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Influence of Torrefaction Temperature and Climatic Chamber Operation Time on Hydrophobic Properties of Agri-Food Biomass Investigated Using the EMC Method

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Abstract: Due to the tendency for excessive moisture adsorption by raw, unprocessed biomass, various methods of biomass valorization are in use, allowing for the improvement of physical–chemical biomass properties, including hydrophobicity. One of the methods is torrefaction, which changes the hydrophilic properties of the biomass to hydrophobic. Therefore, in this study, the influence of the torrefaction temperature and the exposure time to moisture adsorption conditions on the hydrophobic properties of waste biomass from the agri-food industry (lemon peel, mandarin peel, grapefruit peel, and butternut-squash peel) were analyzed. The torrefaction was carried out at the following temperatures: 200, 220, 240, 260, 280, 300, and 320 °C. The hydrophobic properties were determined by using the EMC (Equilibrium Moisture Content) method, conducting an experiment in the climatic chamber at atmospheric pressure, a temperature of 25 °C, and relative humidity of 80%. The total residence time of the material in the climate chamber was 24 h. It was shown that the torrefaction process significantly improves the hydrophobic properties of waste biomass. Concerning dried raw (unprocessed) material, the EMC (24 h) coefficient was 0.202 ± 0.004 for lemon peels, 0.223 ± 0.001 for grapefruit peels, 0.237 ± 0.004 for mandarin peels, and 0.232 ± 0.004 for butternut squash, respectively. After the torrefaction process, the EMC value decreased by 24.14–56.96% in relation to the dried raw material, depending on the type of organic waste. However, no correlation between the improvement of hydrophobic properties and increasing the torrefaction temperature was observed. The lowest values of the EMC coefficient were determined for the temperatures of 260 °C (for lemon peel, EMC = 0.108 ± 0.001; for mandarin peel, EMC = 0.102 ± 0.001), 240 °C (for butternut-squash peel, EMC = 0.176 ± 0.002), and 220 °C (for grapefruit peel, EMC = 0.114 ± 0.008). The experiment also showed a significant logarithmic trend in the dependence of the EMC coefficient on the operating time of the climatic chamber. It suggests that there is a limit of water adsorption by the material and that a further increase of the exposure time does not change this balance.

Keywords: agri-food residues; torrefaction; moisture adsorption; hydrophobicity; waste biomass valorization

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1. Introduction

The need to limit the use of fossil fuels, dictated by the prospect of their exhaustion, and the growing challenges related to the implementation of a zero-emission economy leave us looking for alternative solutions to meet environmental problems, while also ensuring the demand for electricity and heat [1]. One of the prospective sources of energy that can meet these expectations is waste biomass, which includes, among other things, agri-food residues, wood industry residues (logging and processing), residues from urban green areas, or roadside vegetation [2]. It is estimated that the annual potential of waste biomass is 140 Gt [3,4], and the problem of its management related to such a high biomass stream means that the energetic use of this raw material may turn out to be an interesting choice.

The use of waste biomass, compared to traditional agricultural and forest industry raw materials, brings many benefits. First of all, they are related to the limitation of logging. Despite the implementation of increasingly restrictive forest management rules and strict harvesting requirements [5], the production of biofuels can significantly affect the deforestation of large areas of land, especially in developing countries [6,7]. In addition, increasing the degree of use of waste biomass does not adversely affect the competition for soil resources where the cultivation of edible food for humans or farm animals is carried out [8]. Currently, about 13% of the world's land is used for the production of biofuels and textiles [9]. Hence, strategies for more efficient management and use of biomass, especially with regard to waste biomass, can soften this competition for soil resources, thus avoiding increasing pressures on natural resources and ecosystems [10–12]. The reuse of the waste biomass from the food-processing sector helps not only with the energy crisis; it also allows for solving the problems with waste landfilling and the emissions of greenhouse gases, and improving the environment through the ecological balance. Their usage allows for the implementation of the circular economy. Food-waste biomass is an alternative source of energy with the possibility of application in co-firing [13,14]. The residues, such as straw, hay, and wooden biomass, in a few years, cannot be enough to meet the energy needs of the energy mixes. Therefore, nowadays, it is highly recommended to use the food-waste biomass for the transformation of the challenges related to climate and the environment [15]. It is estimated by the European Commission that the annual food-waste production is about 88 million tons [16]. Therefore, it is important to analyze the new alternative possibilities of biomass to be ready for industrial implementation.

Unfortunately, due to its unfavorable physical and chemical parameters, biomass waste requires valorization. This is because raw waste biomass is often characterized by, among other things, low bulk density [17], low energy concentration per volume unit [18], unfavorable grinding properties, the need for high energy inputs [19], or high moisture content [20]. Finally, an important parameter from the point of view of harvesting, handling, transport, and storage of biomass is its high ability to adsorb moisture and degradation [21].

Currently, the literature distinguishes several ways of testing the ability of biomass to adsorb moisture, the most popular of which are the immersion method, the contact angle test, the water drop penetration time (WDPT) test, and the EMC (Equilibrium Moisture Content) method. When using the immersion method, the material is immersed in deionized water at a constant temperature. After the specified time, the amount of water adsorbed by the test sample is measured [22]. The measurement of the contact angle consists in determining, using specialized equipment, the angle formed by the tangent to the surface of the measuring drop deposited on the surface of a solid body at the point of contact of three phases: solid, liquid, and gas. Depending on the value of the contact angle, the ability of the substance to adsorb moisture is determined [23]. The WDPT method consists of placing a drop of water on the surface of the dried material and measuring the time needed for it to be adsorbed into the substance. The droplet infiltration time reflects the time it takes for the surface tension to become higher than that of the droplet [24]. Still, most attention is paid to the method in which the EMC coefficient is determined when testing the moisture adsorption capacity; the EMC coefficient is the value of the relative humidity obtained in the given conditions of temperature, pressure, and humidity. The tests are carried out in climatic chambers, allowing us to set and control the desired process conditions in a limited space. Measurements of the mass change (resulting from the adsorption of moisture from the environment by the material) are made at specified intervals.

Depending on the material's susceptibility to moisture adsorption, it is referred to as hydrophilic (susceptible to moisture adsorption) or hydrophobic (not susceptible to moisture adsorption). Unfortunately, raw, unprocessed biomass possesses hydrophilic properties and is therefore susceptible to the effects of weather conditions [25]. In addition, the organic matter in biomass can be quickly decomposed, and its storage in inappropriate conditions may result in the decomposition of the material, related to biological degradation, due to the activity of microorganisms [26,27]. For this reason, methods of valorization of waste biomass are sought that will reduce the biomass ability to adsorb moisture from the environment, while improving the physical–chemical properties of the biomass.

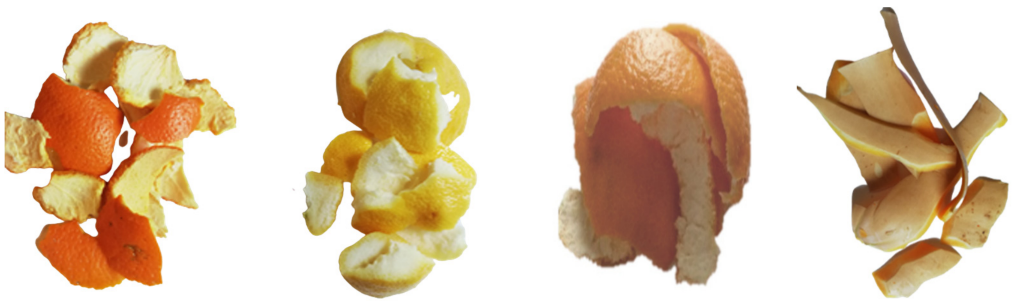
One of the promising methods of biomass valorization is torrefaction, a thermochemical process consisting of mild thermal treatment, occurring typically between 200 and 320 °C, at atmospheric pressure, under inert gas flow for several tens of minutes [28]. Torrefaction, in addition to improving properties such as high moisture content, low energy density, and poor flowability, also makes the biomass hydrophobic [29]. So far, studies carried out by many authors have shown that, as a result of the torrefaction process, a significant improvement in the hydrophobic properties of the biomass has been achieved. These results were confirmed, inter alia, by Baronti et al. [30], using the WDPT method; Alvarez et al. [31], using the contact angle test method; or Yan et al. [32], using the EMC method.

It should be mentioned that, during the estimation of the EMC coefficient, the experiment is carried out for variable humidity, thanks to which moisture sorption isotherms are obtained that are used to describe the relationship between water content and equilibrium humidity at equilibrium state [33] by applying different equations [21]. However, little attention is paid to the determination of this parameter, analyzing the time significance when conducting the experiment under conditions of constant humidity. The exposure time of the material to adsorb moisture can be important in this subject. Moreover, there is not too much data in regard to what extent the time significance influences the hydrophobicity of biomass materials subjected to the torrefaction process. Taking these arguments into account (applying the EMC method), this work aimed (i) to evaluate the effect of the torrefaction temperature of waste biomass on the water adsorption propensities, (ii) to investigate the influence of the torrefied material exposure time under test conditions on the degree of water adsorption, and (iii) to determine the Equilibrium Moisture Content Index.

2. Materials and Methods

2.1. Materials

In the research, the waste biomass from the agri-food industry were used. Four types of organic residues were evaluated (Figure 1): mandarin peel, lemon peel, grapefruit peel, and butternut-squash peel.



(**a**) (**b**) (**c**) (**d**)

Figure 1. Biomass waste used in the experiment: (**a**) mandarin peel, (**b**) lemon peel, (**c**) grapefruit peel, and (**d**) butternut-squash peel.

2.2. Samples Preparation and Torrefaction Procedure

Before the torrefaction process, all materials were dried for 24 h, at 105 °C, in a Drying Chamber KBC–65 W (WAMED, Warszawa, Poland), in order to obtain the same analytical state. After the drying process was completed, the materials were crushed in an LMN 400 (TESTCHEM, Pszów, Poland) knife mill with a sieve size of 1 mm. For the thermal conversion, the shredded organic waste in the amount of 50 g was put into the muffle furnace SNOL 8.2/1100 (SNOL, Utena, Lithuania). The mass of the samples was determined by using the RADWAG AS 220.R2 (RADWAG, Radom, Poland) scale. The torrefaction of the waste biomass was carried out in the commonly used conditions at the following temperatures: 200, 220, 240, 260, 280, 300, and 320 °C, for a period of 60 min [34,35]. Carbon dioxide from the gas cylinder was used to maintain an inert atmosphere in the reaction chamber.

2.3. Physical Properties Analysis

To characterize the basic properties of the alternative fuel from waste biomass, a proximate analysis was carried out. The analysis included the determination of moisture content (MC), ash content (AC), volatile matter content (VMC), fixed carbon content (FCC), and the higher heating value (HHV). The analysis was carried out according to the standards (Table 1). Each measurement was repeated three times, and the statistical analysis was applied.

Table 1. Standards and methods used during the proximate analysis.

Parameter	Standard/Method
Moisture content (MC)	PN EN ISO 18134-2:2017-03E
Higher heating value (HHV)	PN EN ISO 18125:2017-07
Volatile matter content (VMC)	PN EN ISO 18123:2016-01
Ash content (AC)	PN EN ISO 18122:2015
Fixed carbon content (FCC)	ASTM D-3172-73

2.4. Hydrophobic Properties Analysis

The evaluation of the hydrophobic properties was performed applying the Equilibrium Moisture Content (EMC) method [36]. In this method, the ability of the material to perform water adsorption (in the defined/controlled conditions of the environment) is measured. The less water is adsorbed by the tested material the more hydrophobic it is. Thus, in terms of hydrophobic properties, the lower value of the EMC coefficient is, the better. The Equilibrium Moisture Content (EMC) assay was calculated by using the following formula:

$$EMC_p = \frac{m_p}{m_t} \quad (1)$$

where EMC_p is the Equilibrium Moisture Content (EMC_p); m_p is an increase in mass of the sample compared to the initial state (after the specified time of climatic chamber operation time) (kg), and m_t is the initial mass of the sample in the dry state (kg).

The determination of moisture adsorption degree of the tested materials was performed in the Climatic Chamber WK111 340 (WEISS Technik, Liedekerke, Belgium). The technical data of the climatic chamber are presented in Table 2. The test conditions were set at 25 °C, 80% relative humidity, and normal pressure. Before starting the experiment, 1 g of each investigated material was placed in ceramic crucibles and dried in a laboratory drier for 24 h, at the temperature of 105 °C. Then, the crucibles were covered with lids and placed in a desiccator to minimize environmental influences on the sample. Finally, the weighed crucibles with the material (without lids) were placed in the climatic chamber, and the mass increase was measured at appropriate time intervals. Measurements were made at the following time intervals: 4 measurements every 15 min, 4 measurements every 30 min, 5 measurements every one hour, and one measurement after 24 h from the beginning of the tests. Each measurement was repeated three times.

Table 2. Technical parameters of Climatic Chamber WK111 340.

Parameter	Value
Volume	335 dm^3
Temperature range	−10 °C/+90 °C
0Electric parameters	3/-/PE, AC 220 V ± 10%, 60 Hz
Cooling	R134 A, 0.6 kg
HP max.	25 bar
Nominal output power	1.7 kW
Nominal current	10 A
Weight	410 kg
Dimensions	780 mm × 1775 mm × 1480 mm

2.5. Logarithmic Function Fitting (EMC Kinetics)

Due to the tendency of the EMC coefficient to the equilibrium value (using the Solver add-in), the values of the coefficients of determination (R^2) and the coefficients a and b were determined in accordance with the logarithmic trend line expressed by the following formula:

$$EMC_p = a \cdot ln(CCOT) + b \quad (2)$$

where $CCOT$ is the climatic chamber operation time (h), and a and b are constant values of functions (-).

2.6. Statistical Analysis

The obtained research data were analyzed by descriptive statistics, taking into account the mean values and standard deviation for all tests. Moreover, a statistical analysis (at p-value < 0.05) involving a two-way analysis of variance (ANOVA) was performed. The test was focused on the elaboration of statistical significance of the influence of the torrefaction temperature and the climatic chamber operation time on the hydrophobic properties of the materials under analysis. Additionally, for the considered materials, the interaction between these parameters was investigated as well. The differences between the levels of factors were determined by a post hoc test, using a Tukey (HSD) test. The statistical analysis was developed in statistical software STATISTICA (StatSoft—DELL Software, TX, USA).

3. Results and Discussion

3.1. Proximate Analysis of the Torrefied Waste Biomass

The analysis of the moisture content in the tested materials did not show any dependencies between materials and temperature. The torrefied waste biomass was characterized by moisture content from 1 to 10%.

When analyzing the ash content (Figure 2) in the torrefied fruit waste biomass, we observed the increase of AC as the torrefaction temperature increased. The average ash content in the mandarin, lemon, and grapefruit peels ranged from 4 to 12% with increasing torrefaction temperature. The highest content of the ash was observed for butternut-squash peels and ranged from 8 to 20%. Similar changes of ash content in waste biomass after the torrefaction process were observed for other organic materials [37,38].

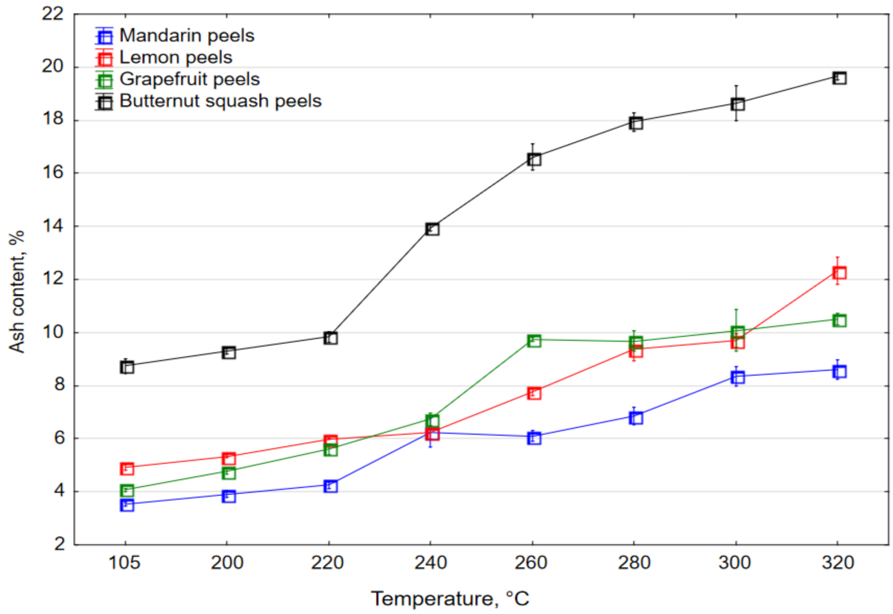


Figure 2. Ash content in the torrefied waste biomass.

During the torrefaction process, a significant amount of volatile matter devolatilized from the waste biomass. As the torrefaction temperature increases, the volatile matter loss is greater (Figure 3). For the untreated waste biomass, the VMC was approximately 80%. However, for the torrefied biomass at 320 °C, the VMC was ca. 40%. No significant differences in VMC between the materials were observed.

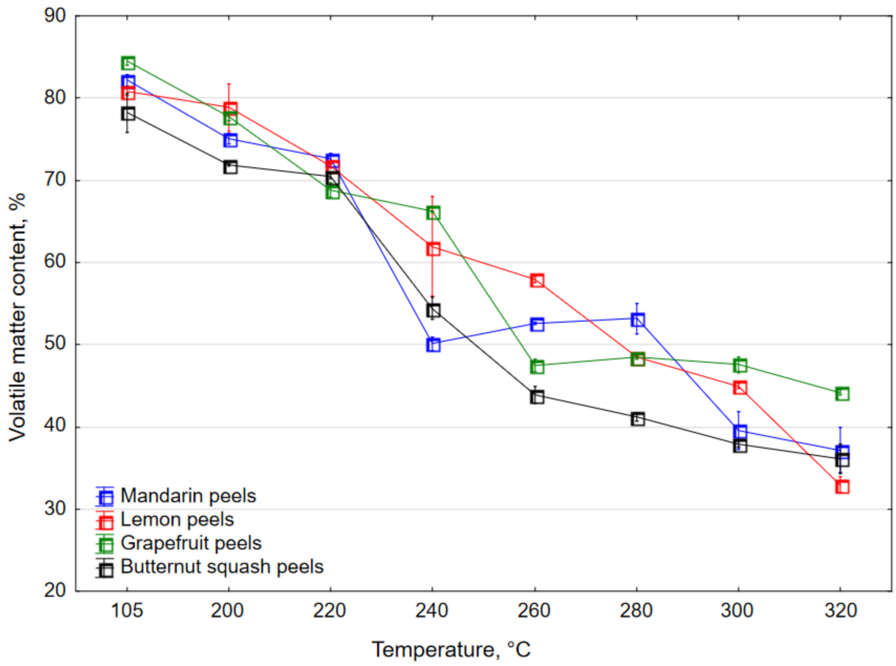


Figure 3. Volatile matter content in the torrefied waste biomass.

One of the basic fuel properties is the content of the fixed carbon, which indicates the carbonization rate of the material and affects the caloric value. The fixed carbon content was calculated as the difference between unity and MC, AC, and VMC. For the tested materials, it was from ca. 10% (for dried raw material) up to 50% (for torrefied biomass at 320 °C) (Figure 4).

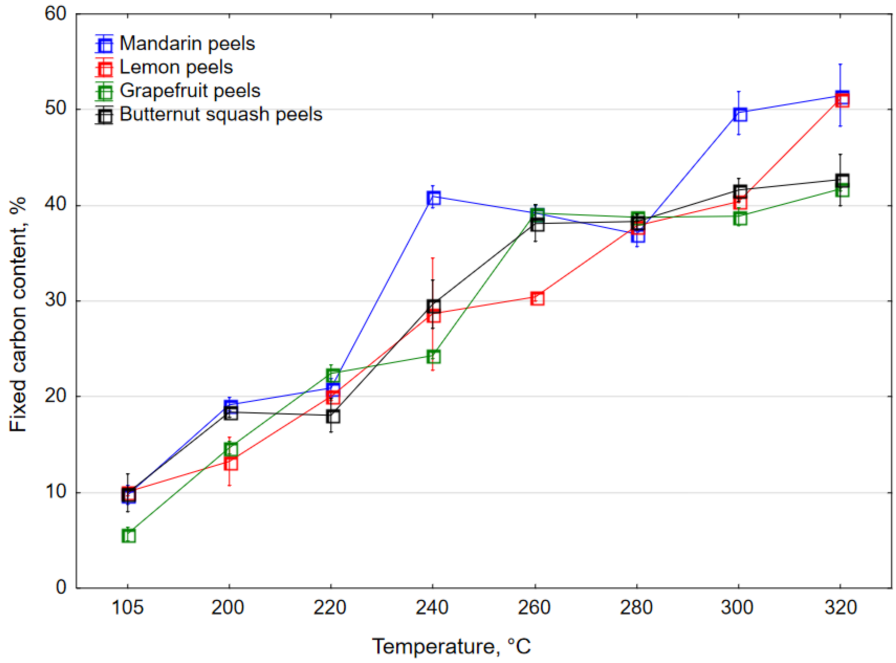


Figure 4. Fixed carbon content in the torrefied waste biomass.

Considering the heating value of the dried raw and torrefied waste biomass, the HHV of the untreated waste biomass was similar to the other biomasses, such as straw or hay, and it was approximately 16 MJ·kg^{-1} (Figure 5). As the torrefaction temperature increased, the HHV also increased, and for the biomass torrefied at 320 °C, it was approximately 26 MJ·kg^{-1}, which can be comprised of the HHV of the coal [39].

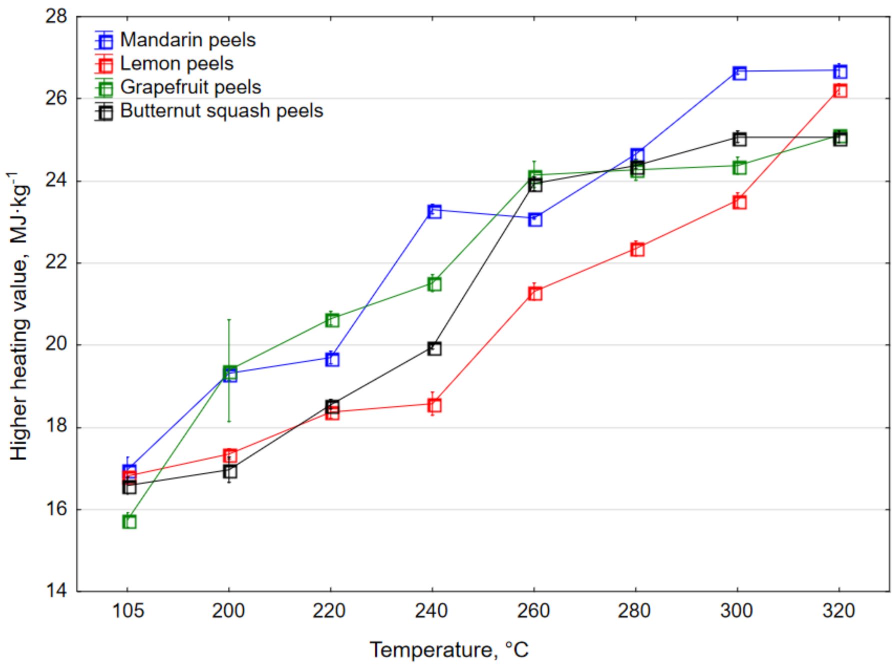


Figure 5. Higher heating value of the torrefied waste biomass.

The details of the proximate analysis for the investigated materials are shown in Supplementary Materials Table S1.

3.2. *Impact of the Torrefaction Temperature and Climatic Chamber Operation Time on Hydrophobic Properties*

Tables 3–6 show the influence of the climatic chamber operation time and the torrefaction temperature on the value of the EMC coefficient for the organic waste used in the tests.

Table 3. Influence of the climatic chamber operation time (CCOT) and the torrefaction temperature on the value of the EMC coefficient for lemon peel.

	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
0.25 h	0.026	0.022	0.022	0.020	0.025	0.024	0.021	0.020
SD	0.003	0.001	0.005	0.003	0.002	0.006	0.001	0.003
0.5 h	0.042	0.034	0.035	0.031	0.038	0.038	0.034	0.032
SD	0.005	0.001	0.005	0.004	0.002	0.008	0.001	0.003
0.75 h	0.054	0.043	0.043	0.038	0.047	0.048	0.043	0.041
SD	0.006	0.001	0.005	0.004	0.003	0.008	0.001	0.004
1 h	0.067	0.052	0.052	0.046	0.054	0.056	0.052	0.049
SD	0.007	0.002	0.005	0.005	0.002	0.009	0.001	0.005
1.5 h	0.082	0.064	0.064	0.055	0.064	0.067	0.064	0.061
SD	0.008	0.002	0.005	0.005	0.002	0.007	0.002	0.005
2 h	0.095	0.075	0.074	0.063	0.07	0.075	0.072	0.069
SD	0.009	0.002	0.005	0.006	0.002	0.006	0.002	0.006
2.5 h	0.105	0.083	0.081	0.068	0.075	0.080	0.079	0.076
SD	0.010	0.002	0.005	0.006	0.002	0.005	0.002	0.005
3 h	0.114	0.090	0.088	0.074	0.078	0.085	0.085	0.082
SD	0.010	0.002	0.005	0.006	0.002	0.004	0.002	0.005
4 h	0.129	0.102	0.098	0.082	0.084	0.09	0.091	0.089
SD	0.010	0.002	0.005	0.005	0.002	0.002	0.001	0.005
5 h	0.141	0.112	0.107	0.089	0.088	0.095	0.096	0.094
SD	0.011	0.002	0.005	0.006	0.002	0.002	0.001	0.004
6 h	0.150	0.120	0.113	0.094	0.091	0.097	0.099	0.098
SD	0.011	0.002	0.006	0.005	0.002	0.001	0.001	0.004
7 h	0.158	0.126	0.118	0.099	0.094	0.100	0.102	0.101
SD	0.010	0.002	0.006	0.005	0.002	0.001	0.001	0.003
8 h	0.164	0.132	0.122	0.103	0.096	0.101	0.104	0.103
SD	0.010	0.002	0.006	0.005	0.001	0.001	0.001	0.003
24 h	0.202	0.164	0.145	0.128	0.108	0.113	0.117	0.118
SD	0.004	0.001	0.006	0.002	0.001	0.003	0.001	0.002

Table 4. Influence of the climatic chamber operation time (CCOT) and the torrefaction temperature on the value of the EMC coefficient for grapefruit peel.

	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
0.25 h	0.035	0.030	0.033	0.029	0.036	0.035	0.035	0.039
SD	0.001	0.002	0.003	0.001	0.003	0.002	0.003	0.002
0.5 h	0.055	0.046	0.049	0.045	0.053	0.050	0.051	0.058
SD	0.001	0.002	0.003	0.002	0.004	0.004	0.006	0.002
0.75 h	0.072	0.058	0.059	0.055	0.066	0.062	0.063	0.071
SD	0.002	0.003	0.004	0.002	0.005	0.004	0.006	0.002
1 h	0.085	0.067	0.068	0.063	0.073	0.070	0.071	0.080
SD	0.001	0.005	0.004	0.002	0.005	0.004	0.006	0.003
1.5 h	0.105	0.082	0.077	0.072	0.085	0.081	0.081	0.092
SD	0.002	0.003	0.004	0.002	0.005	0.005	0.008	0.002
2 h	0.122	0.094	0.084	0.079	0.091	0.087	0.087	0.099
SD	0.002	0.003	0.004	0.002	0.004	0.005	0.008	0.002

Table 4. *Cont.*

	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
2.5 h	0.134	0.102	0.089	0.084	0.096	0.091	0.093	0.104
SD	0.002	0.003	0.005	0.002	0.003	0.005	0.007	0.002
3 h	0.144	0.109	0.093	0.088	0.100	0.095	0.095	0.108
SD	0.002	0.003	0.004	0.001	0.003	0.005	0.011	0.001
4 h	0.160	0.120	0.099	0.094	0.106	0.100	0.100	0.113
SD	0.002	0.003	0.004	0.001	0.003	0.006	0.010	0.001
5 h	0.173	0.129	0.101	0.098	0.110	0.103	0.104	0.117
SD	0.001	0.003	0.008	0.002	0.003	0.007	0.011	0.001
6 h	0.183	0.136	0.104	0.098	0.113	0.106	0.106	0.119
SD	0.001	0.003	0.008	0.006	0.002	0.007	0.011	0.001
7 h	0.191	0.141	0.104	0.103	0.114	0.105	0.108	0.120
SD	0.001	0.003	0.009	0.002	0.003	0.012	0.011	0.001
8 h	0.197	0.145	0.105	0.105	0.116	0.106	0.110	0.122
SD	0.001	0.003	0.009	0.002	0.002	0.012	0.012	0.001
24 h	0.223	0.162	0.114	0.115	0.124	0.115	0.120	0.130
SD	0.001	0.002	0.008	0.003	0.002	0.012	0.012	0.001

Table 5. Influence of the climatic chamber operation time and the torrefaction temperature on the value of the EMC coefficient for mandarin peel.

	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
0.25 h	0.032	0.032	0.031	0.054	0.03	0.031	0.032	0.036
SD	0.002	0.001	0.002	0.031	0.001	0.002	0.002	0.001
0.5 h	0.054	0.05	0.048	0.071	0.045	0.047	0.049	0.055
SD	0.002	0.002	0.001	0.030	0.001	0.003	0.003	0.001
0.75 h	0.069	0.061	0.059	0.083	0.055	0.059	0.06	0.069
SD	0.002	0.002	0.001	0.030	0.001	0.003	0.003	0.001
1 h	0.083	0.071	0.068	0.091	0.062	0.067	0.068	0.077
SD	0.002	0.002	0.001	0.031	0.001	0.002	0.003	0.001
1.5 h	0.102	0.084	0.081	0.102	0.072	0.078	0.078	0.089
SD	0.003	0.002	0.002	0.031	0.001	0.003	0.002	0.002
2 h	0.118	0.095	0.09	0.108	0.078	0.084	0.085	0.096
SD	0.003	0.001	0.002	0.032	0.001	0.003	0.001	0.001
2.5 h	0.132	0.103	0.098	0.113	0.082	0.089	0.089	0.100
SD	0.004	0.001	0.002	0.032	0.001	0.003	0.001	0.002
3 h	0.143	0.11	0.104	0.116	0.085	0.092	0.092	0.103
SD	0.004	0.001	0.003	0.032	0.001	0.003	0.001	0.002
4 h	0.161	0.119	0.112	0.121	0.089	0.096	0.096	0.108
SD	0.003	0.002	0.002	0.033	0.001	0.003	0.001	0.002
5 h	0.174	0.126	0.119	0.123	0.091	0.099	0.098	0.11
SD	0.003	0.004	0.002	0.033	0.001	0.003	0.001	0.002
6 h	0.184	0.132	0.124	0.125	0.093	0.100	0.100	0.112
SD	0.002	0.004	0.002	0.033	0.001	0.002	0.001	0.003
7 h	0.193	0.137	0.127	0.127	0.094	0.101	0.101	0.113
SD	0.001	0.003	0.002	0.032	0.001	0.001	0.001	0.003
8 h	0.200	0.141	0.131	0.128	0.095	0.101	0.102	0.114
SD	0.001	0.003	0.002	0.032	0.001	0.001	0.001	0.003
24 h	0.237	0.156	0.145	0.134	0.102	0.107	0.108	0.120
SD	0.004	0.013	0.002	0.034	0.001	0.003	0.001	0.004

Table 6. Influence of the climatic chamber operation time (CCOT) and the torrefaction temperature on the value of the EMC coefficient for butternut-squash peel.

	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
0.25 h	0.022	0.023	0.023	0.030	0.032	0.031	0.036	0.032
SD	0.002	0.002	0.003	0.001	0.001	0.002	0.001	0.005
0.5 h	0.036	0.037	0.037	0.046	0.050	0.049	0.055	0.049
SD	0.002	0.003	0.004	0.001	0.002	0.004	0.001	0.007
0.75 h	0.048	0.049	0.049	0.060	0.064	0.063	0.071	0.062
SD	0.003	0.004	0.004	0.002	0.002	0.005	0.002	0.008
1 h	0.058	0.058	0.058	0.069	0.073	0.073	0.082	0.074
SD	0.003	0.004	0.005	0.002	0.001	0.005	0.004	0.009
1.5 h	0.076	0.075	0.073	0.085	0.089	0.085	0.099	0.089
SD	0.004	0.004	0.006	0.002	0.002	0.005	0.004	0.010
2 h	0.09	0.088	0.085	0.096	0.099	0.099	0.110	0.099
SD	0.005	0.005	0.006	0.002	0.001	0.006	0.003	0.010
2.5 h	0.103	0.099	0.095	0.106	0.108	0.108	0.118	0.108
SD	0.005	0.005	0.007	0.002	0.001	0.005	0.003	0.009
3 h	0.113	0.108	0.103	0.113	0.115	0.114	0.124	0.115
SD	0.006	0.005	0.007	0.001	0.001	0.005	0.003	0.009
4 h	0.130	0.122	0.115	0.124	0.124	0.125	0.134	0.124
SD	0.006	0.005	0.008	0.002	0.001	0.005	0.003	0.008
5 h	0.145	0.133	0.126	0.132	0.132	0.132	0.141	0.131
SD	0.007	0.006	0.007	0.002	0.001	0.004	0.003	0.008
6 h	0.152	0.140	0.134	0.138	0.138	0.138	0.147	0.137
SD	0.009	0.006	0.007	0.002	0.001	0.004	0.003	0.007
7 h	0.166	0.149	0.140	0.144	0.143	0.143	0.152	0.143
SD	0.007	0.005	0.007	0.002	0.001	0.004	0.003	0.007
8 h	0.175	0.155	0.146	0.148	0.147	0.147	0.156	0.147
SD	0.006	0.005	0.006	0.002	0.001	0.004	0.003	0.007
24 h	0.232	0.19	0.178	0.176	0.177	0.177	0.184	0.178
SD	0.004	0.002	0.003	0.002	0.001	0.003	0.003	0.004

In the case of lemon peel, after the first measurement (CCOT = 15 min), the highest moisture increase was observed for the material dried at 105 °C, non-torrefied (EMC = 0.026 $\pm$ 0.003). Slightly lower values were observed for the torrefied material in 260 °C (EMC = 0.025 $\pm$ 0.002) and 280 °C (EMC = 0.024 $\pm$ 0.006). The highest hydrophobicity was observed for materials torrefied at 320 and 240 °C (EMC = 0.02 $\pm$ 0.003). Thus, it can be seen that the EMC coefficient values after the first measurement cycle did not differ significantly from each other. However, along with the extension of the climatic chamber operation time, higher differences in the EMC coefficient were noted between the torrefied and non-torrefied material. After the last measurement cycle (CCOT = 24 h), the increase in moisture of the dried lemon peel was at the level of EMC = 0.202 $\pm$ 0.004. The material torrefied at 260 °C (EMC = 0.108 $\pm$ 0.001) was characterized by the highest hydrophobicity, achieving nearly half the value of moisture increase. Slightly higher increases were also recorded for torrefied materials at 280 °C (EMC = 0.113 $\pm$ 0.003), 300 °C (EMC = 0.117 $\pm$ 0.001), and 320 °C (EMC = 0.118 $\pm$ 0.002). Slightly greater increases were seen in torrefied lemon peel at 200 °C (EMC = 0.164 $\pm$ 0.001), 220 °C (EMC = 0.145 $\pm$ 0.006) and 240 °C (EMC = 0.128 $\pm$ 0.002).

A slightly different dependence of the initial increase in moisture content was observed for grapefruit peel, where the highest increase (EMC = 0.039 $\pm$ 0.002) was indicated in the material torrefied at the highest temperature (320 °C). The value higher or equal to that for the non-torrefied material (EMC = 0.035 $\pm$ 0.001) was also noted in three other case: for the torrefaction temperature of 260 °C (EMC = 0.036 $\pm$ 0.003), 280 °C (EMC = 0.035 $\pm$ 0.003), and 300 °C (EMC = 0.035 $\pm$ 0.003)). In other cases, (torrefaction temperature 200 °C–240 °C) the EMC coefficient value was in the range of 0.029–0.033. However, as in the case of lemon peel, as the climatic chamber operation time was extended, non-torrefied material absorbed more and more water, while the water adsorption by torrefied materials deteriorated. The dried grapefruit peel after the last measurement cycle (CCOT = 24 h) was characterized by the EMC index = 0.223 ± 0.001. A much greater hydrophobicity was observed for materials torrefied at 220 °C (EMC = 0.114 ± 0.008), 240 °C (EMC = 0.115 ± 0.003), and 280 °C (EMC = 0.115 ± 0.012). Slightly larger increases were recorded for torrefaction at 300 °C (EMC = 0.120 ± 0.012), 320 °C (EMC = 0.130 ± 0.001), and 200 °C (EMC = 0.162 ± 0.002).

The initial level of moisture adsorption for mandarin peels was similar to that of lemon and grapefruit peels. Only in one case—for the material torrefied at 240 °C—a much higher value of moisture adsorption (EMC = 0.054 ± 0.031) was recorded than for other temperatures. The non-torrefied material dried at 105 °C was characterized by EMC = 0.032 ± 0.002. Similar values of the index were also obtained by torrefied materials (EMC varied from 0.030 to 0.036). Extension of the CCOT resulted in higher differences in hydrophobicity between non-torrefied and torrefied material. The mandarin peel dried at 105 °C, after the last measurement cycle (CCOT = 24 h), was characterized by the EMC coefficient at the level of 0.237 ± 0.004. The highest hydrophobicity was observed for materials torrefied at 260 °C (EMC = 0.102 ± 0.001), 280 °C (EMC = 0.107 ± 0.003), and 300 °C (EMC = 0.108 ± 0.001), where the moisture adsorption decreased by more than 50%, as compared to non-torrefied material. Materials torrefied at other temperatures were also characterized by much lower moisture adsorption coefficients: for 320 °C, the EMC = 0.12 ± 0.004; for 240 °C, the EMC = 0.134 ± 0.034; for 220 °C, the EMC = 0.145 ± 0.002; and for 200 °C, the EMC = 0.156 ± 0.013.

In the case of testing the increase in moisture adsorption after the first measurement cycle (CCOT = 0.25 h) for butternut-squash peel, a slightly different situation than for other organic waste was observed. The dried material adsorbed the least amount of moisture and was characterized by the EMC coefficient at the level of 0.022 ± 0.002, while the highest moisture adsorption capacity was noted for the material torrefied at 300 °C (EMC = 0.036 ± 0.001). High increases in humidity also applied to the temperatures of 320 °C (EMC = 0.032 ± 0.005), 260 °C (EMC = 0.032 ± 0.001), 280 °C (EMC = 0.031 ± 0.002), and 240 °C (EMC = 0.030 ± 0.001). Slightly higher increases in the amount of water concerned materials torrefied at the two lowest temperatures: 200 °C (EMC = 0.023 ± 0.002) and 220 °C (EMC = 0.023 ± 0.002). As the operating time of the climatic chamber was extended, more and more moisture was detected for the non-torrefied material, while the torrefied materials were characterized by a more hydrophobic structure. After the last measurement cycle (CCOT = 24 h), the dried butternut-squash peel was characterized by the value of the coefficient EMC = 0.232 ± 0.004. For materials torrefied at temperatures of 220, 240, 260, 280, and 320 °C, a much smaller increase in moisture adsorption was noted (EMC = 0.176 − 0.178 ± 0.002). A slightly greater increase was recorded at the temperature of 300 °C (EMC = 0.184 ± 0.003) and 200 °C (EMC = 0.190 ± 0.002). Therefore, it should be noted that the butternut-squash peel was characterized by smaller differences between the hydrophobic properties in relation to the torrefied and non-torrefied material, as compared to other organic waste.

The research showed that non-torrefied materials were characterized by a much higher ability to adsorb moisture from the environment. Meanwhile, the torrefied waste biomass obtained hydrophobic properties, which significantly increased the resistance of materials to water adsorption. However, it should be marked that the most important factor is to perform the torrefaction process, as it causes significant changes in the abilities of water adsorption. The temperature of the torrefaction, in fact, is not as critical as the performance of the thermal treatment of the waste biomass (torrefaction) itself. The differences caused by the temperature (in the examined range) in the resistance to water adsorption are smaller. Moreover, the results clearly indicated that the increase of the torrefaction temperature is not always in line with the best hydrophobic propensities. It depends on the type of biomass and probably on its many other properties.

In terms of the CCOT, the initial water adsorption by all the samples results rather in surface wetting. The water, in the case of torrefied biomass, does not bond internally with a material. The results should be considered as consistent with other literature reported. In the previous work [37], by performing an alternative test (the Water Drop Penetration Time—WDPT), it was shown that the torrefaction of materials changes the hydrophilic properties of biomass to extremely hydrophobic, preventing water droplets from penetrating the surface of the material. The experiment carried out in a climatic chamber confirms these results. After the last measurement cycle, the increase in the moisture content (water adsorption) of the torrefied material, as compared to the non-torrefied material, was lower at 46.53% (for lemon peel), 48.88% (for grapefruit peel), 56.96% (for mandarin peel), and 24.14% (for butternut-squash peel). However, it was noticed that the materials torrefied at 260 °C (lemon peel, mandarin peel), 220 °C (grapefruit peel), and 240 °C (butternut-squash peel) showed the lowest increase in water adsorption for specified materials. So far, mainly the improvement of hydrophobic properties of biomass with increasing torrefaction temperature has been reported [37]. Similar conclusions were observed by Acharjee et al. [40], who obtained a decrease in the EMC coefficient with an increase in the torrefaction temperature, conducting tests at a constant temperature (30 °C) and humidity (11.3% and 83.6%). Materials subjected to the torrefaction process at higher temperatures and a longer residence time in the reaction chamber were characterized by a lower moisture ratio uptake [41].

The mechanism of improving hydrophobic properties, along with increasing the temperature of the torrefaction process, is associated with the degradation of hydroxyl groups, which is responsible for binding moisture in the material. This is because of the availability of hydroxyl groups in hemicelluloses and the amorphous reign of the cellulose chains. Their presence makes the biomass hygroscopic and makes it much more susceptible to water attraction. As a result of the thermal processing of materials (torrefaction and pyrolysis), these compounds undergo thermal decomposition. Thereby, the capture of the water molecules is impossible in order to prevent the formation of hydrogen bonds [42]. It was found that the dehydration reaction is common during the torrefaction process, and the formation of carbon dioxide is mainly due to the decarboxylation of unstable carboxyl groups in the hemicellulose structure [43]. These factors were the basis for recognizing the relationship between the presence of hydroxyl groups and the thermal treatment of the material, which was described by Chen et al. [44]. As a result of increasing the temperature of the torrefaction process, more O-H bonds are dissolved and dehydrated, thanks to which the material becomes hydrophobic [45,46]. Additionally, it is also worth noting that the lower saturated moisture content of the torrefied biomass may, to some extent, be the result of tar condensation in the pores of the thermally treated biomass. Such a phenomenon significantly hinders the transfer of moist air through a solid, which prevents water vapor condensation [42]. The presence of condensed apolar tar on a solid, such as torrefied biomass, prevents condensation of water vapor inside the pores [47].

The obtained results are therefore the basis for the search for further correlations between the chemical and physical properties of the material and its hydrophobicity. Recently, Korshunov et al. [48] noticed that the hydrophobic properties of biomass depend on its porosity. They found that the contact angle of the droplet with a material's surface decreases with increasing measurement time, due to the penetration of water droplets inside the dried material. This speed decreases if torrefaction was applied.

From an economic point of view, obtaining a higher degree of hydrophobicity at a lower torrefaction temperature is highly desirable in order to minimize the production costs resulting from the higher electricity/energy consumption. Hence, further elucidation of the determinants influencing the hydrophobic behavior of materials seems to be crucial.

3.3. Trend Lines Parameters

Figure 6 shows the kinetics of EMC growth as a function of the climatic chamber operation time (CCOT), expressed as logarithmic trend lines. It can be seen that the value of the EMC coefficient tends to stabilize (the curve flattens). For all organic waste, in the initial period, the increase in moisture adsorption is dynamic, and then it decreases, reaching the limit, meaning that it reaches the thermodynamic equilibrium. This phenomenon is quite common in the literature that studies water adsorption by materials of various types. Chen et al. [49], examining the water adsorption capacity of bamboo and low-density polyethylene composites with bamboo charcoal addition in the immersion test, observed that the samples rapidly adsorbed water in the early stages of immersion. This process slowed down until the equilibrium conditions were reached. Similar conclusions were defined by others: Tiebie et al. [50], examining the kinetics of water adsorption by coffee powder (test duration time was 90 min); Mu et al. [51], investigating the characteristics of biocomposites (water adsorption study exceeding 50 days); and Tamrakar and Lopez-Anido [52], examining water-adsorption behavior and durability of extruded wood polypropylene composite (WPC) material used in Z-section sheet piles. The shape of the water-adsorption curves is usually constant, regardless of the test time (it is also dependent on some other factors, such as material shape, structure, type, and sample size).

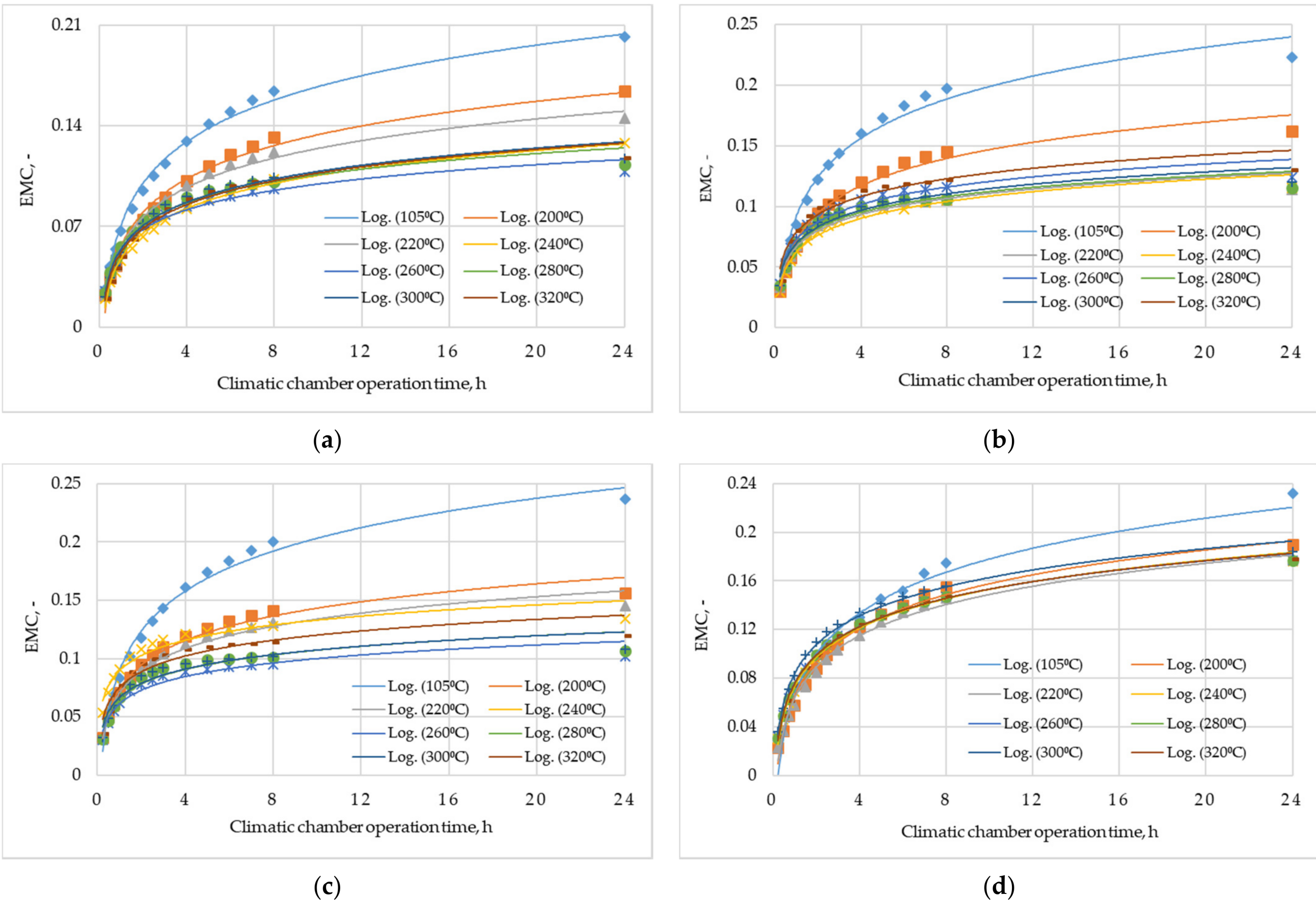


Figure 6. Kinetics of the EMC coefficient increase (logarithmic trend lines) as a function of the climatic chamber operation time: (**a**) lemon peel, (**b**) grapefruit peel, (**c**) mandarin peel, and (**d**) butternut-squash peel.

Table 7 shows the evaluated parameters of the *a* and *b* coefficients for the modeled logarithmic trend lines. The highest matches with respect to the experimental data concerned butternut-squash peel (average R^2 = 0.9907), and the lowest concerned mandarin peels (average R^2 = 0.9456). In the other cases, the coefficient of determination (R^2) was 0.9594 (average) for grapefruit peel and 0.9834 (average) lemon peel. Therefore, it can be concluded that the logarithmic model used indicates a high degree of matching the estimated parameters to the experimental data.

Table 7. Determined parameters *a* and *b* and the coefficient of determination (R^2) for logarithmic trend lines.

Waste	Coefficient	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
Lemon peel	a	0.0417	0.0335	0.0296	0.0250	0.0195	0.0213	0.0234	0.0239
	b	0.0713	0.0565	0.0560	0.0480	0.0546	0.0570	0.0543	0.0520
	R^2	0.9886	0.9850	0.9881	0.9919	0.9835	0.9721	0.9759	0.9822
Grapefruit peel	a	0.0466	0.0326	0.0191	0.0201	0.0208	0.0188	0.0196	0.0213
	b	0.0918	0.0718	0.0668	0.0623	0.0727	0.0690	0.0696	0.0786
	R^2	0.9827	0.9819	0.9481	0.9714	0.9524	0.9401	0.9597	0.9388
Mandarin peel	a	0.0494	0.0302	0.0276	0.0188	0.0168	0.0180	0.0177	0.0195
	b	0.0895	0.0738	0.0704	0.0898	0.0614	0.0659	0.0666	0.0753
	R^2	0.9881	0.9824	0.9813	0.9292	0.9361	0.9139	0.9217	0.9117
Butternut-squash peel	a	0.0485	0.0401	0.0370	0.0346	0.0335	0.0339	0.0343	0.0337
	b	0.0663	0.0654	0.0636	0.0733	0.0760	0.0752	0.0837	0.0756
	R^2	0.9752	0.9864	0.9887	0.9928	0.9963	0.9954	0.9934	0.9965

3.4. Main Results of Statistical Analysis

The conducted statistical analysis (applying two-way analysis of variance ANOVA) confirmed that there is a strong dependence (p-value < 0.05 for all organic waste) between the climatic chamber operation time (CCOT) and the Equilibrium Moisture Content assay (EMC). Likewise, changing the torrefaction temperature (TT) of torrefied materials significantly affects the EMC as well.

Appendix A shows significant differences for the EMC variable for the effect of torrefaction temperature (TT), according to Tukey's test (HSD), for biomass waste. The conducted analysis showed a statistically strong relationship between dried raw and torrefied waste for all groups in terms of moisture adsorption. It was noted that the impact of the torrefaction temperature itself is smaller, as evidenced by statistically insignificant differences in a large proportion of these cases.

Appendix B shows significant differences for the EMC variable for the effect of climatic chamber operation time (CCOT), according to Tukey's test (HSD), for biomass waste. In the vast majority of cases, a significant time relationship was demonstrated, with the exception of a few statistically insignificant cases.

With the combination of the torrefaction temperature parameters and the operating time of the climatic chamber (TT·CCOT), a significant influence (p-value < 0.05 for all organic waste) on the EMC index increase was observed. Detailed data for the analysis of variance are presented in Table 8.

Table 8. Results of analysis of variance (two-way ANOVA) for the dependent variable (EMC).

Effect	SS	df	MS	F	*p*
Lemon peel					
Intercept	2.205	1	2.205	100829.9	0.00
Climatic Chamber Operation Time (CCOT)	0.350	13	0.026	1233.5	0.00
Torrefaction Temperature (TT)	0.047	7	0.006	308.4	0.00
Interaction (TT·CCOT)	0.025	91	0.0002	12.6	0.00
Error	0.004	224	0.00002		
Grapefruit peel					
Intercept	3.060	1	3.060	124952.6	0.00
Climatic Chamber Operation Time (CCOT)	0.298	13	0.022	936.6	0.00
Torrefaction Temperature (TT)	0.086	7	0.012	503.3	0.00
Interaction (TT·CCOT)	0.042	91	0.0004	19.1	0.00
Error	0.005	224	0.00002		

Table 8. *Cont.*

Effect	SS	df	MS	F	*p*
Mandarin peel					
Intercept	3.134	1	3.134	23974.6	0.00
Climatic Chamber Operation Time (CCOT)	0.295	13	0.022	173.6	0.00
Torrefaction Temperature (TT)	0.098	7	0.014	108.1	0.00
Interaction (TT·CCOT)	0.054	91	0.0005	4.5	0.00
Error	0.029	224	0.0001		
Butternut-squash peel					
Intercept	3.776	1	3.776	171662.2	0.00
Climatic Chamber Operation Time (CCOT)	0.642	13	0.049	2248.0	0.00
Torrefaction Temperature (TT)	0.008	7	0.001	53.8	0.00
Interaction (TT·CCOT)	0.014	91	0.0001	7.3	0.00
Error	0.004	224	0.00002		

SS—a value of variability (sum of squares of all deviations), df—degree of freedom, MS—mean square (variance value), F—a value of F-statistic, *p*—probability value.

4. Conclusions

Due to the unfavorable physical, chemical, and hydrophilic properties of raw biomass, different low-energy-consuming solutions are offered to obtain a valuable stable and high-quality fuel. One of the proposed alternatives is biomass torrefaction, which improves the energy density, as well as the hydrophobicity, of the material. The hydrophobic properties are of special importance, as they enable the safe and long-term storage of torrefied biomass, thus preventing its degradation.

The article analyzed the impact of the torrefaction temperature and the operation time of the climatic chamber on the hydrophobic properties of selected agri-food waste (lemon peel, mandarin peel, grapefruit peel, and butternut-squash peel). It was shown that torrefaction significantly improves the hydrophobic properties of biomass. With regard to dried non-torrefied material, the EMC coefficient decreased by a maximum of 56.96–24.14%, depending on the type of organic waste.

There was also no evidence of an increase in hydrophobic properties (lowering of the EMC coefficient) with increasing torrefaction temperature. The lowest EMC index was obtained at different temperatures for different waste biomass. It is an important observation influencing the economics of torrefaction and optimization of production processes, guaranteeing a reduction in energy expenditures, and thus lowering the costs of torreficates production. Significant differences in the kinetics of adsorbing moisture were also observed, along with increasing the operation time of the climate chamber.

The results also create a space for further research on the determinants of improving the hydrophobic properties of biomass during torrefaction, especially in terms of the physical and chemical properties of materials.

Supplementary Materials: The following are available online at https://www.mdpi.com/article/10.3390/en14175299/s1, Table S1: Proximate analysis of the investigated raw and torrefied food waste biomass.

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Appendix A

Statistically significant differences are marked in bold.

Table A1. Significant differences for the EMC variable for the effect of torrefaction temperature (TT) according to Tukey's test (HSD) for lemon peels.

	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
105 °C		**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**
200 °C	**0.000032**		**0.001419**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**
220 °C	**0.000032**	**0.001419**		**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**
240 °C	**0.000032**	**0.000032**	**0.000032**		0.811593	**0.000033**	**0.000071**	0.056986
260 °C	**0.000032**	**0.000032**	**0.000032**	0.811593		**0.001715**	**0.020818**	0.811593
280 °C	**0.000032**	**0.000032**	**0.000032**	**0.000033**	**0.001715**		0.997598	0.196947
300 °C	**0.000032**	**0.000032**	**0.000032**	**0.000071**	**0.020818**	0.997598		0.606686
320 °C	**0.000032**	**0.000032**	**0.000032**	0.056986	0.811593	0.196947	0.606686	

Table A2. Significant differences for the EMC variable for the effect of torrefaction temperature (TT) according to Tukey's test (HSD) for grapefruit peels.

	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
105 °C		**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**
200 °C	**0.000032**		**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.028198**
220 °C	**0.000032**	**0.000032**		**0.019725**	**0.000032**	0.538435	0.062399	**0.000032**
240 °C	**0.000032**	**0.000032**	**0.019725**		**0.000032**	**0.000036**	**0.000032**	**0.000032**
260 °C	**0.000032**	**0.000032**	**0.000032**	**0.000032**		**0.000046**	**0.001866**	**0.000032**
280 °C	**0.000032**	**0.000032**	0.538435	**0.000036**	**0.000046**		0.969003	**0.000032**
300 °C	**0.000032**	**0.000032**	0.062399	**0.000032**	**0.001866**	0.969003		**0.000032**
320 °C	**0.000032**	**0.028198**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	

Table A3. Significant differences for the EMC variable for the effect of torrefaction temperature (TT) according to Tukey's test (HSD) for mandarin peels.

	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
105 °C		**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**
200 °C	**0.000032**		0.298847	0.320611	**0.000032**	**0.000032**	**0.000032**	**0.027590**
220 °C	**0.000032**	0.298847		**0.000174**	**0.000032**	**0.000035**	**0.000038**	0.982673
240 °C	**0.000032**	0.320611	**0.000174**		**0.000032**	**0.000032**	**0.000032**	**0.000033**
260 °C	**0.000032**	**0.000032**	**0.000032**	**0.000032**		0.326179	0.243517	**0.000032**
280 °C	**0.000032**	**0.000032**	**0.000035**	**0.000032**	0.326179		1.000000	**0.000365**
300 °C	**0.000032**	**0.000032**	**0.000038**	**0.000032**	0.243517	1.000000		**0.000693**
320 °C	**0.000032**	**0.027590**	0.982673	**0.000033**	**0.000032**	**0.000365**	**0.000693**	

Table A4. Significant differences for the EMC variable for the effect of torrefaction temperature (TT) according to Tukey's test (HSD) for butternut-squash peels.

	105 °C	200 °C	220 °C	240 °C	260 °C	280 °C	300 °C	320 °C
105 °C		**0.000032**	**0.000032**	**0.000032**	**0.001633**	**0.000163**	**0.001633**	**0.000347**
200 °C	**0.000032**		**0.000236**	0.066560	**0.000084**	**0.000757**	**0.000032**	**0.000347**
220 °C	**0.000032**	**0.000236**		**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**
240 °C	**0.000032**	0.066560	**0.000032**		0.610168	0.914717	**0.000032**	0.837568
260 °C	**0.001633**	**0.000084**	**0.000032**	0.610168		0.999305	**0.000032**	0.999954
280 °C	**0.000163**	**0.000757**	**0.000032**	0.914717	0.999305		**0.000032**	1.000000
300 °C	**0.001633**	**0.000032**	**0.000032**	**0.000032**	**0.000032**	**0.000032**		**0.000032**
320 °C	**0.000347**	**0.000347**	**0.000032**	0.837568	0.999954	1.000000	**0.000032**	

Appendix B

Statistically significant differences are marked in bold.

Table A5. Significant differences for the EMC variable for the effect of climatic chamber operation time (CCOT) according to Tukey's test (HSD) for lemon peels.

	0.25 h	0.5 h	0.75 h	1 h	1.5 h	2 h	2.5 h	3 h	4 h	5 h	6 h	7 h	8 h	24 h
0.25 h		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
0.5 h	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
0.75 h	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
1 h	**0.000023**	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
1.5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
2 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.000063**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
2.5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000063**		**0.000862**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
3 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000862**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
4 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.000063**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000063**		**0.004724**	**0.000023**	**0.000023**	**0.000023**
6 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.004724**		0.064472	**0.000024**	**0.000023**
7 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	0.064472		0.452332	**0.000023**
8 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000024**	0.452332		**0.000023**
24 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	

Table A6. Significant differences for the EMC variable for the effect of climatic chamber operation time (CCOT) according to Tukey's test (HSD) for grapefruit peels.

	0.25 h	0.5 h	0.75 h	1 h	1.5 h	2 h	2.5 h	3 h	4 h	5 h	6 h	7 h	8 h	24 h
0.25 h		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
0.5 h	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
0.75 h	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
1 h	**0.000023**	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
1.5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
2 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.001161**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
2.5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.001161**		0.065416	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
3 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	0.065416		**0.000028**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
4 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000028**		**0.018850**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.018850**		0.255247	**0.000361**	**0.000023**	**0.000023**
6 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	0.255247		0.844798	**0.034334**	**0.000023**
7 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000361**	0.844798		0.945591	**0.000023**
8 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.034334**	0.945591		**0.000023**
24 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	

Table A7. Significant differences for the EMC variable for the effect of climatic chamber operation time (CCOT) according to Tukey's test (HSD) for mandarin peels.

	0.25 h	0.5 h	0.75 h	1 h	1.5 h	2 h	2.5 h	3 h	4 h	5 h	6 h	7 h	8 h	24 h
0.25 h		**0.000031**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
0.5 h	**0.000031**		**0.017836**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
0.75 h	**0.000023**	**0.017836**		0.242620	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
1 h	**0.000023**	**0.000023**	0.242620		**0.015568**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
1.5 h	**0.000023**	**0.000023**	**0.000023**	**0.015568**		0.339811	**0.000568**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
2 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	0.339811		0.823644	**0.038723**	**0.000026**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
2.5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000568**	0.823644		0.962901	**0.022289**	**0.000047**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
3 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.038723**	0.962901		0.722052	**0.022289**	**0.000184**	**0.000025**	**0.000023**	**0.000023**
4 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000026**	**0.022289**	0.722052		0.962901	0.285118	**0.028893**	**0.001917**	**0.000023**
5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000047**	**0.022289**	0.962901		0.996898	0.771570	0.277744	**0.000023**
6 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000184**	0.285118	0.996898		0.999912	0.960372	**0.000035**
7 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000025**	**0.028893**	0.771570	0.999912		0.999987	**0.000842**
8 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.001917**	0.277744	0.960372	0.999987		**0.014872**
24 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000035**	**0.000842**	**0.014872**	

Table A8. Significant differences for the EMC variable for the effect of climatic chamber operation time (CCOT) according to Tukey's test (HSD) for butternut-squash peels.

	0.25 h	0.5 h	0.75 h	1 h	1.5 h	2 h	2.5 h	3 h	4 h	5 h	6 h	7 h	8 h	24 h
0.25 h		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
0.5 h	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
0.75 h	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
1 h	**0.000023**	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
1.5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
2 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
2.5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.000025**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
3 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000025**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
4 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**
5 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**		**0.000201**	**0.000023**	**0.000023**	**0.000023**
6 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000201**		**0.000045**	**0.000023**	**0.000023**
7 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000045**		**0.007099**	**0.000023**
8 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.007099**		**0.000023**
24 h	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	**0.000023**	

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