



Article

Performance Improvement of Paper Dye-Sensitized Solar Cell Using H₂/Ar-Treated n-Type Semiconducting Carbon-Nanotube Composite Paper

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Abstract

This paper presents paper-based dye-sensitized solar cells (paper DSSCs) fabricated using carbon nanotube (CNT) composite paper produced from mixtures of CNT and pulp dispersions. DSSC is composed of a dye-adsorbed semiconducting electrode, a counter electrode, and an electrolyte. In this study, our DSSC is constructed using n-type semiconducting CNT composite paper as the semiconducting electrode, metallic CNT composite paper as the counter electrode, and ordinary paper for keeping the electrolyte. In our previous study, potassium hydroxide was used to convert semiconducting CNT composite paper to n-type, but the performance was limited. Therefore, we aim to achieve a more stable and higher-performing paper DSSC by annealing the semiconducting CNT composite paper in a hydrogen–argon atmosphere to induce n-type properties. For this, CNT composite paper was prepared using the cationic surfactants DODMAC (dimethyl octadecyl ammonium=chloride, cationic surfactant) and DDAC as dispersing agents. The fabricated DSSCs were evaluated in terms of photoelectric conversion efficiency and fill factor (FF). As a result, DSSCs using DODMAC increased the efficiency from $5.04 \times 10^{-3}\%$ to $13.37 \times 10^{-3}\%$ and the FF from 0.13 to 0.21. When DDAC was used, the efficiency increased to $17.11 \times 10^{-3}\%$ and the FF improved to 0.27.

Keywords: carbon nanotube (CNT); carbon nanotube composite paper; dye-sensitized solar cell (DSSC); paper DSSC; cationic surfactant; hydrogen–argon treatment

1. Introduction

In recent years, environmental concerns have led to growing interest in renewable energy sources, including wind power, hydro power, and solar power. Consequently, research on photovoltaics and solar cells has been increasingly active [1]. Among various types of solar cells, we focus on dye-sensitized solar cells (DSSCs) [2,3]. DSSCs generate electricity through the redox reactions of dyes and offer the advantages of low cost and easy of fabrication [4]. In this study, we aim to develop a new DSSC using carbon nanotube (CNT) composite paper as a distinctive paper DSSCs. CNTs were first discovered in 1991 [5], and numerous studies have been conducted since then. Due to their nanoscale dimensions, CNTs are difficult to handle individually, which complicates their application in electronic devices. In this study, CNT composite paper, developed in 2008, was fabricated by mixing CNTs with pulp—the raw material for paper—using a method inspired by traditional Japanese washi papermaking techniques [6]. Since CNTs exhibit semiconducting or metallic



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properties depending on their structure, it is possible to produce semiconducting or metallic CNT composite paper by combining pulp with CNTs of the corresponding type [7]. We have successfully fabricated incipient paper DSSCs using CNT composite paper with each property for the cathode and anode [8]. Since n-type semiconducting electrodes are typically employed for the anode of the conventional DSSCs, it is desirable for the semiconducting CNT composite paper to exhibit n-type properties to manufacture paper DSSCs. However, semiconducting CNTs are known to generally exhibit p-type behavior in air [9]. To address this, we previously succeeded in producing n-type semiconducting CNT composite paper using potassium hydroxide (KOH) and crown ether, although issues such as long-term stability remained [10]. This was caused by the swelling of the CNT composite paper and changes in its internal structure due to the use of liquid for doping. Therefore, in this study, a liquid-free n-type doping method, i.e., a hydrogen-argon (H_2/Ar) annealing method [11,12], was adopted to evaluate its feasibility and the performance of paper-based DSSCs using the n-type composite paper. In the reference study that guided the annealing process in this work, CNTs were reported to maintain their n-type properties for approximately two years under H_2/Ar annealing. This duration was described as the longest reported in the literature, suggesting that H_2/Ar annealing provides superior long-term stability compared to other gas-phase n-type doping methods. For the CNT composite paper, this method involves inducing n-type behavior by annealing CNT composite paper in which the cationic surfactant dimethyl octadecyl ammonium=chloride (DODMAC) is used as a dispersant. Although this approach improved the performance compared with conventional liquid-based n-type doping methods, there remained room for further improvement due to the relatively low dispersibility of the cationic surfactant [13]. Based on this, in the present study, we focused on a cationic surfactant, DDAC, and evaluated the performance of paper DSSCs using n-type CNT composite paper prepared with this surfactant as a dispersant.

2. Materials and Methods

In this study, two types of single-walled CNTs—SGCNT (SG101, Zeon corporation, Tokyo, Japan) as a metallic CNT and (6,5)-chirality CNT (SG65i, CHASM, Boston, MA, USA) as a semiconducting CNT—were used to fabricate the CNT composite papers. The metallic CNT composite paper is used for the cathode (positive electrode), and the semiconducting CNT composite paper is used for the anode (negative electrode) of our paper DSSC. The CNT composite papers are prepared using a method based on traditional Japanese *washi* papermaking technique, as described in Introduction. Figure 1 illustrates the fabrication process of the CNT composite paper. The procedures for metallic and semiconducting CNT composite papers are identical. As one example, the procedure for preparing metallic CNT composite paper is as follows. First, 50 mg of SGCNTs and 50 mg of sodium dodecyl sulfate (SDS) as a dispersant are mixed with 15 mL of pure water. This mixture is then sonicated by ultrasonication for 60 min to obtain a CNT dispersion. Next, 100 mg of pulp is added to 100 mL of pure water and stirred using an agitator until the pulp clumps break up and are dispersed to make a pulp dispersion. The CNT dispersion and the pulp dispersion are then mixed and sonicated for 30 min by ultrasonication. The resulting mixture is placed in a papermaking apparatus, shaped and dried using a heat press. Then, the obtained sample is cut into $2 \times 2.5 \text{ cm}^2$ rectangles. The prepared metallic CNT composite paper and semiconducting CNT composite papers are shown in Figure 2. The specific amounts of each CNT composite paper used for fabricating our paper DSSC are listed in Tables 1 and 2.

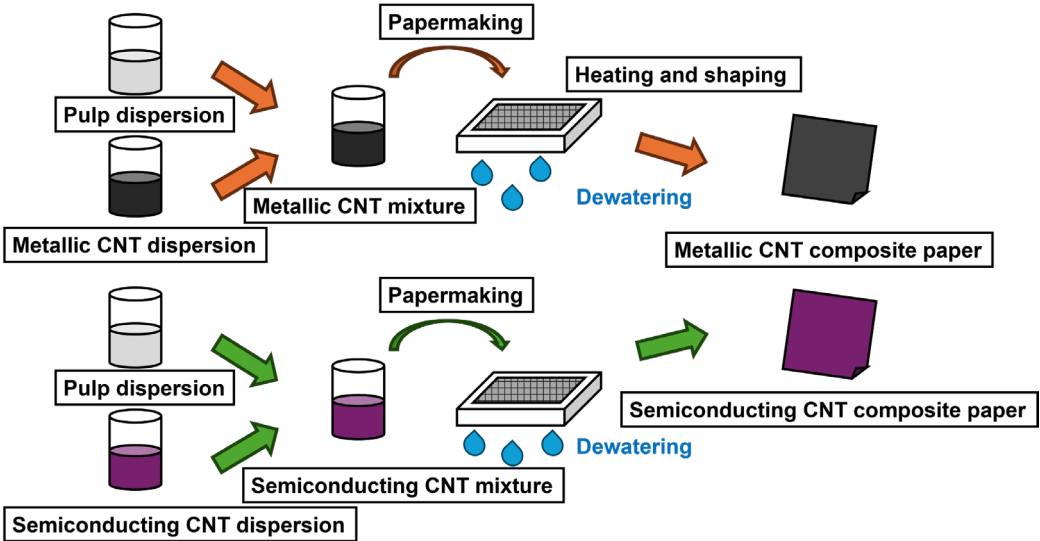


Figure 1. Manufacturing process of CNT composite paper.

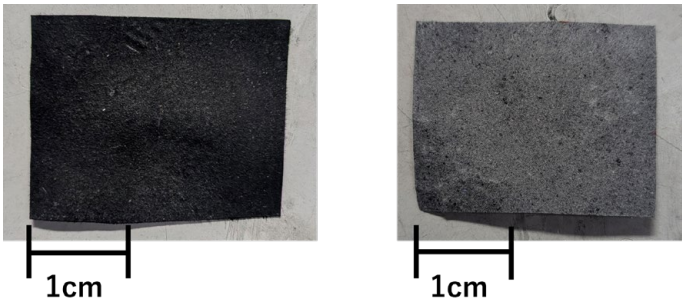


Figure 2. Metallic CNT composite paper (left) and semiconducting CNT composite paper (right).

Table 1. Amounts of materials for metallic CNT composite paper.

For Pulp Dispersion		For CNT Dispersion		
Pulp	Pure water	SGCNT	SDS	Pure water
100 mg	15 mL	50 mg	50 mg	15 mL

Table 2. Amounts of materials for semiconducting CNT composite paper.

For Pulp Dispersion			For CNT Dispersion		
Pulp	Pure water	(6,5)-chirality	DODMAC or DDAC	Purple sweet potato dye	Pure water
100 mg or 200 mg	15 mL	6 mg	30 mg	60 mg	15 mL

Semiconducting CNT composite papers were prepared using 100 mg and 200 mg of pulp, respectively. As described in Introduction, semiconducting CNTs generally exhibit p-type characteristics in air due to oxygen adsorption; however, n-type CNT are required for the development of CNT-based DSSCs. Because n-type semiconducting electrodes are typically employed for conventional DSSCs. Therefore, in this study, the fabricated semiconducting CNT composite paper was attempted to be made n-type, and the characteristics of DSSCs with n-type anodes were investigated. In our previous studies, a 1:1 mixture of KOH solution and crown ether solution was used as the doping agent. It has been reported that the surface of CNTs can be n-type doped using KOH due to the excess electrons

supplied by potassium. In addition, using crown ether as an auxiliary doping agent can encapsulate cations within the crown ether, thereby stabilizing the doping process [14]. In our previous study, n-type doping was carried out by immersing the semiconducting CNT composite papers in the doping solution. However, this doping method caused the composite paper to swell due to solution impregnation, leading to shape changes, and also presented issues, particularly regarding the long-term stability of its n-type semiconducting properties. Therefore, in this study, we attempt n-type doping by annealing under H₂/Ar atmosphere, referencing the method described in Ref. [11]. This method is expected to improve the long-term stability of the n-type semiconducting properties in the composite paper. First, to prepare the semiconducting CNT composite paper, the cationic surfactant DODMAC as a dispersant is used. The procedure for preparing semiconducting CNT composite paper using DODMAC is described below. To prepare the semiconducting CNT dispersion, 6 mg of (6,5)-chirality CNTs and 30 mg of DODMAC are added to 15 mL of pure water. Because a low temperature is preferable for dispersion by DODMAC, the mixture is sonicated for 90 min while the bottle containing the solution is cooled to approximately 20 °C, as shown in Figure 3. Next, 60 mg of purple sweet potato dye is added to the prepared CNT dispersion, and the CNT dispersion is made by ultrasonication for 60 min. Figure 4 shows the CNT dispersion prepared by this method and the resulting CNT composite paper produced from it. From this, although further investigation is needed to optimize the quantities, it was confirmed that CNTs can be effectively dispersed using DODMAC as the dispersant, and CNT composite paper can be successfully fabricated. Next, semiconducting CNT composite paper was prepared using dodecyl dimethyl ammonium=chloride (DDAC), a cationic surfactant, as the dispersant. The same quantities and preparation procedure as described above were employed. Ultrasonic dispersion with DDAC was carried out without cooling. This is because no reports on the temperature dependence of the dispersion ability were found for DDAC. As a result, it was confirmed that CNT composite paper can be successfully fabricated using DDAC as the dispersant. Next, the semiconducting CNT composite paper is H₂/Ar-annealed to induce n-type characteristics. The procedure for n-type conversion via annealing is illustrated in Figure 5. First, the semiconducting CNT composite paper is placed in an infrared heating furnace, which is filled with 1000 sccm of Ar. The temperature is gradually raised until the furnace reaches approximately 110 °C. Then, approximately 1000 sccm of H₂ is supplied to the furnace. After 2 min, the temperature is lowered, and the composite paper is removed when it reaches about 40 °C. In this manner, the semiconducting CNT composite paper is converted to n-type. Since no significant difference in performance was observed compared with longer annealing times, an annealing time of 2 min was adopted in this study. In addition, the annealing temperature was selected based on previous reports, which indicated that n-type behavior was not observed below approximately 80 °C. In addition, when the annealing temperature exceeds approximately 150 °C, the paper substrate tends to char or burn, making electrical measurements difficult. Therefore, 110 °C was chosen as an appropriate temperature. After annealing, the paper is cut into the desired sizes. To prepare the paper DSSC, the metallic and semiconducting CNT composite papers are cut into 2 × 2.5 cm² pieces, and ordinary paper is prepared and cut into 2 × 2 cm² sections. A sufficient amount of electrolyte is applied onto the ordinary paper. The electrolyte solution is prepared by mixing 0.26 g of iodine, 1.66 g of potassium iodide, and 20 mL of ethylene glycol. These components are layered as shown in Figure 6. A 2 × 2 cm² area is assembled so that the semiconducting and metallic CNT composite papers each extend outward by 0.5 cm for connection to the external circuit.

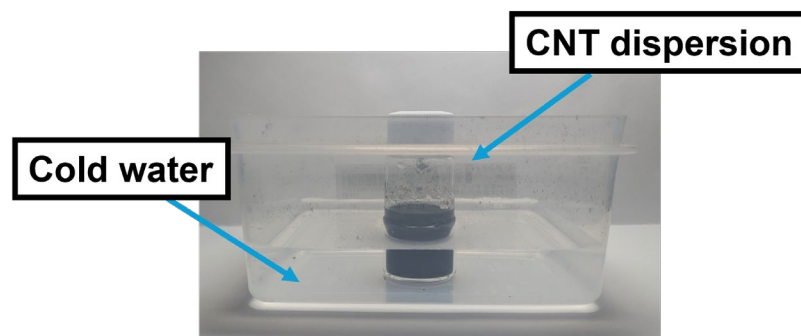


Figure 3. Manufacturing process of CNT composite paper.

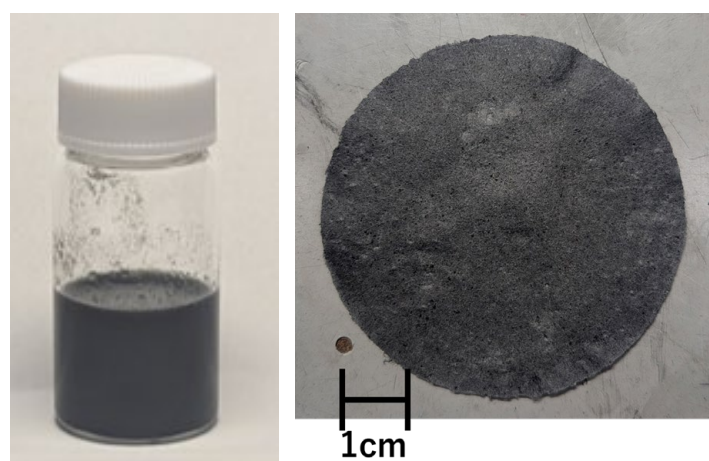


Figure 4. Prepared CNT dispersion with DODMAC (left) and semiconducting CNT composite paper made from it (right).

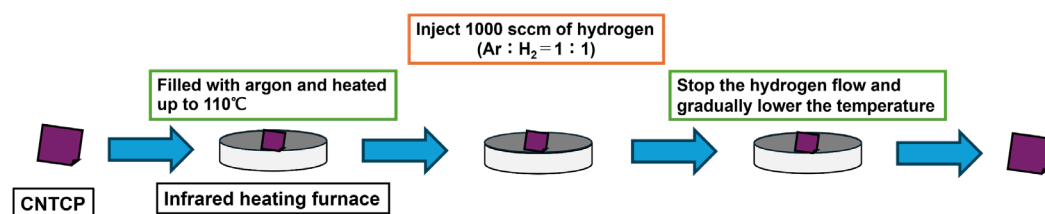


Figure 5. Annealing process for converting semiconducting CNT composite paper to n-type.

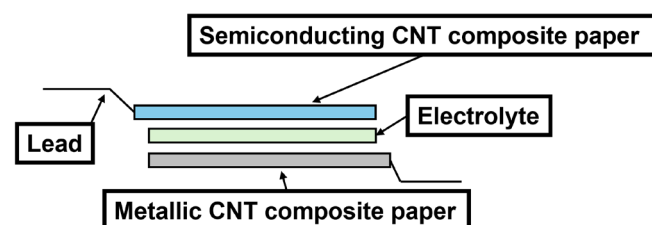


Figure 6. Manufacturing process of CNT composite paper.

The fabricated paper DSSC is connected to leads and then vertically exposed to artificial sunlight with an intensity of 1000 W/m^2 , as shown in Figure 7. A semiconductor parameter analyzer (4200A-SCS, Semiconductor Characterization System, Keithley, Solon, OH, USA) is used to measure the I-V characteristics.

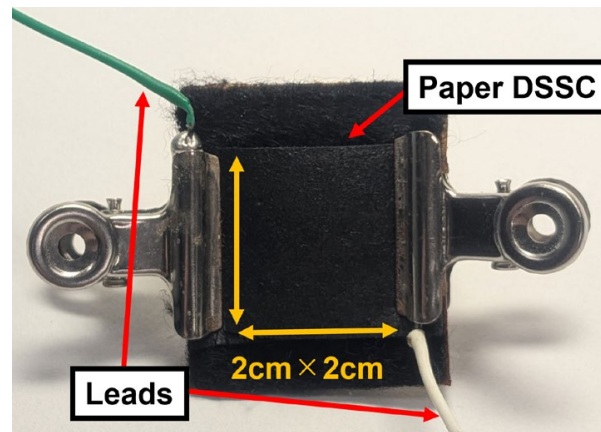


Figure 7. Paper DSSC for measurement setup.

The performance of the DSSC can be assessed by measuring the short-circuit current I_{sc} and open-circuit voltage V_{oc} and calculating fill factor FF and photoelectric conversion efficiency η . Typical I-V characteristics of the DSSC are shown in Figure 8 [15]. FF can be determined using the maximum power P_{max} , which corresponds to the largest rectangle that fits under the I-V curve. The Photoelectric conversion efficiency can be calculated using Equation (1), where E is the irradiance, which is 1000 W/m^2 in this study, and S is the light-receiving area of the power-generating element, which is $2 \times 2 \text{ cm}^2$ of the paper DSSC. FF can also be obtained by Equation (2).

$$\eta = \frac{P_{max}}{E \times S} \times 100\% \quad (1)$$

$$FF = \frac{P_{max}}{V_{oc} \times I_{sc}} \quad (2)$$

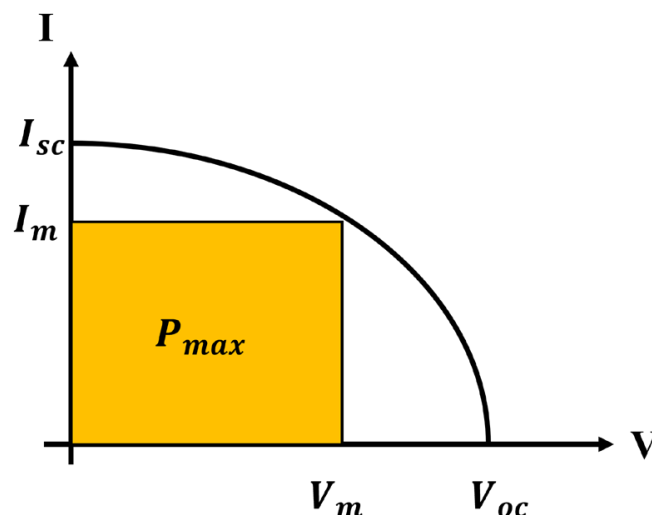


Figure 8. Typical I-V characteristics of paper DSSC.

3. Results and Discussion

Our paper DSSCs were fabricated using the method described in the previous section, and their performance was evaluated. Figure 9 presents the Seebeck effect measurement results for semiconducting CNT composite paper with 200 mg of pulp prepared using DODMAC, while Figure 10 presents those prepared using DDAC. Hall measurements are generally required to evaluate intrinsic material properties. However, Hall measure-

ments ideally require a homogeneous, planar sample. In contrast, CNT composite paper possesses a unique structure in which the CNT network is intricately entangled within a three-dimensional network of insulating fibers (paper fibers). This structural complexity makes it difficult to accurately evaluate its properties using conventional Hall measurement techniques. Therefore, in this study, the electrical characteristics were evaluated by measuring the Seebeck coefficient. In addition, Figure 11 presents the measurement results of paper DSSCs when the pulp of semiconducting CNT composite paper is 200 mg. Figure 12 shows the results for paper DSSCs with 100 mg of pulp. In addition, the dark current measurement results of the paper DSSCs fabricated using each dispersant are also included in the figure. Furthermore, the detailed characteristics of each device are summarized in Tables 3 and 4. For both pulp amounts of 100 mg and 200 mg, the n-type paper DSSCs using the H₂/Ar-annealed CNT composite paper with DDAC exhibited the highest values of conversion efficiency and I_{sc} . The next highest values were observed for n-type paper DSSCs using the H₂/Ar-annealed CNT composite paper with DODMAC, while the lowest values were obtained for n-type paper DSSCs using the CNT composite paper treated with KOH. These results indicate that the photoelectric conversion efficiency and I_{sc} of n-type paper DSSCs using H₂/Ar-annealed CNT composite paper are higher than those of n-type paper DSSCs using CNT composite paper treated with KOH.

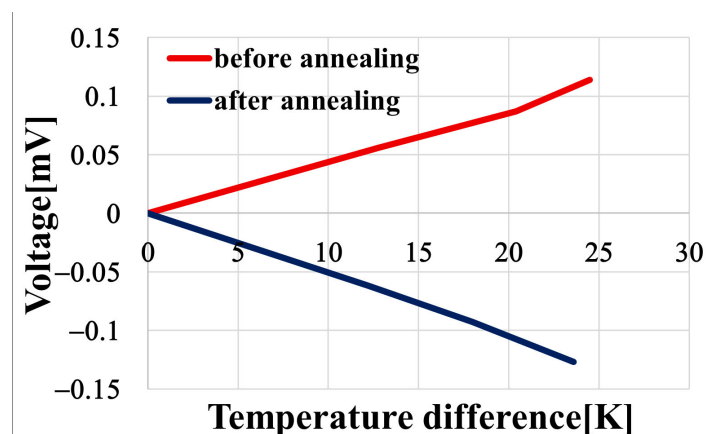


Figure 9. Measurement results of Seebeck effect for semiconducting CNT composite paper with 200 mg of pulp using DODMAC.

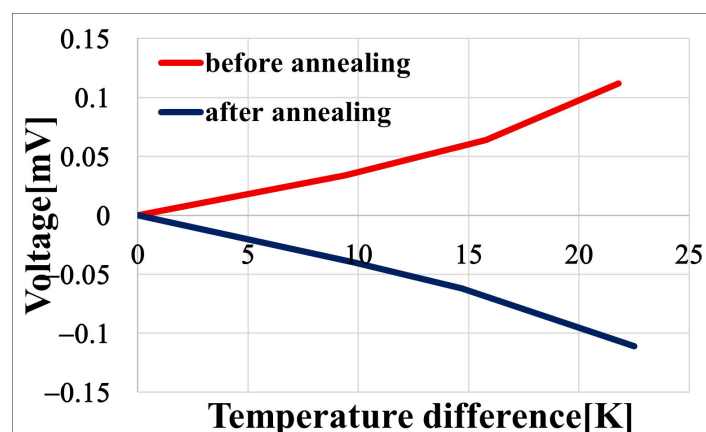


Figure 10. Measurement results of Seebeck effect for semiconducting CNT composite paper with 200 mg of pulp using DDAC.

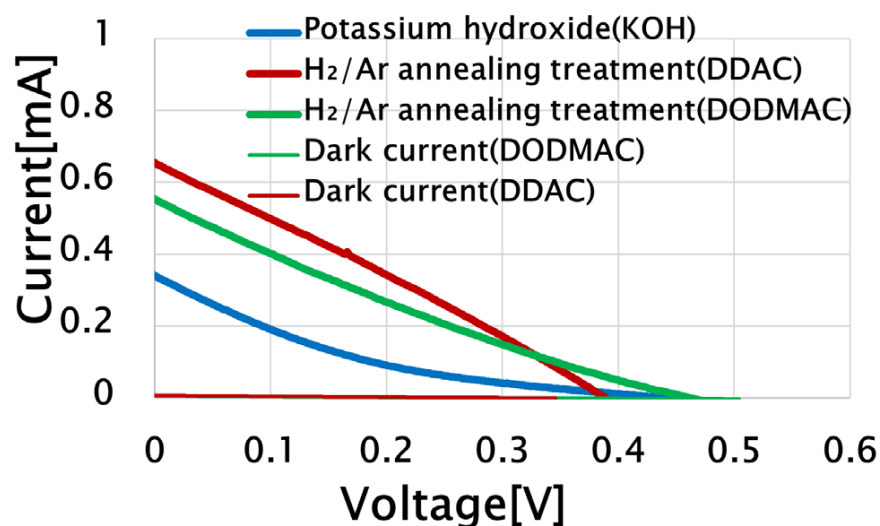


Figure 11. I-V characteristics of paper DSSC based on 200 mg of pulp used semiconducting CNT composite paper. Two dark current curves almost overlap.

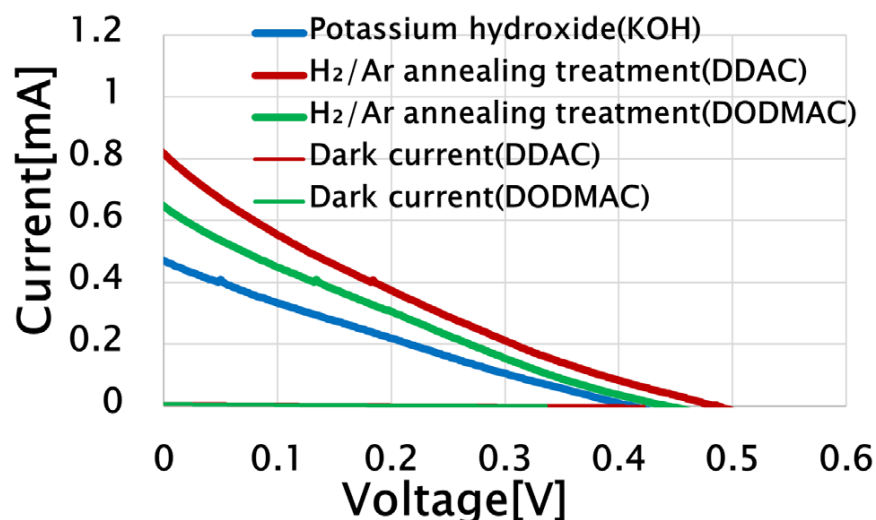


Figure 12. I-V characteristics of paper DSSC based on 100 mg of pulp used semiconducting CNT composite paper. Two dark current curves almost overlap.

Table 3. Characteristics of paper DSSCs prepared with 200 mg of pulp.

	I_{sc} [mA]	V_{oc} [V]	FF	η [$\times 10^{-3}$]
DDAC	0.65	0.39	0.27	17.11
DODMAC	0.55	0.46	0.21	13.37
KOH	0.34	0.44	0.13	5.04

Table 4. Characteristics of paper DSSCs prepared with 100 mg of pulp.

	I_{sc} [mA]	V_{oc} [V]	FF	η [$\times 10^{-3}$]
DDAC	0.82	0.49	0.19	18.75
DODMAC	0.65	0.44	0.21	15.32
KOH	0.47	0.41	0.23	10.97

When CNT composite paper is treated with KOH, the paper swells because it is immersed in the solution. For a pulp amount of 200 mg, the thickness of the CNT composite

paper increased from 0.12 mm to 0.19 mm before and after n-type doping with KOH. Furthermore, even for a pulp amount of 100 mg, the thickness increased from 0.08 mm to 0.16 mm. When the CNT composite paper absorbs liquid, the distance between the pulp fibers increases. As a result, the CNT network within the CNT composite paper becomes separated or interrupted, leading to a decrease in conductivity. On the other hand, regarding the annealing process, since it is a liquid-free n-type conversion method, the CNT composite paper does not swell. Therefore, this is considered to be the reason for the observed high photoelectric conversion efficiency and I_{sc} . Furthermore, since the absorption of liquid by the CNT composite paper reduces the mechanical strength of the paper, from the perspective of DSSC durability, H_2/Ar annealing method can be considered a more suitable n-type doping method.

Even with the same H_2/Ar annealing method, DSSCs using CNT composite paper with DDAC exhibited higher photoelectric conversion efficiency and I_{sc} than those using CNT composite paper with DODMAC. The sheet resistance of each CNT composite paper fabricated with a pulp amount of 200 mg was measured, and the CNT composite paper prepared using DDAC exhibited a sheet resistance of 15 $\Omega/sq.$ to 40 $\Omega/sq.$ The sheet resistance of each CNT composite paper fabricated with a pulp amount of 200 mg was measured. The CNT composite paper prepared using DDAC exhibited a resistance of 15 $\Omega/sq.$ to 40 $\Omega/sq.$, whereas that prepared using DODMAC showed a resistance of 100 $\Omega/sq.$ to 200 $\Omega/sq.$ This is believed to be due to the difference in the dispersibility of the dispersants. When the dispersibility is high, CNTs form a uniform network within the CNT composite paper. In contrast, when the dispersibility is low, the CNT network becomes non-uniform and local aggregation occurs. As a result, the electrical resistance of the composite paper increases. These factors are believed to have contributed to the differences in the short-circuit current and conversion efficiency of the paper DSSCs.

From these results, we believe that n-type conversion by hydrogen annealing is an excellent process for semiconducting CNT composite papers and is useful for application development, including paper DSSC. In addition, using DDAC as a dispersant enables the achievement of higher photoelectric conversion efficiency. Based on the above results, the usefulness of H_2/Ar annealing for n-type conversion of semiconducting CNT composite paper has been confirmed. Furthermore, the potential for improving the performance of paper- DSSC using this composite paper has been confirmed, providing a solution to a challenge that remained unresolved in our previous research.

4. Conclusions

This study aimed to convert the semiconducting properties of the CNT composite paper to n-type by annealing it under H_2/Ar atmosphere, and to further enhance the performance of paper DSSCs using this composite paper. For this purpose, As the first step in converting the semiconducting CNT composite paper to n-type, we employed the cationic surfactant DODMAC and DDAC. It was confirmed that the CNT composite papers could be fabricated using these dispersants. Then, fabricated samples were annealed in the infrared heating furnace under H_2/Ar atmosphere to investigate n-type conversion. Finally, the new paper DSSCs were fabricated using these composite papers, and their photoelectric conversion efficiency and FF were measured. As a result, the photoelectric conversion efficiency and FF of the H_2/Ar -treated n-type paper DSSC were higher than those of that our previous n-type paper DSSC treated with KOH. Specifically, for the DODMAC-used semiconducting CNT composite paper with 200 mg of pulp, the photoelectric conversion efficiency increased from $5.04 \times 10^{-3}\%$ to $13.37 \times 10^{-3}\%$, and FF increased from 0.13 to 0.21 compared with the previous paper DSSC with KOH. In the case of 100 mg of pulp, the photoelectric conversion efficiency increased from $10.97 \times 10^{-3}\%$ to $15.32 \times 10^{-3}\%$,

but FF decreased from 0.23 to 0.21. Furthermore, DDAC-used semiconducting CNT composite paper with 200 mg of pulp, the photoelectric conversion efficiency increased from $5.04 \times 10^{-3}\%$ to $17.11 \times 10^{-3}\%$ and FF increased from 0.13 to 0.27 compared with the previous one. In the case of 100 mg of pulp, the photoelectric conversion efficiency increased from $10.97 \times 10^{-3}\%$ to $18.75 \times 10^{-3}\%$, but FF decreased from 0.23 to 0.19. FF and conversion efficiency have not yet reached sufficient levels. This is mainly attributed to the unique internal structure of the CNT composite paper, and further performance improvement is expected by identifying and optimizing these factors in future work. Further improvement in device performance is expected through optimization of the H₂:Ar ratio. The photoelectric conversion efficiency and FF of previously reported DSSCs on paper substrates are 1.21% and 0.33, respectively [16]. Other studies have also investigated DSSCs on paper substrates [17]. Although our current values have not yet reached these levels, we believe that this work represents a step toward the practical application of paper DSSCs.

Author Contributions: All authors contributed equally. Conceptualization, C.S. and T.O.; methodology, C.S.; validation, C.S. and T.O.; formal analysis, C.S.; investigation, C.S. and T.O.; resources, C.S. and T.O.; data curation, C.S.; model, C.S.; writing—original draft preparation, C.S.; writing—review and editing, T.O.; visualization, C.S. and T.O.; supervision, T.O.; funding acquisition, T.O.; project administration, T.O. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data that support the findings of this study are available from the corresponding author upon reasonable request.

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

Abbreviations

The following abbreviations are used in this manuscript:

DSSC	Dye-sensitized solar cell
CNT	Carbon nanotube
KOH	potassium hydroxide
SDS	Sodium dodecyl sulfate
DODMAC	Dimethyl octadecyl ammonium=chloride
DDAC	Dodecyl dimethyl ammonium=chloride
sccm	Standard cubic centimeter per minute (standard cc/min)
FF	Fill factor

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