

Article

Surface Free Energy Analysis Using the Washburn Capillary Rise Method to Improve the Accuracy of Measuring Carbon Fiber Interfacial Properties [†]

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Highlights

What are the main findings?

- The Washburn capillary rise method was optimized by selecting a capillary-rise interval with minimal deviation to improve the reliability of carbon fiber wettability measurements;
- Among various fiber lengths, 2-inch carbon fibers showed the smallest deviation in contact angle and surface free energy due to favorable axial alignment and reduced pore heterogeneity.

What are the implications of the main findings?

- Controlling the carbon fiber length and packing structure is critical for obtaining accurate and reproducible surface free energy measurements using the Washburn method.
- The proposed measurement strategy provides a reliable criterion for evaluating carbon fiber surface wettability, contributing to improved understanding and optimization of fiber–matrix interfacial interactions in composites.

Abstract

The wettability of a carbon fiber surface is an important factor that determines the strength of its bonding with matrices, and hence, an optimized criterion is required to accurately measure the wettability. In this study, the Washburn capillary rise method was used to select the capillary constant with the minimal deviation among various carbon fiber lengths, and it was applied to determine the contact angle and surface free energy of each carbon fiber length according to the wetting liquid. The smallest deviation in the contact angle was observed for a carbon fiber length of 2 inches, and this observation was attributed to the pores in the fibers and the orientation of the carbon fibers packed inside the column. By reducing the number of pores and achieving favorable packing, the surface free energy of carbon fibers can be measured with a high degree of accuracy, contributing to an improved understanding of fiber–matrix interactions.



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Keywords: carbon fiber; Washburn capillary rise method; capillary constant; contact angle; surface free energy

1. Introduction

Carbon fiber is a high-performance material that offers several advantages, such as high specific strength, high elastic modulus, remarkable wear resistance, and lightness, and is an ideal reinforcing material for fabricating fiber-reinforced composites. Because of these advantages, carbon fiber-reinforced plastic is used in various fields such as sports, transportation, mobility, and aerospace [1–4]. However, because of the chemically inert and dispersive nature of carbon fibers, the interfacial adhesion between a carbon fiber and matrix is limited, and thus, the interfacial adhesion with the matrix can be improved by enhancing the wetting properties of the carbon fiber through surface treatment [5–9]. Thus, the surface wettability of carbon fibers, one of the most crucial surface characteristics, plays an important role in determining the quality of the final composite interface [10–12]. Consequently, an optimized metric is required to accurately measure the surface wettability of carbon fibers.

In general, the wettability of a solid surface is evaluated by measuring the contact angle, which is the angle formed by a liquid in contact with the surface. Various methods, including the sessile drop [13–15], Wilhelmy plate [16–20], Washburn capillary rise (WCR) [21–23], and dynamic contact angle (DCA) [24–29] methods, are used to measure the contact angle. Among them, the sessile drop method is the most commonly used for contact angle measurements; in this method, the contact angle is measured by dropping a liquid droplet onto a surface and subsequently measuring the angle formed between the liquid–gas and liquid–solid interfaces. However, because the surface of a carbon fiber is uneven, and its diameter is small (generally, $\sim 7\ \mu\text{m}$), using the sessile drop method to measure the contact angle formed by a liquid droplet on a carbon fiber is very difficult. Therefore, several studies have been conducted to develop experimental methods for measuring the static and dynamic contact angles between carbon fibers and liquids [30–35]. The single fiber measurement method using a DCA analyzer has been widely adopted because of its simple experimental approach; however, it is sensitive to various conditions, such as carbon fiber diameter and the surrounding environment (temperature, time, volume, density, gravity, surface roughness, laboratory environment, etc.).

The WCR method can be used to measure the surface free energy of carbon fiber. This method involves filling a column with a powder and allowing a liquid to penetrate through the pores of the powder. By measuring the wetting speed, the contact angle between the filled particles and liquid can be determined, rendering it an effective method for measuring carbon fiber wettability [36–38]. Generally, the WCR method involves filling the inside of the column with a powder; however, as a fiber is considered a porous medium, it can be approximated as a capillary bundle. In other words, carbon fiber wettability can be evaluated through permeability and capillary tests [39,40]. In the WCR method, chopped carbon fibers act as a porous medium; however, the packing density and orientation of fibers strongly affect the capillary rise behavior [41,42]. When fibers are excessively short or long, the resulting pore structure becomes non-uniform due to random entanglement or bridging, respectively. This structural inhomogeneity can cause deviations in the effective pore radius, thereby impacting the contact angle calculation. Similar observations have been reported in studies where pore morphology and anisotropic packing significantly influenced the accuracy of wettability measurements using the Washburn approach [23,40].

Therefore, reducing the influence of irregular pore formation through controlled fiber packing is critical to improve the reproducibility of surface free energy calculations.

In this study, the WCR method was used to select the capillary constant interval with minimal deviation along the carbon fiber length, and it was applied to evaluate the contact angle and surface free energy along the carbon fiber length according to the wetting liquid.

2. Materials and Methods

2.1. Materials

The primary materials used for contact angle measurements were polyacrylonitrile (PAN)-based carbon fibers without any surface treatment or sizing (H2550, 12K, Hyosung Adv. Mater., Seoul, Republic of Korea) and n-hexane ($\text{CH}_3(\text{CH}_2)_4\text{CH}_3$, 95%, Daejung, Siheung, Republic of Korea). Diiodomethane (CH_2I_2 , 99%, Sigma-Aldrich®, St. Louis, MO, USA) and distilled water were used as the wetting liquids. Table 1 lists the surface free energy of the liquids, along with the polar and dispersive components of the surface free energy.

Table 1. Characteristics of test liquids.

Test Liquid	γ_L (mN/m)	γ_L^d (mN/m)	γ_L^p (mN/m)
n-hexane	18.4	18.4	0
Distilled water	72.8	21.8	51
Diiodomethane	50.8	50.42	0.38

To evaluate the surface free energy of carbon fiber, fibers of various lengths (0.5, 1, 2, and 3 inches) and uncut fibers were prepared. Before conducting measurements, each fiber was oven-dried at 80 °C for 24 h to minimize the effect of foreign substances and moisture on the results. The dried fibers were stored in a desiccator and equilibrated in the measurement environment prior to analysis. All measurements were performed sequentially within a short period under stable laboratory conditions at 25 °C and a relative humidity of 40–50%.

2.2. Carbon Fiber Contact Angle Measurement

The wettability of the prepared carbon fiber samples was evaluated using the Washburn capillary rise method with a tensiometer (K100, Krüss GmbH, Hamburg, Germany) equipped with a powder sample column, and a schematic of the experimental setup is shown in Figure 1. To prevent fiber loss during measurement, a stainless steel mesh and filter paper were installed at the bottom of the column. For each test, 2 g of carbon fibers of a given cut length (0.5, 1, 2, or 3 inches, and uncut) were packed into a cylindrical column with an inner diameter of 1.2 cm and a length of 5 cm (corresponding to a volume of approximately 5 mL) without compression. The sample column was automatically lowered until it contacted the liquid surface, and the mass uptake was recorded in real time over a 300 s interval. Each measurement was repeated at least five times to ensure reproducibility. The Washburn equation [43] was used to measure the contact angle:

$$h^2 = \frac{R\gamma_L \cos \theta}{2\eta} t \quad (1)$$

where h , R , γ_L , θ , t , and η are the height of the liquid rising with respect to time, effective pore radius of the packed carbon fiber column, liquid surface tension, contact angle between the solid and liquid, contact angle, measurement time, and liquid viscosity, respectively. Using Equation (1), the average pore radius can be calculated by measuring the wetting height of solids and fully wet liquids such as n-hexane (i.e., $\theta = 0^\circ$ or $\cos \theta = 1$) over time. The following assumptions were adopted for measuring the contact angle between carbon

fiber and liquid using the Washburn equation: First, the particle size and filling level are constant; second, the liquid passing through the column is under turbulent flow; and third, the influence of gravity can be neglected. The relationship between the average pore radius (R) in Equation (1) and the capillary constant (C) can be derived as follows. The mass (m) of the wetting liquid absorbed by the packed column is related to the liquid rise height (h) and the cross-sectional area (A) of the column as

$$m = \rho Ah \quad (2)$$

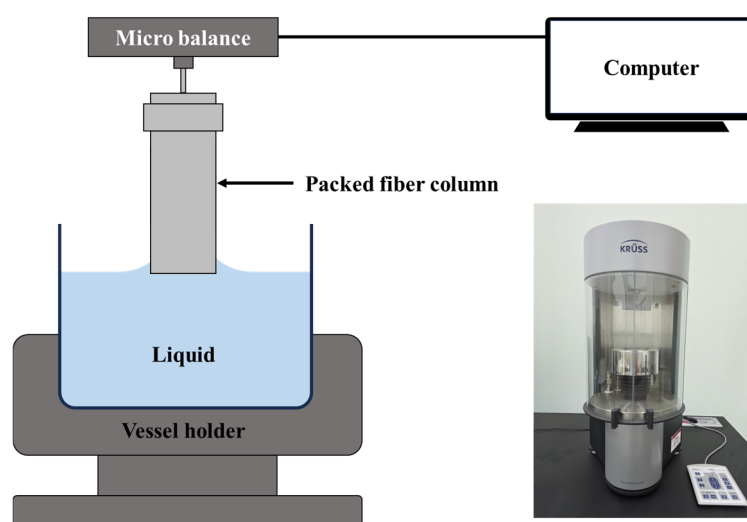


Figure 1. Schematic and photograph of the experimental setup used for contact angle measurements via the Washburn capillary rise method.

By substituting h from Equation (1),

$$m^2 = \rho^2 A^2 h^2 = \rho^2 A^2 \frac{R \gamma_L \cos \theta}{2\eta} t \quad (3)$$

The capillary constant C is therefore defined as

$$C = \frac{1}{2} A^2 R \quad (4)$$

Accordingly, the final relationship is given by Equation (5):

$$\frac{m^2}{t} = \frac{C \rho^2 \gamma_L \cos \theta}{\eta} \quad (5)$$

where m is the mass of the wetting liquid that wets the carbon fiber inside the column, C is the capillary constant, and ρ is the density of the wetting liquid. First, the viscosity, density, and surface tension of n-hexane were recorded. Next, the wetting mass of the liquid was plotted with respect to time, and the slope of this graph was obtained. Subsequently, the capillary constant was calculated using Equation (5). To determine the contact angle between the liquid and filled particles, first, the capillary constant is calculated using the aforementioned procedure; next, the density, viscosity, and surface tension of the liquid used to measure the contact angle are evaluated, and finally, the variations in the wetting mass of the wetting liquid with time are assessed. Physical properties of the test liquids were referenced from the Krüss K100 database and used for all calculations involving the Washburn equation.

The Owens and Wendt equation [44] (Equation (6)) and Young's equation (Equation (7)) were used to derive Equation (8). The contact angle measured on the packed carbon fiber column using the wetting liquid was used in Equation (8) to derive the surface free energy:

$$\gamma_{SL} = \gamma_S + \gamma_L - 2\left(\gamma_S^d \gamma_L^d\right)^{1/2} - 2\left(\gamma_S^p \gamma_L^p\right)^{1/2} \quad (6)$$

$$\gamma_S - \gamma_{SL} = \gamma_L \cos \theta \quad (7)$$

$$\gamma_L(1 + \cos \theta) = 2\left(\gamma_S^d \gamma_L^d\right)^{1/2} + 2\left(\gamma_S^p \gamma_L^p\right)^{1/2} \quad (8)$$

where γ_{SL} is the solid–liquid interfacial free energy, γ_S is the solid surface free energy, γ_L is the surface free energy of the liquid, γ_S^d is the dispersive component of the surface free energy of the solid, γ_L^d is the dispersive component of the surface free energy of the liquid, γ_S^p is the polar component of the surface free energy of the solid, and γ_L^p is the polar component of the surface free energy of the liquid.

3. Results

3.1. Evaluation of Capillary Constant Using Complete Wetting Liquid

Figure 2 shows the mass of n-hexane absorbed and the capillary constants for various carbon fiber lengths. Figure 2a presents the wetting graphs in the time range of 0–300 s, indicating that most samples absorb n-hexane for approximately 40 s. The graph for the 0–20 s period, where a constant slope is observed, was selected for analysis, and the capillary constant in this time range was determined through linear fitting, as shown in Figure 2b. Figure 2c displays the capillary constants obtained for various carbon fiber lengths, confirming that the capillary constant tends to increase with increasing fiber length. For the CF-0.5 sample, the shorter length of the entangled carbon fibers inside the column led to the formation of relatively larger pores compared to the other samples, resulting in the slowest wicking speed and the lowest capillary constant. As the fiber length increased, smaller pores were formed, enhancing the wicking speed of the wetting liquid and thereby increasing the capillary constant. However, a considerable variation in capillary constants was observed across all samples. This is likely due to the inconsistent pore size and distribution formed by randomly packed carbon fibers inside the column. Therefore, the capillary wicking section should be set to a small interval to reduce this deviation.

To reset the capillary wicking interval, the obtained Washburn curves are divided into three sections (Figure 3). The first is the surface-tension section, which is ascribed to the surface tension of the wetting solution at the moment of its contact with the column. The second is the capillary-rise section, wherein the wetting solution fills the pores between the carbon fibers via capillary action. The third is the wetting-liquid-filling section, wherein the wetting solution initially drawn into the pores via capillary action steadily fills the pores. Although previous studies have divided the Washburn curve into three characteristic regions, including an initial surface tension phase, a capillary rise phase, and a saturation phase [23,40], the present study focuses on the capillary rise section (0–0.4 s) to minimize deviation and capture the most consistent wicking behavior across fiber lengths. This procedure reveals that the most reliable data are obtained when the capillary constant in the capillary-rise section, where the wetting liquid is sucked into the pores between the carbon fibers, is measured. Figure 4 shows the reset capillary wicking interval and the resulting capillary constants. As shown in Figure 4a, the CF-long fiber exhibits surface-tension, capillary-rise, and wetting sections, whereas in the remaining samples, capillary-rise and wetting sections form without a surface-tension section. In contrast to shorter carbon fibers, CF-long fibers show a surface-tension section. This is attributed to the dominance of the capillary-rise section over the surface-tension section due to the presence of microscopic

pores. Therefore, the capillary constant from 0 s to 0.4 s is obtained to normalize the capillary wicking interval of all the samples. A comparison between Figures 4b and 2c indicates that the capillary constant shows a similar trend, and the deviation from 0 to 0.4 s is smaller than that from 0–20 s. This result is attributed to the similar lengths of the carbon fibers and column, because of which most of the carbon fibers within the column align axially, thereby minimizing the deviation in the capillary constant.

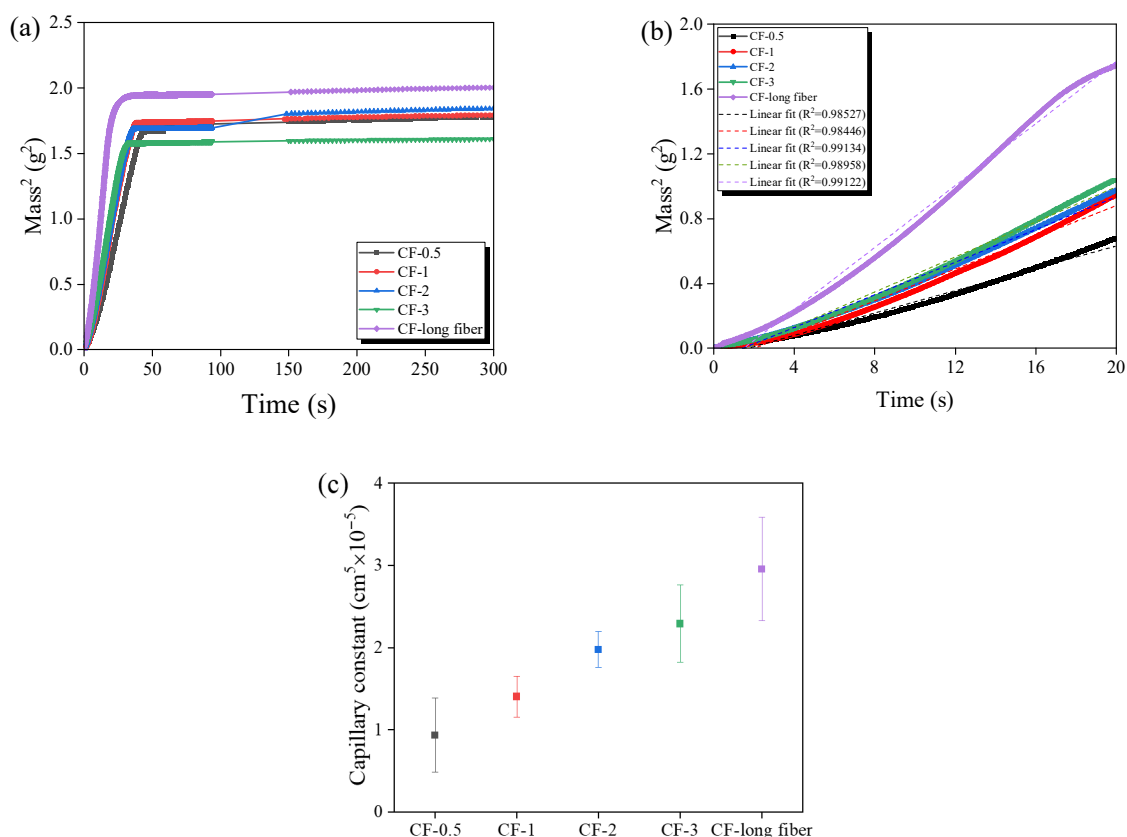


Figure 2. Washburn curves and variations in capillary constants with different carbon fiber lengths over the time ranges of (a) 0–300 s and (b) 0–20 s. (c) Variation in capillary constant with carbon fiber length (calculated from the 0–20 s range).

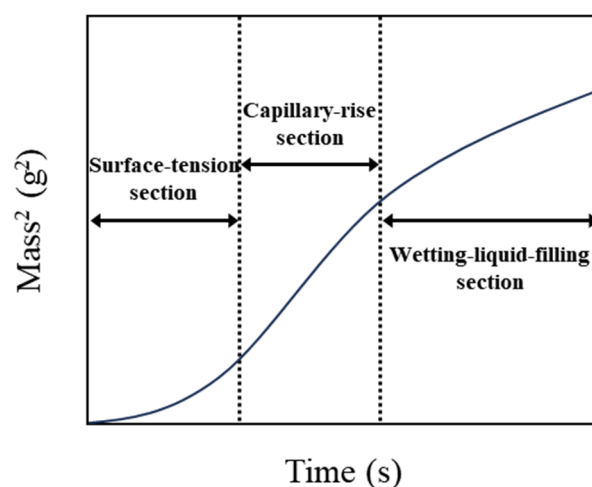


Figure 3. Three stages in the Washburn curve during capillary wicking.

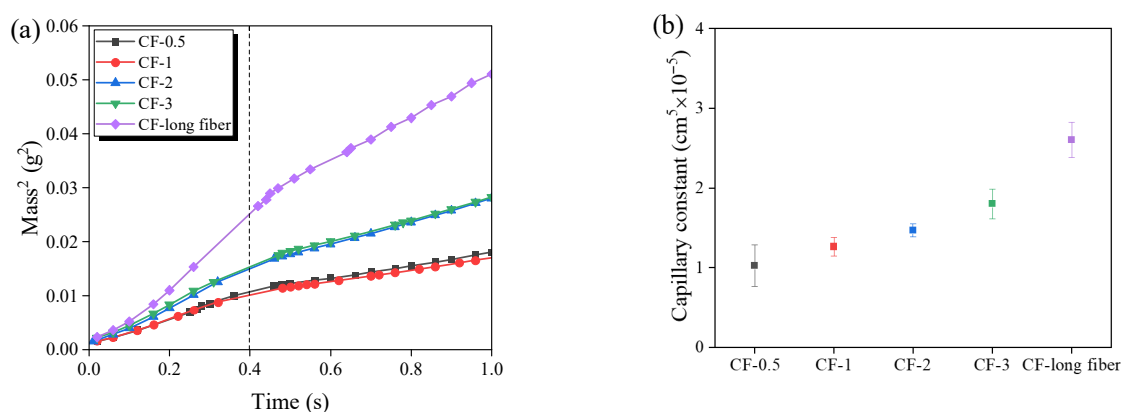


Figure 4. (a) Variation in Washburn curves obtained using n-hexane as the wetting liquid for different carbon fiber lengths in the time range of 0–1 s. (b) Variation in capillary constant with carbon fiber length (calculated from the 0–0.4 s range).

3.2. Contact Angle Measurement and Surface Free Energy Calculation According to Carbon Fiber Length

To investigate the variation in surface free energy with the length of the carbon fiber samples, contact angle measurements were performed using distilled water and diiodomethane as wetting liquids. Figure 5 shows the sorption graph and contact angle obtained using distilled water for the time range of 0–0.4 s, derived using the capillary constant obtained in this time range.

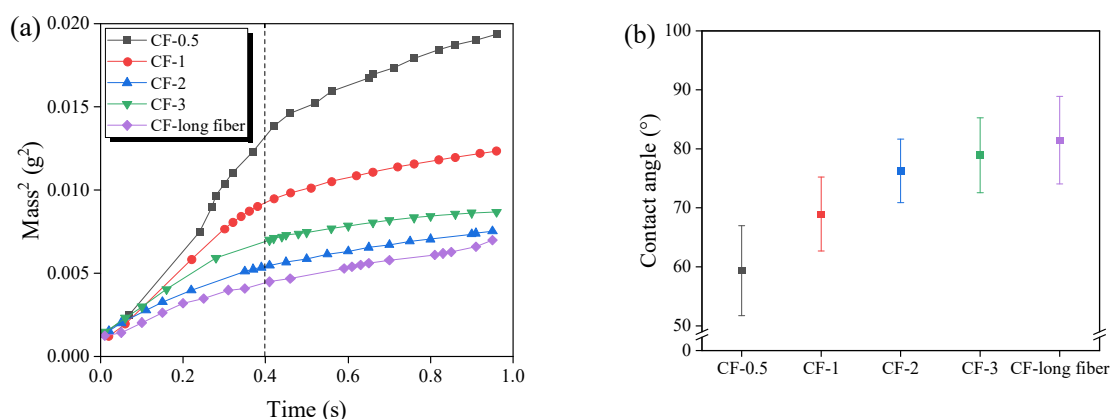


Figure 5. (a) Variations in Washburn curves obtained using distilled water as the wetting liquid for various carbon fiber lengths in the time range of 0–1 s. (b) Variation in contact angle with carbon fiber length (calculated from the 0–0.4 s range).

The results obtained using distilled water differed significantly from those obtained using n-hexane, primarily due to interfacial interactions. As a non-polar solid, carbon fiber exhibits low affinity toward polar liquids such as distilled water, resulting in repulsive interfacial forces that impede capillary uptake. When the packing density of carbon fibers increases, the pore structure becomes more compact, which reduces the penetration rate of distilled water and lowers the slope of the Washburn curve, thereby increasing the contact angle.

Conversely, shorter carbon fibers tend to pack more randomly, forming larger and irregular pores that facilitate faster liquid uptake. This leads to an increased slope and subsequently a lower apparent contact angle. Among all tested samples, the CF-2 sample exhibited the smallest deviation in contact angle. This is attributed to the fact that the 2-inch fiber length closely matches the length of the sample column, resulting in favorable

axial alignment and a more uniform internal structure. A similar trend was observed in the experiments using n-hexane as the wetting liquid.

Figure 6 shows the sorption graph and contact angle obtained using diiodomethane as the wetting liquid. In contrast to distilled water, the contact angle of diiodomethane is obtained within the 0–10 s interval. Because of the high wicking speed and density of diiodomethane in the 0–0.4 s interval, the $\cos \theta$ value exceeds 1 (Equation (5)), and the contact angle cannot be determined. Therefore, the contact angle is measured by choosing the interval wherein $\cos \theta$ does not exceed 1 (0–10 s). Similarly to the contact angle measured using distilled water, that measured using diiodomethane as the wetting liquid decreases as the length of the carbon fibers decreases. Notably, the 2-inch carbon fibers, whose length is the closest to the column length, exhibit minimal deviation in their contact angle. Table 2 presents the capillary constants and contact angles measured for different carbon fiber lengths.

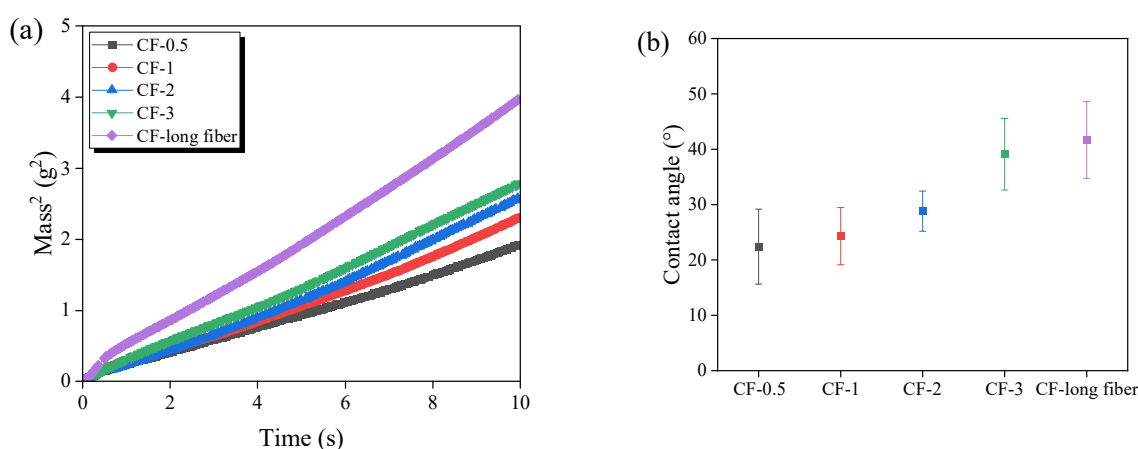


Figure 6. (a) Variations in Washburn curves obtained using diiodomethane as the wetting liquid for various carbon fiber lengths in the time range of 0–10 s. (b) Variation in contact angle with carbon fiber length (calculated from the 0–10 s range).

Table 2. Variations in the capillary constant and contact angle of the wetting liquids with the carbon fiber length (capillary constant calculated from the 0–0.4 s range).

Sample Name	Capillary Constant ($\text{cm}^5 \times 10^{-5}$)	Contact Angle (°)	
		Distilled Water	Diiodomethane
CF-0.5	1.02	59.36 ± 7.63	22.42 ± 6.76
CF-1	1.26	68.96 ± 6.27	24.31 ± 5.15
CF-2	1.47	76.28 ± 5.38	28.81 ± 3.62
CF-3	1.80	78.92 ± 6.34	39.11 ± 6.46
CF-long fiber	2.60	81.48 ± 7.42	41.69 ± 6.95

The surface free energies calculated using distilled water and diiodomethane are presented in Table 3 and Figure 7. The most stable and consistent results were obtained for the 2-inch carbon fiber sample, whereas larger deviations in surface free energy were observed when the fiber length was either shorter or longer than 2 inches. This trend is attributed to variations in contact angle that arise from differences in fiber packing structure and pore distribution within the column. As shown in Figure S1, short or excessively long fibers tend to form internal entanglement or bridging structures, while the 2-inch fibers exhibit relatively uniform axial alignment within the column. This structural difference explains the reduced pore heterogeneity and improved reproducibility of the 2-inch sample. In particular, non-uniform fiber alignment and inconsistent pore sizes introduce variability

in liquid wicking behavior, ultimately affecting the accuracy and repeatability of surface energy measurements [23,40]. In practice, inherent heterogeneities in pore size distribution, pore connectivity, and local fiber orientation remain, particularly when working with polar or high-surface-tension liquids such as distilled water or diiodomethane. These liquids are more sensitive to local variations in the pore structure, and even small differences in packing can lead to measurable changes in wicking behavior, contact angle, and surface free energy. Among all samples, the 2-inch carbon fibers exhibited the smallest deviation, which is attributed to the reduced pore size variation and the favorable axial alignment of fibers within the column. Therefore, to minimize pore heterogeneity and obtain consistent measurements of surface energy, it is crucial to control fiber length and ensure uniform alignment during packing.

Table 3. Polar components, dispersive components, and surface free energies for various carbon fiber lengths.

Sample Name	γ_s^d (mN/m)	γ_s^p (mN/m)	γ_s
CF-0.5	39.19 ± 3.98	13.10 ± 4.01	52.29 ± 6.71
CF-1	43.17 ± 2.16	7.25 ± 3.41	50.42 ± 3.05
CF-2	42.25 ± 1.98	4.52 ± 2.50	46.77 ± 2.38
CF-3	37.67 ± 3.66	4.73 ± 3.02	42.40 ± 3.57
CF-long fiber	36.61 ± 4.34	4.18 ± 3.27	40.79 ± 4.13

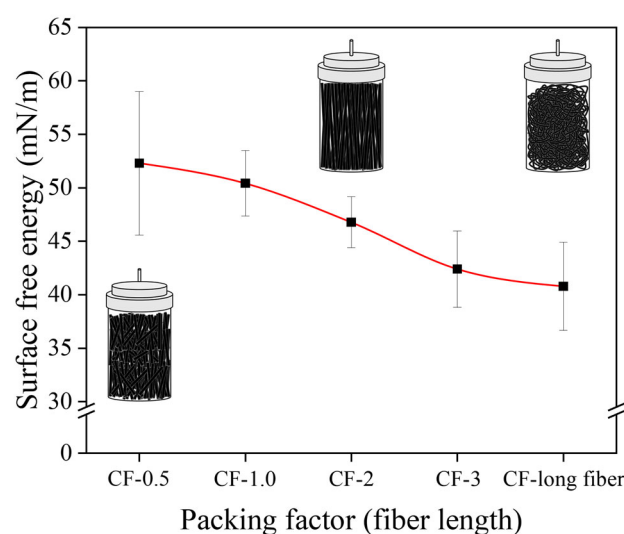


Figure 7. Deviations in surface free energy as a function of carbon fiber length.

4. Conclusions

In this study, the variations in the capillary constant, contact angle, and surface free energy of carbon fibers with different lengths were systematically investigated. When using n-hexane as a wetting liquid, significant deviations in the capillary constant were observed in the wetting-liquid-filling section due to inconsistent pore size and distribution within the packed column. By selecting the capillary-rise section as the measurement interval, the deviation was significantly reduced. Furthermore, contact angle measurements using distilled water and diiodomethane showed the smallest deviation for 2-inch carbon fibers, which was attributed to the alignment of fibers along the column axis due to their similar length to the column. This favorable packing also resulted in the most consistent surface free energy values. These findings demonstrate that aligning carbon fibers axially and minimizing pore heterogeneity during sample preparation are essential for achieving reliable and reproducible surface energy measurements using the Washburn method.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/fib14020016/s1>.

Author Contributions: Conceptualization, W.H.; methodology, W.H. and Y.C.C.; validation, D.-K.K. and W.H.; formal analysis, D.-K.K.; investigation, D.-K.K. and W.H.; data curation, D.-K.K.; writing—original draft preparation, D.-K.K.; writing—review and editing, W.H., Y.C.C., K.-W.K. and B.-J.K.; visualization, D.-K.K.; supervision, K.-W.K. and B.-J.K.; project administration, B.-J.K.; funding acquisition, W.H. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The original contributions presented in this study are included in the article/Supplementary Materials; further inquiries can be directed to the corresponding authors.

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

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