



# Physico-chemical characterisation of plant particles with potential to produce biobased building materials

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28 Water absorption kinetics were consistent with the plant chemical composition and the open  
29 porosity which highlight a fair understanding of inter-dependence of particles physical  
30 properties.

31 **Keywords:** density; porosity; sorption; bio-based aggregate; building material

32

## 33 1. Introduction

34 The renovation and construction of buildings reducing the global warming potential can be  
35 partly addressed by using low-environmental-impact materials such as raw biobased  
36 materials. In France, hemp and straw are the most well-known plants used for building  
37 applications (Colinart et al., 2020), hemp crop represented 17000 ha in 2018 (FAOSTAT) and  
38 it generated about 50000 t of hemp shiv. Hemp production can be increased due to its  
39 usefulness for farmers that goes toward low impact agriculture schemes. A way to enhance  
40 the sustainability could be diversify the use of different biobased particles in construction  
41 rather than the use of only one kind of plant particle. Firstly, this may allow the use of plant  
42 particles available at local scale. Secondly, it could reduce the risk of competition with other  
43 ways for the valorisation of the same plant particles (litter, bioenergy, etc). Laborel et al.  
44 (2016) in their review about earth construction highlighted the great diversity of bio-  
45 aggregates (plant and animal) used worldwide. This review identified cereal straws (wheat,  
46 barley, oat, lavender (Giroudon et al., 2019)), wood aggregates (shavings or fibres), bast  
47 fibres (hemp, flax, jute, kenaf, diss), palm tree fibres (coir, palm, date), wastes and residues  
48 (cassava, millet, cotton, tea, tobacco, grass), leaf fibres (sisal, banana, pineapple), aquatic  
49 plants (phragmite, typha, seaweed) and wool (sheep). Jiang et al. (2020) specifically  
50 characterized hemp shiv, flax shiv, rape shiv and wheat straw as hygric and insulating  
51 construction materials for energy efficient building. Other works assessed the use of maize or  
52 sunflower bark chips (Lagouin et al., 2019), reed (Honoré et al., 2020), miscanthus  
53 (Ntimugura et al., 2020) and rice/rice husk ash (Taye et al., 2021) for bio-based building  
54 materials. In France, the main crops able to produce particles with potential applications in

55 construction are cereals (wheat, barley, oat, rye, rice), oil plants (sunflower, rapeseed),  
56 herbaceous plants (corn, miscanthus, reed) and fibre plants (flax, hemp).

57 Microstructure and biochemistry are **two of the important parameters** to be studied in order to  
58 understand how plant particles would behave when used as a building material. The  
59 microstructure of plant particles can be studied at different scales (Glé et al., 2021). The bulk  
60 density, **which is characterized by the mass of the particles per volume unit**, gives the overall  
61 density of the bulk plant material. It is calculated by measuring the **total** volume occupied by a  
62 certain mass of plant particles. **The total volume is the result of the sum of different volumes:**  
63 **a) the volume occupied by the walls of the particles; b) the volume of air between the particles**  
64 **and c) the volume of occupied air inside the particles. The volume occupied by the walls of**  
65 **the particles (a) could be considered intrinsic of the material. The air-filled volume between**  
66 **the particles (b) is dependent on the arrangement of the particles between them which is**  
67 **influenced by their shape and particle size. Therefore, this volume may be decreased by**  
68 **grinding or milling which could have a positive effect on the arrangement between the**  
69 **particles and as a consequence increase the apparent density. Finally, c) includes the volume**  
70 **accessible from the outside of the particle (open porosity) and the volume not accessible**  
71 **(closed porosity) (Delannoy et al., 2020).**

72 Functionalities are properties induced by the intrinsic characteristics of plant particles. These  
73 characteristics such as the microstructure, chemical composition or water behaviour may lead  
74 to functionalities that can be positive or negative depending on the final use of the plant  
75 particle. A clear example of a functionality related to chemical composition that can be  
76 positive or negative as function of the final use is the search for ecological solutions in the  
77 design of particleboard. By using the self-adhesion capacity, it is possible to process particle  
78 boards without the addition of binders, thanks to the action of different parameters such as  
79 solubilization of molecules, temperature and pressure which allow the particles to  
80 agglomerate with each other (Lenormand et al., 2017, Pintiaux et al., 2015). However, this  
81 ability to solubilize molecules **could be considered a negative functionality** when the

molecules disturb the setting of lime or cement-based mortars (Diquélou et al., 2015; Wang et al., 2019).

The cell walls of plant particles are mainly composed of a mix of organic macromolecules (pectins, cellulose, hemicelluloses, lignin, waxes, starch, proteins, aromatic compounds, **short-chain sugars**) and a minority of mineral molecules such as ashes. Cell walls can therefore be considered as biochemically complex composites. Cellulose molecules, hemicelluloses and pectins are polysaccharides (polymers of carbohydrates) with hydrophilic **properties**. The water-soluble compounds generally correspond to pectins and **short-chained** soluble molecules. The proportions of organic macromolecules vary according to the botanical species and the location in the plant. For example, sunflower pith stands out from the others with a proportion of water-soluble compounds **higher** than 50% (Chabriac et al., 2016).

In order to clear out what are the essential differences between these plant particles, **a physico-chemical characterization with the same techniques and protocols in order to facilitate the comparison was carried out**. The procedure was applied to 9 different plant particles **that** can be obtained in France, focusing on the particle size, the hygroscopic behaviour, the chemical composition, the densities and the water absorption behaviour.

## 2. Materials and Methods

### 2.1. Materials

Nine different plant particles were studied: **A)** flax (*Linum usitatissimum*) shiv bought on a local market, Linabox (76590 Crosville sur Scie, France); **B)** hemp (*Cannabis sativa L.*) shiv bought in a local market and produced in France; **C)** maize (*Zea mays L.*) bark particles obtained from stems of maize cultivated in Villainville (Seine-Maritime, France) in 2015; **D)** miscanthus (*Miscanthus*) particles obtained on a local market, Miscanplus (28250 Digny, France); **E)** reed (*Phragmites australis*) particles obtained after milling and sieving (the studied fraction corresponded to particles that were retained between 2 and 8 mm sieves) of reed from the Brenne region (Centre-Val de Loire, France); **F)** rice (*Oryza sativa*) husk,

obtained from a high density batch (Balle Concept, 13200 Arles, France) that was sieved with a sieve of 10 mm, vacuuming once to remove heavy parts (grains and others) and re-sieved with a sieve of 10 mm; **G**) sunflower (*Helianthus annuus*) bark and sunflower pith (**H**) obtained from sunflower stem kindly provided by the farm cooperative *Oxyane* and **I**) wheat (*Triticum*) particles obtained from wheat cultivated in Normandie in 2017.

The employed hemp and flax shiv were obtained by raw plant transformation by a large scale defibrator plant that was hammer milled during the defibrating process. Maize bark and wheat studied particles were obtained after milling employing a cutting mill (Retsch SM100, Germany) equipped with a standard sieve with 10 mm of mesh size. All different samples were sieved employing a standard sieve of 0.5 mm in order to eliminate dust that could have influence on the subsequent studies.

#### *Location of Figure 1*

### **2.2. Chemical Composition**

Chemical composition of samples, content of ash, lignin, cellulose, hemicellulose and soluble compounds in neutral detergent was determined following the Van Soest method (AFNOR, 1997, Viel et al., 2018) employing an adapted machine (FibercTR 8000, Foss, Denmark). To carry out this measurement all samples were previously dried until constant weight at 40°C using an oven (Memmert, Germany) and then milled employing a micro impact mill (Culatti, Switzerland), the rotor speed was adjusted to 7 and a standard sieve of 1 mm of mesh size was employed. All measurements were carried out at least in triplicate.

### **2.3. Particle size characterization**

Particle size distribution of all samples was determined by image analysis employing the protocol described by Amziane & Collet (2017) slightly modified. Images of each sample were taken employing a vision system (Keyence, CA-MX500M) placing their particles on a plate (CA-DSW15) where a maximum of 200 particles were placed to take the photos. Size of

particles was then determined employing a scale factor of 0.0609 mm/pixel employing the associated software of the machine. At least 1000 particles of each sample were measured. Experimental data was modelled employing the error function in order to confirm if this function can be a good model to predict experimental particle size distribution of samples. The model is defined as, Eq. (1):

$$P = \frac{1}{2} \left( 1 + \operatorname{erf} \left( \frac{\ln(x) - \mu}{\sqrt{2}\sigma} \right) \right) \quad (1)$$

where P is the cumulative frequency,  $\operatorname{erf}$  the error function ( $\operatorname{erf}(z) = \frac{2}{\sqrt{\pi}} \int_0^z e^{-t^2} dt$ ), x the variable (in this case width or length) and  $\mu$  and  $\sigma$  are model parameters corresponding to mean and standard deviation of the variable's natural logarithm.

## **2.4. Bulk and true density**

### **2.4.1. Bulk Density**

Two different bulk densities of samples previously dried at 40°C until constant weight were calculated employing different methods: apparent and compacted density. Apparent density ( $\rho_A$ ) was calculated by measuring the height of a known quantity of sample placed in a cylinder that was upended ten times (Amziane & Collet 2017). For compacted density ( $\rho_C$ ) a known quantity of samples was placed in a cylinder then it was manually compressed and finally the volume of the sample was determined (Glé et al., 2021). All measurements were carried out at least in triplicate.

### **2.4.2. True Density**

True density ( $\rho_T$ ) of samples was calculated after measurement of sample volume by gas displacement using a gas pycnometer (Pycnomatic Evo, ThermoScientific) with argon as employed gas. The gas was introduced into a small vessel (21.42 cm<sup>3</sup>) at a constant temperature of 23°C where the sample was previously placed. The pressure (P) was set at 1

163 bar (with highest and lowest limit of 1.5 bar and 0.5 bar, respectively) considering that sample  
164 is in equilibrium when  $\Delta P$  between different measurements is lower than 0.00020 bar and  
165 volume standard deviation lower than 0.050 %. True density was then calculated by ratio of  
166 sample mass (previously determined) and sample volume determined by gas displacement.  
167 Each sample was measured at least in duplicate.

168 Once both true and apparent density were obtained, open porosity was calculated as follows,  
169 Eq. (2):

170

$$171 \quad \theta_{open} = 1 - \frac{\rho_A}{\rho_T} \quad (2)$$

172

173 where  $\theta_{open}$  corresponded to open porosity (-) and  $\rho_A$  and  $\rho_T$  to, respectively, apparent and true  
174 density ( $\text{kg/m}^3$ ) of samples determined as previously explained.

175

## 176 2.5. Water sorption isotherms

177 Water sorption isotherms of samples were determined at 23°C using a Dynamic Vapour  
178 Sorption machine (SPS-Sorption Test System, proUmid), employing nitrogen as gas in order  
179 to control relative humidity ( $\varphi$ ). At least duplicate samples ( $219 \pm 34$  mg as average sample  
180 weight) were measured. Different equilibrium points were determined corresponding to a  
181 fixed relative humidity: 0, 5, 10, 15, 20, 30, 35, 50, 75, 85 and 90 % for adsorption  
182 experiments, and 0, 15, 30, 35, 50, 75, 85, 90 % for desorption experiments. Samples  
183 employed for adsorption experiments were previously air-dried until constant weight in an  
184 oven (Mettler, Germany) at 40°C. Desorption samples were previously exposed to a relative  
185 humidity of 90% for hydration until they reached a constant weight.

186 GAB (van den Berg & Bruin (1981), Eq. (3)) and BET models (Brunauer *et al.*, 1938, Eq. (4))  
187 were employed to model water sorption experimental data (equilibrium moisture content,  $X_{eq}$   
188 versus  $\varphi$ ):

189

$$X_{eq} = \frac{X_m K C \phi}{(1-K\phi)(1-K\phi+CK\phi)} \quad (3)$$

191

$$X_{eq} = \frac{X_m C \phi}{(1-\phi)(1+(C-1)\phi)} \quad (4)$$

193

194 where  $X_m$  is the monolayer moisture content (kg water/kg dried solid, **dry basis: d.b.**),  $C$   
 195 (dimensionless, -) and  $K$  (dimensionless, -) is a parameter related to the heat of sorption of the  
 196 multilayer in GAB model. The values of GAB model parameters give different information.  $C$   
 197 parameter is related to the water bound in the monolayer, the higher  $C$ , the stronger water is  
 198 bound in the monolayer and the higher the difference in enthalpy between the monolayer  
 199 molecules and multilayer molecules (Quirijns et al., 2015).  $K$  is called a correction factor,  
 200 since it corrects the properties of the multilayer molecules relative to the bulk liquid. When  $K$   
 201 approaches one, there is almost no distinction between multilayer molecules and liquid  
 202 molecules. In that case the water molecules beyond the monolayer are not structured in a  
 203 multilayer, but have the same characteristics as the molecules in the bulk liquid. The more the  
 204 sorbed molecules are structured in a multilayer, the lower the value for  $K$  (Quirijns et al.,  
 205 2015). Finally,  $X_m$  is a measure of the availability of active sites for water sorption by the  
 206 material. It has to be noted that BET was applied in the range of relative humidity from 0.05  
 207 to 0.35 in order to determine the specific surface area of samples (**Collet et al., (2008)**)  
 208 assuming that  $X_m$  of BET model represents the quantity of water molecules that covers the  
 209 entire surface as a monolayer and no diffusion of water through the material **has** taken place.  
 210 Experimental data was modelled using these two methods employing Solver function of Excel  
 211 software. In the case of BET model, once model parameters were obtained, specific surface  
 212 area was calculated as follows, Eq. (5):

213

$$a_s = \frac{X_m L a_m}{M_w} \quad (5)$$

215

216 where  $a_s$  is the specific surface area of the sample ( $\text{m}^2/\text{g}$ ),  $L$  the Avogadro constant ( $6.023 \cdot 10^{23} \text{ mol}^{-1}$ ),  $a_m$  cross sectional area of water molecule ( $1.08 \cdot 10^{-19} \text{ m}^2$ ) and  $M_w$  molecular weight  
 217 of water molecules ( $18 \text{ g/mol}$ ).  
 218

219

## 220 **2.6. Water absorption kinetics**

221 Water absorption kinetics of different samples were determined at 5, 15, 60, 480, 1440, 2880  
 222 min (t) employing a protocol based on the one previously published by Amziane & Collet  
 223 (2017) with some modifications. Samples ( $M_0 \approx 1 \text{ g}$ ) previously dried at  $40^\circ\text{C}$  until constant  
 224 weight using an oven (Mettler, Germany) were put in a water bath (water temperature  
 225  $23.0 \pm 0.5^\circ\text{C}$ ) during different periods of time employing a tea strainer ball. Once the desired  
 226 time of immersion was achieved, samples were removed and filtered by vacuum filtration  
 227 with a Büchner funnel using a paper filter, in order to eliminate the excess of water on the  
 228 surface of the material, and finally weighed to determine the mass of the sample after  
 229 immersion ( $M_t$ , g). Afterwards, samples were dried in an oven (Mettler, Germany) at  $105^\circ\text{C}$   
 230 until constant weight ( $M_{105^\circ\text{C}}$ , g). Each experimental data point of water absorption kinetics  
 231 were carried out at least in triplicate.

232 In order to analyse the water absorption kinetics different parameters were calculated: water  
 233 absorption related to initial dry mass ( $WA_{M_0}$ , kg water/kg initial dry mass, Eq. (6)); water  
 234 absorption related to final dry mass ( $WA_{M_f}$ , kg water/kg final dry mass, Eq. (7), which takes  
 235 into account the mass lost due to leaching during immersion) and solid loss during immersion  
 236 (SL, kg lost dry solid/kg final dry mass, Eq. (8)):

237

$$238 \quad WA_{M_0} = \frac{M_t}{M_0} \quad (6)$$

239

$$240 \quad WA_{M_f} = \frac{M_t}{M_{105^\circ\text{C}}} \quad (7)$$

241

$$242 \quad SL = \frac{M_0 - M_{105^\circ\text{C}}}{M_{105^\circ\text{C}}} \quad (8)$$

243

244 These parameters were finally modelled employing a function developed by Nagy and Vas  
245 (Nagy and Vas 2003) that gives an asymptotically correct approximation of the capillary  
246 water uptake process for both  $t \rightarrow 0$  and  $t \rightarrow \infty$ , Eq. (9):

247

$$248 \quad \Delta m(t) = \Delta m_{\infty} (1 - e^{-At^{\frac{1}{2}B}})^B \quad (9)$$

249

250 where A and B are constant parameters,  $\Delta m_{\infty}$  is the modelled parameter related to equilibrium  
251 (when  $t \rightarrow \infty$ ) and  $\Delta m(t)$  is the experimental data of water absorption kinetics ( $W_{A_{M0}}$ ,  $W_{A_{MF}}$   
252 or SL).

253

## 254 **2.7. Scanning electro microscopy (SEM)**

255 Images of samples were taken employing scanning electro microscopy (JSM-IT100 LA,  
256 JEOL, Japan) under vacuum conditions (40 Pa) and an accelerating voltage of 15 kV.

257

## 258 **2.8. Statistical analysis**

259 The goodness of the adjustment of each model was determined based on coefficient of  
260 determination ( $R^2$ ) and root mean square error ( $E_{RMS}$ , Eq. (10)):

261

$$262 \quad E_{RMS} = \left[ \frac{1}{N} \sum_{i=1}^N (X_{\text{exp}} - X_{\text{cal}})^2 \right]^{1/2} \quad (10)$$

263

264 where N is the number of data points,  $X_{\text{exp}}$  the experimental data point and  $X_{\text{cal}}$  the calculated  
265 data point using the corresponding model. The values of the parameters of each model were  
266 obtained with an automated least square minimization approach.

267

## 268 **3. Results and Discussion**

### 3.1. Chemical Composition

Table 1 highlights the heterogeneity of the studied materials. In the case of soluble compounds, the range values varied from 6.0 up to 38.6 % d.b., sunflower pith had the highest value whereas sunflower bark presented the lowest one. Most of the studied samples presented values of soluble compounds around 10 % d.b.. Hemicellulose ranged between 5.0 to 34.8 % d.b and in most of the studied samples it was the second most important fraction on the materials composition. Cellulose is the main component of all studied samples except for sunflower bark and pith where the highest percentage corresponded respectively to lignin and soluble compounds. The cellulose values varied from 23.3 to 49.0 % d.b.. Lignin represented between 3.3 to 41.7 % d.b.. Finally, ashes varied from 2.4 % for miscanthus to 13.7 % d.b. for rice husk. Except for sunflower pith and rice husk, the percentage of ashes on the studied samples always represented the smallest part of their composition.

The composition of the different studied plant particles was similar of the one previously published in literature by different authors even if in certain cases the comparison is difficult due to the use of different methods. Flax shiv (Mahieu et al., 2019), hemp shiv (Viel et al., 2018), maize bark (Zhang et al., 2018), miscanthus (Ntimugura et al., 2020), reed (Honoré et al., 2020), rice husk (Chandrasekhar et al., 2003), sunflower bark and pith (Chabriac et al., 2016) and wheat (Viel et al., 2018).

For a specific plant, its chemical composition may vary as function of the geography, the weather and the maturity of the plant (Viel et al., 2018). The botanical species and the location in the plant can also influence the chemical composition. In this sense, the difference on chemical composition between sunflower bark and pith could be explained by their location. Sunflower bark, which is obtained from outer part of the stem (mainly responsible of the structural stability of the plant), had a higher proportion of hemicellulose, cellulose and lignin because they are polymers with a very important supportive structural function. On the other hand sunflower pith, obtained from the inner part of the stem where the structural function is less important, presented a lower proportion of these polymers and a higher

296 proportion of non-structural components (soluble compounds). Hemp and flax shiv show  
297 similar compositions (mainly composed of cellulose), which may also be linked to the fact  
298 that they are obtained from the same location of the plant stem. Chemical composition of  
299 wheat particles and maize bark particles are similar, with high contents of hemicellulose and  
300 cellulose, and intermediate contents of soluble compounds. For miscanthus and reed particles  
301 chemical composition is similar, with high contents of hemicellulose and cellulose, and low  
302 contents of soluble compounds. Compositions of sunflower pith, sunflower bark and rice husk  
303 particles differ from all others with, respectively, high content of soluble, lignin and ash.

304

### 305 *Location of Table 1*

306

### 307 **3.2. Particle size characterization, density and porosity**

308 Particle size characterization of different samples showed that most of them can be  
309 satisfactorily fitted employing a lognormal model for both length and width parameters  
310 ( $R^2 > 0.993$  and  $E_{RMS} < 0.027$ ). The exception corresponded to rice husk length where the  
311 lognormal model was unable to fit rice husk length, particularly at lower sizes, Table 2.  
312 Taking into account that the selected plant particles may come from different parts of the  
313 plant: 1) the inner part of the stem (flax shiv, hemp shiv, sunflower and maize pith); 2) the  
314 outer part of the stem (sunflower and maize bark); 3) the whole stem (fragments of the stems  
315 obtained after grinding: wheat or miscanthus) or 4) the husks (envelopes) of grains of rice and  
316 the differences in terms of shape and texture could be remarkable, from elongated and rigid  
317 (i.e. sunflower bark, miscanthus) to thin and deformable (i.e. flax shiv, wheat or reed) or  
318 spherical/cubic and sponge-like (i.e. sunflower pith particles) the use of the same model to  
319 manage their particle size distribution may prove to be useful. Largest and smallest particles  
320 for both length and width were the same: sunflower bark showed the largest mean values  
321 (10.7 mm for length and 3.9 for width) hemp shiv being the smallest studied particle with a  
322 mean value of 4.5 mm for length and 1.2 mm for width. Several particles were ground up

and/or sieved during the transformation process, so the particle size distribution is not an intrinsic property of plant particles.

Regarding density values, it has been found that in the case of bulk density sunflower bark showed the highest value followed by miscanthus, hemp shiv, rice husk and flax shiv, Table 2. The lowest values corresponded to sunflower pith, wheat, maize bark and reed. The range of values for the studied samples varied between 21 kg/m<sup>3</sup> and 157 kg/m<sup>3</sup>, which is the result of differences between intrinsic properties of each material and of the grinding and/or sieving process. These values are in accordance with those previously published by Chabriac et al. (2016), which studied hemp shiv, sunflower bark and pith, flax shiv and rapeseed.

### ***Location of Table 2***

In the case of true density, sunflower pith was also the material with the lowest density. However, it was observed that for the other studied samples, true density did not follow the same trend of bulk density. For example, flax shiv and sunflower bark presented the highest true density whereas they showed intermediate bulk densities. This fact reflects that bulk density can be influenced by other properties of the material such as particle size and shape that could lead to different arrangement of the material in the bulk. Specifically, in the case of sunflower bark this difference can be related to the higher particle size in the bulk which could lead to an arrangement in the bulk with higher void volume leading to a lower bulk density. In this sense, this is corroborated by the higher porosity obtained for this material. Even if grinding and sieving processes affect the particle shape and arrangement, a slight linear relationship ( $R^2 < 0.87$ ) between the porosity and the bulk density was found: the higher the porosity, the lower the bulk density. Figure 1, shows as a qualitative example, the different microstructures of each plant particle obtained employing SEM.

### **3.3. Water sorption isotherms**

Water adsorption-desorption isotherms of all studied samples were satisfactorily modelled employing GAB model (Eq. 3) ( $R^2 > 0.9994$ ;  $E_{RMS} < 0.003$ ; Table 3) which confirms the type II isotherm (Collet et al., 2008), Figure 2a and 2b.

#### *Location of Table 3 and Figure 2*

At high relative humidities ( $>0.80$ ), three main groups of plant particles could be established. Sunflower pith, maize bark and hemp shiv presented equilibrium moisture contents  $>0.24$  kg water/kg initial dry solid; wheat and sunflower bark showed  $X_{eq}$  between 0.20 and 0.22 and rice husk, miscanthus, reed and flax shiv were the ones with lower values (0.16-0.18 kg water/kg initial dry solid). In the case of adsorption process, hemp shiv was the sample with a stronger water bound in the monolayer followed by flax shiv and wheat whilst miscanthus and sunflower bark were the samples with the weaker water bound. Regarding  $X_m$ , it has been noticed that sunflower (bark and pith) had the higher values indicating a higher availability of active sites for water adsorption being hemp shiv, wheat and maize bark the ones with the lower values. This trend was corroborated by  $a_s$  values obtained employing Eq. (5), Table 3. This fact makes hemp shiv the sample with the lower quantity of available active sites of all studied samples but, at the same time, the one with the stronger bounded water. Moreover,  $K$  value was near to 1 which means that water molecules that are not present in the monolayer have almost the same behaviour as liquid molecules. During desorption process all equilibrium moisture contents were higher compared to adsorption process for each sample. In general, the samples with lower values for adsorption process remained the same in the case of desorption process. However, in the case of flax shiv and miscanthus the  $X_m$  for desorption process were the highest between all samples. This fact could be related to the compositions of these materials, Table 1, which presented the lower values of ash content having, consequently, a higher proportion of other compounds that could help to retain or absorb water.

### 3.4. Water absorption kinetics

Experimental data of water absorption kinetics of all studied samples presented a similar behaviour, Figure 3a. At the beginning of the immersion (first 500 min) samples quickly absorb water and then an asymptotic process of water gain takes place until achieving the equilibrium (48 h). A similar kinetic was the one observed for solid loss (SL) where it was observed that the main SL during immersion takes place at the beginning of the immersion process, Figure 3b. This phenomenon explains the difference between  $W_{A_{M0}}$  and  $W_{A_{MF}}$  kinetics. As it can be observed, at the beginning of the immersion process samples absorbed water and leach water-soluble compounds that lead to an increasing difference between  $W_{A_{M0}}$  and  $W_{A_{MF}}$ . Once the equilibrium of SL is achieved and the leaching of soluble compounds becomes almost negligible the difference between  $W_{A_{M0}}$  and  $W_{A_{MF}}$  remains constant. Experimental data was satisfactorily modelled employing Nagy and Vas model, Eq. (9) for WA parameters ( $R^2 > 0.98$ ). In the case of SL modelling the fitting was not satisfactory ( $R^2 > 0.83$ ), Table 4.

#### *Location of Table 4 and Figure 3a and 3b*

Results revealed that sunflower pith was the sample with the highest water absorption capacity (35.8 kg water/kg final dry solid) far higher than the other studied samples. The range of values for the other studied samples varied from 2.3 for rice husk up to 7.1 (kg water/kg final dry solid) for maize bark. Moreover, sunflower pith and rice husk were the samples with a higher absorption rate (they reach the equilibrium water absorption capacity faster than other ones), being hemp shiv, miscanthus and sunflower pith the ones with the lower water absorption rate. This behaviour could be related to both chemical composition and porosity of bulk material. In the case of hemp shiv and miscanthus they all have the lower values of open porosity which could lead to a slower water absorption process. This is not the case of sunflower pith, the explanation in this case may be found with the higher content of lignin and the lower content of other compounds known to be hydrophilic.

Finally, Nagy and Vas model parameters related to equilibrium ( $SL_{\infty}$ ) were in accordance with the trend obtained by Van Soest method for soluble compounds. However, their values were systematically lower than those obtained by the Van Soest method. It can be explained by the fact that during water absorption experiments only water-soluble compounds are leaching from the sample and consequently determined, compared to Van Soest method where the most part of soluble compounds (not only water-soluble) are quantified. The solid loss may be related mainly to soluble compounds that are eliminated from the samples during immersion.

#### 4. Conclusions

Nine biobased particles available on French territory with an actual or potential use in construction were studied. Their microstructure, densities, water behaviour and chemical composition was compared. The novelty of this study lies in three essential contributions. Firstly, all plant particles were characterized with exactly the same procedure and operator, which allows a fair comparison between them in order to clear out their differences. Secondly, results of particle size distribution (log-normal distribution model), water sorption isotherms (GAB model) and water absorption kinetics (Nagy and Vas model) were satisfactorily modelled using the same model for the different samples which could be useful for the prediction or estimation of these properties for future applications. Finally, the water absorption kinetics observed were found to be consistent with the chemical composition of the plant particles and the open porosity, which highlight a fair understanding of inter-dependence of particles physical properties.

Applications of the plant particle could be the use of the plant particle alone (i.e as a raw insulation) or mixed with a binder such as clay (Colinart et al., 2020) or lime, to make biobased concrete (light), wattle and daub (medium) or coating (heavy). Based on our results, it can be concluded that there are no best or worst particles, but different particles with varying degrees of difference depending on the studied property. The knowledge of such

430 differences between particles allows professional construction workers to select the best  
431 particle for their need, or alternatively, to adapt its practices depending on the **plant** particle  
432 locally available. For instance, quantity of **soluble compounds** might be an issue when using  
433 lime binder. Water absorption rate explains the workability or texture evolution when the  
434 particle is mixed with a binder in a mixer, or the hardening behaviour for sprayed biobased  
435 concrete. Sorption in the medium relative humidity range illustrates the water regulation  
436 capabilities of the material. **From a practical point of view it is useful to evaluate if the use of**  
437 **a different plant particle in a biobased concrete would affect its water regulation capabilities.**  
438 This study is a first step since it was carried out on only one batch for each type of plant.  
439 Further studies might be focused on the variability of each plant types in order to assess the  
440 range of dispersion for each studied characteristic. Plant diversity allows a wide range of  
441 methods in construction. **Each construction applications (particle boards, lime-based**  
442 **mortars/renders, earth-based mixes, etc.) has its own characteristics and interactions with**  
443 **plant properties.** The multitude of specifications echoes the diversity offered by plants. By  
444 working according to defined specifications, the diversity and complexity of plant particles  
445 become a source of potential.

446

#### 447 **CRedit authorship contribution statement**

448 Santiago Arufe: Conceptualization; Investigation; Validation; Writing original draft, review  
449 and editing.

450 Arthur Hellouin de Ménibus: Conceptualization; Investigation; Validation; Writing review  
451 and editing.

452 Nathalie Leblanc: Investigation; Validation; Writing review and editing.

453 Hélène Lenormand: Conceptualization; Investigation; Validation; Writing review and editing.

454

#### 455 **Declaration of Competing Interest**

456 The authors report no declarations of interest.

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464

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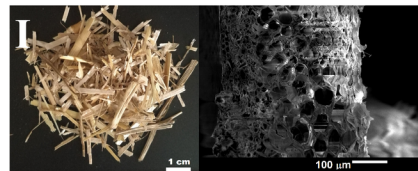
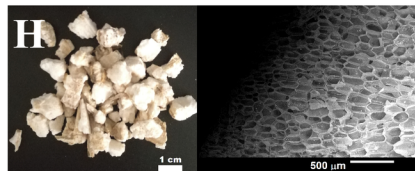
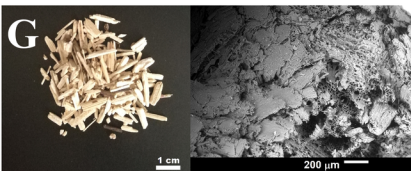
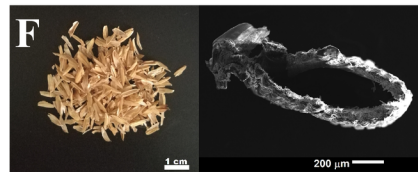
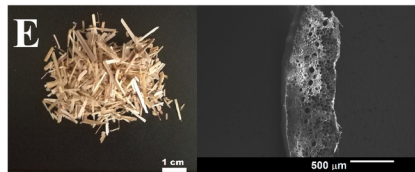
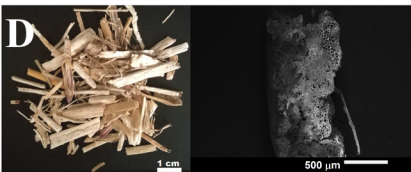
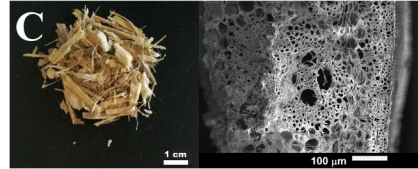
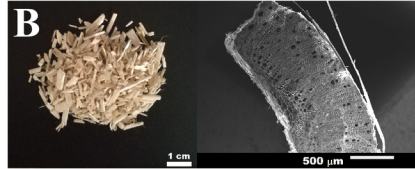
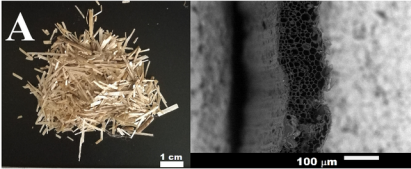
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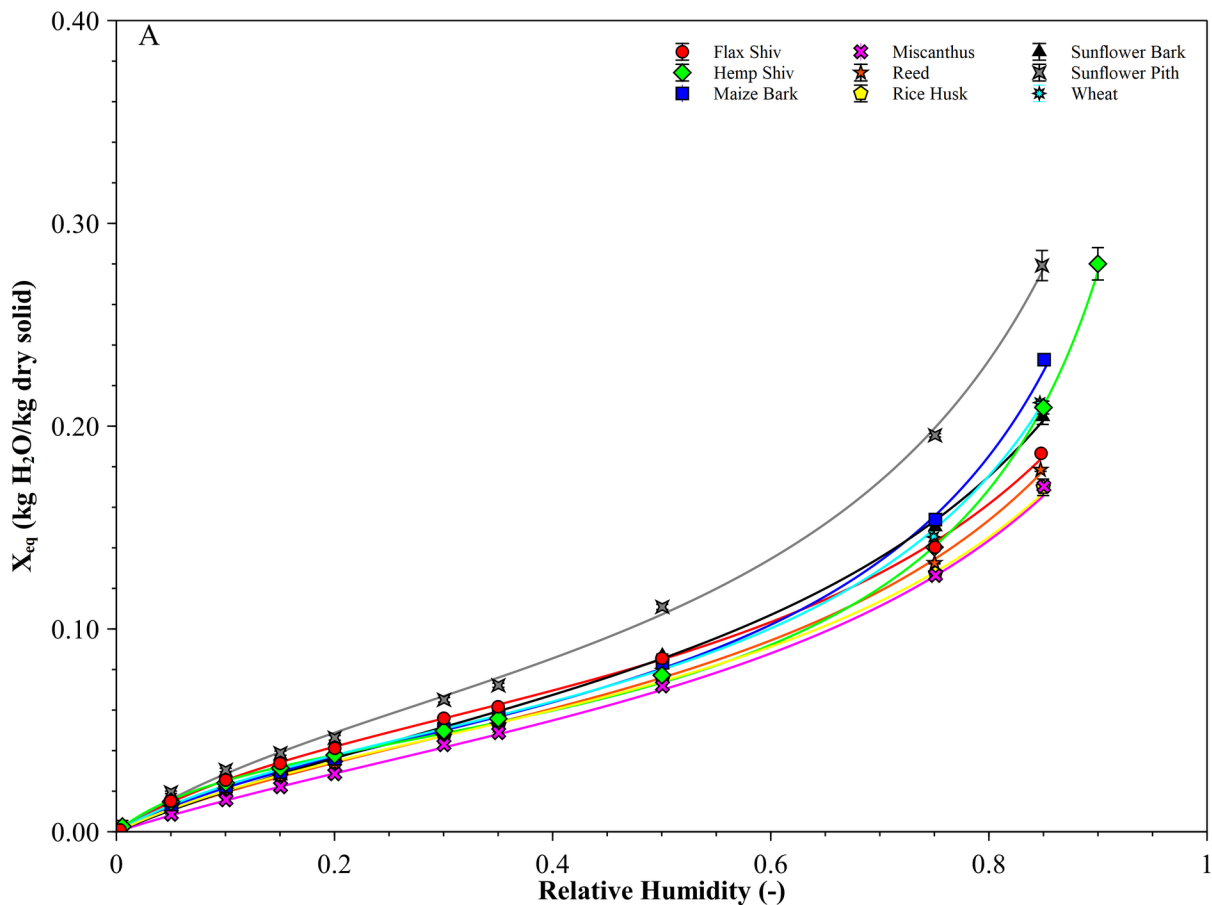
# **Caption for Figures**

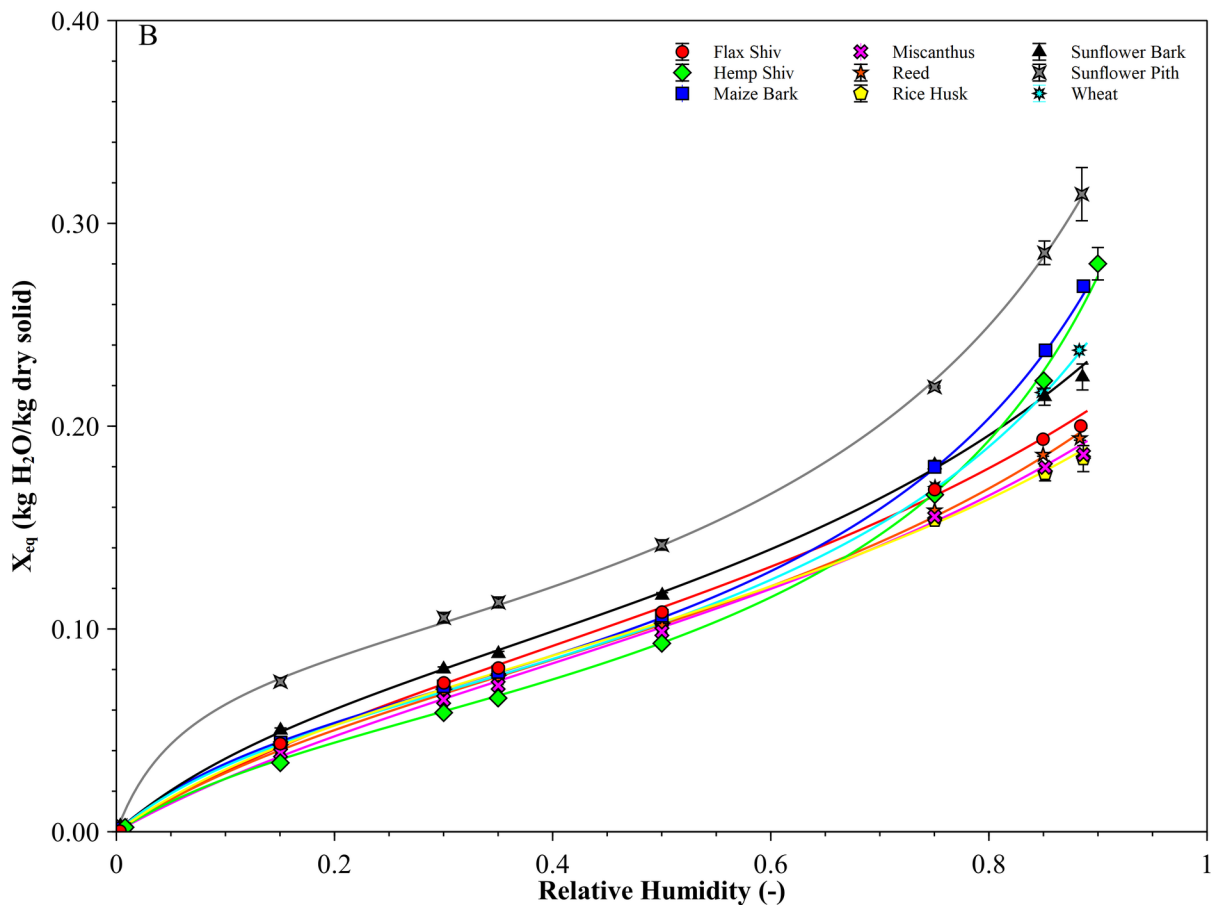
**Figure 1.** Images of the studied plant particles. A: Flax Shiv; B: Hemp Shiv; C: Maize Bark; D: Miscanthus; E: Reed; F: Rice Husk; G: Sunflower Bark; H: Sunflower Pith; I: Wheat.

**Figure 2.** Water adsorption (A) and desorption (B) isotherms of the studied systems. Line corresponds to GAB model, Eq. (3).

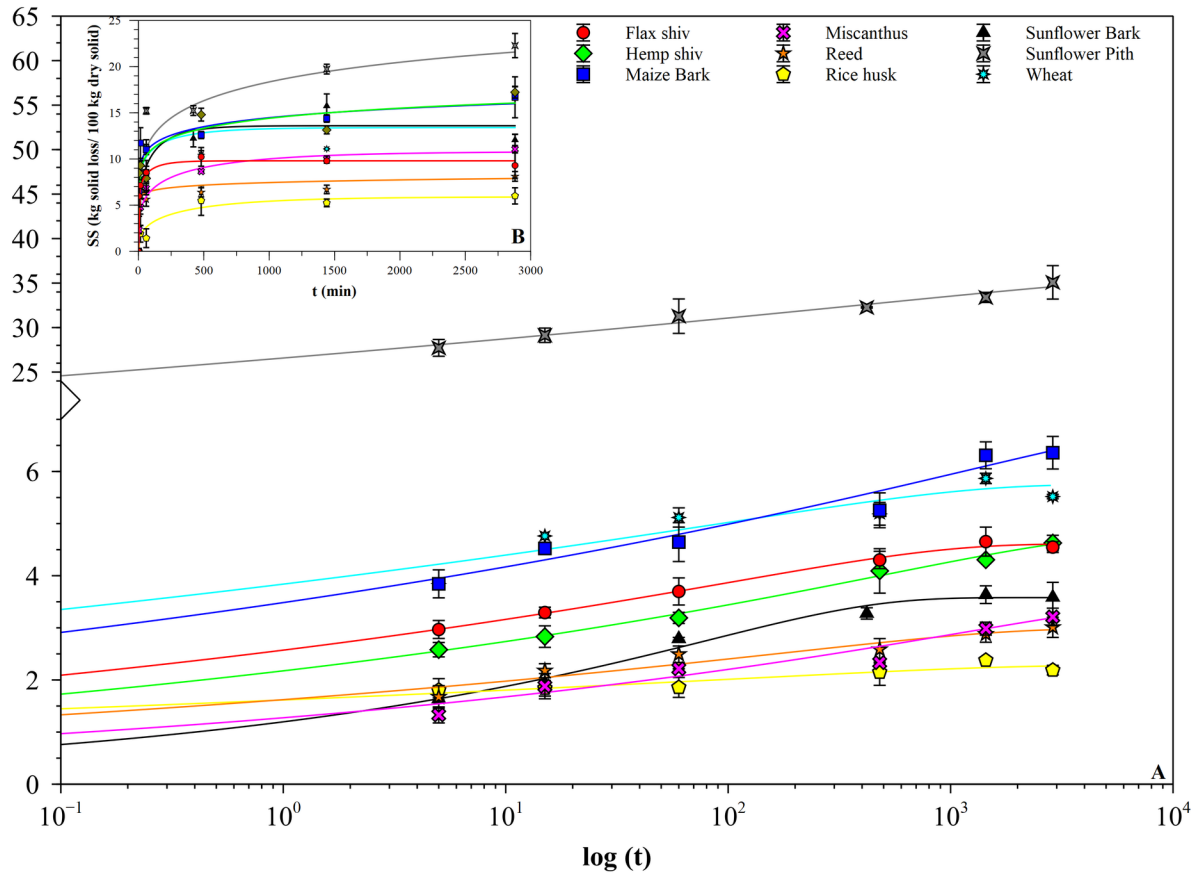
**Figure 3.** Water absorption (A) and solid loss kinetics (B) of the studied systems. Line corresponds to Nagy and Vas model, Eq. (9).







W<sub>A<sub>MF</sub></sub> (kg water/kg final dry solid)



**Table 1.** Chemical composition of different studied plant particles.

<b>Sample</b>	<b>Soluble (% d.b.)</b>	<b>Hemicellulose (% d.b.)</b>	<b>Cellulose (% d.b.)</b>	<b>Lignin (% d.b.)</b>	<b>Ash (% d.b.)</b>	<b>X<sub>initial</sub> (% d.b.)</b>
Flax Shiv	9.1±1.9	17.6±2.4	43.5±4.7	27.1±4.5	2.7±0.9	4.7±0.7
Hemp Shiv	17.2±1.1	21.6±1.8	49.0±2.8	8.1±0.6	4.1±1.6	3.1±0.4
Maize Bark	15.3±0.2	34.8±0.9	42.8±1.0	3.3±0.2	3.8±0.2	2.8±0.1
Miscanthus	7.2±0.5	27.8±1.4	43.6±9.8	19.0±10.8	2.4±0.1	3.5±1.5
Reed	9.6±1.5	28.3±1.3	47.6±1.2	10.5±1.5	4.0±0.1	2.4±0.1
Rice Husk	7.7±0.3	19.8±0.5	42.1±0.6	16.8±0.2	13.7±0.1	1.9±0.1
Sunflower Bark	6.0±2.2	18.1±2.8	30.3±6.2	41.7±8.5	3.8±0.5	6.5±0.1
Sunflower Pith	38.6±1.1	5.0±1.9	23.3±6.8	21.4±6.4	11.7±0.3	8.7±0.3
Wheat	10.5±0.1	34.1±4.5	45.3±4.3	6.2±0.3	3.9±0.1	5.5±0.1

% d.b.: g/100 g dry solid; X<sub>initial</sub>: Humidity of samples before starting the tests.

**Table 2.** Parameters of Lognormal function, Eq. (1), corresponding to systems length and width frequency distribution, apparent ( $\rho_A$ ), compacted ( $\rho_C$ ) and true ( $\rho_T$ ) densities and open porosity ( $\theta=1-\rho_A/\rho_T$ ), Eq. (2).

Sample	Length				Width				Density (kg/m <sup>3</sup> )			Open Porosity (-)
	$\mu$	$\sigma$	$R^2$	$E_{RMS}$ (-)	$\mu$	$\sigma$	$R^2$	$E_{RMS}$ (-)	$\rho_B$	$\rho_C$	$\rho_T$	$\theta$
Flax Shiv	2.070	0.320	0.999	0.008	0.265	0.330	0.999	0.009	101±5	144±4	1406±19	0.928
Hemp Shiv	1.503	0.546	0.999	0.011	0.157	0.565	0.998	0.013	114±4	167±6	1247±41	0.909
Maize Bark	1.633	0.633	0.999	0.010	0.446	0.760	0.999	0.008	67±2	112±3	1075±6	0.938
Miscanthus	1.812	0.829	0.998	0.014	0.288	1.036	0.997	0.015	128±5	186±9	940±9	0.864
Reed	2.057	0.679	0.998	0.017	0.283	0.639	0.997	0.010	81±2	134±3	1016±11	0.920
Rice Husk	2.077	0.129	0.985	0.040	0.840	0.305	0.997	0.015	112±2	147±4	1328±26	0.916
Sunflower Bark	2.373	0.385	0.997	0.017	1.364	0.330	0.999	0.010	157±9	176±6	1395±12	0.887
Sunflower Pith	1.641	0.395	0.998	0.014	1.278	0.400	0.999	0.009	21±3	31±4	754±25	0.973
Wheat	2.233	0.596	0.999	0.011	0.557	0.731	0.993	0.027	45±4	85±5	1163±13	0.961

**Table 3.** GAB model, (Eq. 3), parameters for water sorption isotherms of studied plant particles.

Agroresource	Adsorption					Desorption					$a_s$ (m <sup>2</sup> /g) Eq. (5)
	C (-)	K (-)	X <sub>m</sub> (d.b.)	R <sup>2</sup> (-)	E <sub>RMS</sub> (d.b.)	C (-)	K (-)	X <sub>m</sub> (d.b.)	R <sup>2</sup> (-)	E <sub>RMS</sub> (d.b.)	
Flax Shiv	7.0±0.1	0.812±0.004	0.061±0.001	0.9995	0.002	5.2±0.4	0.607±0.038	0.111±0.010	0.9994	0.003	178.9±0.6
Hemp Shiv	10.8±1.3	0.941±0.008	0.043±0.001	0.9997	0.002	6.3±1.3	0.863±0.034	0.064±0.007	0.9997	0.002	156.6±8.0
Maize Bark	6.0±0.2	0.919±0.006	0.052±0.001	0.9998	0.002	8.2±0.4	0.834±0.010	0.072±0.002	0.9999	0.001	168.8±0.1
Miscanthus	3.6±0.1	0.805±0.001	0.060±0.001	0.9996	0.002	4.8±0.4	0.612±0.031	0.103±0.008	0.9994	0.003	170.1±1.8
Reed	5.0±0.1	0.829±0.001	0.057±0.001	0.9997	0.001	5.9±0.1	0.660±0.013	0.092±0.002	0.9997	0.002	158.2±1.1
Rice Husk	5.7±0.2	0.817±0.015	0.055±0.001	0.9997	0.001	6.5±0.1	0.618±0.023	0.096±0.003	0.9997	0.002	172.3±2.4
Sunflower Bark	4.1±0.1	0.820±0.009	0.068±0.001	0.9997	0.002	7.0±0.1	0.684±0.003	0.099±0.001	0.9997	0.002	196.9±5.8
Sunflower Pith	5.7±0.1	0.884±0.018	0.073±0.003	0.9997	0.002	20.2±1.1	0.811±0.030	0.090±0.005	0.9998	0.002	205.2±1.6
Wheat	6.7±0.2	0.900±0.002	0.052±0.001	0.9998	0.001	7.8±0.2	0.796±0.004	0.074±0.000	0.9999	0.000	160.8±0.1

d.b.: g water/g dry solid

**Table 4.** Nagy and Vas model parameter, Eq. (9), for different plant particles.

Parameter	Flax shiv	Hemp shiv	Maize Bark	Miscanthus	Reed	Rice Husk	Sunflower Bark	Sunflower Pith	Wheat
$WA_{F\infty}$ (kg water/ kg d.s.)	4.6	4.8	7.1	3.6	3.0	2.3	3.6	35.8	5.7
$A_{WAF}$	0.033	0.007	0.003	0.003	0.019	0.032	0.039	0.009	0.036
$K_{WAF}$	0.089	0.100	0.078	0.119	0.086	0.048	0.197	0.034	0.059
$R^2$	0.999	0.998	0.991	0.976	0.983	0.989	0.993	0.999	0.983
$E_{RMS}$ (kg water/ kg f.d.s.)	0.1	0.1	0.2	0.2	0.1	0.1	0.1	0.4	0.2
$WA_{0\infty}$ (kg water/ kg i.d.s.)	4.1	3.7	5.8	3.1	2.6	2.1	3.0	29.0	4.9
$A_{WA0}$	0.027	0.035	0.003	0.003	0.170	0.055	0.051	0.009	0.057
$K_{WA0}$	0.078	0.110	0.065	0.112	0.187	0.036	0.182	0.012	0.050
$R^2$	0.999	0.998	0.992	0.982	0.986	0.991	0.988	1.000	0.979
$E_{RMS}$ (kg water/ kg i.d.s.)	0.0	0.1	0.1	0.1	0.1	0.1	0.1	0.4	0.2
$SL_{\infty}$ (kg solid/ kg i.d.s.)	9.8	17.9	18.4	10.8	9.1	5.9	13.6	25.4	13.4
$A_{SL}$	0.124	0.003	0.002	0.011	0.001	0.009	0.034	0.002	0.038
$K_{SL}$	0.162	0.121	0.104	0.229	0.056	0.332	0.287	0.181	0.143
$R^2$	0.994	0.927	0.941	0.975	0.960	0.918	0.868	0.940	0.838
$E_{RMS}$ (kg solid/ kg i.d.s.)	0.3	1.4	1.3	0.6	0.5	0.6	1.9	1.9	2.0

kg i.d.s.: kg of initial dry solid; kg f.d.s.: kg of final dry solid