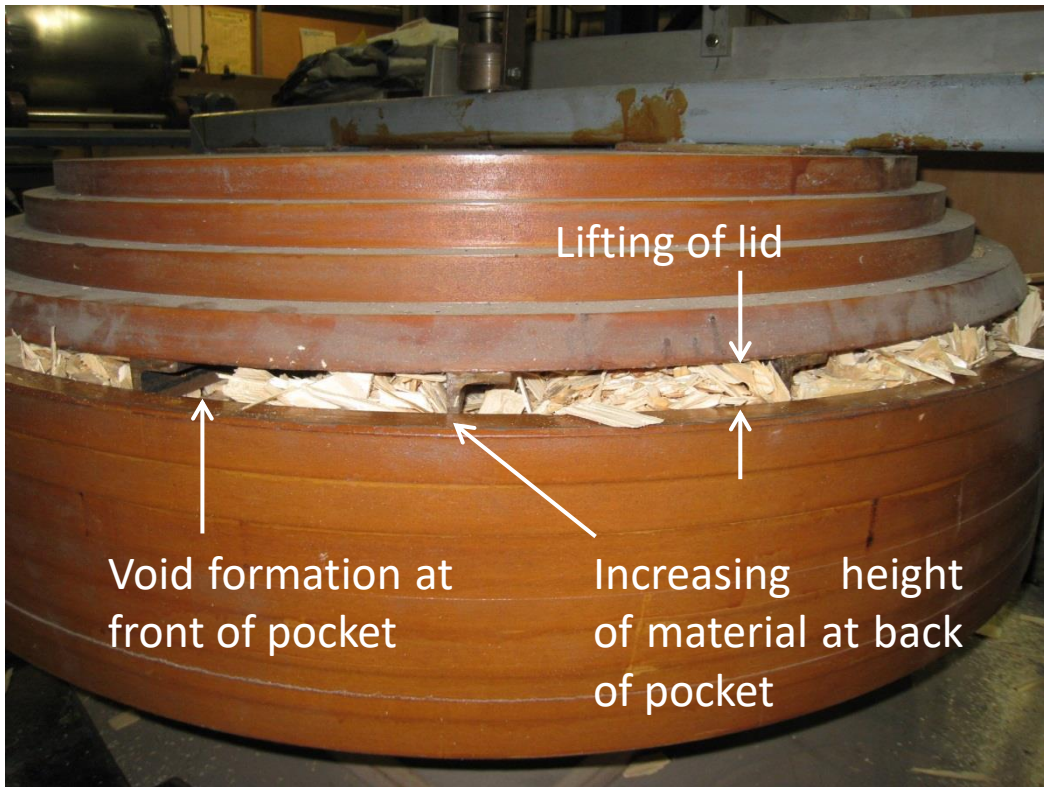
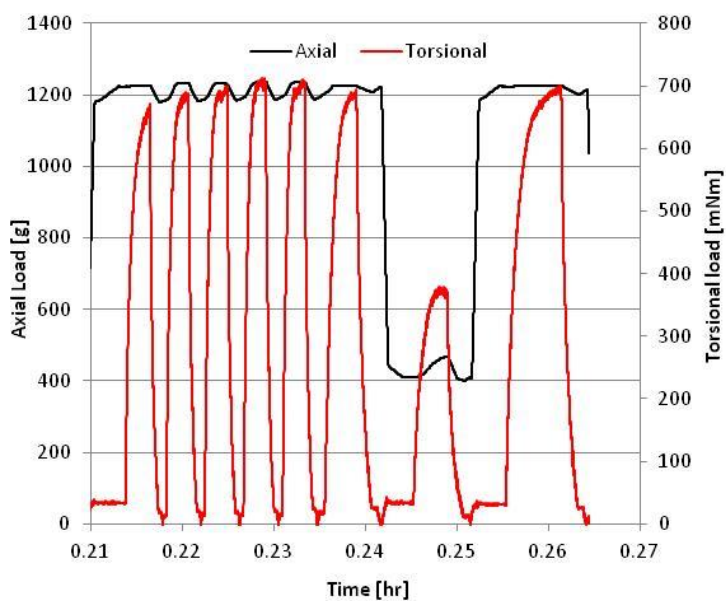
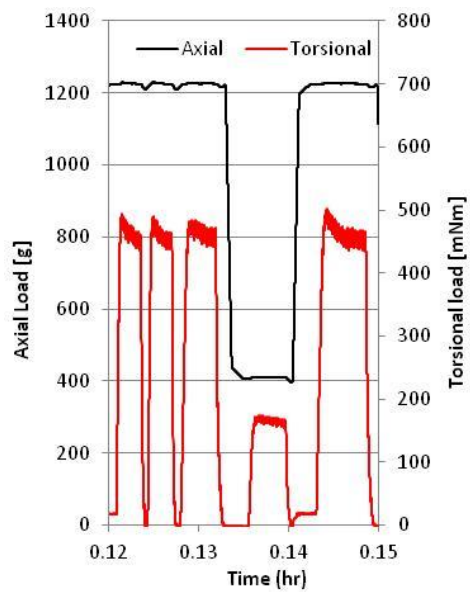


Figure 7. Photograph of the large annular shear cell showing the formation of voids at the front of the lid pockets for the wood chips



a)



b)

Figure 8. Torsional and axial load traces from the Brookfield Powder Flow Tester (PFT) for a) wood powder and b) sand.

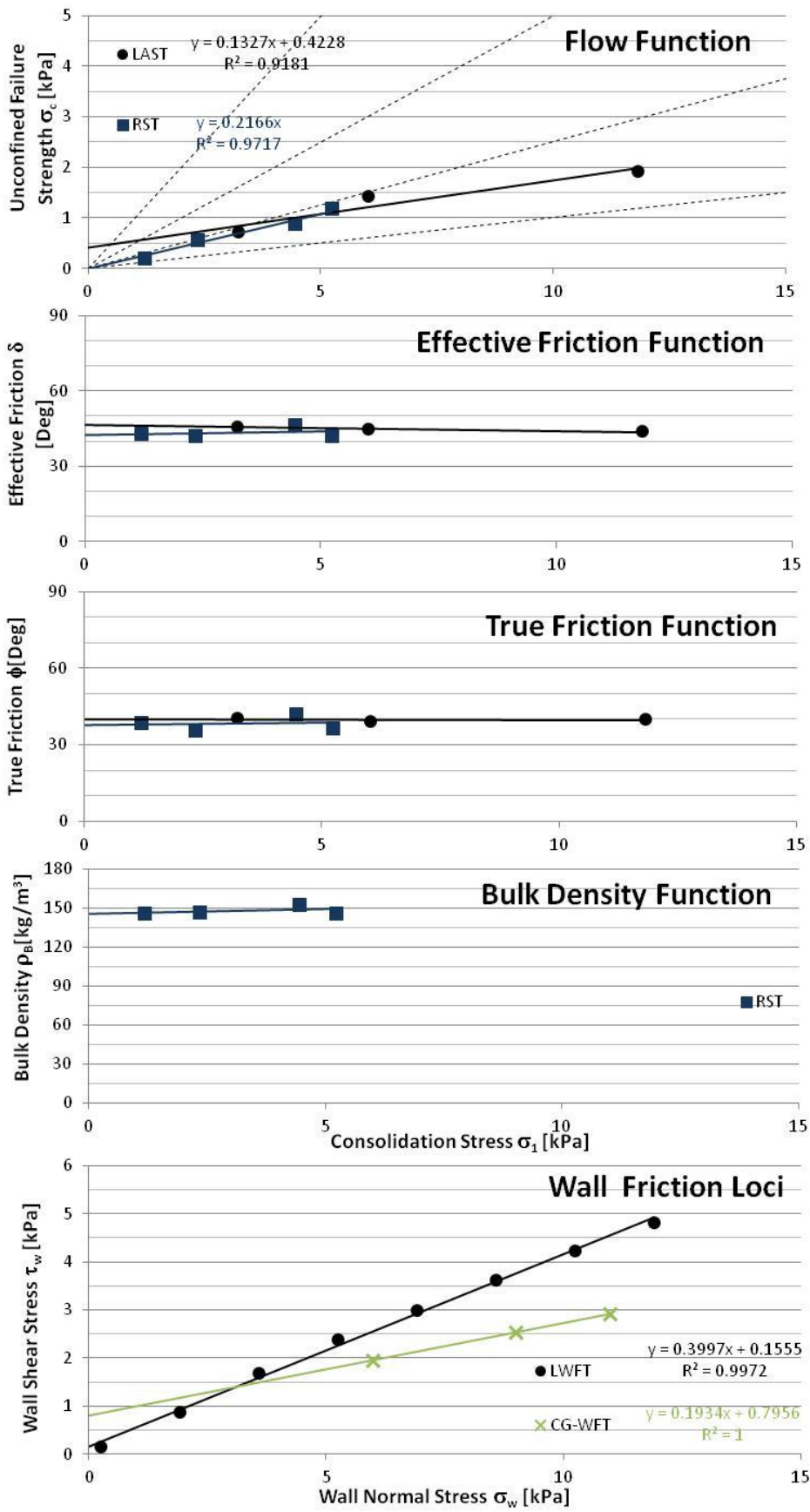


Figure 9. Bulk flow properties measured for wood chips using a range of shear testers.

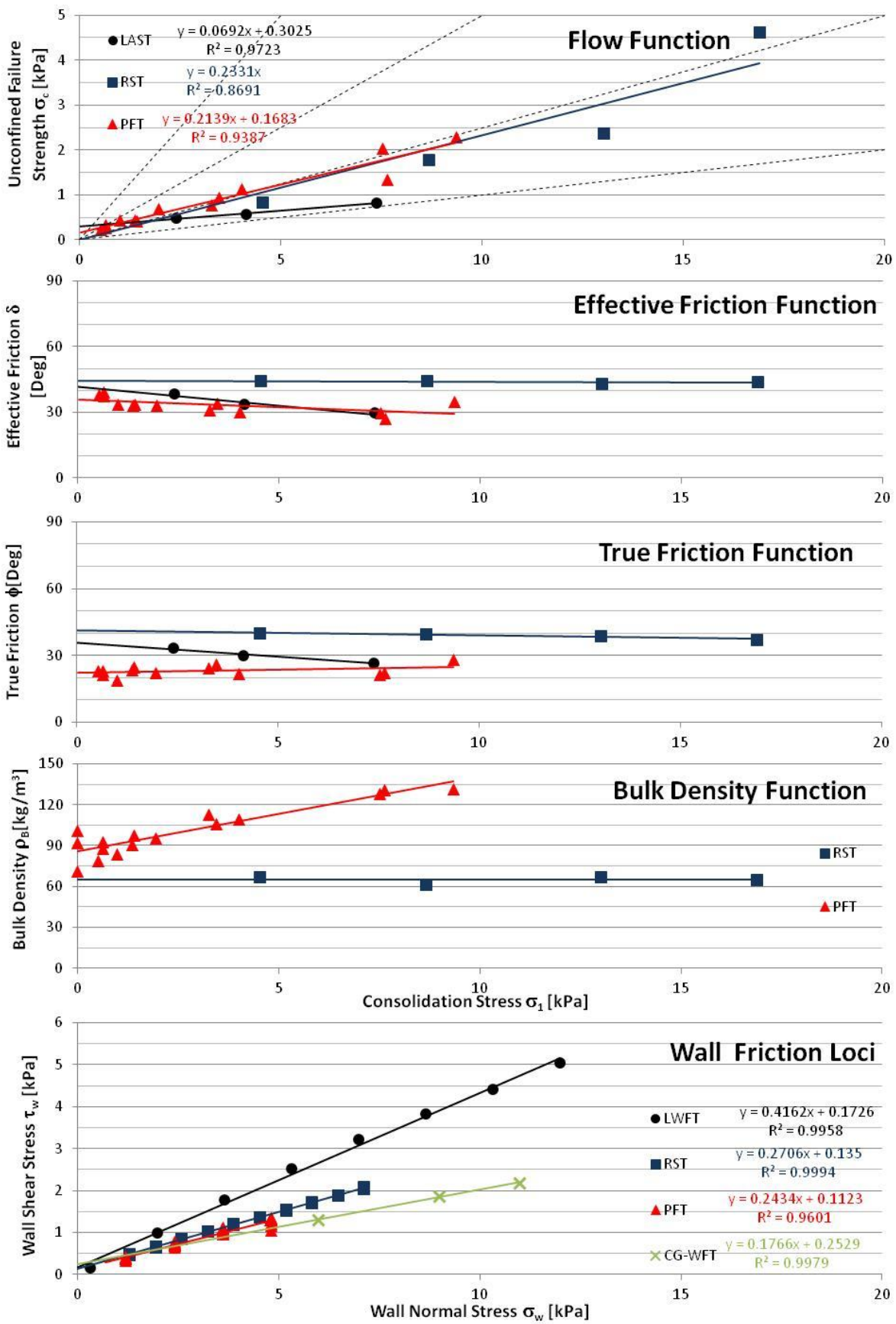


Figure 10. Bulk flow properties measured for the reed canary grass (RCG) using a range of shear testers.

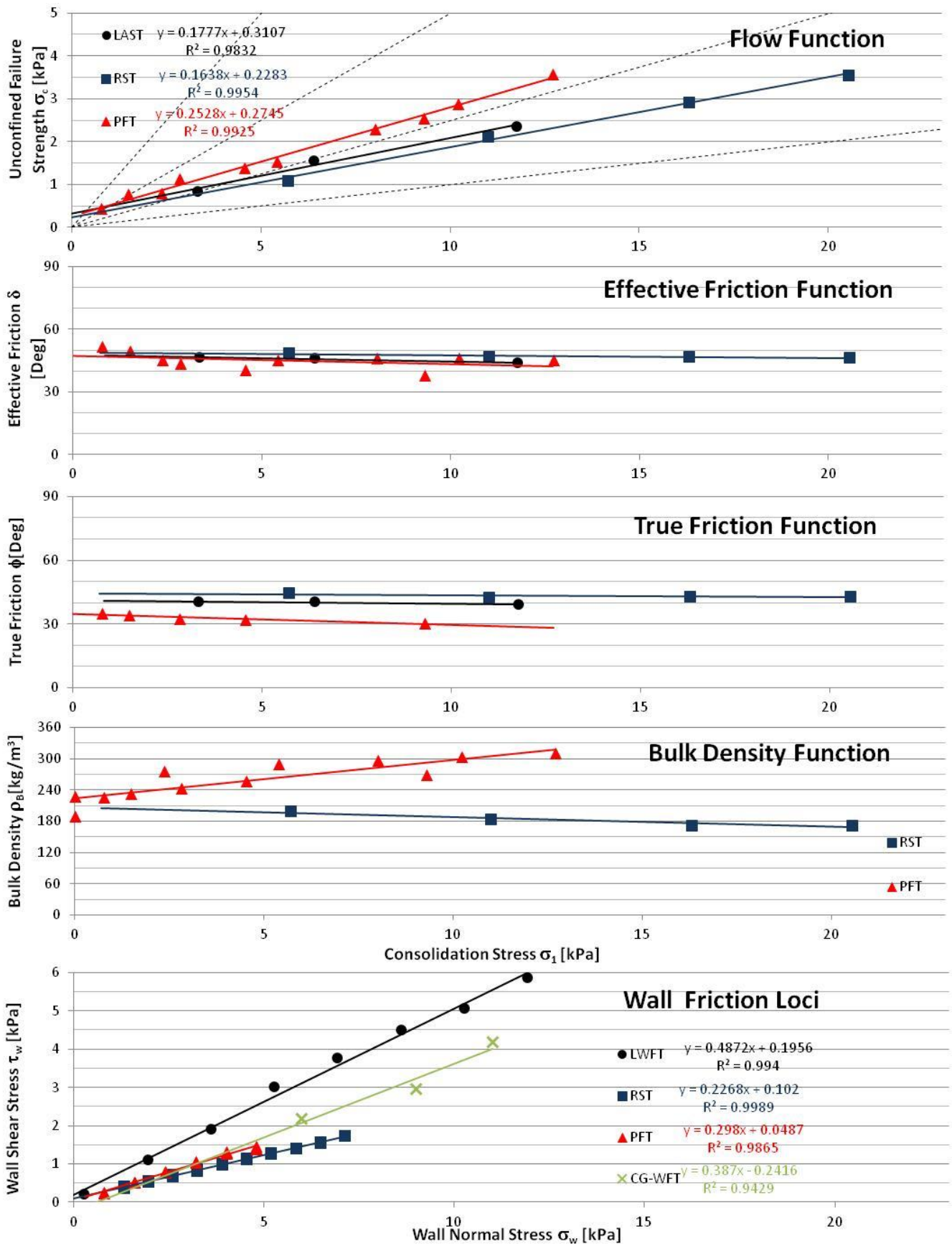


Figure 11. Bulk flow properties measured for wood powder using a range of shear testers.

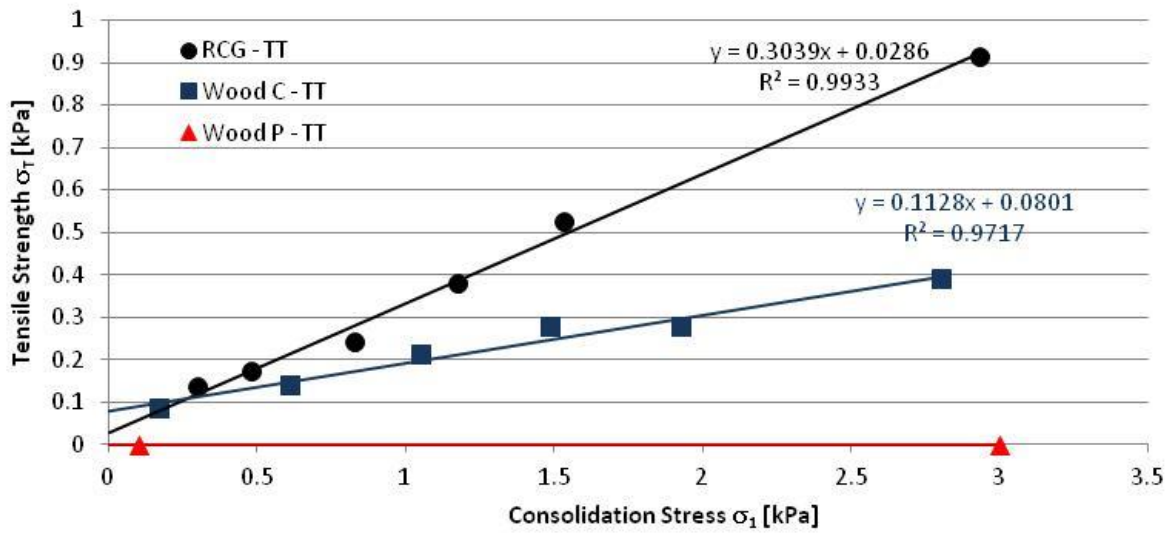
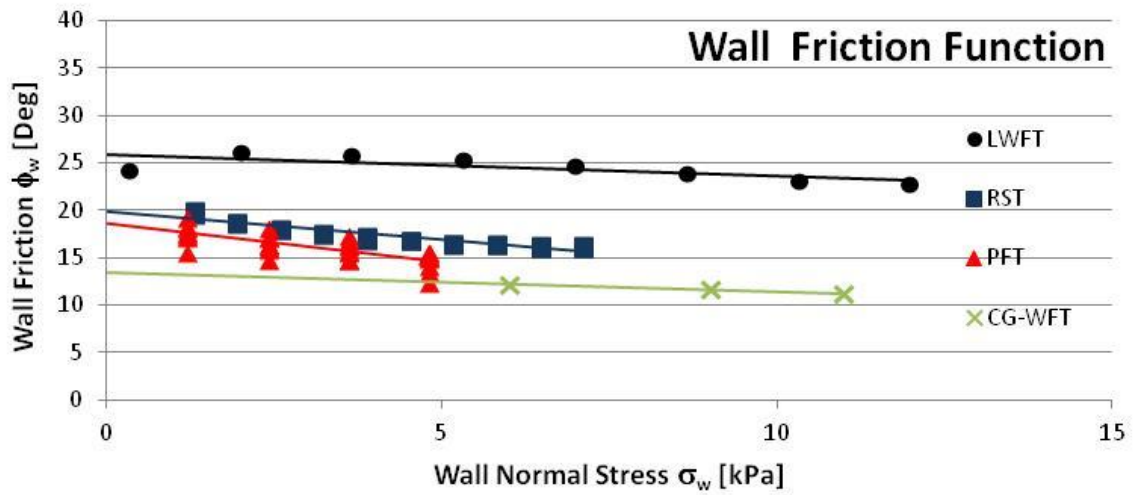
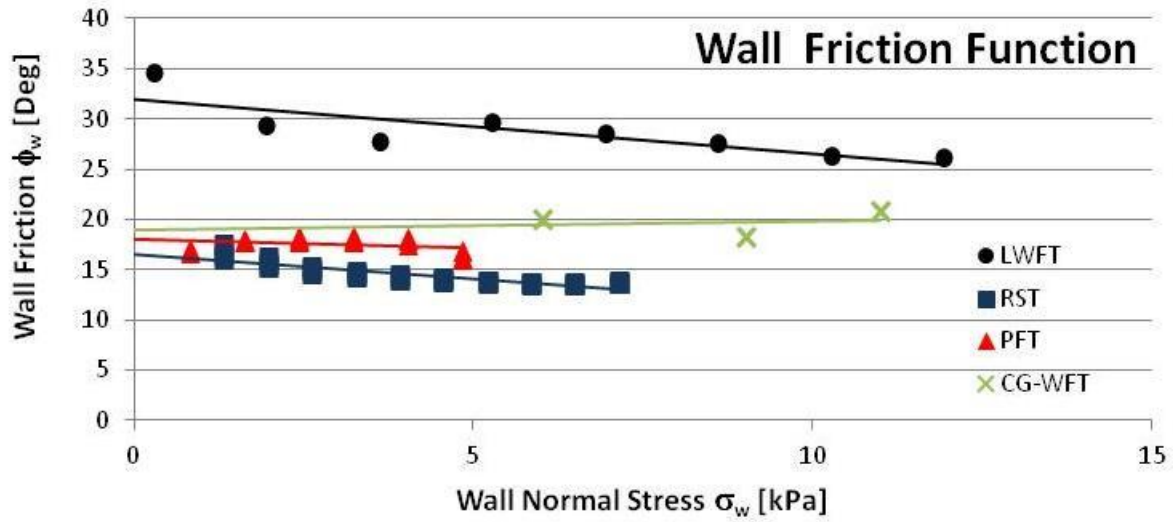


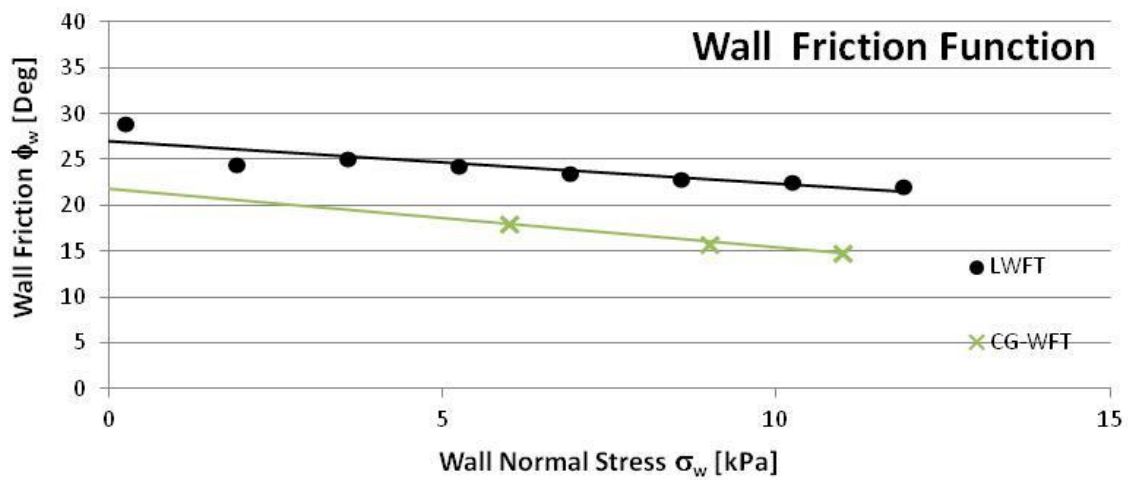
Figure 12. Tensile strength functions measured for the three biomass materials in the Wolfson Centre tensile tester.



a)



b)



c)

Figure 13. Wall friction functions measured in a range of different shear testers for a) reed canary grass, b) wood powder and c) wood chips.

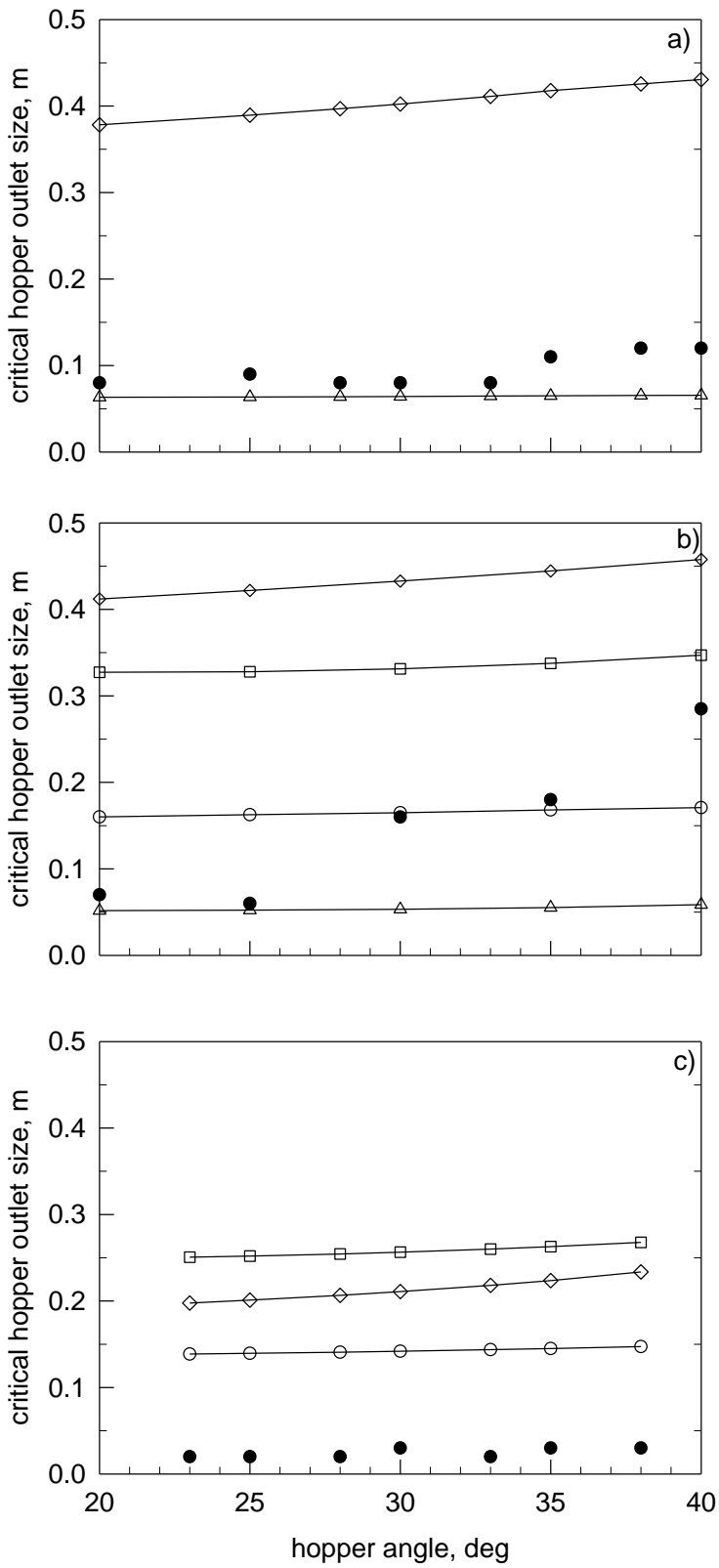
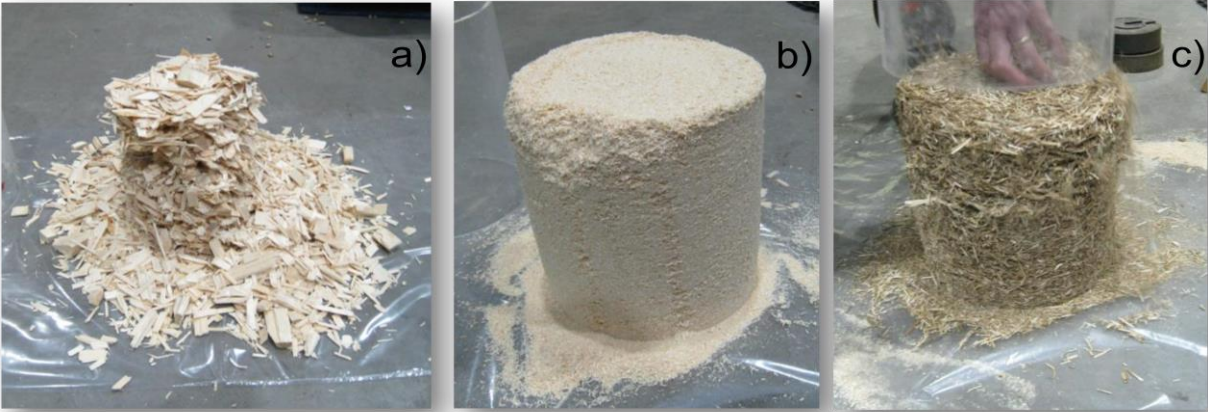


Figure 14. Critical hopper outlet size for arching: a) wood chips; b) reed canary grass; c) wood powder; ●, experiments; ○, theory with RST data; □, theory with PFT data; ◇ theory with LAST data; ▽, theory with tensile test data.



Graphical abstract



a) wood chips, b) wood powder, c) reed canary grass