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# 1 Hygrothermal properties of clayey plasters with olive fibers

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### 13 Abstract

14

This research deals with how to use agricultural waste materials in constructions considering that the 15 development of innovative materials has to respond to both environmental and energy issues. The 16 leaves and the small branches derived from the pruning of olive trees were incorporated, after drying, 17 into a clay-sand mixture to obtain a bio-based plaster. Earthen samples, containing different 18 19 percentages of olive fibers, were prepared and tested to investigate their hygrothermal behaviour. Thermal conductivity and the effects of several parameters on thermal performance were analyzed. 20 21 The evaluation of the hygric behaviour was based on the measurement of the water vapour diffusion 22 resistance factor and of the isothermal sorption curves. Results showed that the addition of olive fibers in the earth matrix enhances the insulating and hygric performances. The ability of the material to 23 24 exchange moisture was studied by calculating the ideal Moisture Buffering Value (MBV<sub>ideal</sub>). Results 25 showed that the effects of moisture adsorption/desorption improved with the increase of the fibers 26 content.

28 Keywords: hygrothermal properties, clay, vegetable fibers, thermal properties, hygric properties

### 29 **1. Introduction**

30

31 Considering the development of society and people's ecological awareness, a sustainable and healthy 32 indoor environment is increasingly attracting the attention of the research. Thus, renewable and 33 environment-friendly materials are highly demanded [1]. Particularly, in recent years, there has been 34 a revival of interest in earthen construction materials. The main advantages of the use of earth are 35 related to the significant reduction in environmental impact. Clay is an abundant local resource with 36 very low embodied energy, which is cheap and easy to work [2]. According to Aymerich et al. [3] 37 earth-based materials can also offer very high levels of thermal comfort due to their high humidity 38 absorption/desorption rates, heat storage capacities and sound transmission properties. Natural fibres 39 are added to the mix, enhancing the physical performances as the reduction of density. It is important 40 to underline that the fibers are renewable and they have a sustainable life cycle [4].

This study investigates hygric and thermal properties of the clay-based plasters incorporating natural fibers as olive pruning waste. According to Meli et al. [5], earth plasters outperform conventional industrial plasters. On one hand, producing plasters from unbacked clay and sand requires a relatively small amount of energy compared to that required to produce conventional lime and cement plasters, since the production of these materials requires very high temperatures. On the other hand, transport represents an important proportion of the overall impact of earth plasters. Clay can be considered a local source available on the same site in which building will be realized.

Clay plasters are especially suitable for interior surfaces, to create comfortable and healthy spaces with a minimal impact on the environment. Many studies deal with the use of natural fibers as replacement to synthetic fibers in reinforced composites. Several aggregates derived from plants such as coconut fibers [6], hemp [7], straw [8], flax fibers [9], date palm trees fibers [10], or bamboo [11] have already been studied due to their attractive features, such as good mechanical properties, low cost, low density, low thermal conductibility, durability and recyclability. Radazzo et al. [12] carried 54 out specific laboratory tests on commercially available pre-mixed earthen plasters. The aim of the 55 research was to verify how mineralogical and textural features might affect the performances of the 56 final products in terms of moisture absorption and thermal conductivity. Results showed that the 57 thermal conductivity was strictly connected to the grain shape, grain size and porosity. Moreover, the 58 most significant increase in moisture content occurs for the mixtures characterized by a higher amount 59 of fibers.

60 Palumbo et al. [13] evaluated the thermal conductivity, the thermal diffusivity, the water vapour 61 permeability and the moisture buffering of plasters incorporating two types of vegetable materials 62 such as barley straw and corn pith. It was observed that the thermal conductivity of the clay materials 63 decreased with the addition of the vegetable fibers due to the reduction of density. The incorporation 64 of corn pith caused the most significant reduction of thermal conductivity. The vegetable materials had a limited effect on the hygric properties. They provoke a moderate increase of the water vapour 65 66 permeability and the moisture buffering capacity. Ashour et al. [14] measured the thermal conductivity of three different types of fibers as a reinforcement for natural plasters. Wheat straw, 67 barley straw and wood shavings were used. As expected, the thermal conductivity of all materials 68 69 decreased with increasing fibers content and increased with the addition of sand. The results 70 demonstrated that the plaster reinforced with barley straw fibers had the lowest values of thermal 71 conductivity. Plasters with wood shaving fibers had the lowest values of thermal insulation due to their highest density. 72

Currently hemp fibers are the most used for sustainable construction materials. Mazhoud et al. [15] investigated hygric and thermal properties of two hemp-lime plasters with different sizes of hemp shiv. Results showed that the hemp-lime plaster with the smallest hemp shiv was the best hygric regulator. The fiber size was also the basis of the studies conducted by Benmansoura et al. [16]. They investigated mortars formed with sand, cement and different sizes of date palm fibers. The addition of fibers in the mortar matrix decreased its density. Therefore, the first advantage of the use of natural fibers was the decreasing of its thermal conductivity. On the other hand results showed that the

83	2. Experimental procedure
82	the higher thermal conductivity values.
81	capacity of water absorption and the finer fibers adsorbed more water than the larger fibers, showing
80	thermal conductivity increased with water adsorption. The date palm fibers showed an enormous

## 83 2. Experimental proces

84 *2.1 Materials* 

85

Four different clayey plasters with olive fibers were studied. Different percentages of clay, sand,
gravel and fibers were used. The composition of the cohesive soil is as follows: quarry fines, quartzite
grit sand (<2mm), quartzite grit gravel (2-4mm), hydrated lime and water.</li>

89 The clay used has sandy fraction (1%), silt (28 %) and clay (71 %). Carbonates were equal to 22 %.

90 The clay granulometric analysis is shown in Figure 1.

91

92

93 The porosity of the clay and the fibers (olive leaves and branches) was measured by a Helium gas
94 Pycnometer ULTRAPYC 1200-e Quantachrome; the results are shown in Table 1.

95

Table 1: Porosity and true density of the clay and the olive pruning waste.

Raw materials	True density	Porosity
	$\rho_{true}  [kg/m^3]$	n [-]
Olive waste	1251	0,23
Clay	2859	0,37

96

97 The microstructural morphology and chemical composition of the used clay and of the clayey olive 98 plaster were characterized by Zeiss EVO MA 10 Environmental Scanning Electron Microscopy 99 (SEM) with Oxford Instruments Inca model spectrometer for Energy Dispersive X-Ray Spectrometry 100 (EDS). Figure 2 shows the scaly crystal structure of the clay, whereas Figure 3 shows the 101 microstructural morphology of the plaster.

102
103

104	EDS (Energy Dispersive Spectroscopy on the SEM) was used to identify the main elemental
105	composition: Figure 4 and Figure 5 represent the spectrum of clay and clay-olive plaster, respectively.
106 107 108	Clay exhibited a higher content of SiO <sub>2</sub> than the clayer placter (Fig. 4.5), in fact in the first case the
100	City exhibited a higher content of $510_2$ than the eraycy plaster (Fig. 4-5), in fact in the first case the
109	SIO <sub>2</sub> content was on average around 55.62% and in the second case was on average around 41.70%.
110	This is likely due to the higher sand content in the mixture.
111	Clay, sand and gravel were mixed with leaves and branches derived from the pruning of olive trees.
112	According to Laborel-Préneron et al. [17], the aggregates were sized to obtain a homogeneous
113	mixture that would be easy to apply on the wall. The average size of the fibers was about 2 cm as it
114	is shown in Figure 6.
115	The SEM was also used to analyze the microstructural morphology of an olive leaf. Figure 7 shows
116	that the olive leaf appears covered by numerous starred petals that guarantee ultraviolet radiation
117	protection and maintain a thin layer of damp air to the surface.
117 118	protection and maintain a thin layer of damp air to the surface.
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117 118 119 120 121	protection and maintain a thin layer of damp air to the surface. 2.2 Sample preparation
117 118 119 120 121 122	protection and maintain a thin layer of damp air to the surface. 2.2 Sample preparation The blends were prepared using a forced-action rotary paddle mixer before adding the required water
<ol> <li>117</li> <li>118</li> <li>119</li> <li>120</li> <li>121</li> <li>122</li> <li>123</li> </ol>	protection and maintain a thin layer of damp air to the surface. 2.2 Sample preparation The blends were prepared using a forced-action rotary paddle mixer before adding the required water content for dynamic compaction. Different weight percentages of clay, sand, gravel were mixed with
<ol> <li>117</li> <li>118</li> <li>119</li> <li>120</li> <li>121</li> <li>122</li> <li>123</li> <li>124</li> </ol>	protection and maintain a thin layer of damp air to the surface. <i>2.2 Sample preparation</i> The blends were prepared using a forced-action rotary paddle mixer before adding the required water content for dynamic compaction. Different weight percentages of clay, sand, gravel were mixed with four percentages (4%, 6%, 8%, 12%) of olive pruning waste fibers; the proportions are reported in
<ol> <li>117</li> <li>118</li> <li>119</li> <li>120</li> <li>121</li> <li>122</li> <li>123</li> <li>124</li> <li>125</li> </ol>	protection and maintain a thin layer of damp air to the surface. 2.2 Sample preparation The blends were prepared using a forced-action rotary paddle mixer before adding the required water content for dynamic compaction. Different weight percentages of clay, sand, gravel were mixed with four percentages (4%, 6%, 8%, 12%) of olive pruning waste fibers; the proportions are reported in Table 2. Considering the hygric and the thermal tests shown in the paragraphs below specimens were
<ol> <li>117</li> <li>118</li> <li>119</li> <li>120</li> <li>121</li> <li>122</li> <li>123</li> <li>124</li> <li>125</li> <li>126</li> </ol>	protection and maintain a thin layer of damp air to the surface. 2.2 Sample preparation The blends were prepared using a forced-action rotary paddle mixer before adding the required water content for dynamic compaction. Different weight percentages of clay, sand, gravel were mixed with four percentages (4%, 6%, 8%, 12%) of olive pruning waste fibers; the proportions are reported in Table 2. Considering the hygric and the thermal tests shown in the paragraphs below specimens were prepared by casting the mixture into Styrofoam molds with different sizes. For each percentage of
<ol> <li>117</li> <li>118</li> <li>119</li> <li>120</li> <li>121</li> <li>122</li> <li>123</li> <li>124</li> <li>125</li> <li>126</li> <li>127</li> </ol>	protection and maintain a thin layer of damp air to the surface. 2.2 Sample preparation The blends were prepared using a forced-action rotary paddle mixer before adding the required water content for dynamic compaction. Different weight percentages of clay, sand, gravel were mixed with four percentages (4%, 6%, 8%, 12%) of olive pruning waste fibers; the proportions are reported in Table 2. Considering the hygric and the thermal tests shown in the paragraphs below specimens were prepared by casting the mixture into Styrofoam molds with different sizes. For each percentage of olive pruning six samples for the hygrothermal measurements were prepared. The specimens were

129 15 cm x 15 cm x 4 cm were prepared for thermal tests and according to UNI EN 1015-19 [18] three

130 different cylindrical specimens of 15 cm diameter and 1 cm thickness were realized for water vapour

- 131 permeability tests (Figure 8).
- 132

Mixture code	Clay [%]	Sand [%]	Gravel [%]	Fibers [%]
4	38	56	2	4
6	38	54	2	6
8	37	53	2	8
12	35	51	2	12

Table 2: Composition of the olive-clay plasters (% b.w.).

- 133
- 134

### 135 **3. Experimental tests**

### 136 *3.1 Thermal performances*

137

138 The thermal properties (thermal conductivity, thermal diffusivity and specific heat capacity) were139 experimentally evaluated.

Prior to testing the thermal properties, all specimens were oven dried at 105 °C until they achieved constant mass  $\pm 0.5\%$  before cooling to ambient temperature in desiccators containing silica gel. The dry-state thermal conductivity and thermal diffusivity were measured using an ISOMET 2104, a transient plane source device.

The measurement is based on the analysis of temperature response of the analyzed material to heat flow impulses. The advantage of this technique is that it is possible to perform rapid measurements (typically about 10/15 min) on relatively small samples. Thermal conductivity, thermal diffusivity and volumetric heat capacity were obtained from the measurement. The specific heat capacity was evaluated from volumetric heat capacity and density. 149 *3.2 Hygric properties* 

150

151 Water vapour permeability was measured according to UNI EN 1015-19 [18] using the cup method.

- 152 Saturated salt solutions of Potassium Nitrate (KNO<sub>3</sub>) were used for wet-cup test (93,2% RH) and
- 153 Lithium Chloride (LiCl) saturated solutions for dry-cup test (12,4% RH).
- 154 Each cylindrical specimen was wax sealed on the top of a PVC vessel containing salt solution with 1
- cm thickness air layer between the water surface and internal sample surface. The assembly was then
  placed in the Perani AC520 climate chamber set to 20 °C and 50% RH.
- 157 The assemblies weight was recorded by a Mettler Toledo PB3002 balance ( $\pm 0.01$  g accuracy) until 158 constant time variation of mass ( $\Delta G/\Delta \tau$ ) was achieved. The vapour pressure gradient between 159 climatic chamber environment and cup ( $\Delta p$ ) was estimated. For each specimen, the vapour 160 permeance was calculated as follows:

$$\Lambda = \frac{1}{\frac{A \cdot \Delta p}{(\Delta G / \Delta \tau)} - R_A}$$
(1)

161 The water vapour permeability was achieved from the following equation:  $\delta_p = \Lambda \cdot d \qquad (2)$ 

162

163 The water vapour resistance factor was estimated as:

$$\mu = \frac{1,94 \cdot 10^{-10}}{\delta_p} \tag{3}$$

164

- 165 where  $1,94 \cdot 10^{-10}$  was the air permeability at 20 °C.
- 166 *3.3 Porosity*
- 167

168 A ULTRAPYC 1200-e Quantachrome Helium gas Pycnometer was used to determine bulk porosity

169 (n):

170

$$n = \frac{V_{tot} - V_{true}}{V_{tot}} \tag{4}$$

Because of its ideal gas behaviour, helium is the preferred gas used for this analysis. V<sub>true</sub> was measured from the measured drop in pressure when the gas (helium) was allowed to expand into a chamber containing about 20 g of sample and a second chamber of fixed, known (via calibration) internal volume. Thus the true density ( $\rho_{true}$ ) was calculated as follows:

$$\rho_{true} = \frac{m}{V_{tot} - V_{pores}} = \frac{m}{V_{true}} \tag{5}$$

176

178

## 177 *3.4 Hygroscopic sorption properties*

The hygroscopic sorption properties of porous building materials can be measured by the salt 179 solutions in desiccators or in climatic chamber [19]. In this study, a Perani AC520 climatic chamber 180 181 was used. Three representative samples were tested after oven drying at 105 °C for 48 hours. The 182 specimens were placed in the climatic chamber at 20 °C and 30% RH. Their weight was recorded daily, until constant mass was achieved. According to UNI EN 12571 [19], a specimen was 183 184 considered to be in steady-state when the weight loss between two successive measurements, with a 185 time interval of at least 24 h, remained less than 0.1%. Values at 30%, 50%, 65%, 80%, 93% RH 186 were performed.

The moisture content at 97% RH was measured according to the salt solutions in the desiccator [19]. The specimens were placed in a desiccator containing a Potassium Sulphate ( $K_2SO_4$ ) saturated solution on the bottom. During the test the desiccator was kept in the climatic chamber at 20 °C. The specimen's weight was recorded daily until achieving constant equilibrium mass. The water content (w) was calculated as the amount of absorbed water per dry volume (kg/m<sup>3</sup>) with an experimental error of 3%:

193

$$w = \frac{m - m_0}{V} \tag{6}$$

194 The sorption isotherms  $w(\phi)$  describing the equilibrium moisture content is a function of relative 195 humidity ( $\phi$ ) and was represented by the following relation:

$$w = \exp\left(a \cdot \varphi^b + c \cdot \varphi^d\right) \tag{7}$$

197 where a, b, c, d were constant to be determined with a multiple regression on the experimental data.

# 198 *3.5 Ideal Moisture Buffer Value*

199

The ideal Moisture Buffer Value (MBV<sub>ideal</sub>) as defined in [20] was calculated. This value constitutes one theoretical possibility to express the ability of a material to absorb or release moisture when the humidity of air changes in the environment that surrounds it. The ideal value of MBV was calculated from the following equation:

$$MBV_{ideal} \approx \frac{G(t)}{\Delta RH} = 0,00568 \cdot p_{sat} \cdot b_m \cdot \sqrt{t_p}$$
(8)

204 where  $b_m$  is the moisture effusivity:

$$b_m = \sqrt{\frac{\delta_p \cdot \rho \cdot \frac{\partial u}{\partial \varphi}}{p_{sat}}} = \sqrt{\frac{\delta_p \cdot \xi}{p_{sat}}}$$
(9)

## 205 **4. Results and discussion**

### 206 4.1 Thermal conductivity, thermal diffusivity and specific heat capacity

207

208 Table 3 gives the average values of the density and the thermal properties, i.e. thermal conductivity,

- thermal diffusivity and specific heat capacity.
- 210

Table 3: Thermal and physical properties.

Mixture code	Bulk density	Thermal	Thermal	Specific heat	
	ho [kg/m <sup>3</sup> ]	conductivity	diffusivity	capacity	
		$\lambda$ [W/mK]	$\alpha [\mathrm{m^2/s}]$	c [J/kgK]	
CO4	1669	0,593	0,387×10 <sup>-6</sup>	849,57	

CO6	1599	0,532	0,365×10 <sup>-6</sup>	869,65
CO8	1497	0,458	0,320×10 <sup>-6</sup>	908,61
CO12	1409	0,428	0,295×10 <sup>-6</sup>	958,91
	206 208 2012	206     1599       208     1497       2012     1409	206       1599       0,532         208       1497       0,458         2012       1409       0,428	$206$ 15990,5320,365×10^{-6} $208$ 14970,4580,320×10^{-6} $2012$ 14090,4280,295×10^{-6}

Thermal conductivity versus density is presented in Figure 9. The curve shows a decrease of thermal conductivity with a decrease of the density. Plasters with a higher content of fibers had a lower thermal conductivity, i.e. a greater thermal insulation capacity.

In Figure 10, thermal conductivity of tested olive-clay plasters was compared to other bio-based materials. Experimented materials properties are in accordance with the performances measured by Minke [21] and that reported in UNI EN 1745 [22].

#### 4.2 Influence of the percentage of olive fiber on the density

Laborel-Préneron et al. [17] stated that the density is an interesting property because of its correlation with thermal characteristics. As expected when increasing the fiber content a decrease in the composite dry density can be noted. Figure 11 shows the effect of olive fiber content on bulk density of the plasters. The density strongly decreases as the percentage of olive fiber increases from 4% to 12%.

#### 4.3 Hygric properties

### Figure 12 shows the water vapour mass loss of a specimen (CO4P-A) during a water vapour permeability test. As shown it takes about ten days for reaching constant mass variation.

- Table 4 shows results of cup method measurements as water vapour diffusion resistance factor.

Table 4: Water vapour diffusion resistance factor.

Mixture code	Water vapour diffusion		
	resistance factor $\mu$ [-]		
	Wet cup test	Dry cup test	
4	12.5	25,0	
6	15.1	24,8	
8	14.1	23,1	
12	13.4	22,1	

237 In Figure 13, water vapour diffusion resistance factor for olive-clay were compared to values obtained

for other bio-based materials with equivalent density [13]. It can be observed the similar trend of the

239 water vapour diffusion resistance factor by varying the density.

### 240

### 241 *4.4 Porosity*

### Table 5 gives the average values of the total open porosity for each mixture.

243

Table 5: Average values of the bulk density, true density and total open porosity.

Mixture	Bulk density	True density	Porosity
	$ ho_{bulk}  [kg/m^3]$	$\rho_{true}  [kg/m^3]$	n [-]
4	1669	2721	0,38
6	1599	2683	0,39
8	1497	2724	0,42
12	1409	2601	0,44

244

## 245 *4.5 Hygroscopic sorption properties*

246

247 Figure 14 shows the sorption isotherms obtained from adsorption-desorption test according to

equation 7 with the a, b, c, d constant values reported in table 6.

249

Table 6: Constant values according to the Eq.7

Mixture	Constant			
	a	b	c	d
4	1.30	2.69	2.84	0.17
6	1.44	2.82	2.77	0.22
8	1.41	3.63	2.97	0.21
12	1.33	4.57	3.14	0.30

It can be observed that the sorption isotherms of the four materials displayed a similar slope up to 80% RH. As the relative humidity increased above 80%, the moisture content adsorbed increased and the curves tends to rise rapidly. These results are in accordance with the observation of Cagnon et al. [23] who showed that when 100% RH was approached, significant capillary condensation occurred and led to a vertical asymptote on the sorption-desorption isotherms. This behaviour was observed especially for the specimens with higher content of fibers (C08-CO12), due to the highest porosity of these materials.

258

Figure 15 and Figure 16 show the water content  $(w_{80})$  adsorbed at 80% RH and the sorption capacity

260  $\xi$ , the slope of the sorption curves at 80% RH.

261 It can be noted that when using higher content of fibers a better sorption capacity is achieved.

262 263

### 264 *4.6 Ideal Moisture Buffer Value*

265 Table 7 gives the ideal MBV values corresponding to each mixture.

266

Table 7: Ideal Moisture Buffer Value.

Mixture code Ideal Moisture Buffer Value

 $MBV_{ideal}$  [g/(m<sup>2</sup> %RH) @8/16h]

CO4	1,708
CO6	1,709
CO8	1,710
CO12	1,739

The results show an improved moisture exchange performance by increasing fibers content. According to the classification of the moisture buffer performance proposed by NORDTEST protocol [20] this material is classified "good" (1<MBV<2). Thanks to this characteristic, the studied clayey plaster can be considered positively to moderate indoor air relative humidity amplitudes, and as a consequence, to improve occupant's hygrothermal comfort sensation.

- 272 **5.** Conclusions
- 273

In this study leaves and small branches derived from the pruning of olive trees were incorporated, after drying, into a clay-sand mixture to obtain a bio-based plaster. Earthen samples, with different percentages of olive fibers, were prepared and tested to investigate their hygrothermal behaviour. The results achieved from the test measurements show that the addition of olive fibers in the earth matrix resulted in a linear reduction in the density and in an increase of porosity. This means a reduction in thermal conductivity and therefore a more insulating behaviour of the material.

Experimental tests have also allowed to characterize the hygric properties of the materials. The hygrothermal properties are in agreement with that of other bio-based plasters with similar density. The addition of the olive fibers doesn't produce significant differences in the sorption curves up to 80% RH. As the relative humidity increased above 80%, the moisture content adsorbed increased especially for the specimens with higher content of fibers (C08, CO12).

Results in terms of the ideal Moisture Buffer Value (MBV<sub>ideal</sub>) show that all mixtures allow to obtain
a plaster with a good ability to exchange moisture with neighboring environment, when there is a

daily cyclic variation of relative humidity. This performance has positive consequences on the indoorair quality and occupant's comfort.

Further studies are ongoing in order to improve the hygrothermal properties of bio-based plasterswith agro-waste.

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### 352 Appendix: nomenclature

Α	specimen area	$m^2$
$b_m$	moisture effusivity	kg/m <sup>2</sup> Pa·s <sup>0,5</sup>
С	specific heat capacity	J/kgK
d	specimen tickness	m
G	moisture uptake	kg/m <sup>2</sup>
m	weight mass	kg

$m_0$	initial weight mass	kg
<b>MBV</b> <sub>ideal</sub>	Ideal Moisture Buffer Value	$kg/(m^2\% RH)$
n	porosity	-
р	pressure	Ра
<i>p</i> sat	saturation vapour pressure	Ра
R <sub>A</sub>	water vapour diffusion resistance in the gap in the plastic cup	m <sup>2</sup> sPa/kg
RH	relative humidity	%
t <sub>p</sub>	period	S
и	moisture content	kg/kg
V	volume	m <sup>3</sup>
W	moisture content	kg/m <sup>3</sup>
W80	moisture content at 80 % RH	kg/m <sup>3</sup>
α	thermal diffusivity	m <sup>2</sup> /s
$\delta_p$	water vapour permeability	kg/msPa
$\Delta G/\Delta \tau$	flow of water vapour over time	kg/s
Δp	difference of the water vapour pressure between the saline solution and the air of environment	Pa
λ	thermal conductivity	W/mK
Λ	vapour permeance	kg/m <sup>2</sup> sPa
μ	water vapour diffusion resistance factor	_
ξ	slope of the sorption curves at 80 % RH	-
$ ho_{bulk}$	bulk density	kg/m <sup>3</sup>
$\rho_{true}$	true density	kg/m <sup>3</sup>
τ	period	h
ф	relative humidity	-