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Article

Drying Behavior of Flax Stems at Different Degrees of Dew Retting Under Simulated Rainfall: Implications for Smart Agriculture and Sensor Development

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Abstract: Dew retting of flax stems is a key agricultural process which facilitates fiber extraction from parent stems. If sensors are to be developed to monitor the degree of retting for optimal fiber extraction, then stem characteristics such as water uptake and loss need to be accurately known. Here, the moisture uptake and drying behavior of short flax stem samples in different degrees of dew retting is studied. Their drying is characterized in laboratory conditions following simulated light and heavy rainfall. The data were accurately fitted with the Page model of moisture loss. It was observed that the drying rate constant of small flax stem samples is smaller for simulated light rain compared to simulated heavy rain. Also, over-retted stem samples dry more rapidly compared to under-retted samples. The findings suggest that this is due to the degradation of the external tissue of the stems observed in the over-retted samples. The results could be useful for laboratory testing of flax stems and designers of emerging technological sensor-based tools used to monitor the degree of retting in controlled conditions.

Keywords: flax stems; dew retting; moisture content; drying; Page model; digital agriculture



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1. Introduction

Dew retting of flax in the field is an important process which facilitates the extraction of flax fibers from their parent stems [1–4]. During dew retting, the pulled stems are deliberately exposed to the elements to stimulate biochemical processes which break down pectin linking the fiber bundles to the core and epidermis of the stem [5,6]. Dew retting usually takes place in summer and early autumn, meaning stems can be exposed to rain. This means that they can also contain excess water because of rainfall exposure. Due to the chemical composition of the flax fiber, water can diffuse between the amorphous regions of the cellulose and hemicellulose and thus be absorbed [7]. The amount of water in the flax stems has an influence on their mechanical properties. This means that any variations in water content can significantly affect stiffness and strength of flax stems. In a study, Baley et al. [8] found that the tensile strength of elementary flax fibers decreases after drying. Nair et al. [9] showed an increase in the tensile strength of flax stems following microwave conductive drying and hot air drying. These mechanical properties are important in the gathering of stems following retting and the monitoring of the degree of retting. Thus, knowledge of the drying behavior of flax stems following rain is important both for the ultimate gathering of stems once retting is deemed over, and also for emerging technologies which could monitor the degree of retting in real time using sensor-based tools where small

samples are used. In order to better understand the behavior of water content in small flax stem samples, an in-depth investigation of the water content in these stems is therefore essential. Such an investigation has the potential to refine the operation and timing of monitoring tools. However, in this context, the literature survey shows that there have been previous studies of the real-time measurement of the water content of fruity plants [10–14] but very few on green flax. Recently, Gibaud et al. [15] carried out a study to determine the differences between the characteristics of a flax stem in its green state and the same stem when dried, thus investigating the effects of water variation. In this study, the drying of short (1 cm long) flax stems in different degrees of retting was monitored. The drying of flax stems was studied following simulated light (5 min) and heavy (12 h) rain. The data were fitted to the Page model [12].

2. Materials and Methods

Figure 1 illustrates the overall methodology and steps of the study.

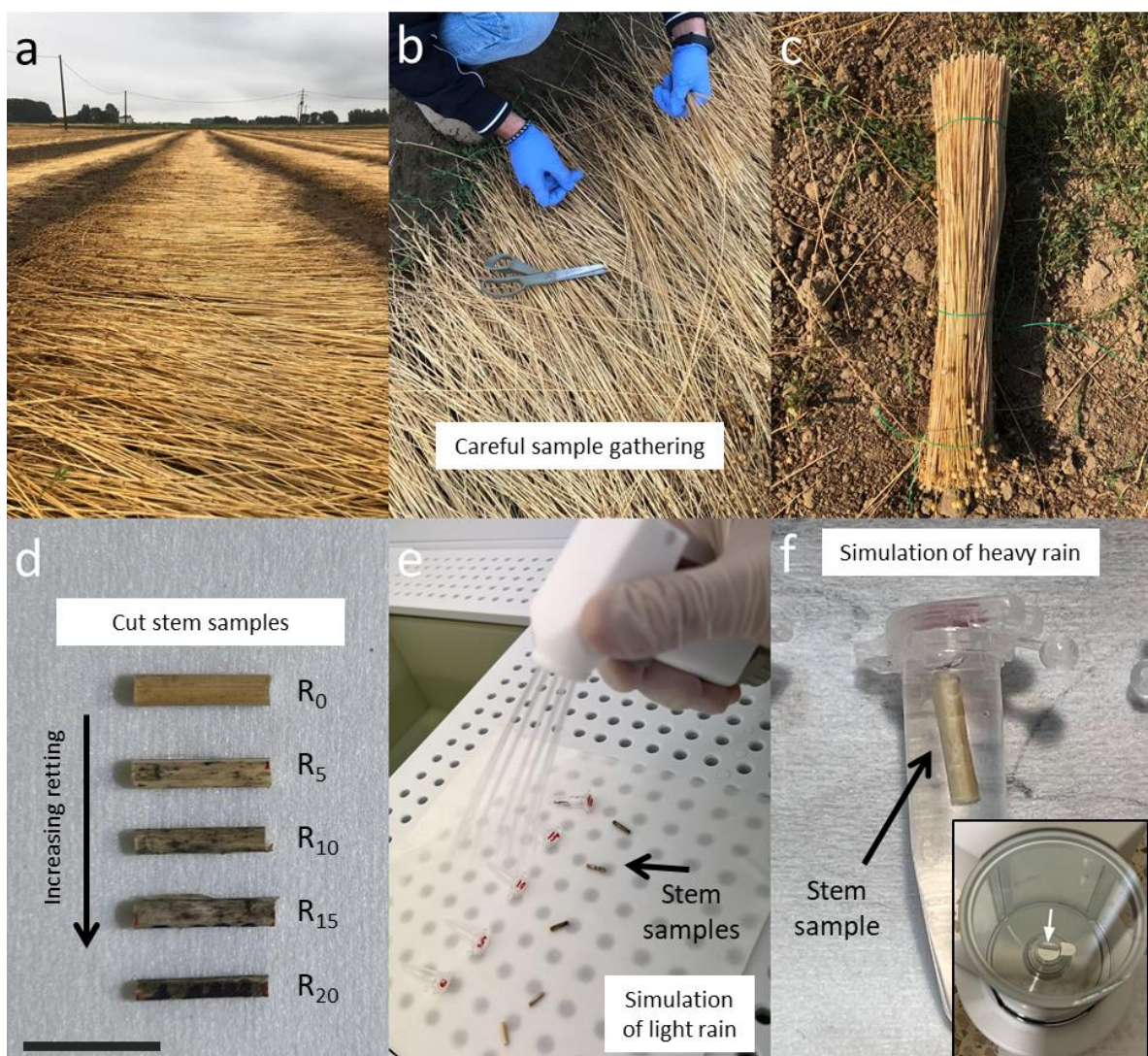


Figure 1. Overall methodology and steps of the study. (a) Flax stems dew retting in the field. (b) Careful choice of undamaged stems. (c) Flax stem bundle taken to our laboratory. (d) Examples of the carefully cut samples of flax stems (1 cm in length) at various degrees of retting ($R_0 = 0$ days, $R_5 = 21$ days, $R_{10} = 35$ days, $R_{15} = 56$ days, and $R_{20} = 91$ days). (e) Simulation of light rain and (f) simulation of heavy rain in the laboratory. The scale bar in (d) equals 1 cm. The inset to (f) shows the flax stem sample (indicated by the white arrow) being measured by the precision scales.

Figure 1a shows the flax stems dew retting in the field in summer 2022. Figure 1b,c show the careful stem gathering into bundles. Figure 1d shows the samples used in the study; they are approximately 1 cm long. Figure 1e,f show how light and heavy rain were simulated for the study. Precision scales were used to monitor the water loss (due to evaporation) from the stem samples in a controlled environment. Note that the drying of the stems was water loss via evaporation at fixed temperature and humidity.

2.1. Sample Gathering

Retted flax stems (Family: Linaceae, Genus: *Linum*, Species: *L. usitatissimum*, Variety: Felice) were collected from a flax production field run by the Van Robaeys Frères (VRF) company, located near Killeme in northern France. The flax samples were collected twice per week during the 91-day retting period of summer 2022 starting from the 6th of July until the 4th of October. The weather data of this period is included in the Supplementary Information—see Figure S1. This involved an under-retting period (0–56 days), an optimum retting point as defined by an artisan and an over-retting period (56–91 days). In this study, R_0 denotes the initial stage of retting immediately after harvesting (0 days); R_5 corresponds to a point three weeks after harvesting and the beginning of retting (21 days); R_{10} denotes a period of five weeks after harvesting (35 days); R_{15} represents the optimum retting time (56 days) which occurs eight weeks after harvesting. Finally, R_{20} denotes a retting stage thirteen weeks after harvesting (91 days), characterized by an advanced stage of retting.

2.2. Sample Preparation

Flax stem samples were always taken from the middle of the long flax plant stem shown in Figure 1c. Small (1 cm long) samples were cut using a razor blade using an in-house tool used to hold the stems during cutting in order to minimize damage. Details of the techniques can be found elsewhere [16–18]. The 1 cm long flax stem samples were observed for damage using a digital optical microscope (Keyence, France). The mass of each individual sample was determined using an UP-6U Ultra Micro Balance (Mettler Toledo, France).

2.3. Drying Experiments: Bundles of Long Flax Stems

To compare with the results obtained on short flax stem samples using precision measurements, rudimentary experiments were initially carried out on long flax stem bundles. The flax stem bundles were initially weighed and then subjected to a 5 min shower to simulate a light summer rain. During the shower, the stems were a little spread out rather than in a tight collection bundle—again, to simulate field conditions during retting. The flax stems were then weighed again to determine their water absorption. Following this, the weight of the stems was recorded as a function of time. The scales had a precision of 0.1 g. The room temperature was measured to be ~ 20 °C. Note that some stems are in contact with each other, as would be the case in the field. Evidently, for a bundle, the drying behavior is complicated by a number of factors, e.g., stem uniformity, contact of stems forming capillary bridges, and defects in stems.

2.4. Drying Experiments: Short Flax Stem Samples

The precision experiments on small flax stem samples were carried out in a controlled environment ($T = 21 \pm 1$ °C, $RH = 40 \pm 5\%$). Note that during flax drying in the retting period, the RH can vary significantly [19]. In addition, most studies of flax stems and fibers consider a high humidity [20,21]. However, for laboratory testing conditions, the relative humidity is likely to be lower. Therefore, in this study, to obtain precise results, experiments were conducted in typical laboratory temperature and humidity conditions.

For this part of the study, the mass of each individual small flax sample was accurately determined using an UP-6U Ultra Micro Balance (Mettler Toledo, France). The micro balance has a repeatability of 0.25 µg at low load and a sensitivity stability of 0.0001%. The mass of a typical sample is 30 mg. The small sample experiment was performed as follows. Following the cutting of the stems, their weight was accurately determined prior to exposure to water (simulated rainfall). Each sample was then exposed to two types of simulated rain. In order to simulate a light shower, the sample was sprayed with a hand-held shower for 5 min, see Figure 1e. To simulate heavy rainfall, the samples were soaked in 1000 µL of water for 12 h, see Figure 1f. Following each simulated rain, the sample was placed on the precision scales and the moisture content of the sample was measured by recording its weight change with time.

2.5. Modeling of the Moisture Content of Flax Stems During Drying

The dimensionless moisture ratio (MR) of a flax stem is given by

$$MR = \frac{m(t) - m_e}{m_0 - m_e} \quad (1)$$

where $m(t)$ is variation in the weight of the stem sample with time, m_e is the final constant weight of the stem sample, and m_0 is the initial stem sample weight at time zero. There is experimental evidence for flax [9] that suggest that the moisture ratio can be approximated by the following relationship during drying:

$$MR = e^{-kt^n} \quad (2)$$

where k is the drying rate constant (time^{-1}) and n is a dimensionless drying fitting factor. This is the Page model of moisture loss [12].

It is possible to obtain these factors k and n by numerically fitting the function in Equation (2) to the data using the least squares method. This numerical approach enables one to calculate the coefficient of determination R^2 of the fit.

3. Results

3.1. Long Flax Stem Bundles

Flax stem bundles were weighed and then subjected to a simulated rain (see Materials and Methods). Subsequently, the weight of the bundle was then recorded every 5 min and the data were analyzed, see Materials and Methods. Figure 2 shows plots of the mass and the computed moisture ratio of a flax stem bundle as a function of time following a 5 min light shower experiment. The moisture ratio is fitted using the Page model, indicated by the red curve in Figure 2.

Let us consider Figure 2. First, the moisture content of the flax stems is seen to increase after rain. In this example, the initial dry weight of the bundle was 19.6 g. Following a 5 min shower, this value rises to 33.3 g, Figure 2a. This corresponds to a water uptake of 13.7 g of moisture. Second, the moisture content slowly reduces due to evaporation over time. The computed moisture ratio (MR), using Equation (1), is observed to fall according to a smooth curve with time, Figure 2b. The average error bar for the data are 0.04. For the simulated heavy rain, the dry stems initially weighed 10 g. After simulated heavy rain they weighed 28 g. This is an uptake of 18 g, which is lost over the subsequent 5 h. This corresponds to a comparable evaporation evolution, as with the light rain. The data appear to fit well with the Page model discussed previously, see Equation (2). The drying rate constant k and the dimensionless drying fitting factor n are extracted from the numerical fit (Figure 2b). The results of this part of the study are summarized in Table 1.

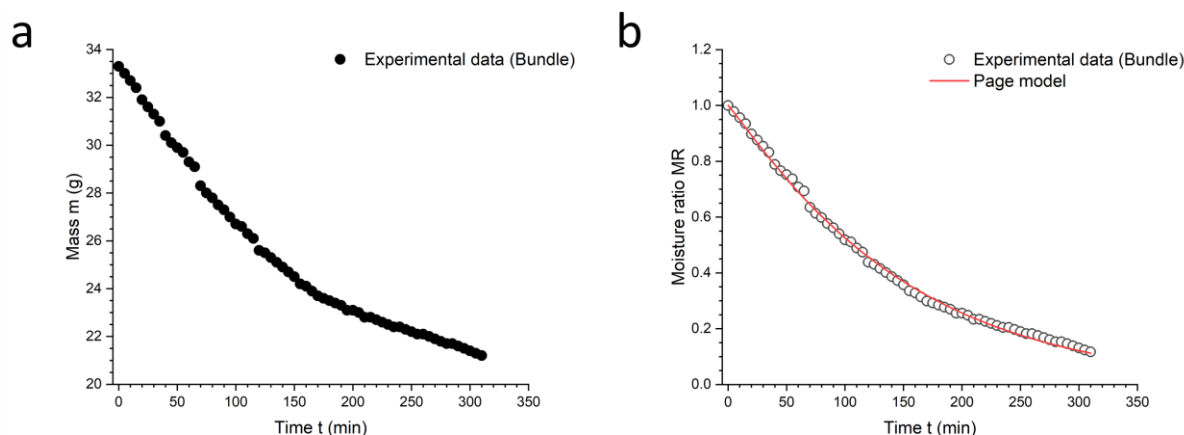


Figure 2. A plot of the mass and moisture ratio of a flax stem bundle as a function of time following exposure to a 5 min simulated rainfall. (a) The mass of the flax bundle versus time, and (b) the computed moisture ratio of the bundle fitted with the Page model (indicated by the red curve). The bundle was composed of 77 stems.

Table 1. A summary of the fitting parameters for the drying of flax stem bundles. *r* is the moisture uptake ratio, *n* is a unitless drying constant, *k* is the drying rate constant (time⁻¹), and *R*² is the coefficient of determination.

Parameter	Value
<i>r</i> (light rain)	1.70 ± 0.04
<i>r</i> (heavy rain)	2.8 ± 0.1
<i>n</i>	1.02 ± 0.08
<i>k</i> (min ⁻¹)	5.41 ± 1.37 × 10 ⁻³
<i>k</i> (h ⁻¹)	0.35 ± 0.03
<i>R</i> ²	0.995

3.2. Short Flax Stem Samples Exposed to Light Rain

In this part of the study, short single flax stem samples were studied. Flax stems samples of 1 cm length were prepared and subjected to a 5 min shower, see Materials and Methods. Figure 3 shows plots of the mass and moisture ratio of small flax stem samples as a function of time following the simulated rainfall. The data are fitted with the Page model of evaporation, indicated by the red curves.

Let us consider Figure 3. First, the moisture content of the flax stems is seen to increase following light rain (Figure 3). For example, the initial dry weight of the flax sample taken from the Retting stage *R*₀ was 13.82 mg. Following a 5 min shower, this rises to 23.44 mg. This corresponds to a water uptake of 9.62 mg of moisture. Similar scenario appeared when stimulating a light shower rain for 5 min on flax stem samples taken from the retting stages *R*₅, *R*₁₀, *R*₁₅, and *R*₂₀ (Figure 3a). The water uptake values are summarized in Table 2. Second, the moisture content reduces due to evaporation over 200 min. The moisture content clearly falls according to a smooth curve with time. Figure 3b–d show the moisture content behavior of flax stem samples taken from different retting stages (*R*₀ to *R*₂₀). Moreover, the Page model fits well the experimental moisture data. The numerical fit in (Figure 3b–f) permit the extraction of the drying rate constant *k* and the dimensionless drying fitting factor *n*. The results are summarized in Table 2.

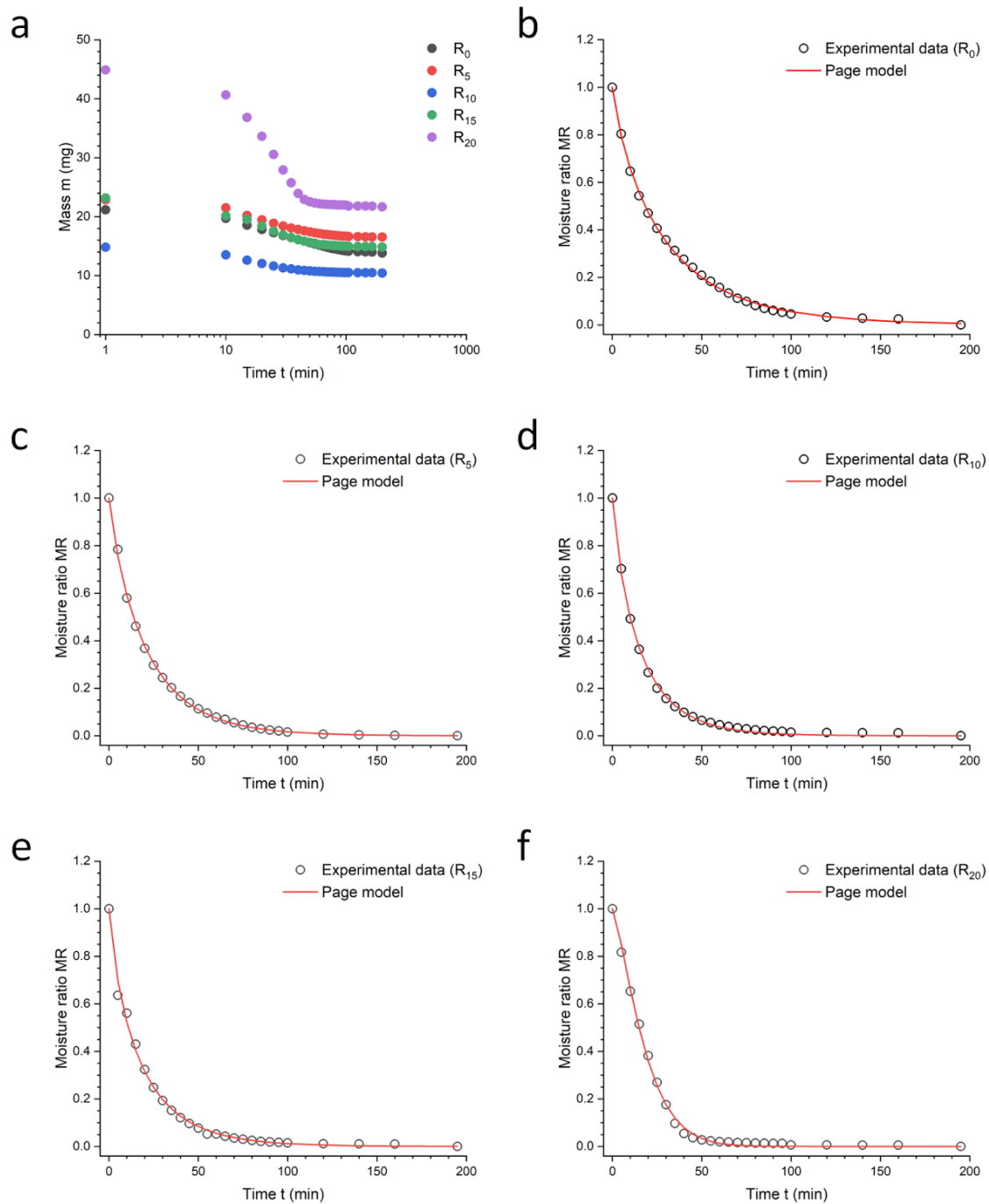


Figure 3. Plots of the mass and moisture ratio of small flax stem samples as a function of time following a simulated 5 min rainfall. (a) The mass of the flax stem samples as a function of drying time. Plots of the moisture ratio versus time for retting stage/length: (b) R₀ (0 days), (c) R₅ (21 days), (d) R₁₀ (35 days), (e) R₁₅ (56 days), and (f) R₂₀ (91 days). The red curves indicate fitting the moisture ratio experimental data using the Page model.

Table 2 shows that the average drying rate constant in the case of light rain measures $6.81 \times 10^{-2} \pm 3.16 \times 10^{-2} \text{ min}^{-1}$ ($2.99 \pm 0.90 \text{ h}^{-1}$) and the average ratio of the moisture uptake ratio r is 1.59 ± 0.28 . See Discussion for unit conversion in the Page model.

Table 2. Summary of the fitting parameters of the Page model and the water uptake values obtained from the study following light rain (5 min). r is the moisture uptake ratio, n is a unitless drying constant, k is the drying rate constant (time^{-1}), and R^2 is the coefficient of determination.

Parameter	R_0	R_5	R_{10}	R_{15}	R_{20}
r	1.53	1.38	1.42	1.56	2.07
n	0.84	0.89	0.86	0.83	1.31
k (min^{-1})	5.96×10^{-2}	6.77×10^{-2}	9.75×10^{-2}	9.54×10^{-2}	2.01×10^{-2}
R^2	0.999	0.999	0.998	0.996	0.998

3.3. Short Flax Stem Samples Exposed to Heavy Rain

In this part of the study, single flax stem samples were studied. The small flax stem samples were soaked in 1.5 mL volume of water for 12 h to simulate heavy, persistent rain. Figure 4 shows plots of the mass and the moisture ratio of small flax stem samples as a function of time following simulated heavy rain.

Let us consider Figure 4. First, the moisture content of the flax stems is seen to increase after one night of simulated heavy rain (Figure 4). For example, following the simulated rain, the weight of a flax stem sample taken from R_0 rises from 13.82 mg to 39.23 mg. This corresponds to a water uptake of 25.31 mg of moisture. A similar scenario appeared when stimulating a heavy shower rain for 12 h on flax stem samples taken from the retting stages R_5 , R_{10} , R_{15} , and R_{20} (Figure 4). The water uptake values are summarized in Table 3. Second, the moisture content reduces due to evaporation over 220 min. The moisture content clearly falls according to a smooth curve with time. Figure 4b–d show the moisture content behavior of flax stem samples taken from different retting stages (R_0 to R_{20}) following a heavy rain for 12 h. Moreover, the moisture content behavior fits well to the numerical model discussed previously. The numerical fitting in (Figure 4a–f) permits the extraction of the drying rate constant k and the dimensionless drying fitting factor n for this experiment. The results are summarized in Table 3.

Table 3. Summary of the fitting parameters of the Page model and the water uptake values obtained from the study following heavy rain (12 h). r is the moisture uptake ratio, n is a unitless drying constant, k is the drying rate constant (time^{-1}), and R^2 is the coefficient of determination.

Parameter	R_0	R_5	R_{10}	R_{15}	R_{20}
r	2.56	2.68	2.88	2.58	2.56
n	1.11	1.11	1.15	1.23	1.22
k (min^{-1})	1.34×10^{-2}	1.10×10^{-2}	1.51×10^{-2}	1.32×10^{-2}	1.94×10^{-2}
R^2	0.999	0.996	0.999	0.999	0.998

Table 3 shows that the average drying rate constant in the case of heavy rain measures $1.44 \times 10^{-2} \pm 3.13 \times 10^{-3} \text{ min}^{-1}$ ($1.77 \pm 0.72 \text{ h}^{-1}$), and the average ratio of the water uptake ratio r is 2.65 ± 0.14 . See Discussion for unit conversion in the Page model.

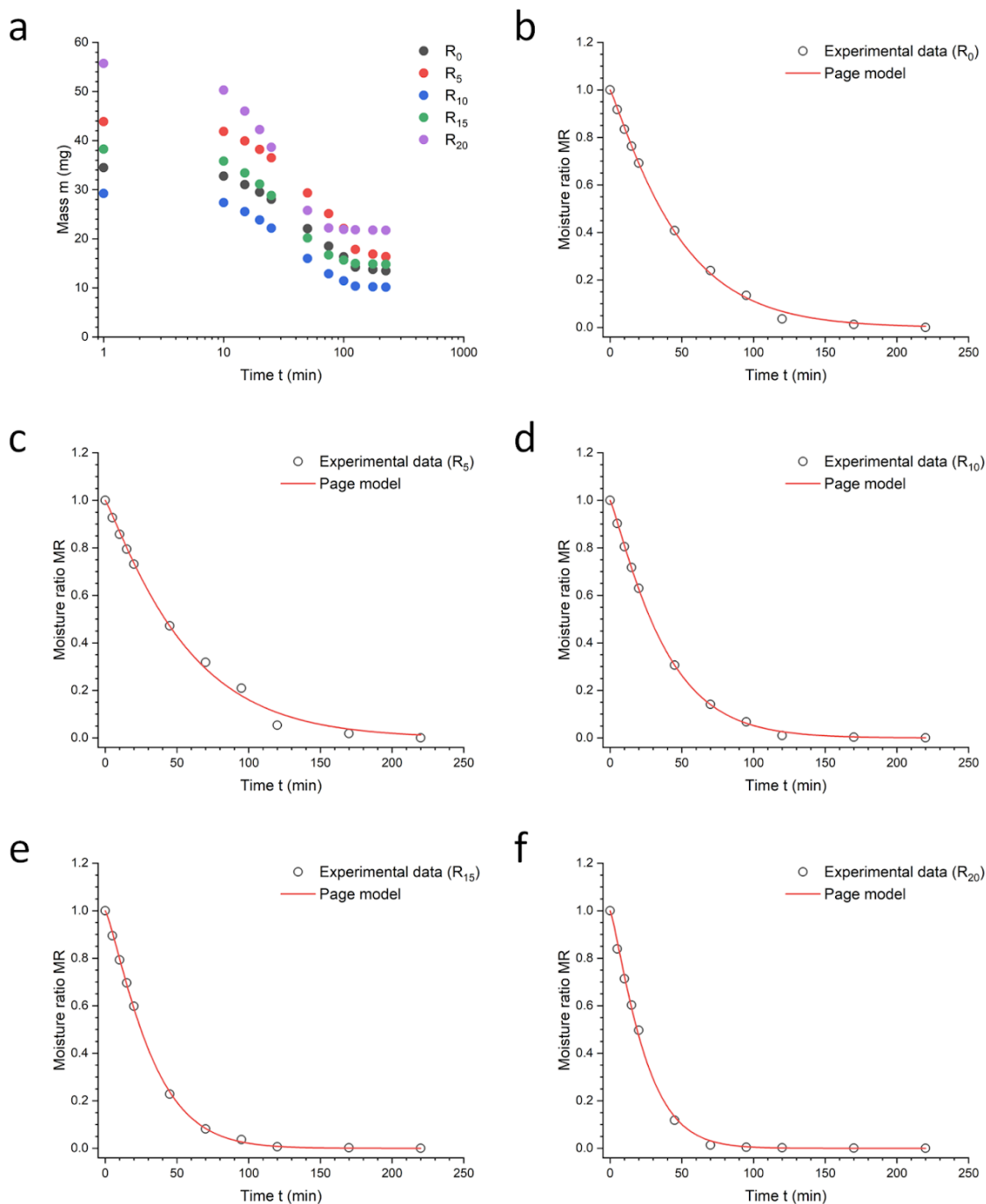


Figure 4. Plots of the mass and moisture ratio of small flax stem samples as a function of time following a simulated 12 h rainfall. (a) The mass of the flax stem samples as a function of drying time. Plots of the moisture ratio versus time for retting stage/length: (b) R₀ (0 days), (c) R₅ (21 days), (d) R₁₀ (35 days), (e) R₁₅ (56 days), and (f) R₂₀ (91 days). The red curves indicate fitting the moisture ratio experimental data using the Page model.

4. Discussion

In terms of flax drying experiments, most studies on flax straw and fibers were conducted at high humidity, whereas the present article considers typical laboratory temperature and humidity (20 °C and 40%) [20,21]. First, the moisture uptake by the stems (long bundles and short single stems) for light and heavy rain can be compared. For long stems in bundles, a value of r equal to 1.7 ± 0.04 and 2.8 ± 0.1 for light and heavy rain were found, respectively. For short flax stem samples, values of r equal to 1.59 ± 0.28

and 2.65 ± 0.14 were found. These values are comparable. The slightly higher values observed for the bundles are likely to be due to capillary bridges between stems. It can be concluded therefore that the moisture uptake is approximately independent of sample length and that any effects related to the ends of cut stems is insignificant for moisture uptake. Next, the drying behavior of the stems (long bundles and short single stems) for light and heavy rain can be compared. The average drying rate constant of long flax stem bundles exposed to light rain is $5.41 \pm 1.37 \times 10^{-3} \text{ min}^{-1}$ ($0.35 \pm 0.03 \text{ h}^{-1}$). The average drying rate constant of short flax stems exposed to light rain is $6.81 \times 10^{-2} \text{ min}^{-1}$ ($2.99 \pm 0.9 \text{ h}^{-1}$). The average drying rate constant of short flax stems exposed to heavy rain is $1.44 \times 10^{-2} \text{ min}^{-1}$ ($1.77 \pm 0.72 \text{ h}^{-1}$). The findings suggest that flax stems exposed to light rain dry faster than flax stems exposed to heavy rain. The results also suggest that short, individual flax stems dry faster than long flax stems in bundles. This can be explained by the stem proximity in the bundles leading to reduced evaporation. In terms of how the stem drying behavior varies with retting stage, there appears to be a trend for short stem samples exposed to light rain where the drying rate constant increases until R_{20} where the outer tissue is very damaged or absent due to over retting [1,3]. For short stem samples exposed to heavy rain, this trend is present but less visible, with the drying coefficient remaining relatively constant. However, interestingly the drying rate constant for the R_{20} (significantly over-retted) samples (light and heavy rain) is comparable, $\sim 2 \times 10^{-2} \text{ min}^{-1}$. This can be explained due to the degradation of the external tissue. The findings of the study can be compared to those of others. Hailemariam and Woldeyes [22] measured the moisture content of manually prepared short (2 cm) green flax stem samples by complete removal of water at high temperature. A moisture content of 11% was found. However, the moisture content was not considered when wetting stems, e.g., rain, or as a function of retting. Nair et al. [9] fitted the Page model to experimental results of drying flax stems in bundles. A drying rate constant k of $2.75 \times 10^{-2} \text{ min}^{-1}$ and an n equal to 1.1 for microwave-assisted drying at $40 \text{ }^\circ\text{C}$ and a drying rate constant k of $1.8 \times 10^{-2} \text{ min}^{-1}$ and a value of n equal to 1.03 for hot air-assisted drying at $40 \text{ }^\circ\text{C}$ was identified. Ghazanfari et al. [23] successfully fitted the Page model to their experimental results of the drying of flax fibers at $30 \text{ }^\circ\text{C}$. A drying rate constant k of 0.35 h^{-1} and n equal to 1.54 was computed. This value is comparable to $k = 0.35 \text{ h}^{-1}$ for flax stem bundles measured here. To help a comparison of the results to those of other studies, the drying rate constants k evaluated from the measurements are also given above in h^{-1} in brackets. As a note of caution, one must take care when converting drying rate constant units, e.g., from min^{-1} to hours^{-1} . This is due to time being raised to a power (n) in the Page model given in Equation (2). The n value must be taken into consideration for each studied sample of the retting stages because the data does not exhibit a linear plot.

5. Conclusions

The study successfully characterized the drying behavior of flax stems at different stages of retting using precise methods that avoided the complications typically associated with large bundles of stems. The Page model accurately fitted all experimental data gathered, both long stem in bundles and short individual stem samples. The modeling enabled the extraction of the drying rate constant k and the dimensionless drying factor n . These parameters can be compared for different samples, following different rain conditions, and as a function of retting stage. The findings show that the drying rate constant of flax stem samples is smaller for light rain compared to heavy rain, and the over-retted stem samples dry more rapidly compared to under-retted samples due to the degradation of the external tissue. In addition, the results highlighted significant differences in water uptake

following light and heavy rainfall, which has implications for agricultural practice, either traditional or using new emerging sensor technologies.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/agriculture15040395/s1>, Figure S1: Shows the weather data for Killem, France during the period of 6th July to the 1st October. The retting period (as defined by VRF) was from 6th July to the 31st August. The optimum retting day (29 August) is defined by the orange colour. The over retting period was from the latter date to the 1st of October. (a) Shows the data of air temperature, (b) Rainfall (precipitation), (c) Wind speed, and (d) The direct sunlight (no cloud cover). Data available on: <https://www.infoclimat.fr/climatologie-mensuelle/000R5/juillet/2022/bergues.html>.

Author Contributions: A.R.: Conceptualization, methodology, validation, investigation, data curation, writing—original draft, writing—review and editing, visualization. L.B.: Writing—review and editing, supervision, project administration, funding acquisition. S.A.: Conceptualization, methodology, validation, investigation, writing—original draft, writing—review and editing, supervision, project administration. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Data underlying the results presented in this article may be obtained from the authors upon reasonable request.

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Conflicts of Interest: The authors declare no conflicts of interest.

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