

Article

Development of Transpiration-Type Thermoelectric-Power-Generating Material Using Carbon Nanotube Composite Papers with Capillary Action and Heat of Vaporization

Yudai Kamekawa ^{1,*}, Koya Arai ^{2,†} and Takahide Oya ^{1,†}

¹ Graduate School of Engineering Science, Yokohama National University, Yokohama 240-8501, Japan; oya-takahide-vx@ynu.ac.jp

² Innovation Center, Mitsubishi Materials Corporation, Saitama 330-8508, Japan; araikoya@mmc.co.jp

* Correspondence: kamekawa-yudai-zy@ynu.jp

† These authors contributed equally to this work.

Abstract: A transpiration-type thermoelectric-power-generating paper based on previously developed carbon nanotube (CNT) composite paper, which is a composite material of CNTs and pulp that can generate thermoelectric power, was developed. The newly developed thermoelectric-power-generating material does not require an external high-temperature heat source due to the ability of paper to absorb liquid through capillary action and heat of vaporization generated when the liquid evaporates. The aim of this study is to investigate the feasibility of realizing the transpiration-type thermoelectric-power-generating paper. To begin with, the type of paper used as raw material for the composite paper was examined, and the fabrication process was modified in order to obtain more efficient liquid absorption based on capillary action. Then, the absorbing ability of the liquid was evaluated. Next, the feasibility of thermoelectric power generation using the heat of vaporization was confirmed. Moreover, for more efficient thermoelectric conversion, multisheet structures were also studied. Through several experiments, the material's feasibility was verified, and it was confirmed that more power can be easily obtained through the use of multiple sheets. Specifically, a single sample spontaneously generated a temperature difference of up to 1.7 °C due to the heat of vaporization, generating an electromotive force of 36 μV. From the sample with a five-sheet structure, an electromotive force of 356 μV was obtained at a temperature difference of 2 °C. This material can be used in watery environments, such as rivers, lakes, and hot springs, and is expected to become a new energy-harvesting device.

Keywords: carbon nanotube; thermoelectric power generation; carbon nanotube composite paper; transpiration; capillary action; heat of vaporization



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1. Introduction

Much of the energy consumed is wasted as heat, which, together with the depletion of energy resources, has become a major environmental problem. As a solution to this problem, there is research being conducted on thermoelectric power generation technology that converts waste heat into electricity [1,2]. Metals such as bismuth and tellurium are currently used as materials for thermoelectric power generation; however, they are rare, toxic, fragile, and heavy [3–8]. We developed a thermoelectric-power-generating material that uses our previously developed carbon nanotube composite paper (CNTCP) consisting of CNTs and pulp [9] to address the above issues. It is known that CNTs exhibit high electrical and thermal conductivity [10,11] as well as high thermoelectromotive force [12]. The thermoelectromotive force observed from a thermoelectric material for each 1 K (1 °C) temperature difference is called the Seebeck coefficient. The Seebeck coefficient of bismuth and tellurium alloys is 200 μV/K, and that of CNTs has been shown to be up to 170 μV/K [12]. However, since CNTs usually exist in powder form and are difficult to handle, they are mainly used

by compositing with other materials to make composite materials. Many practical CNT-based materials have been developed, including buckypaper [13], as well as CNT-based thermoelectric-power-generating materials [14]. Many useful devices that use CNTCP have been developed, including dye-sensitized solar cells [15,16], soft actuators [17], electromagnetic shielding [18,19], and transistors [20]. The first thermoelectric-power-generating paper appeared in 2017 [21]. Previous studies confirmed that the thermal conductivity of CNTCP is low, about 1.5 W/m·K [22]. Since low thermal conductivity and high electrical conductivity are generally desired for thermoelectric devices, it can be concluded that CNTCP is suitable for thermoelectric power generation. Research has also made it possible to fabricate high-performance thermoelectric-power-generating paper [23].

In the natural world, plants absorb water from the ground in the process of transpiration [24–28], and use the heat of vaporization [29] when they release it from their leaves [30–32]. In other words, plants create temperature differences through transpiration. We propose that by combining the “capillary action” [33,34] of paper [35–37], base material of CNTCP, and “heat of vaporization,” a temperature difference can be obtained between the edge of the paper soaked in liquid and the opposite edge where the liquid evaporates, as if transpiring, thus requiring no heat source. This represents the world’s first form of thermoelectric power generation and energy harvesting that is possible using CNTCP, which have the functions of both CNTs and paper.

We evaluated the ability of CNTCP to absorb liquid, especially water, and generate a temperature difference using the heat of vaporization in order to confirm its feasibility as transpiration-type thermoelectric-power-generating paper. We also explored methods to improve sample performance and power generation ability through the use of multiple CNTCPs.

2. Experimental Section

2.1. CNTCP Fabrication Method

The procedure for fabricating CNTCP is based on the traditional Japanese *washi* paper fabrication method [9,23]. First, the pulp and CNT dispersions are prepared and mixed. The pulp dispersion is a mixture of pulp, which is the raw material of paper, in pure water, and the CNT dispersion is CNTs dispersed in pure water with a dispersant by ultrasonication (Figure 1a). Next, as shown in Figure 1b, we use a fine net to strain the water from CNTCP. Finally, as shown in Figure 1c, we carry out heat pressing to shape the paper. By following this process, CNTCP can be fabricated quite easily. In previous studies [9,15,17], we have already confirmed that CNTs indeed exist in paper by using scanning electron microscopy (SEM), Raman spectroscopy, and other methods. In this study, we used 20 mL of pure water, 15 mg of single-walled CNTs (ZEONANO SG101, Zeon Nano Technology Co., Ltd., Tokyo, Japan), and 100 mg of sodium dodecyl sulfate (SDS) as the dispersant to prepare the CNT dispersion. We chose SG101-CNTs because we were able to obtain good Seebeck coefficients of about 30 $\mu\text{V}/\text{K}$ for CNTCP (under dry condition) in our previous study [23] and used it in this study. We used 100 mL of pure water and 100 mg of pulp to prepare the pulp dispersion. With the above quantities, a $3.0 \times 1.5 \text{ cm}^2$ sheet of paper can be fabricated. Although the obtaining values will vary somewhat depending on the chosen fabrication conditions, CNTCP with an electrical conductivity of around $1.5 \times 10^3 \text{ S/m}$, a thermal conductivity of around 1.5 W/m·K as described in Introduction, a Seebeck coefficient of around 35 $\mu\text{V}/\text{K}$, a power factor of around 1.9 $\mu\text{W}/\text{m}\cdot\text{K}^2$, and a thickness of around 0.2 mm can be easily prepared by using the fabrication method described above.

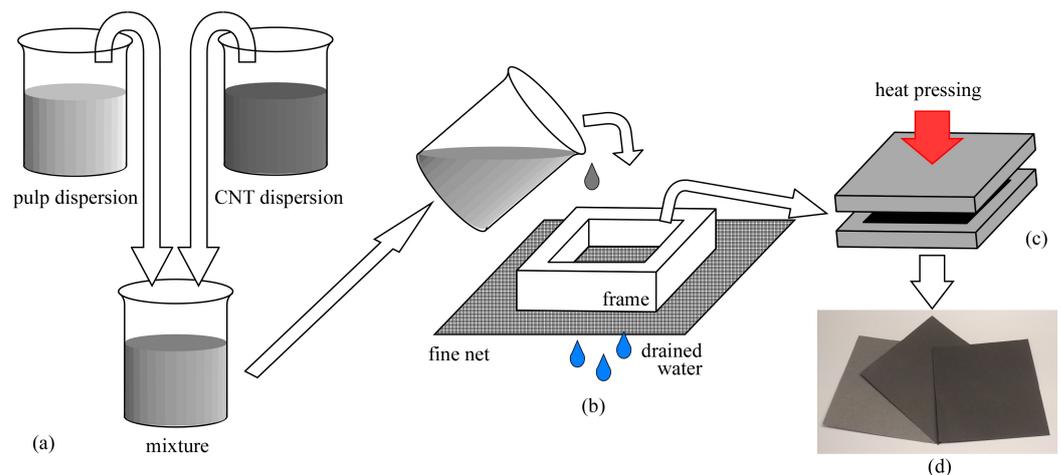


Figure 1. CNTCP fabrication method: (a) preparing mixed solution, (b) straining water with fine net, and (c) heat pressing. (d) CNTCP samples (color differences were caused by amounts of CNT they contained) (from Ref. [23] under License CC BY 4.0).

2.2. Evaluation of Liquid Absorption Ability

To generate electricity, it is first necessary to confirm the ability of CNTCP to absorb liquid and obtain sufficient performance. Therefore, we evaluated several CNTCP samples of different pulp materials and drying methods to determine the optimal absorption ability of CNTCP. Previous CNTCP samples were fabricated using eucalyptus pulp and a heat press as the drying method. In this study, we fabricated CNTCP samples using eucalyptus or rayon as the pulp material and a heat press or oven as the drying method. By changing the above conditions, four types of CNTCP samples were fabricated, as shown in Table 1. These samples were placed in a bottle filled with pure water, as shown in Figure 2. The absorption abilities under different conditions were compared by measuring the changes in overall weight.

Table 1. Pulp material and drying method of samples. Material and drying method for sample 1 were the same as in previous study.

Sample No.	Pulp Material	Drying Method
1	Eucalyptus	Heat pressing
2	Eucalyptus	Oven
3	Rayon	Heat pressing
4	Rayon	Oven

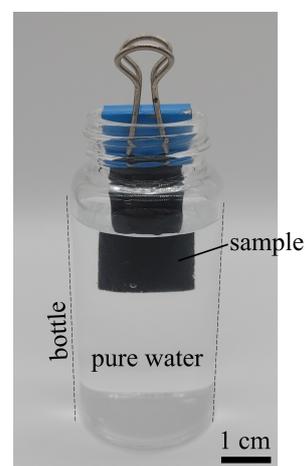


Figure 2. For evaluating liquid absorption ability.

2.3. Measuring Transpiration-Type Thermoelectric Power Generation

To conduct thermoelectric power generation without a heat source, one end (bottom) of a sample was placed below the water level in a jar, and the other end (top) was fixed above the water level, as shown in Figure 3. The temperature at the top was expected to be lower than at the bottom due to the heat of vaporization caused by the evaporation of water absorbed through capillary action. To confirm the occurrence of temperature differences within the sample, two-channel thermometer probes (LR5021, HIOKI E.E. CORPORATION, Ueda, Japan) were placed at the top and bottom of the sample, and the temperature of the sample surface was observed with a thermal imaging camera (FLIR One Pro, Teledyne FLIR LLC, Wilsonville, OR, USA). We did not force the liquid to evaporate by blowing air on the sample to obtain more heat of vaporization. The temperature at the bottom of the sample is calculated as T_b [$^{\circ}\text{C}$], and that at the top is calculated as T_t [$^{\circ}\text{C}$]. The Seebeck coefficient is calculated from the difference in temperature and the generated electromotive force.

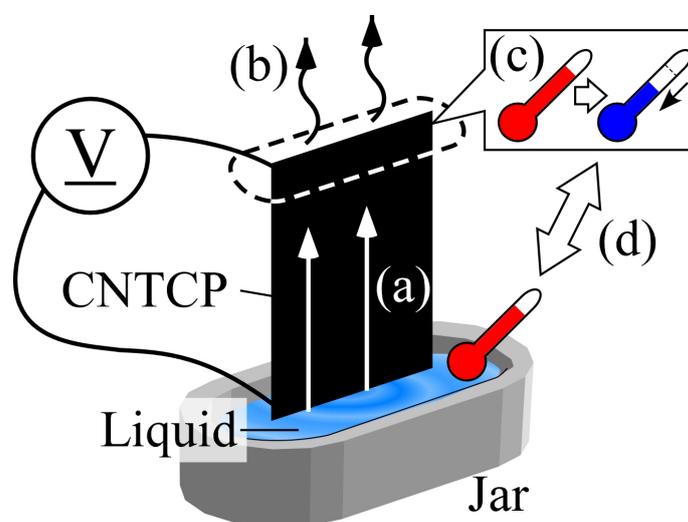


Figure 3. Principle of transpiration-type thermoelectric power generation. Flow to generate thermoelectric power: (a) liquid absorption \rightarrow (b) evaporation of absorbed liquid \rightarrow (c) generation of vaporization heat due to evaporation of absorbed liquid and resulting temperature decrease \rightarrow (d) generation of temperature differences at both ends of the sample and resulting Seebeck coefficient.

2.4. Examination to Improve Electromotive Force by Using Multiple Sheets

Generally, thermoelectric-power-generating material can be connected electrically in series and thermally in parallel to obtain more output from a single heat source. Taking advantage of the easy cutting and pasting properties of CNTCP, a multisheet structure was constructed, as shown in Figure 4. CNTCP consisting of multiwalled CNTs (MWCNTCP) was connected in series to the SG101-CNT composite paper (SGCNTCP) to form our transpiration-type thermoelectric-power-generating paper. The Seebeck coefficient of MWCNT tended to be lower than that of SGCNT. When fabricating thermoelectric-power-generating material, p-type and n-type semiconductors are generally combined; however, this can be achieved with two different materials with different Seebeck coefficients. The stability of n-type semiconducting CNTCP remains an issue, so we combined SGCNTCP and MWCNTCP. We used MWCNTs (NC7000, Nanocyl SA, Sambreville, Belgium) and eucalyptus pulp to fabricate MWCNTCP. The fabrication method is the same as that mentioned in Section 2.1. The Seebeck coefficient of MWCNTCP was around $15 \mu\text{V}/\text{K}$. Conductive carbon tape was also used to connect SGCNTCP to MWCNTCP.

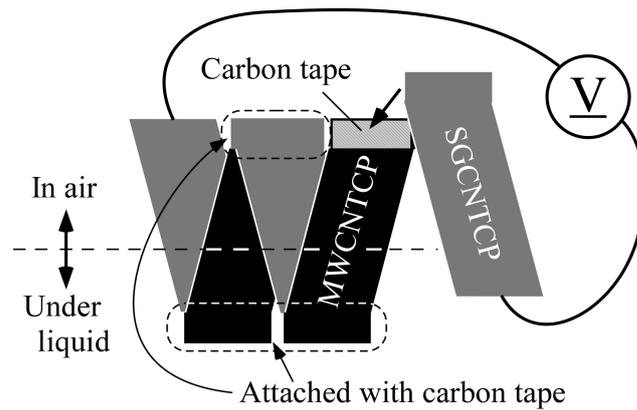


Figure 4. Schematic of transpiration-type power-generating paper with a multisheet structure.

3. Results

3.1. Absorption Ability of CNTCP

We prepared samples 1 to 4 on the basis of the CNTCP fabrication method described in Section 2.1 and under the conditions listed in Table 1. Figure 5 shows photos of the fabricated samples. These CNTCPs were evaluated for their water absorption ability. Figure 6 shows the results of the measurements described in Section 2.2 for 30 min. For comparison, the measurement results without CNTCP are also shown. This suggests that CNTCP made from rayon pulp has excellent absorption ability. It was also found that the absorption ability further improved by drying in an oven instead of using a heat press. This suggests that heat pressing reduces the space inside CNTCP, suppressing capillary action.

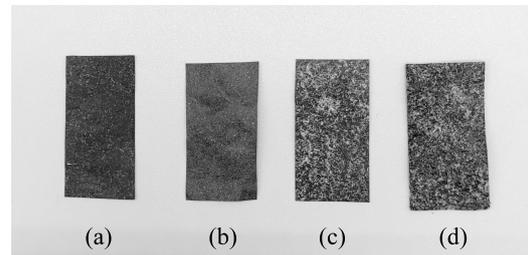


Figure 5. Fabricated CNTCP samples to evaluate their absorption ability. Samples were fabricated under conditions listed in Table 1: (a) sample 1, (b) sample 2, (c) sample 3, and (d) sample 4. Each sample was prepared as a $3.0 \times 1.5 \text{ cm}^2$ sheet (actual size varied due to manual preparation).

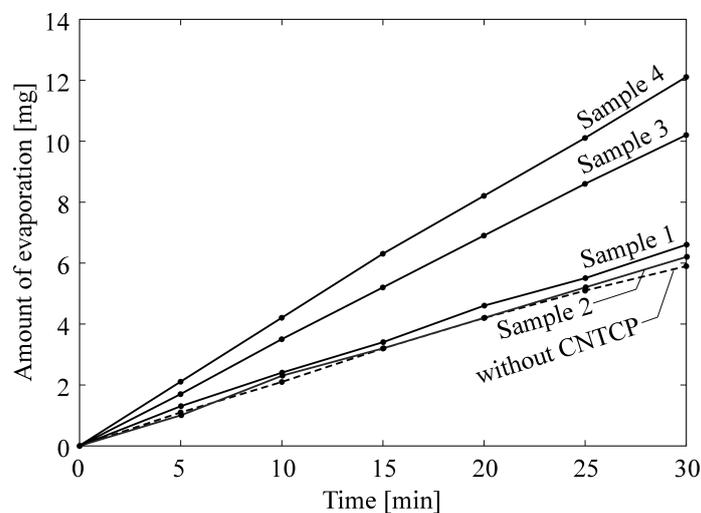


Figure 6. Measuring water absorption ability of CNTCP under different conditions.

3.2. Evaluation of Transpiration-Type Thermoelectric-Power-Generating Paper

On the basis of the results in Section 3.1, a CNTCP sample was prepared by using rayon pulp and drying it in an oven to fabricate our transpiration-type thermoelectric-power-generating paper. Figure 7 shows an example of the results of the temperature difference generated in a sample by capillary action and the generation of vaporization heat, using a thermal imaging camera. The result confirmed that the sample spontaneously generated a temperature difference as desired. Figure 8 shows the temperature change during 15 min of measurement at the top and bottom of the sample and the change in generated electromotive force. From the results, a maximum temperature difference of 1.7 °C was generated between the top and bottom (water surface) of the sample due to the heat of vaporization at room temperature, generating an electromotive force of 36 μV . The Seebeck coefficient at this time is estimated to be about 21.2 $\mu\text{V}/\text{K}$. Figure 9 shows a correlation diagram between the generated temperature difference and the electromotive force. The data indicate that the sample generates an electromotive force that depends on the temperature difference it spontaneously generates. Therefore, these suggest the feasibility of a new type of thermoelectric generation device that uses capillary action and heat of vaporization to spontaneously generate a temperature difference and generate electricity.

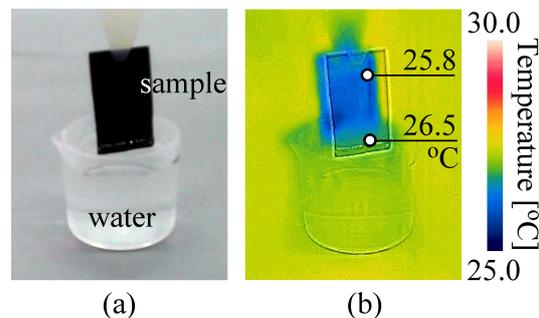


Figure 7. (a) Experiment of temperature difference generation by heat of vaporization and (b) thermal image of a sample.

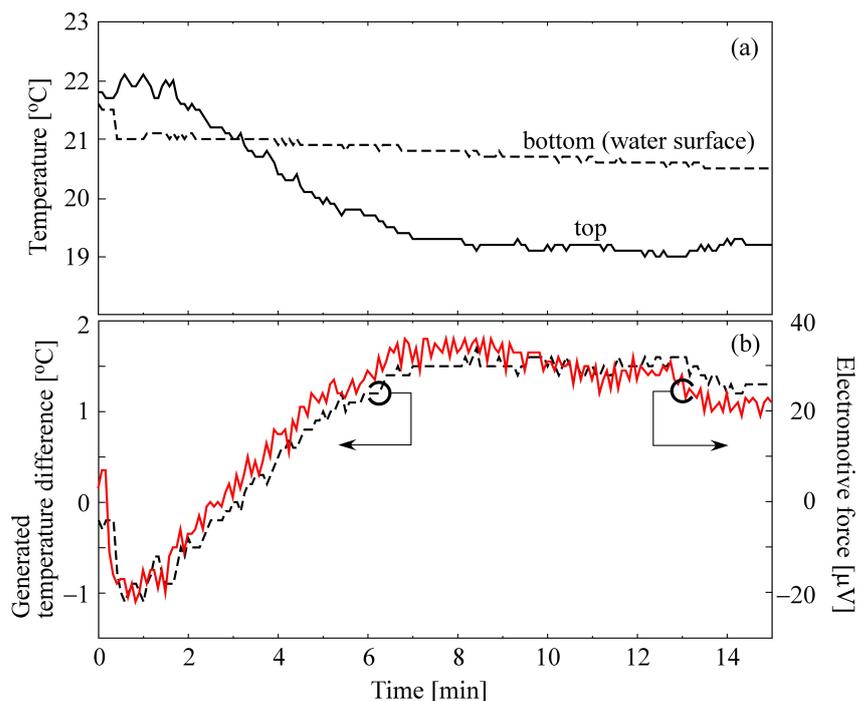


Figure 8. (a) Temperature change at the top (T_t) and bottom (T_b) of the sample and (b) generated temperature difference ($T_b - T_t$) and electromotive force as a function of time.

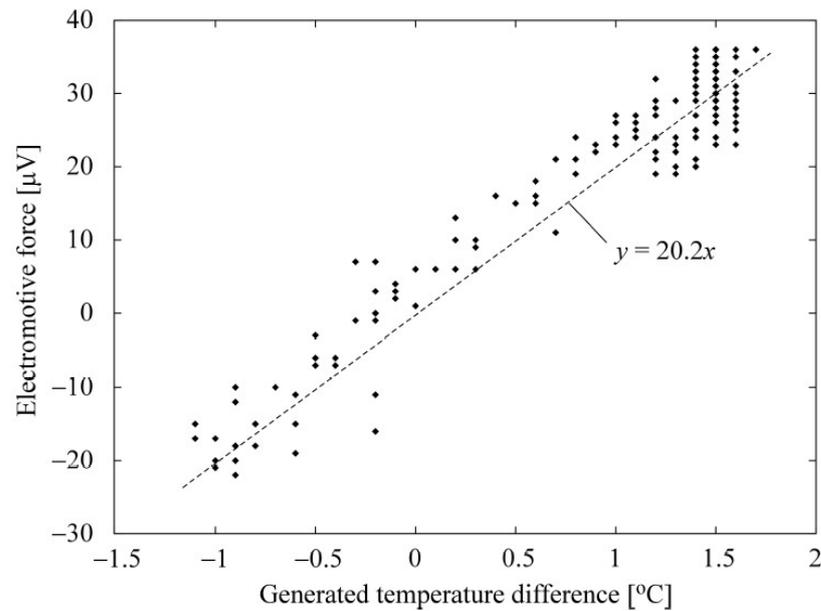


Figure 9. Correlation diagram between generated temperature difference and electromotive force. The dashed line indicates curve fitting.

3.3. Multisheet Structure of Thermoelectric-Power-Generating Paper

Figure 10 shows our multisheet transpiration-type thermoelectric-power-generating paper fabricated by the procedure described in Section 2.4. It is lightweight and flexible even with multiple sheets due to the CNTCP properties. Table 2 lists the measurement results when three to five SGCNTCP sheets were used with MWCNTCPs placed between each SGCNTCP, as explained in Section 2.4. Conductive carbon tape was used to connect each CNTCP. We confirmed that the output obtained by increasing the number of sheets was larger: $137.5 \mu\text{V}$ per 1°C temperature difference for that of three sheets and $178.0 \mu\text{V}$ per 1°C temperature difference for that of five sheets. This indicates the feasibility of our multisheet transpiration-type thermoelectric-power-generating paper.

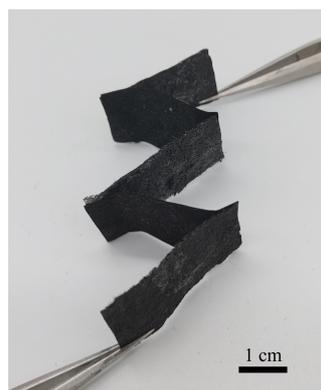


Figure 10. Fabricated multisheet transpiration-type thermoelectric-power-generating paper.

Table 2. Results of transpiration-type thermoelectric power generation from a developed sample.

Numbers of SGCNTCPs	T_b ($^\circ\text{C}$)	T_t ($^\circ\text{C}$)	V (μV)
3	20.4	18.8	220
5	21.8	19.8	356

4. Discussion

4.1. Absorption Ability of CNTCP

Based on the experimental results presented in Section 3.1, CNTCP has the ability to absorb water, and rayon pulp, which has excellent absorption properties, can improve the water absorption ability of CNTCP. It was also found that drying in an oven, rather than using a heat press, was more effective in improving water absorption. Here, we used SEM to observe and evaluate the cross-sectional shape and thickness of the samples (Figure 11). From the results, it was found that the samples with eucalyptus pulp (samples 1 and 2) had similar cross sections regardless of the drying method. This suggests that the shape was determined to some extent at the point of dehydration in the papermaking process (Figure 1b). In both cases, there is little internal space for the occurrence of capillary action, which is also consistent with the results of Figure 6. On the other hand, for the samples with rayon, it was confirmed that clear differences in cross-sectional shape and thickness appeared as expected, depending on the drying method. In particular, it can be said that the sample dried by oven (sample 4) has an overall internal space configuration that is better suited for the occurrence of capillary action. In other words, this is thought due to the fact that the use of a heat press causes the pores inside CNTCP with rayon to collapse, making capillary action less likely to occur (sample 3). Therefore, it is important to select pulp material with good absorption properties and an appropriate fabrication method that does not collapse the internal pores to fabricate transpiration-type thermoelectric-power-generating paper.

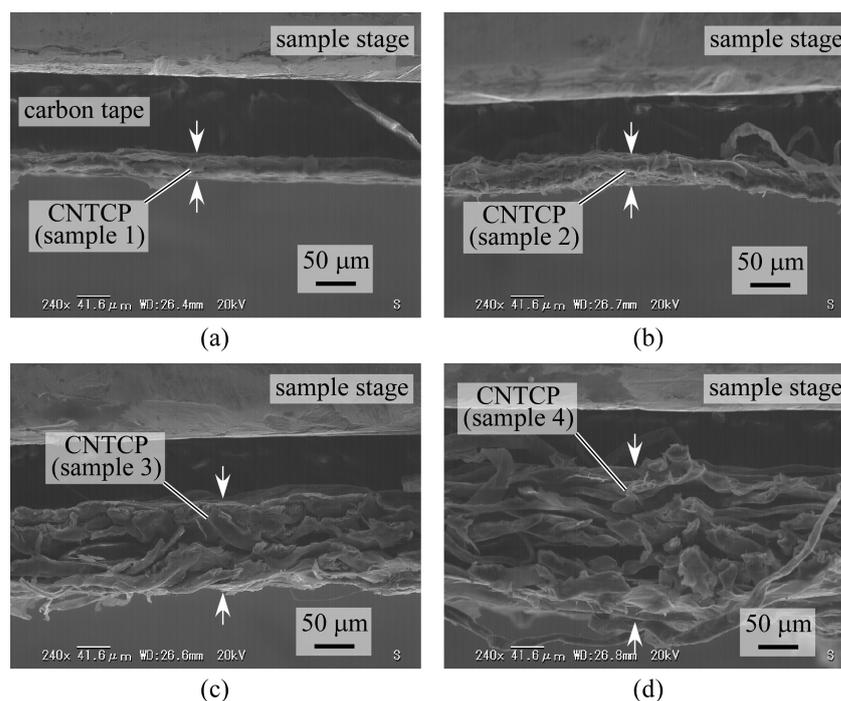


Figure 11. SEM images of samples (cross-sectional view). (a) sample 1; (b) sample 2; (c) sample 3; and (d) sample 4.

4.2. Transpiration-Type Thermoelectric-Power-Generating Paper

The experimental results presented in Section 3.2 indicate that SGCNTCP spontaneously produced a temperature difference of 1.7 °C and generated an electromotive force of 36 μV. However, a slightly lower Seebeck coefficient (about 21 μV/K) was confirmed when compared with that in a dry environment (about 35 μV/K), as shown in our previous study. This may be because the absorbed water may have slightly affected the thermoelectric performance of the CNTs in CNTCP. In contrast, the resistance of the samples did not change much from 10.7 Ω in the dry condition to 9.6 Ω in the wet condition, suggest-

ing that the effect of water on the resistance could be negligible under the conditions of these experiments. The temperature difference due to the spontaneously generated heat of vaporization was about 1.7 °C. Ideally, a larger temperature difference is desirable. One possible solution for this would be to increase the amount of evaporation. To achieve this, the surface area of the area to be evaporated should be increased. This could be achieved by improving the structural component of the CNTCP fabrication process. The evaporation process depends on the surrounding environment, such as room temperature and humidity. Therefore, how to achieve evaporation that is less affected by the surrounding environment will be considered as the next step of this study. Thermoelectric conversion efficiency should also be considered. Generally, the maximum conversion efficiency in thermoelectric conversion elements is given by the following equation:

$$\zeta = \frac{T_H - T_L}{T_H} \frac{\sqrt{1 + Z \frac{T_H + T_L}{2}} - 1}{\sqrt{1 + Z \frac{T_H + T_L}{2} + \frac{T_L}{T_H}}}, \quad (1)$$

$$Z = \frac{S^2 \sigma}{\kappa}, \quad (2)$$

where T_H is the temperature on the high temperature side of the element (T_t in this paper) and T_L is on the low temperature side (T_b in this paper). S indicates Seebeck coefficient, σ indicates electrical conductivity, and κ indicates thermal conductivity. If T_H is 294 K (about 21 °C) and T_L is 292 K (about 19 °C), the maximum conversion efficiency is roughly estimated to be $2.3 \times 10^{-5}\%$. Although this value is small as a conversion efficiency value, we believe that it is reasonable, considering that the environment is at room temperature. However, thermoelectric devices that spontaneously generate a temperature difference are affected by the surrounding environment as described above, and there are issues such as how to consider the “energy of spontaneously generated temperature difference” that need to be considered. We will seek to work on this issue and resolve it in the near future.

4.3. Multisheet Structure

As shown in Section 3.3, the multisheet structure succeeded in obtaining a large electromotive force even with a small temperature difference. In other words, we believe that we have demonstrated the effectiveness of such a structure. The averaged Seebeck coefficients of the used SGCNTCP and MWCNTCP were 28.3 $\mu\text{V}/\text{K}$ and 14.9 $\mu\text{V}/\text{K}$, respectively. Therefore, we can expect an electromotive force of 55.1 $\mu\text{V}/\text{K}$ for the three sheets and 81.9 $\mu\text{V}/\text{K}$ for the five sheets. However, a larger generated electromotive force than expected from the subtraction of the Seebeck coefficients of the used SGCNTCP and MWCNTCP was obtained. As explained in Section 2.4, MWCNTCP sheets are placed between SGCNTCP sheets in series. Since the MWCNTCP used for this study was fabricated by a previous fabrication process, its absorption capacity is the same as that of sample 1 in Figure 6; thus, it does not absorb much water. The fact that it does not absorb much water means that the amount of water evaporating at the top of the MWCNTCP is also considered to be small; accordingly, the temperature change due to heat of vaporization is small. Therefore, MWCNTCP does not function as a thermoelectric material, but only as a conductor for SGCNTCP. This behavior is very convenient for transpiration-type thermoelectric-power-generating paper. As one example of an application, we here consider the operation of an electronic device via a boost converter. To operate the practical step-up DC/DC converter (AP4473, Asahi Kasei Microdevices Corporation, Tokyo, Japan), an open-circuit voltage of 50 mV and an internal resistance of 50 Ω are required. Therefore, about 140 pairs of a five-sheet structure are needed to produce this open-circuit voltage. However, the internal resistance of the five-sheet structure is 262 Ω , so the total internal resistance in this case is about 37 k Ω . Thus, the internal resistance of the current sheet must be reduced by a factor of about 740, which we believe can be achieved by stacking and widening the CNTCP in addition to decreasing its internal resistance. Moreover, since thermoelectric conversion is proportional to the

square of the temperature difference, using hot water instead of room temperature water, for example, is expected to improve the output and make it more feasible.

5. Conclusions

We developed a “transpiration-type thermoelectric-power-generating paper” based on our previously developed CNTCP. It is a new type of thermoelectric-power-generating material that does not require an external high-temperature heat source due to its ability to absorb liquid through the capillary action of paper and heat of vaporization generated when liquid evaporates. The aim of this study was to investigate the feasibility of realizing the transpiration-type thermoelectric-power-generating paper. At first, the type of paper used as raw material for the composite paper was examined, and the fabrication process was modified in order to obtain more efficient liquid absorption based on capillary action. Then, we chose rayon pulp to make CNTCP and changed the drying method in the fabrication process. Next, the feasibility of thermoelectric power generation using the absorbing ability of the liquid and the heat of vaporization was confirmed. Moreover, for more efficient thermoelectric conversion, multisheet structures were also studied. The results of several experiments above indicate that a sample that we fabricated spontaneously produced a temperature difference of 1.7 °C due to the heat caused by the vaporization of the absorbed water, and showed a good electromotive force (36 μ V). When many CNTCP sheets can be easily connected, a larger electromotive force can be obtained. Therefore, we verified this material’s feasibility and confirmed that more power can be easily obtained through the multisheet structure. The material can be used in watery environments, such as rivers, lakes, and hot springs, and is expected to become a new energy-harvesting material.

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Conflicts of Interest: Author Koya Arai is employed by the company Mitsubishi Materials Corporation. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Abbreviations

The following abbreviations are used in this manuscript:

CNT	carbon nanotube
CNTCP	carbon nanotube composite paper
SEM	scanning electron microscope
SDS	sodium dodecyl sulfate
MWCNTCP	CNTCP consisting of multiwalled CNT
SGCNTCP	CNTCP consisting of SG101-CNT

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