

## Article

# The Exothermic Effects of Textile Fibers during Changes in Environmental Humidity: A Comparison between ISO:16533 and Dynamic Hot Plate Test Method

Faisal Abedin <sup>1,2,\*</sup>  and Emiel DenHartog <sup>1,2</sup> 

<sup>1</sup> Department of Textile Engineering, Chemistry and Science, North Carolina State University, Raleigh, NC 27606, USA

<sup>2</sup> Textile Protection and Comfort Center, North Carolina State University, Raleigh, NC 27606, USA

\* Correspondence: fabedin@ncsu.edu; Tel.: +1-919-746-6970

**Abstract:** The exothermic effects of high regain fiber types have been described before; yet, there have not been reliable tests to demonstrate these effects on the human body. Most test methods focus on steady-state measurements; therefore, these exothermic effects during changes in environmental humidity are typically not analyzed or quantified. We have conducted a set of fabric tests that shows the connection between the exothermic effect of water vapor uptake and its consequence for heat loss through the fabric in transient conditions. We have performed the ISO:16533 standard test, a dynamic hot plate test developed by Naylor to measure the exothermic property of the fabric, and dynamic regain tests to connect the dots between these tests and the water vapor uptake phenomenon. Although the ISO:16533 test method tends to show the temperature increase in fibers, it cannot differentiate between the hygroscopic fiber (wool, viscose, cotton) types ( $p > 0.001$ ). In addition, sensor size and sample folding techniques could impact the temperature increase. On the other hand, the Naylor hot plate test showed a greater difference in heat release among the fiber types (wool showed 20% higher heat release than viscose, 50% more than cotton), although the relative humidity changes in the chamber take time, which might not reflect a step-wise change in humidity. So far, these test methods have proven to be the most reliable for determining the exothermic behavior of textile fiber. However, these test methods still have limitations and cannot simulate realistic environmental conditions considering an instantaneous change in the environment. This paper reflects the comparison between the two test methods and recommends directions to accurately address the theory of water vapor uptake under dynamic conditions.

**Keywords:** hygroscopic fibers; exothermic property; moisture; relative humidity; clothing



**Citation:** Abedin, F.; DenHartog, E. The Exothermic Effects of Textile Fibers during Changes in Environmental Humidity: A Comparison between ISO:16533 and Dynamic Hot Plate Test Method. *Fibers* **2023**, *11*, 47. <https://doi.org/10.3390/fib11050047>

Academic Editor: Damien Soulat

Received: 20 February 2023

Revised: 27 April 2023

Accepted: 16 May 2023

Published: 22 May 2023



**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

## 1. Introduction

Clothing comfort is a subjective concept. Numerous attempts have been made throughout the years to quantify and explain human clothing comfort and related properties using various methods [1]. Haghi referred to clothing as the second skin and described humans as unfinished creatures without clothing, which undoubtedly draws attention to the vitality of clothing in everyday life [2]. Humans, particularly in recent years, have developed an excellent sense of fashion for various occasions or activities such as walking, hiking, cycling, and running, and are conscientious of the garment's comfort and functionality. Sweating is vital to ensure human comfort and is the primary means of transporting moisture from the human body. The amount of sweat released from human skin during exercise is approximately 1000 g/m<sup>2</sup>/h, while it is only 15 g/m<sup>2</sup>/h when the body is at rest. This means that a large amount of moisture may evaporate from the skin in the form of sensible perspiration. It supports the notion that humans are never in a true steady state situation [3,4]. Therefore, clothing fibers may have an impact on human comfort in transient situations by absorbing and desorbing moisture.

The property of absorbing water vapor, regarded as moisture regain, is a significant feature of clothing materials. The vapor absorption may make the fabrics act as a heat reservoir, protecting the body from being exposed to more humid conditions. Gao et al. [5] mentioned in a high-water vapor-pressure environment, hygroscopic clothing absorbs water vapor, which releases latent heat. This phenomenon raises the temperature of clothing material and the surrounding air until an equilibrium is reached. This is called the exothermic behavior of clothing materials. Thus, fibers with high moisture regain would better buffer humidity and temperature variation when switching environments. In addition, the article from [3] provides a schematic diagram on how different fibers interact with environmental moisture.

The property of absorbing water vapor regarded as moisture regain is a valuable feature of some fiber types. The absorption of water vapor may facilitate the fabric to act as a heat reservoir, protecting the body from rapid changes when being exposed to a more humid condition. Morton and Hearle [6] explained the relationships between moisture regain and relative humidity at 20 °C for various textile fibers, whereas Wiegierink illustrated the absorption and desorption of wool fibers between 36 °C and 150 °C [7]. From both explanations, wool appears to have the highest moisture regain among the mentioned fibers and the largest response when switching the environment. Furthermore, temperature is also known to influence the moisture regain of wool fiber. In the range of temperatures between 35 °C and 150 °C, the moisture regain percentage increases when temperature decreases. Darling and Belding reported the moisture adsorption of cotton, wool, and rayon at low temperatures from −20 °F to 40 °F [8]. The study showed that at low temperature, cotton fabric had an apparent drop in moisture regain percentage while wool was more constant. Thus, moisture regain properties gave a good reference when exploring the heat of sorption in a dynamic environment when changing from indoor to outdoor. Several studies [9–11] demonstrated wool as an example of hygroscopic fiber that has a higher diffusion coefficient than other fibers, which may provide an advantage of hygroscopic textile materials, where these materials would take away the perspiration on the skin and perform a buffer with rapid changes in the environment and/or the microclimate through the fibers' exothermic behavior.

The exothermic and endothermic properties of textile materials are observed when there is a change in the external relative humidity (RH), so that the materials can absorb or desorb the moisture, which leads to energy exchange. The exothermic effect of a textile may directly affect its physiological comfort. Clothing creates a microclimate between the skin and the clothing. The surrounding environment and the conditions at the skin surface determine the levels of temperature and humidity of the skin microclimate. For example, a change in ambient humidity from low to high can create a sticky perception of the microclimate; similarly, perspiration generation during exercise can create a damp environment in the microclimate. Clothing may absorb both liquid and moisture vapor; however, depending on the type of clothing worn, vapor transfer via clothing will affect the microclimate, which affects the body's core and skin temperature. The evaporation of sweat from the skin is influenced by several factors, including the thickness, wettability, and permeability of the clothing. In addition to ambient humidity and temperature, microclimate conditions are determined by the sweat evaporation and fabric properties [12–14].

However, no test method currently exists to address such issues in dynamic conditions, specifically during a change in environment. Multiple authors have recognized that the thermophysiological components of clothing comfort cannot be entirely described by laboratory measurements of steady-state heat and moisture vapor resistance [15–20].

Few attempts have been made to quantitatively assess comfort attributes in a changing laboratory environment [21]. Among them, ISO 16533 [22] and the dynamic hot plate method developed by Naylor [23] are the most reliable for assessing these properties. However, these methods still have limitations and may be modified instead. This paper addresses the limitations and recommends changes based on experimentation with the above-mentioned methods.

## 2. Materials and Methods

### 2.1. Materials

#### Test Materials and Physical Properties

Knitted fabrics made from wool (yarn diameter  $\leq 17.5 \mu$ ), cotton, viscose, and polyester were received from Australian Wool Innovation for experimentation. The materials were selected based on their moisture regain behavior. The details of the fabric used in the experiment are listed in Table 1. All of the materials were of a single jersey structure with the exception of polyester. This was chosen so that all of the materials would be uniform in thickness, as fabric thickness has the most significant impact on thermal insulation [24]. We used the test methods to characterize the exothermic behavior of knit fabrics which are commonly used as shirt materials for sports apparel [25].

**Table 1.** Details of the experimental fabrics.

Sl. No.	Sample ID	Fabric Composition	Fabric Structure	Weight (g/m <sup>2</sup> )	Thickness (mm)
1	Wool	100% Merino Wool	Jersey Knit	213	0.69
3	Cotton	100% Cotton	Jersey Knit	188	0.65
4	Polyester	100% Polyester	Rib Knit	199	0.69
5	Viscose	100% Viscose	Jersey Knit	204	0.67

### 2.2. Methods

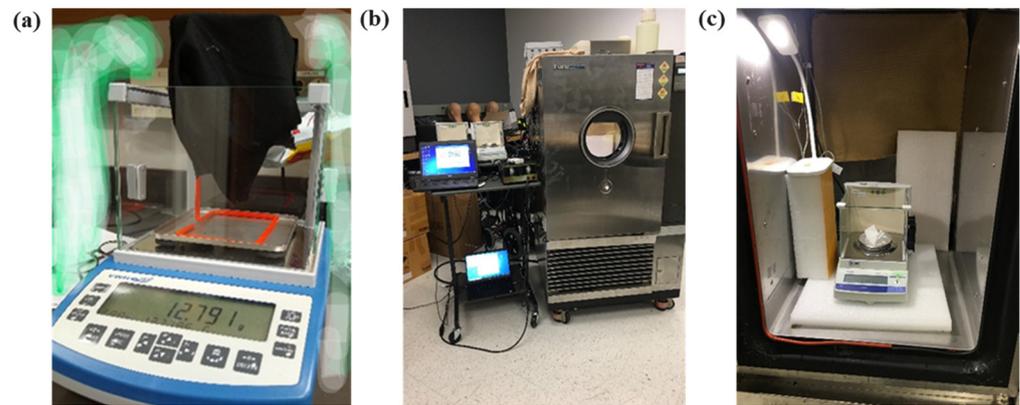
#### 2.2.1. Dynamic Regain Test

The regain of the fiber is considered to be the driving parameter that explains the exothermic effect of the fibers. The dynamic regains test was carried out to see if there were any significant differences in the rate of water vapor uptake from the environment or in the regain rate between the fiber types. Three different principles have been followed to determine the exothermic effect in various ways. For the first two principles, the traditional oven dry method was followed for two different experiments with durations of 3 h and 24 h, respectively.

#### 2.2.2. Oven Dry Method

The fabric samples were cut into square shapes weighing approximately 10 g. After weighing, the fabrics were dried in a blow-dryer oven at 105 °C for 2 h. The fabrics were then placed in a plastic bag immediately and sealed after being left in the oven. The bag was then placed in a desiccator to maintain proper humidity. The experiment was carried out in an air-conditioned lab room where a temperature of 20 °C  $\pm$  0.5 and a RH of 65%  $\pm$  0.5 are maintained. The fabric samples were kept on a stand placed inside a particular type of digital scale with a glass door closed on four sides, which helped to avoid the variation in results due to the airflow from the side (Figure 1). The fabric samples were then allowed to regain moisture from the air for 3 h, and their weight readings were recorded every 5 min. For the other experimental setup, the fabric samples were allowed to regain moisture from the environment for about 24 h. If the initial sample weight is  $w_i$  and the final weight is  $w_f$ , the moisture regain ( $m_r$ ) can be calculated from the following equation:

$$\text{Moisture Regain } (m_r) = \frac{w_f - w_i}{w_i} \times 100\% \quad (1)$$



**Figure 1.** Dynamic regain rate test experimental setup. (a) The digital scale consists of a glass door closed from four sides which will avoid variation in results due to airflow from sides. (b) Environmental chamber set up for 2-phase humidity change experiment. (c) Digital scale placed inside of the climate chamber.

### 2.2.3. Two-Phase Humidity Change

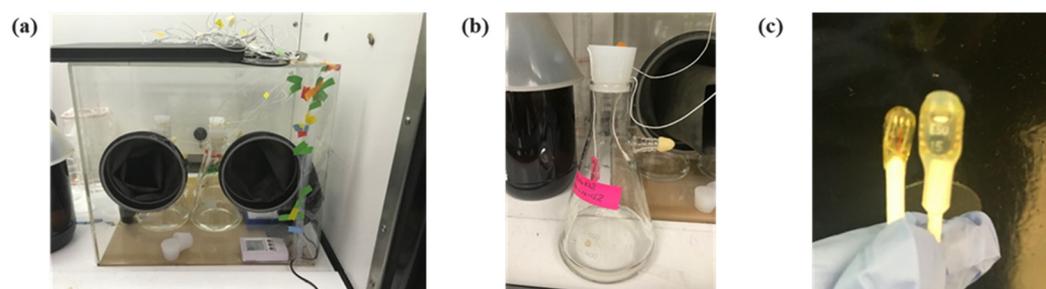
This test was carried out using a small environment chamber and changing the RH from 45% to 85% at two intervals while keeping the temperature constant at 20 °C. The environment chamber (*ESPEC, North America*) can control RH from 10% to 95%. After weighing the samples, oven drying was performed at 105 °C for 2 h. The samples were sealed while leaving the oven and kept in a sealed plastic bag. The plastic bag was always kept in a desiccator to maintain a stable surrounding condition. The chamber's RH was initially set to 45%; the sample was placed on the scale mounted inside the chamber, and the weight was recorded at every 5 min interval. After 3 h, the humidity increased to 85% and the data recording continued for the following 3 h. The fabric underwent two stages following the humidity change; the first transition happened from 0% to 45% RH, and then from 45% RH to 85% RH.

### 2.2.4. ISO 16533 Set Up

ISO 16533 is a standard testing method to measure the exothermic and endothermic properties of textile materials when changing RH. Two constant containers with 65% and 20% concentrated sulfuric acid solution were used to create the 10% and 90% RH environment. The ISO 16533 setup is shown in Figure 2. An MSR sensor (MSR Electronics GmbH, Seuzach, Switzerland) which could measure both temperature and humidity was placed in each of the containers to measure the conditions of the containers. The test specimen was clipped to the MSR sensor to record the temperature change over the transient RH. The test specimen was cut into 5 cm × 5 cm and dried in the oven at 105 °C for two hours, then conditioned in a desiccator until immediately before testing. A sample specimen was folded in half and the sensor probe was placed in the center of the upper half of the area to be tested. The specimen was folded again to wrap the sensor probe. This sample folding technique was followed as per the standard. The fabric was clipped on the sensor and placed in the constant humidity container to condition it for a minimum of 3 h. After more than 3 h of conditioning, all sensors in the two flasks were switched on to record the temperature and RH for 30 min. Then, the plug of the low-humidity chamber with the test specimen was removed and transferred to the high-humidity container. The data recording continued for another 1 h. The temperature change ( $\Delta T_{exo}$ ) can be derived from Equation (1).  $T_{peak}$  could be read from the sensor mounted with the specimen.  $T_{blank}$  could be read from the sensors, without the specimen in the flask.

$$\Delta T_{exo} = T_{peak} - T_{blank} \quad (2)$$

where  $T_{peak}$  is the peak temperature ( $^{\circ}\text{C}$ ), determined using an MSR sensor with the test specimen mounted on the sensor probe in the high humidity container during the test.  $T_{blank}$  is the peak temperature ( $^{\circ}\text{C}$ ), determined using an MSR sensor with the test specimen mounted on the sensor probe while changing from a low humidity container to high humidity container.  $\Delta T_{exo}$  is the difference in the peak temperature ( $^{\circ}\text{C}$ ), determined between a state of fabric temperature with low humidity and after the transition to a high humidity chamber.



**Figure 2.** ISO 16533 setup for measuring the exothermic effect in the fabric. (a) A glove box of dimensions 45 cm  $\times$  45 cm  $\times$  40 cm placed into a fume hood. (b) Constant humidity container with temperature–RH sensor. (c) Size comparison of two MSR sensors, temperature sensor (left), temperature and humidity sensor (right).

Three different principles were followed to check the effect of different sensor sizes (within the ISO-16533 standard) and whether the folding principle has any effect on the exothermic property of the fabric. Two external sensors, both temperature and humidity sensors (larger in size but within the dimension of sensors mentioned by ISO 16533), and only the temperature sensor (smaller in size but within the dimension of sensors mentioned by ISO 16533) have been used to check the effect of sensor size using two different experiments; however, the third experiment was used to check if the folding techniques have an impact on the overall value of the heat gain from the two previous experiments. The details of the experimental procedure are shown in Table 2.

**Table 2.** Summary of the three experiments measuring the exothermic effect by ISO 16533.

Parameter	Experiment 1	Experiment 2	Experiment 3
Sensor Size	Large Temperature–RH sensor (Within the ISO Standard range)	Small Temperature Sensor (Within the ISO Standard range)	Small Temperature sensor (Within the ISO Standard range)
Fold procedure	Same fold as ISO	Same fold as ISO	Both fabrics from Exp 1 and 2 are used and clipped.

### 2.2.5. Dynamic Hot Plate Test

Based on the operation of a sweating-guarded hot plate, Naylor developed a novel approach for measuring the dynamic moisture buffering potential of fabrics by looking at the fabric's exothermic effect during transient humidity change by dynamic hot plate (DHP). The hot plate operation mode is the same as the dry hot plate test, while the RH in the chamber becomes dynamic instead of static. The fabric sample (size  $60 \pm 1 \text{ cm} \times 60 \pm 1 \text{ cm}$ ) is first equilibrated on the hot plate in an environment with low RH (45%). After the heat flux becomes stable, the RH rapidly increases to 85%. With an increase in RH, the fabrics absorb moisture and generate heat, which reduces the heat flux applied to the plate. The area of the transient peak of the heat flux is a measure of the moisture buffering potential. The transient heat flux peak area was calculated using a simple numerical integration over the peak period. The average heat flux value for 10 min before switching the RH was calculated

as the baseline of the steady state at 45% RH. Then, all the difference values between the baseline and each data point were collected and summated. We then multiplied the obtained value by 10 s as the interval of the data record was 10 s, and divided the overall result by the total time of the integration, which was the same for all fabrics. The integration of the heat flux curve thus obtained was used to express the heat release of the humidity change. Three repetitions for each fiber type were taken. The total thermal resistance of the fabric plus the air layer could be calculated using the following equation.

$$R_{ct} = (T_s - T_a)A/H_c \quad (3)$$

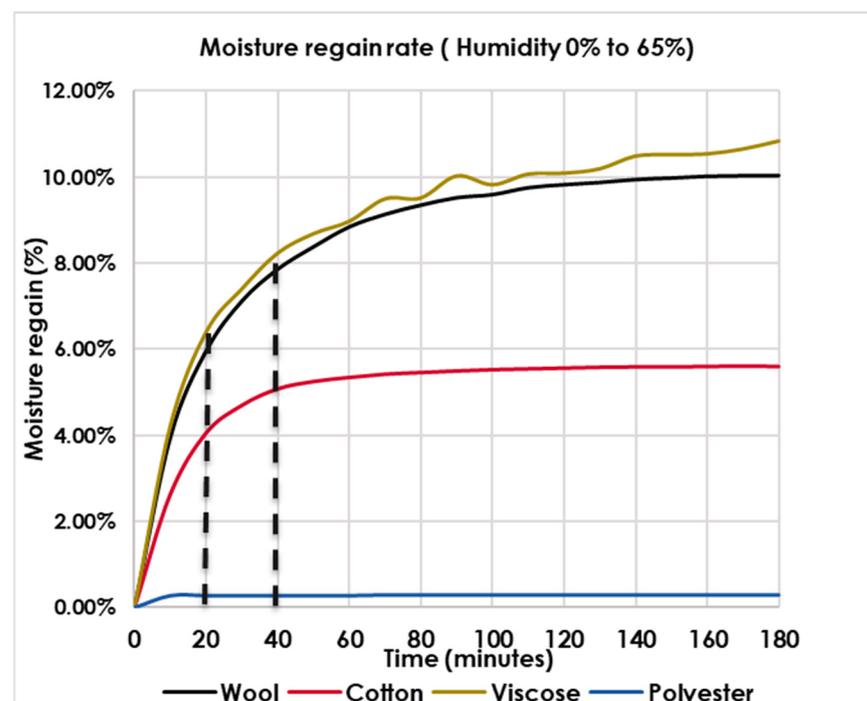
where  $R_{ct}$  is the total resistance to dry heat transfer provided by the fabric system during the steady state and air layer ( $K \cdot m^2/W$ ),  $A$  is the area of the plate test section ( $m^2$ ),  $T_s$  is the surface temperature of the plate ( $^{\circ}C$ ),  $T_a$  is air temperature ( $^{\circ}C$ ), and  $H_c$  is power input (W).

### 3. Test Results and Analysis

#### Dynamic Regain Test.

##### 3.1. Oven Dry Method

Figure 3 shows the moisture regain graph (%) versus time (minutes) for all fabric samples. All fabrics exhibited a similar pattern curve, having two phases. In the first phase, from the start of the test to about 20–30 min, the moisture was absorbed at a high rate due to the high RH gradient in RH (0 to 65%). After this rapid initial phase, the fabrics continued to slowly absorb water vapor from the environment until the end of the experiment. As observed before, not all fabrics reached their expected regain value after 3 h, but the water vapor absorption rate in the wool and viscose samples was greater than in the cotton samples. At 30 min after the first start of the rapid uptake phases, the values for wool, viscose, cotton, and polyester were 7.0%, 7.4%, 4.7%, and 0.3%, respectively; these values are approximately 50% of their expected moisture regain percentage.



**Figure 3.** Moisture regain percentage for different kinds of textile fabrics over time in minutes over 3 h; all samples are oven-dried and then tested in the same environmental conditions with 65% humidity and 200 °C. The two vertical dotted lines indicate the transition of the fabrics to a lower absorption rate at 20 min (cotton) and 40 min (wool and viscose).

The RH gradient between textile samples and the environment started to decrease as time passed. All curves had a “transition point,” after which the moisture regain rate only increased slowly (but steadily). The consequence of this curve flattening, due to the fundamental physics of water vapor diffusion and transport, is that the exothermic effect will also decrease as it is directly related to the water vapor uptake rate (curve steepness). Thus, from these primary curves, an estimate of the duration of the exothermic effects can be derived. Polyester, if it shows any exothermic effect, will have a short effect of up to 10 min max. The effect for cotton is likely to last up to 40 min maximum as the uptake rate (slope) becomes small after that. The expected effect for wool and viscose could be up to 80–100 min (two times as long) and even longer.

Figure 4 further shows the rapid initial increase in regain, after which there is a very long and slow process to reach the final steady state, up to 24 h. With the focus on the regain rate measurement in the first hour, the experiment was conducted at the 1 Hz sampling rate. Figure 5 shows the regain values measured after 24 h of the experiment. With this method, all values appeared consistently slightly below (about 1–2%) the reported values from the literature.

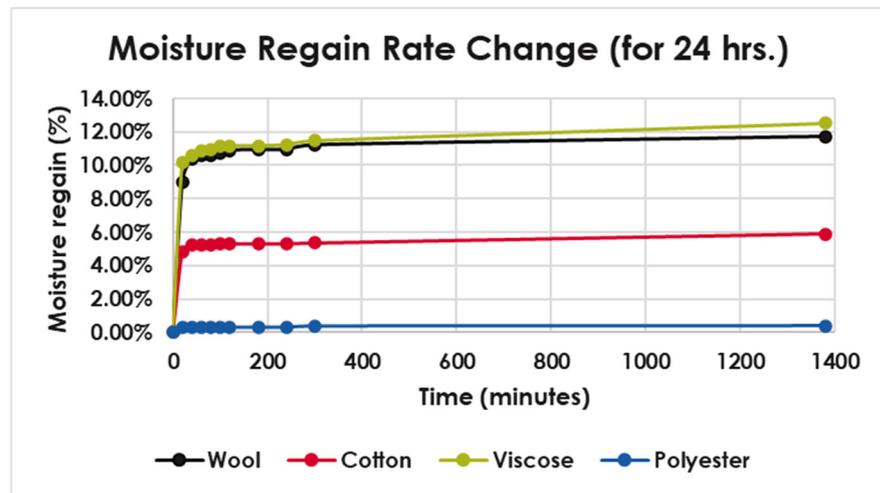


Figure 4. Longer term regain rate for wool, cotton, and viscose fabrics, measured at larger time intervals up to 5 h and after 24 h (the next day). Note the time axis in minutes. The regain rates of detailed measurements were obtained with 1 Hz weight measurements in the first 60 min.

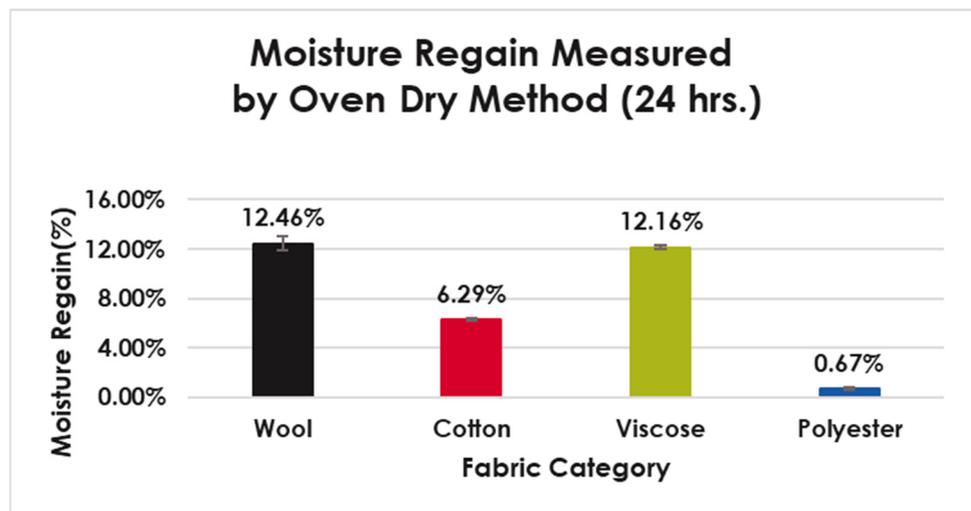


Figure 5. Moisture regain percentage measured by oven dry method (24 h).

### 3.2. Two-Phase Humidity Change

During a two-phase humidity change experiment, shown in Figure 6, all of the curves showed similar patterns based on the absorbency capacity of the fabric. However, it seemed that after 180 min (3 h) of the experiment, all of the fiber types had lower regain values than the literature values. For the initial period of three hours, wool and viscose showed some degree of moisture regaining behavior; however, after switching the humidity to a higher value, the behavior pattern looked more prominent, increasing from a value of 3.46% to almost 10% for wool and 2.66% to almost 9% for viscose. As per usual, polyester did not show any response at all. Cotton had a response between that of wool and polyester. The result indicated that a higher humidity gradient accelerated the moisture absorption behavior of the textile fiber and helped it regain equilibrium faster.

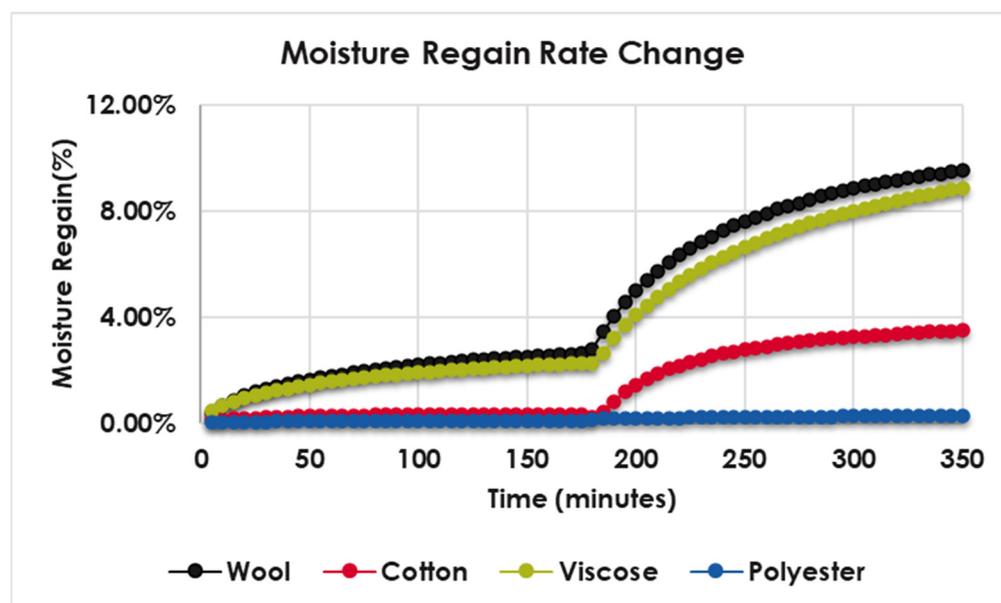


Figure 6. Moisture regain rate calculated from 2-phase regain test by environmental chamber.

From these experiments, it was evident that viscose and wool absorbed the highest amount of moisture, as expected. Both fabrics are hygroscopic in nature. In the case of wool, the  $-NH_2$  and  $-COOH$  groups help to absorb moisture. The morphology of wool is complex and thus water vapor was absorbed slowly; after 3 h of the experiment, the wool had not reached its final equilibrium value. In the case of viscose and cotton, the  $-COOH$  groups help to absorb moisture. The moisture regain rate of viscose was much higher than that of cotton. This might be due to morphological and structural differences. Viscose has a circular cross section and provides more surface area than cotton for moisture absorption [26,27]. Moisture regain rate varies significantly with the type of fabric; polyester absorbs the least amount of moisture. The first phase for polyester is the shortest; on the other hand, the moisture regain rate for viscose and wool is the highest.

Thus, the conclusion of these dynamic regain experiments should be that the initial rapid regain uptake rate will be dominant for the realistic exothermic effects, as that is directly related to the regain rate [3]. This means that although wool and viscose have a higher steady-state regain and their initial regain rate is higher than that of cotton, the differences may be predicted to be, in reality, smaller than their end regain values. Instead of a difference of 6 g of water vapor per 100 g fabric (e.g., 13 for wool versus 7 for cotton), we may only expect a difference in generated heat from 2 g of absorbed water vapor per 100 g fabric (i.e., 6 g versus 4 g). Still, these experiments show that the exothermic effect differs significantly between wool/viscose, cotton, and polyester.

### 3.3. ISO 16533 Test

These three experiments (E1, E2, E3) aimed to determine the optimal protocol for these exothermic tests to detect reliable and significant differences among different types of fiber. The experiments were focused on determining the effect of mass (increasing sample volume) and temperature sensor size. A larger sample (double) folded in the same area was expected to have a larger increase in temperature as measured by the sensor folded within the sample. The smaller temperature sensor was expected to have better intimate contact with the sample and thus better register the temperature changes. Figure 7 indicates that the three experiments on the same type of fabrics had increasing test results in terms of temperature increase. From E1, viscose fabrics had the highest temperature increase of 5.87 °C, while polyester had the lowest or almost no temperature increase. Cotton increased by 4.80 °C when switching the fabric from low to high humidity, while for wool, it was 5.64 °C. In addition, viscose had the highest temperature increase of 6.17 °C and 6.85 °C, respectively, for Experiment 2 and Experiment 3. In comparison, wool had an increase of 5.94 °C and 6.79 °C, and cotton had an increase of 5.29 °C and 6.31 °C, respectively, for E2 and E3. Polyester did not show any responses at all in the three experiments.

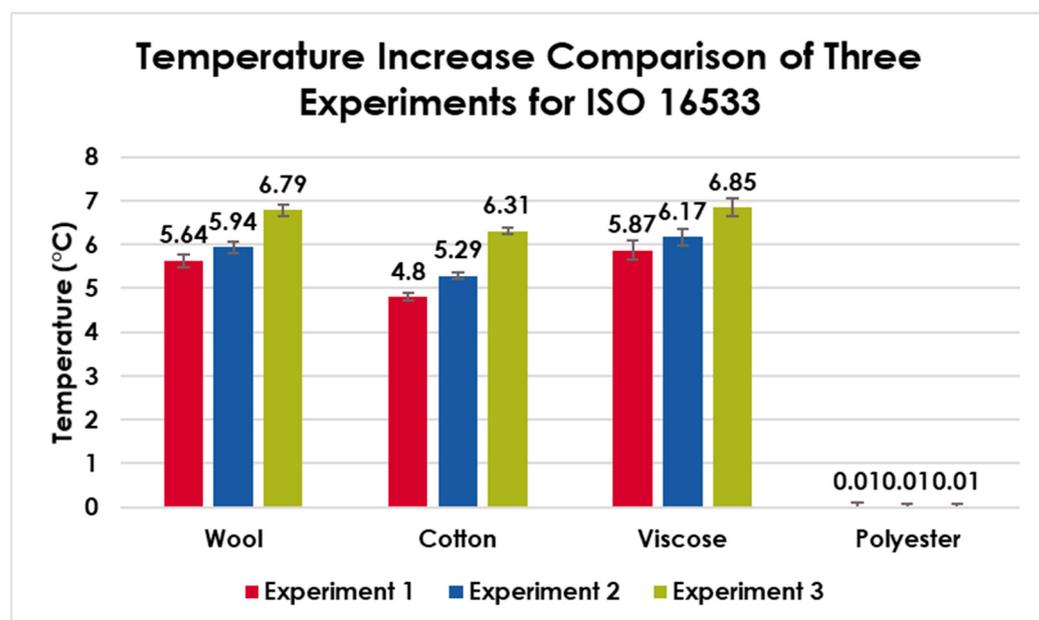


Figure 7. Summary of the three experiments for ISO 16533, with results expressed as mean  $\pm$  S.D.

Tukey's HSD post hoc test was used for each experiment to see if there was a mean difference in temperature increase between the fiber types using different principles. ANOVA yielded statistically significant differences for each pair of fiber type in each experiment, while showing no statistically significant differences between wool and viscose. This means that for the ISO 16533 test, during the step changes in humidity from low to high, the differences in mean temperature increase among each pair of wool, cotton, viscose, and polyester are significant, while the pair wool–viscose did not show any significant results for the increase in mean temperature (Table 3). Even using a small sensor size minimizes the differences between fiber types. In Table 4, it is evident that there are no statistically significant differences between wool, cotton, and viscose in terms of their temperature while changing the humidity from low to high. Increasing thickness yielded significant differences between the fiber types; additionally, in this case, the wool–viscose pair did not show any differences in temperature increase (Table 5). Therefore, ISO 16533 can demonstrate the existence of an exothermic effect, but there is not enough evidence to say that the ISO 16533 test cannot differentiate between fiber types of relatively the same moisture regain percentage, for example, wool (14–19%) and viscose (13%) [6].

**Table 3.** Tukey’s HSD each pair summary for the sensor size effect in the temperature increase for Experiment 1.

Fiber Comparison	Difference	p-Value
Viscose > Polyester	5.9	<0.0001
Wool > Polyester	5.6	<0.0001
Cotton > Polyester	4.8	<0.0001
Viscose > Cotton	1.0	<0.0001
Wool > Cotton	0.83	0.0004
Viscose > Wool	0.23	0.2705

The ‘>’ sign indicates temperature comparison for two different fiber types.

**Table 4.** Tukey’s HSD each pair summary for the sensor size effect in the temperature increase for Experiment 2.

Fiber Comparison	Difference	p-Value
Viscose > Polyester	6.2	<0.0001
Wool > Polyester	5.9	<0.0001
Cotton > Polyester	5.3	<0.0001
Viscose > Cotton	0.88	0.0193
Wool > Cotton	0.65	0.0797
Viscose > Wool	0.23	0.7436

The ‘>’ sign indicates temperature comparison for two different fiber types.

**Table 5.** Tukey’s HSD each pair summary for folding procedure effect in the temperature increase for Experiment 3.

Fiber Comparison	Difference	p-Value
Viscose > Polyester	6.9	<0.0001
Wool > Polyester	6.8	<0.0001
Cotton > Polyester	6.3	<0.0001
Viscose > Cotton	0.54	<0.0001
Wool > Cotton	0.48	<0.0001
Viscose > Wool	0.06	0.0975

The ‘>’ sign indicates temperature comparison for two different fiber types.

### 3.4. Dynamic Hot Plate Test

In this test, the change in the heat flux input of the increase in the hot plate as a function of the humidity is of interest. Fabrics generate heat as humidity increases; therefore, the hot plate’s heat flux input would be reduced to keep the temperature constant. The area of reducing heat flux over time represents the heat that is generated by the fabrics when they absorb moisture from the ambient environment. Since each fabric had a different thermal resistance, all heat flux curves were calibrated to the same baseline. The ambient temperature and RH are shown below in Figure 8. The ambient temperature was stable and well controlled, while the ambient RH took some time to increase. The chamber took 23 min to increase the RH from 50% to 80% and 40 min to reach 85%.

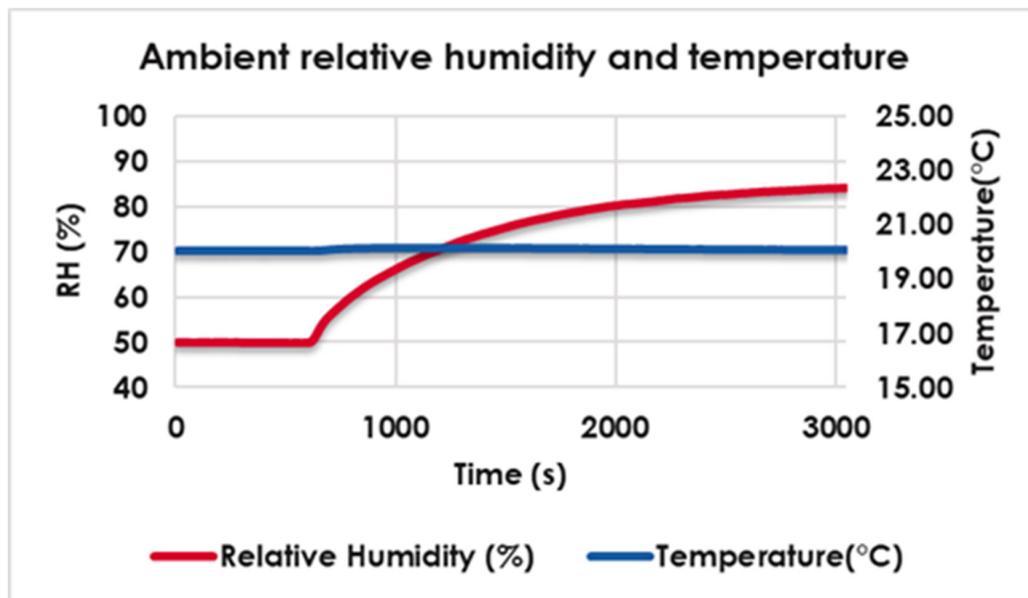


Figure 8. Chamber RH% and temperature during the hot plate test.

The change in the heat flux of the fabrics when ambient RH increased from 45% to 85% is shown in Figure 9. Each curve of the fabric samples showed 10 min (600 s) heat flux at a stable state and 40.8 min (the 2450 s) after an increase in RH. For all fabrics, a decrease in the heat flux could be observed, which means that the fabrics generate heat when switching the RH from low to high. The curves had some noise and fluctuation, especially at the end of each trial, which was caused by the slow increase in RH. Wool fabrics had a relatively larger peak than polyester fabrics and cotton fabrics. Viscose had a peak pattern close to wool fabrics. To further analyze the curve, the peak area was calculated for each fiber type.

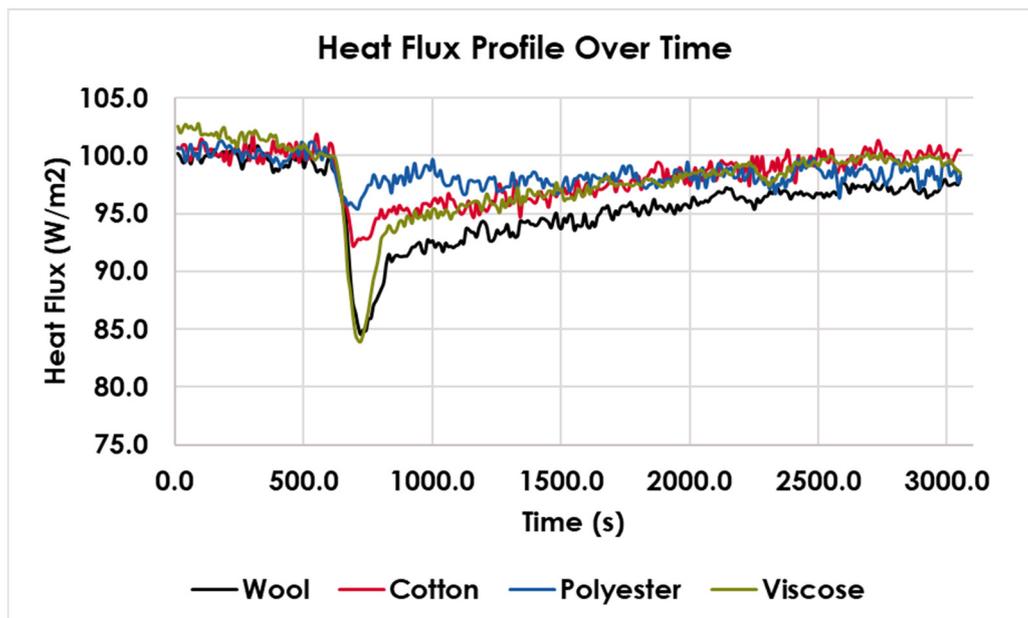


Figure 9. Heat flux change of fabrics when changing from low relative humidity to high relative humidity.

The integration of the heat flux peak over time is the heat generated by the fabrics. Figure 10 shows the integration results of the heat flux curves, and detailed heat release data are shown in Table 6. The average heat flux value for 10 min before switching RH

was calculated as the baseline of steady state at 45% RH. The difference value between the baseline and each data point was determined and multiplied by the 10 s value as the data were recorded at every 10 s interval. The integration area obtained from the heat flux curve would be the heat release due to the humidity change. It is evident that wool and viscose had a higher heat release than cotton and polyester, and had significant differences.

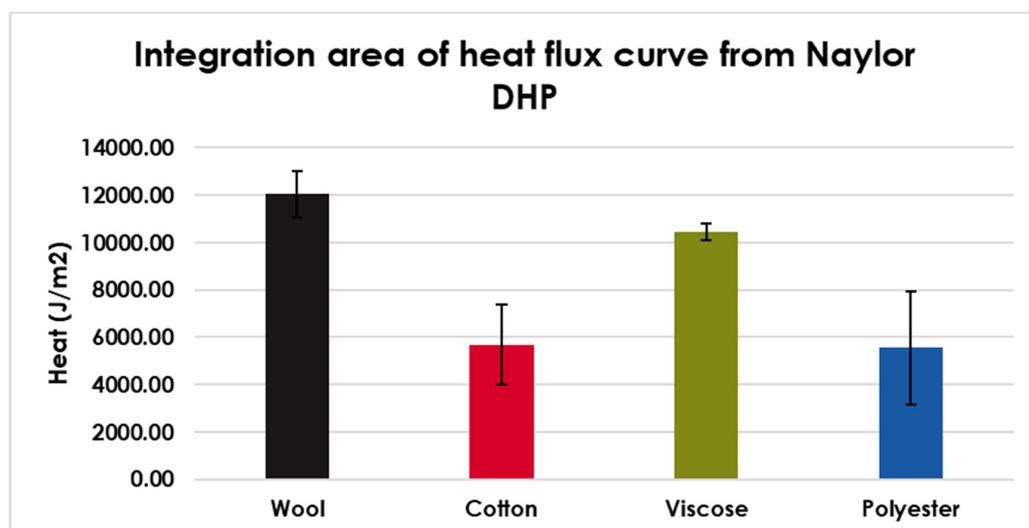


Figure 10. Integration area under the heat flux change curve.

Table 6. Heat release of fabrics from hot plate tests.

Sample ID	Average Heat Release (J/m <sup>2</sup> )	Standard Error
Wool	12,050	973
Cotton	5686	1666
Viscose	10,447	364
Polyester	5541	2408

### 3.5. Comparison of ISO 16533 Test, Dynamic Hot Plate Test, and Relationship with Moisture Regain Rate Test

Both test methods showed the exothermic phenomenon of textile materials when switching the RH from low to high. ISO 16533 used the temperature increase to interpret the exothermic phenomenon, while the DHP test method used heat flux (which can also be converted to generated heat). The temperature increase could only determine the highest temperature in a certain time period but was not able to determine the heat generated in the long term, as the temperature increases were very small after 60 min. Furthermore, ISO 16533 only measured the surface of a single point on the specimen, leading to a great variance (Figure 7). The DHP method, on the other hand, measured the average heat release on a  $60 \pm 1 \text{ cm} \times 60 \pm 1 \text{ cm}$  scale and generated more heat with that much larger sample. However, ISO 16533 had an easy-to-build setup and was easily made and inexpensive. Furthermore, it was easier to control the RH at low temperatures and implement a rapid change in RH ('step change'). The DHP method had higher equipment requirements. The increase in RH was much slower and not as well controlled at lower temperatures. It is envisioned that an additional, in-depth, mathematical analysis of the DHP data, with a slower humidity increase, may be able to separate the rate of change and convert it to data similar to the ISO16533 method.

The goal of ISO 16533 and DHP was to connect the known differences between static fiber properties (regain) and the dynamic exothermic properties of fabrics. As it is widely known and accepted from the literature, the regain for wool (water vapor uptake from

“bone-dry” to 65% RH) is around 13–15 g/100 g dry fiber, whereas the regain for cotton is around 7–8 g/100 g dry fiber. These values could be considered static as they represent the steady state in these fabrics after equilibrating with the environment, which may take many hours, up to 24 h. Water vapor uptake is an exothermic effect; therefore, a larger water vapor uptake by wool, compared to cotton, should result in a significantly higher increase in temperature.

The ISO 16533 method exists as a standard and is relatively simple in its setup and procedure. Yet, there still seem to be some challenges in repeatability depending on container size and the effects of humidity changes. In addition, the current method suggests measuring temperature changes (peak temperature) only, which is shown to be a very poor estimate of exothermic effects. By determining the area under the curve, possibly with some data processing to reduce data noise and adjust for baseline drift, good results are obtained for the different fiber types. Thus, this method provides a direct measure of energy release that can be used to describe the potential for the heating and cooling of the wearer and predict the results for human physiological comfort.

However, the DHP method has the downside of using heat flux and plate temperature as controls, and thus requires advanced modeling to obtain reliable and accurate results. As an alternative, the possibility of running the hot plate tests in a “constant heat flux mode” can be suggested. Under these circumstances, the plate temperature is no longer constant, but the plate generates a constant heat flux, and the corresponding temperature changes will be measured. The downside of this method is that the plate is not equal to human skin, so the resulting temperatures cannot be directly considered equal to changes in skin temperature. However, the temperature changes are likely very strongly correlated, as the temperature change is a fundamental physics response, but the size of the response depends on the exact thermal material properties. As a consequence, such experiments might better reflect our human experimental data. Furthermore, as heat flux is fixed, any resulting transient change in plate temperature (and fabric temperature) is likely to be directly correlated to the exotherm energy released.

There seems to be another challenge with the hot plate data. The fabrics on the hot plate have a temperature between the plates and air temperature, usually around 26–27 °C on the outside and 30–31 °C on the inside of the fabric, thus an average temperature around 28–29 °C. As a consequence, the relative humidity of the fabric is different from the air. With the RH in the air changing from 45% to 85% at 20 °C, the change in RH at the same vapor pressure at 29 °C (fabric temperature) would change from 16.5% to 47.5%, thus exhibiting a much smaller increase in RH (30% change instead of 40% change). Furthermore, this would be in the flatter parts of the standardized regain curve; thus, the expected regain uptake in the experiment was much lower than the expected effect associated with an RH change from 45% to 85%. The actual fabric temperature and associated RH must be calculated to predict exothermic effects. This issue seems not to have been addressed by Naylor in their method and needs further study.

However, this would assume that the water vapor uptake process is instantaneous, and it can be expected from transport physics (diffusion, absorption, and forced convection) phenomena that the process is dynamic in reality (i.e., it will take time to absorb moisture). Thus, dynamic tests should be conducted to determine practically relevant differences between wool, viscose, cotton, and polyester as they happen with time after a change in humidity, as that is what a wearer would perceive, not just the static endpoints.

The fabric test methods described in this paper have not led to a clear and convincing test procedure that demonstrates the difference between hygroscopic fibers for practical applications. This seems to be due to the complex time dependencies and dynamics in this multistep adsorption process. Even with a ‘perfect’ step change in the RH of the environment, the water vapor uptake of the fabric samples, due to the regain effect, is not instantaneous but takes time. The consequence here is that water vapor uptake needs time; after that, temperature build-up and cooling-down are also processes that have specific

time constants. More studies will be needed to develop a set of test methods that allow a good prediction of these effects and their relevance to human thermal comfort.

#### 4. Conclusions

The conclusions are that both the ISO method and the Naylor DHP test have limitations but potentially have the capacity to show fiber-type differences in exothermic response. Further, more detailed data analysis and mathematical modeling of the diffusion processes that guide the water vapor uptake (regain) with its exothermic effects and the convective processes that drive the cooling of the fabric will allow us to reliably determine the differences between fiber types, especially at lower temperatures. Furthermore, the ISO16533 test may allow for variations with larger samples and longer exposure times to determine other fiber-type differences; this slow response in temperature is unusual if measured at room temperature, as the low-temperature responses are much slower than those measured at room temperature. The area under the curve analysis could provide a better idea of the energy release for the ISO method. On the other hand, for the Naylor test, the presence of a temperature gradient affects the actual local RH of the fabric, leading to a much smaller RH change in the fabric, and, therefore, too many smaller measured effects compared to what is expected from the large environmental RH change, and thus these issues need to be addressed.

**Author Contributions:** Conceptualization, F.A. and E.D.; methodology, F.A. and E.D.; formal analysis, F.A.; investigation, F.A.; writing—original draft preparation, F.A.; writing—review and editing, F.A. and E.D.; supervision, E.D.; project administration, E.D.; funding acquisition, E.D. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by Australian Wool Innovation (4500012475), and the APC was funded by Australian Wool Innovation.

**Data Availability Statement:** The corresponding author can provide the data used in this study upon request.

**Acknowledgments:** The authors acknowledge technical support provided by Fangfang Wang and Monika Mali.

**Conflicts of Interest:** The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

#### References

1. Das, A.; Alagirusamy, R. *Science in Clothing Comfort*; Woodhead Publishing Limited: Oxford, UK, 2010.
2. Haghi, A.K. *Heat & Mass Transfer in Textiles*, 2nd ed.; WSEAS Press: Athens, Greece, 2011.
3. Cui, Y.; Gao, S.; Zhang, R.; Cheng, L.; Yu, J. Study on the Moisture Absorption and Thermal Properties of Hygroscopic Exothermic Fibers and Related Interactions with Water Molecules. *Polymers* **2020**, *12*, 98. [[CrossRef](#)]
4. Hai-bo, H.U.; Lu, Q.I. The Development and Application of Hygroscopic and Exothermic Fiber. *Synth. Fiber China* **2010**, *3*, 13–16.
5. Gao, S.; Cui, Y.; Yao, W.; Jing, M.; Liu, L.; Zhang, R. Analysis of thermal and wet comfort properties of hygroscopic and exothermic knitted fabrics. *Text. Res. J.* **2022**, *92*, 2327–2339. [[CrossRef](#)]
6. Hearle, J.W.; Morton, W.E. *Physical Properties of Textile Fibres*, 4th ed.; CRC Press: Boca Raton, FL, USA, 2008.
7. Wiegerink, J.G. Effects of Drying Conditions on Properties of Textile Yarns. *Text. Res.* **1940**, *10*, 493–509.
8. Darling, R.C.; Belding, H.S. Moisture Adsorption of Textile Yarns at Low Temperatures. *Ind. Eng. Chem.* **1946**, *38*, 524–529. [[CrossRef](#)]
9. King, G. Permeability of keratin membranes to water vapour. *Trans. Faraday Soc.* **1945**, *41*, 479–487. [[CrossRef](#)]
10. Li, Y.; Luo, Z.X. Physical Mechanisms of Moisture Diffusion into Hygroscopic Fabrics during Humidity Transients. *J. Text. Inst.* **2000**, *91*, 302–316. [[CrossRef](#)]
11. Mackay, B.H.; Downes, J.G. 27—The kinetics of water-vapour sorption in wool: Part II: Results obtained with an improved sorption vibroscope. *J. Text. Inst.* **1969**, *60*, 378–394. [[CrossRef](#)]
12. Berglund, L.G.; Gonzalez, R.R. Evaporation of sweat from sedentary man in humid environments. *J. Appl. Physiol.* **1977**, *42*, 767–772.
13. Davis, J.-K.; Bishop, P.A. Impact of Clothing on Exercise in the Heat. *Sports Med.* **2013**, *43*, 695–706. [[CrossRef](#)]

14. Pascoe, D.D.; Bellingar, T.A.; McCluskey, B.S. Clothing and exercise. II. Influence of clothing during exercise/work in environmental extremes. *Sport. Med.* **1994**, *18*, 94–108. [[CrossRef](#)] [[PubMed](#)]
15. Barker, R. Evaluating the heat stress and comfort of firefighter and emergency responder protective clothing. In *Improving Comfort in Clothing*; Elsevier: Sawston, UK, 2011; pp. 305–319.
16. Barker, R.L. From fabric hand to thermal comfort: The evolving role of objective measurements in explaining human comfort response to textiles. *Int. J. Cloth. Sci. Technol.* **2002**, *14*, 181–200. [[CrossRef](#)]
17. Huang, J. Sweating guarded hot plate test method. *Polym. Test.* **2006**, *25*, 709–716. [[CrossRef](#)]
18. Huang, J. Review of heat and water vapor transfer through multilayer fabrics. *Text. Res. J.* **2016**, *86*, 325–336. [[CrossRef](#)]
19. Kaplan, S.; Okur, A. A new dynamic sweating hotplate system for steady-state and dynamic thermal comfort measurements. *Meas. Sci. Technol.* **2010**, *21*, 085701. [[CrossRef](#)]
20. Kim, E.A.; Yoo, S.; Kim, J. Development of a Human-Clothing-Environment simulator for dynamic heat and moisture transfer properties of fabrics. *Fibers Polym.* **2003**, *4*, 215–221. [[CrossRef](#)]
21. Naylor, G.R. Measurement of the dynamic moisture buffering potential of fabrics. *Text. Res. J.* **2019**, *89*, 739–747. [[CrossRef](#)]
22. *ISO 16533:2014*; Textiles—Measurement of Exothermic and Endothermic Properties of Textiles under Humidity Change. ISO: London, UK, 2014.
23. Naylor, G.R.; Wilson, C.; Laing, R.M. Thermal and water vapor transport properties of selected lofty nonwoven products. *Text. Res. J.* **2017**, *87*, 1413–1424. [[CrossRef](#)]
24. Cooke, B. 2-The physical properties of weft knitted structures. In *Advances in Knitting Technology*; Au, K.F., Ed.; Woodhead Publishing: Sawston, UK, 2011; pp. 37–47. [[CrossRef](#)]
25. Gao, H.; Deaton, A.S.; Barker, R. A new test method for evaluating the evaporative cooling efficiency of fabrics using a dynamic sweating hot plate. *Meas. Sci. Technol.* **2022**, *33*, 125601. [[CrossRef](#)]
26. Das, B.; Das, A.; Kothari, V.K.; Fanguiero, R.; De Araújo, M. Effect of fibre diameter and cross-sectional shape on moisture transmission through fabrics. *Fibers Polym.* **2008**, *9*, 225–231. [[CrossRef](#)]
27. Jiang, X.; Bai, Y.; Chen, X.; Liu, W. A review on raw materials, commercial production and properties of lyocell fiber. *J. Bioresour. Bioprod.* **2020**, *5*, 16–25. [[CrossRef](#)]

**Disclaimer/Publisher’s Note:** The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.