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Research Article

Production and Properties of Carbon Nanotube/Cellulose Composite Paper

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Multiwalled carbon nanotube/cellulose composite papers have been prepared by mixing the cellulose with MWNT/gelatin solution and drying at room temperature. The CNTs form an interconnected network on the cellulose paper and as a result CNT paper sheet exhibits enhanced electrical properties and thermal stabilities. It is found that both sides of CNT paper sheet have the uniform electrical conductivities. The sheet exhibits strong microwave absorption in the microwave range of 10.5 GHz. The CNT/cellulose paper is as flexible and mechanically tough as the pure cellulose paper. This work provides a novel and simple pathway to make CNT/cellulose sheet as multifunctional biomaterials for electronic, magnetic, semiconducting, and biotechnological applications.

1. Introduction

Composites based on carbon nanotubes (CNTs) are very promising for the continuous growth of telecommunication market due to their many unique chemical and physical properties [1, 2]. Next-generation computer devices, consumer electronics, wireless LAN devices, wireless antenna systems, and cellular phone systems are few portable device applications that require these composite materials, because nanocomposites have the potential to significantly surpass the physical properties of conventional bulk materials. As heavy heat transfer materials have delayed the development of portable devices, it is required to develope light-weight, flexible, inexpensive, and wearable composite materials, which has high heat-conductivity [3–6]. Therefore, to develop the CNT-composite materials, cellulose is considered due to being biosource, light-weight and biologically compatible [7, 8]. The problems in developing the composites would be dispersion of nanotubes in solvents, CNT-polymer interactions, manufacturing cost, and performance of the composite. Cellulose has attracted much attention with expertise in diverse areas [9, 10] as a renewable source-based biodegradable polymer and will be medium for displaying and transmitting information owing to its potential compostibility, mechanical stability under atmospheric conditions, and ability to absorb liquid [11, 12]. Carbon nanotubes (CNTs) are regarded as one of the most versatile additives of composites because it can improve mechanical, thermal, and electrical characteristics of cellulose [13–15]. Especially, homogeneous distribution of CNTs in the cellulose matrix would contribute to improve its characteristics. However, the nonreactive surface and strong agregation properties of CNTs due to van der Waals attractive force have limited the effectiveness of CNTs with cellulose during the time of mixing [16]. Previously it is found that chemical modification of CNT is the most successful dispersion technique which forms carboxyl functional groups on the surfaces and the ends. This carboxyl functional group binds the CNTs with cellulose very tightly. On the other hand, chemical modification would considerably alter their desirable properties. Therefore, CNT dispersion in solvents is prerequisite to utilize the unique multifunctional properties of CNTs in case of preparation of CNT/cellulose composites [17]. For this purpose, in this report, biofriendly material, gelatin is used to disperse the CNTs before mixing with cellulose. Gelatin wraps the surface of CNTs enabling their dispersion in water and does not affect the physical properties of CNTs while enabling their dispersion in aqueous solutions [18]. CNTs form a conducting network using their electron donating and accepting ability in the composite [19–22].

These CNT/cellulose composite materials have some outstanding properties such as mechanical toughness, electrical and thermal conductivity, and the usage of the materials that have been investigated [17, 23–25]. In this study, CNT sheets have been prepared by the paper making process which is on the easy, simple, and readily scalable process. Previous study has also not focused on sustaining the structure of the CNTs for making composite. Here, it is considered by using gelatin to disperse CNT before mixing with cellulose. Besides this, we have concentrated on the good environmental stability of composite and low cost combined with the easiness of preparation. The resulting CNT/cellulose paper sheets show high electrical conductivity, strong microwave absorbing properties, thermal stability, and wettability. To the best of our knowledge, no study has yet investigated the properties of composite by increasing the amount of CNTs. This study reports that the composite properties are improved by increasing amount of CNTs and it can be the product of any size and shape by maintaining the CNT/cellulose ratio. The properties of the composite are reported here.

2. Experimental Methods

To optimize the paper making process for CNT/cellulose composites and the quality of the resultant paper, it is important to improve the interaction between the pulp fibers, gelatin, and multiwalled carbon nanotubes (MWNTs). MWNTs (Sigma-Aldrich, outer diameter = 10–30 nm, inner diameter = 3–10 nm, length = 1–10 μm, and purity > 90%) and gelatin (Wako 1st Grade, appearance: yellowish brown and crystalline powder) are used as received. Cellulose fibers used here are made by pure paper, where cellulose is mixed well in pure water with an electric beater to get homogeneous pulp dispersion in water.

At first, Cellulose suspension is made by soaking the fibers into pure water and blended for 20 minutes. Then, 10 mg of MWNTs and 30 mg of gelatin are sonicated with 10 mL of pure water by using a supersonic homogenizer (Sonic & Materials VC 130, f = 20 kHz, 6 mmϕ probe) at an input power of 20 W for 60 minutes. Different CNT dispersions are prepared by using 5, 10, 15, 20, and 30 mg of MWNTs. The concentration of the gelatin in CNT dispersion is kept fixed at 3 mg/mL. Then 300 mg of cellulose suspension is added to the 10 mL of CNT dispersion and blended for 20 minutes. The final solution is poured into a tray dish and dried at room temperature to get a paper sheet. The paper making process is schematically shown in Figure 1(a). Figure 1(b) shows the photographs of pure cellulose sheet and the CNT/cellulose sheet. The obtained paper sheets have a thickness of 0.2–0.4 mm. The weight ratio of CNT to cellulose is about 1/30, 1/15, and 1/10, when 10, 20, or 30 mg of CNT is added.

Electrical resistance, R, of the sheet is measured by a digital multimeter (Iwatasu VOAC 87, Japan). Two nickel electrodes (thickness of 0.15 mm, purchased from Nilaco, Japan) of width 5 mm are placed on the CNT/cellulose sheet at a distance of 50 mm and a dc voltage V_{in} is applied by a regulated power supply (Kikusui Electronics PAB 350-0.2, Japan) between the two nickel electrodes. The image of the uniform distribution of MWNTs in the CNT/cellulose composite is taken by a SEM (JEOL JSM 6510LV) at an operating voltage of 10 kV. Raman spectra are obtained with a Raman spectrometer (JASCO NR-1800) using a laser for excitation (λ = 532 nm). The thermal stability of the paper sheet is observed by a TG/DTA analyzer (Rigaku Thermo Plus TG8120). The temperature of the CNT/cellulose paper sheet is measured by using a thermometer (K type thermocouples, 0.2 mm in diameter, Custom CT-2000D) and an infrared camera (FLIR i3, emissivity 0.95, focus length = 6.8 mm). A gun-diode microwave transmitter and a recorder (Pasco Scientific WA-9314B, P_{μ} = 10 mW) of 10.5 GHz with horn antenna are used to measure the microwave absorption characteristics of the sheet.

3. Results and Discussion

Figure 2 shows the morphology of the CNT/cellulose paper sheets which is observed by SEM. The paper surface is smooth when no MWNTs are coated as shown in Figure 2(a). In Figures 2(b)–2(d), CNTs cover surface of almost all fibers of the sheet. Some CNTs seem to form bridge-like structure with paper fibers. The SEM images clearly reveal that the interconnected networks are established by the individual CNTs so that numerous electrical paths can be formed.

In addition to the SEM observation, Raman measurement is also carried out to prove that CNTs are present in the paper sheets. Raman peaks of both G-band and D-band generated from CNTs are observed as shown in Figure 3.

Owing to the formation of conducting paths, the paper sheet shows considerable low resistance. Resistance is measured of ten randomly chosen measuring points on both sides (A side and B side) of a CNT/cellulose paper sheet by the needle-electrodes, and the distance between needle-electrodes is kept constant at 20 mm. The distributions of the resistances are shown as histograms in Figure 4. It is found that the electrical conductivity of the CNT/cellulose paper sheets is highly uniform. In case of 5 mg MWNTs added sheet, the A side has resistance, R = 2.61 MΩ (standard deviations, s = ±0.08, electrical conductivity, σ = 1.91 mS/m) and the B side has resistance, R = 2.60 MΩ (standard deviations, s = ±0.05, electrical conductivity, σ = 1.92 mS/m). When 10 mg of MWNTs is added, the A side has resistance, R = 1.14 MΩ (standard deviations, s = ±0.04, electrical conductivity, σ = 4.38 mS/m) and the B side has resistance, R = 1.103 MΩ (standard deviations, s = ±0.05, electrical conductivity, σ = 4.53 mS/m). Therefore, it is noticed that the conductivity can be improved by increasing the amount of CNTs added to the cellulose. Current-voltage, I-V_{in} characteristic curve is measured by using Ni-rod-type electrodes as shown in Figure 5 and the Ohmic conduction on the paper sheet is obtained.

TG/DTA experiments for the cellulose sheet and the CNT/cellulose sheet are carried out at a heating rate of 10°C/min from room temperature to 800°C and the curves are shown in Figure 6. DTA curves show that the thermal

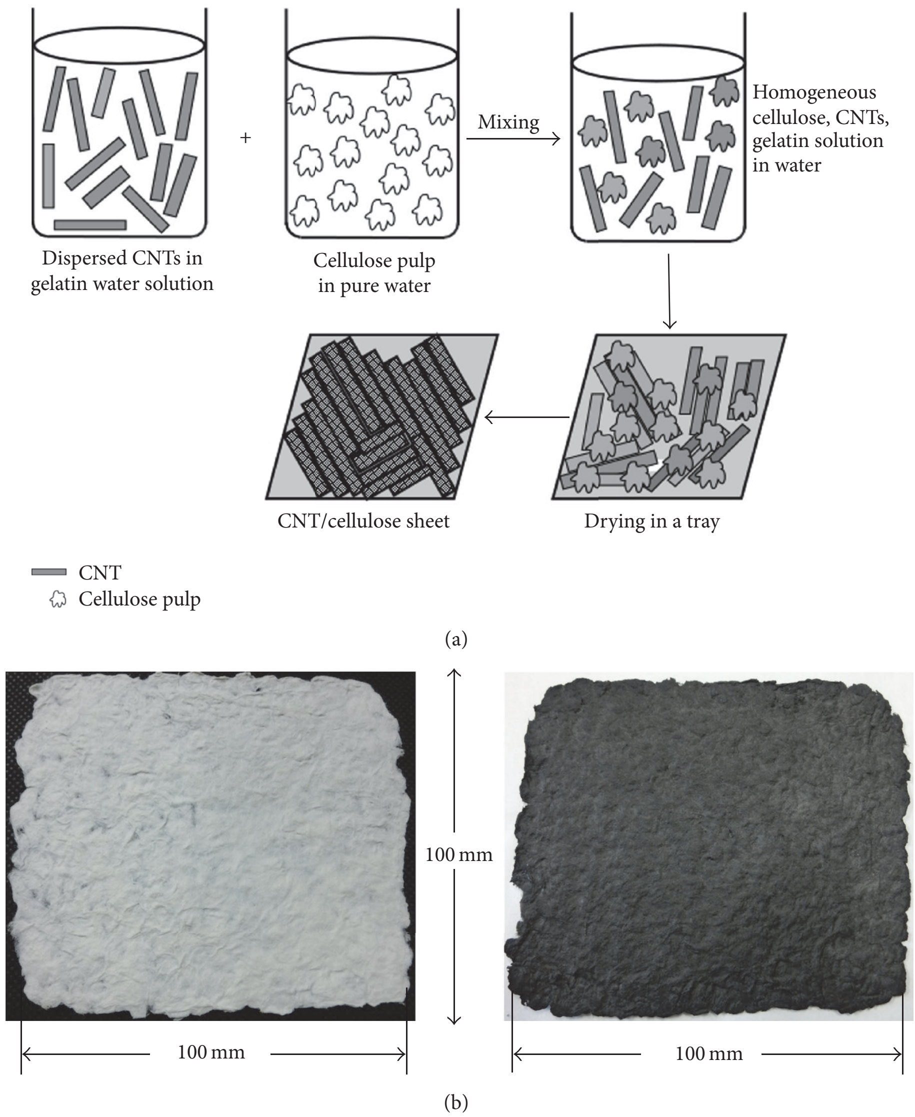

Figure 1: (a) A schematic of the paper making process for the fabrication of CNT/cellulose composite sheets. (b) Photographs of a pure cellulose sheet (left) and a CNT/cellulose sheet (right).

degradation of both sheets occurs in two different steps in air atmosphere. In the first step of degradation, the pure cellulose sheet and the CNT/cellulose sheet begin to lose weight at 332 and 355°C, respectively, and the final combustion in the second step occurs at 409 and 507°C, respectively. The final combustion of the CNT/cellulose sheet delayed by ca. 100°C is clearly observed from the DTA curves, which indicates that the cellulose sheet loses weight faster than the CNT/cellulose sheet. From the TG curves, it is observed that the combustion of CNT/cellulose sheet is 22% less than that of the pure cellulose sheet. This result signifies that the thermal stability of the sheet is improved by adding MWNTs [26, 27]. There is also a hump shape peak at 463°C on the CNT/cellulose curve before the final combination peak which corresponds to the combustion of the gelatin [18].

The flame retardancy test is carried out for both the cellulose and CNT/cellulose sheets. The image of the combustion process is shown in Figure 7. Both sheets with same thickness are hanged on a metal supporter and ignited simultaneously by a gas flame. It is observed that the cellulose sheet burns quickly to be ash completely within 20 s, while the CNT/cellulose takes more than 30 s to burn and turns to charcoal sheet, which indicates that added MWNTs improve the flame retardancy of the sheet. It is conjectured that the flame retardancy of the nanocomposites is strongly affected by MWNTs network structures on the cellulose [28]. The flame retardancy is attractive for the use in industrial applications.

The sheet could be used as a flat-type electric heater. The heating test is carried out by measuring the change in the temperature under the applied voltage, V_{in} = 80–220 V. The schematics of the measurement are shown in Figure 8(a). Three layers of CNT/cellulose sheets ($d = 1.2$ mm) have been used to reduce resistance lowly enough by which

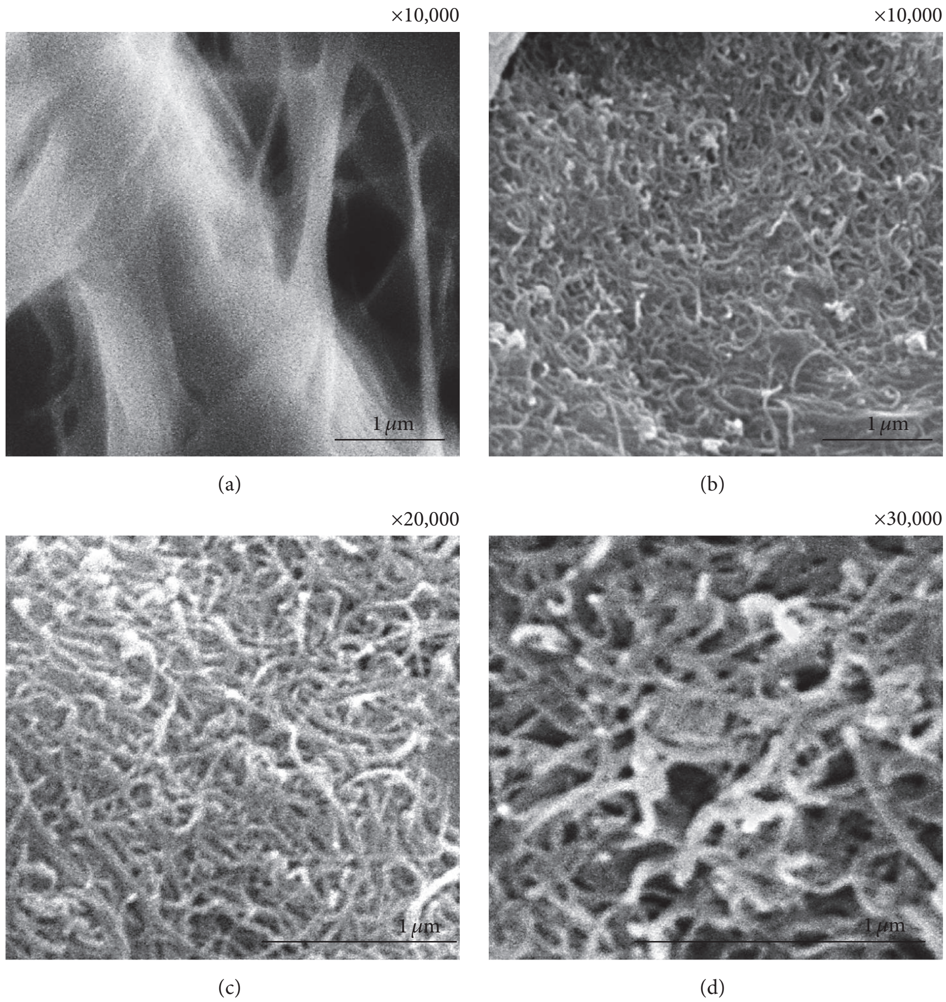

Figure 2: SEM images of (a) the pure cellulose sheet and (b)–(d) the CNT/cellulose sheet at different magnifications taken at an operating voltage of 10 kV.

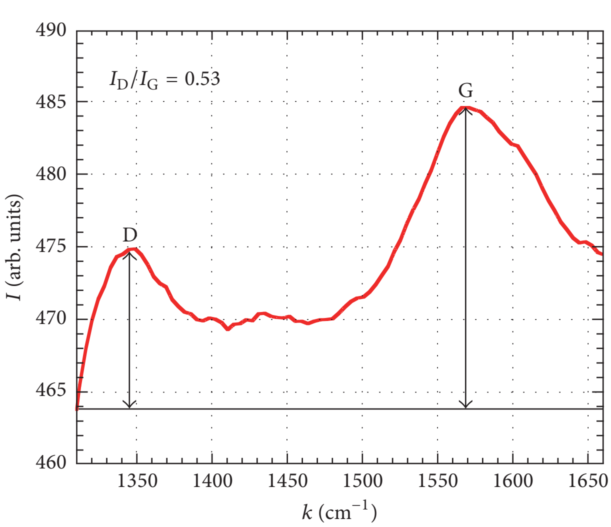

Figure 3: Raman spectrum of the CNT/cellulose paper sheet. The G and D bands show the quality of CNTs.

the electric current through a CNT/cellulose sheet releases heat due to the Joule heating process [29, 30]. Heating and cooling temperatures of the sheet are measured at the centre of the sheet. Figures 8(b) and 8(c) show the time variation of temperature of the CNT/cellulose sheet measured by a thermocouple and by an IR camera during heating and cooling process. It is observed that surface temperature starts to increase with time by applying a dc voltage, and the IR emission from the sheet increases. The surface temperature of the CNT/cellulose sheet reached a maximum temperature of 120°C within 5 minutes depending on the applied voltage. By increasing the number of layers of the CNT/cellulose sheets more, the sheet resistance can be decreased. The cooling rate of the sheets is also measured. During the test, all the samples are heated to 35 minutes and then cooled naturally. It is observed that the temperature decreases exponentially to the room temperature within few minutes. The thermocouple-based measurement shows a little lower value of temperature than the infrared-based measurement.

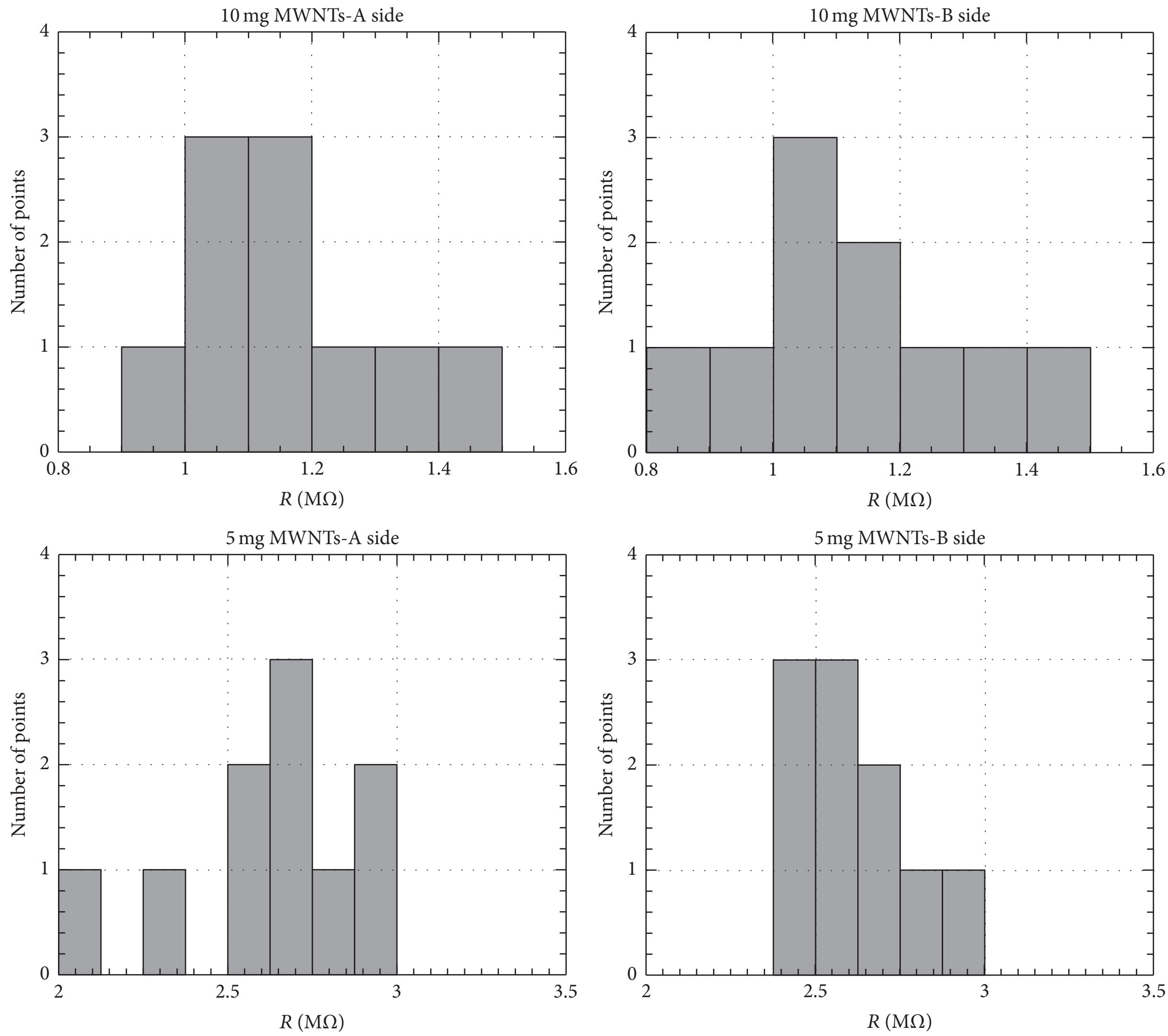

Figure 4: Histograms of measured resistance on the both sides of the CNT/cellulose paper sheet. Resistances are decreased with the increase of MWNTs content.

This difference would arise from the slow response of the thermocouple due to the slow heat propagation [31]. The IR image of the CNT/cellulose sheet is shown in Figure 8(d), which indicates the uniformity of the surface temperature of the area (50 mm × 50 mm). It is clearly shown that the highest temperature area is located at the centre of the sheet. To study the flexibility of the CNT/cellulose sheets, the temperature and current are also measured during bending the sheet more than 90° for 20 times, and the total time elapsed for the whole process is 80 minutes. Figure 8(e) shows the flexible nature of the paper sheet, because repeated bending of paper sheet does not make any significant change of the current conduction through the paper sheet, and temperature also remains almost constant. These results mentioned above indicate that the CNT/cellulose sheet can be used as a flexible electrothermal heating element.

The microwave absorption ability of the CNT/cellulose sheet is measured by a microwave transmitter of constant frequency 10.5 GHz ($\lambda = 2.85$ cm) and emission power P_μ = 10 mW. A schematic representation of the measurement of microwave absorption ability is shown in Figure 9(a). The CNT/cellulose sheet is made with a size of 100 mm × 100 mm × 0.3 mm for this absorption test experiment. The same size of pure cellulose sheet is made for comparison. The pure cellulose sheet and the CNT/cellulose sheet are placed on a thin plastic substrate during the experiment. In this absorption ability experiment, the incident microwave is partly absorbed by the sheet, partly reflected, and partly transmitted through the sheet. To determine the reflection ability of the sheet, the transmitter and the recorder are set in such a way that the incident and reflected wave make equal angles to the surface normal as shown in Figure 9(b).

Figure 10 shows the experimental result of the absorption and transmission ability versus the thickness. By increasing thicknesses, from 0.3 to 1.8 mm, content of MWNTs increases from 30 to 150 mg in the sheet. The absorption increases

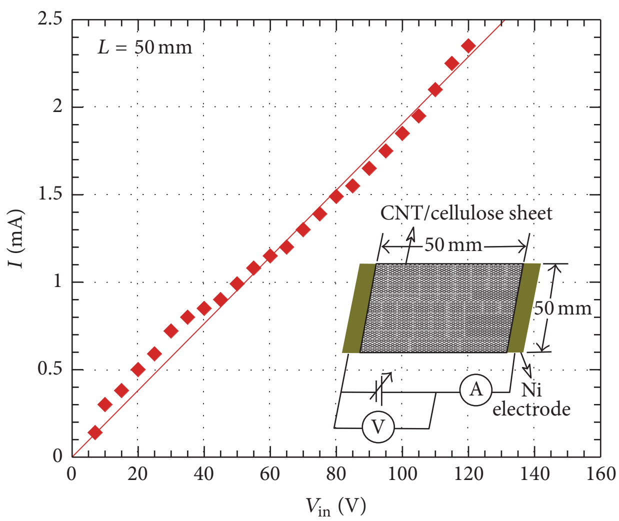

Figure 5: I-V_{in} curve for the sheet showing the linear electric conduction (inset: schematic of the I-V_{in} characteristic measurement setup).

with the increase of MWNTs content. Several sheets of same thickness and same content of MWNTs (30 mg) are used for this experiment. During the experiment, thickness is increased by adding sheets one after one on the plastic sheet. Absorption ability is found to increase with the increase of thickness as well as MWNTs content. This increase of absorption could occur due to the increase of absorption volume of the sheet.

Figure 11 shows that the CNT/cellulose sheets exhibit very small reflection, and the reflection increases with the increase of thickness of the sheet. It is conjectured that interfacial electric polarization improves with the increase of MWNTs content due to the interaction of microwave radiation with the large number of charge dipoles between the MWNTs and cellulose, which would cause reflection [32]. The results can be comparable with the previous examinations with the other composites of MWNTs [33, 34]. This means that there are several considerable factors, like the MWNTs content, thickness of sheet, the interfacial electric polarization, and so on, to design microwave absorbing materials [35]. It is also found that a zigzag setup of 4 sheets of same dimensions, each of which contains 30 mg MWNTs, can absorb about 50% of the incident microwave. Considering the above results, this composite sheet has potential advantages as an electromagnetic shielding material (EMS material) at the microwave region and the ultrahigh frequency (UHF) region.

The aqueous absorption speeds are conducted to understand the microwettability of the composites. Generally cellulose sheets have high absorption speed which in turn degrades their mechanical properties. CNT/cellulose composite shows the significant improvement on the mechanical property without degradation resulted from the addition of carbon nanotubes in the cellulose. In the bio and chemical analyses, liquid-absorption of cellulose sheets is commonly used.

The pure cellulose sheet and the CNT/cellulose sheet (including 10~30 mg of MWNTs) are prepared with a size of 100 mm × 20 mm × 0.3 mm. In this experiment, one end of the sheet sample is immersed in a color ink or a saline liquid. Both cellulose and CNT/cellulose sheets start to absorb aqueous solution immediately after the immersion. The sheets are kept in the solution until they get completely wet. The schematic of the absorption speed measurement is shown in Figure 12(a). The absorption speed is measured from this experiment and compared with those of the pure cellulose sheet. Absorbed saline amount by the sheet is calculated by measuring the weight changes of the sheets after the experiment.

It is evident from Figure 12(b) that the absorption speed of the cellulose sheet is very high in both color ink and saline liquid. On the other hand, the CNT/cellulose sheet has low absorption speed. A high DC voltage of about 300 V is applied through Ni electrodes to the CNT/cellulose sheet to investigate the changes in speed, and it is found that the absorption speed with application of the electric field increases significantly in both cases of color ink and saline liquid. The various voltages are also applied by changing the polarity, and the polarity does not have noticeable effects on absorption speed. It is also evident from Figure 13 that the absorption speed increases with the increase of MWNT content and the applied voltage. This indicates that the absorption speed and absorbed aqueous amount of the CNT/cellulose sheet can be controlled by the MWNT loading. By addition of MWNTs and high voltage in the sheet, the sheet becomes electroconductive and the electric field could control the mobility of ionic liquid. The absorption speed is also related to other factors, for example, cellulose pulp content, and dispersion state of MWNTs in the sheet. By adding MWNTs in the pulp sheets, the absorption speed of liquid increases, but by adding DC voltage the speed can be controlled.

4. Conclusions

This work provides a simple and effective method of preparing CNTs/cellulose sheets. The sheets are physically strong and yet highly flexible. This CNTs/cellulose sheet exhibits improved electrical and thermal properties because of the good interaction between cellulose and MWNTs. Better mechanical properties could be expected at the optimized processing condition. The sheet can be used as flexible electrothermal heating elements due to the thermal conduction. The high microwave absorption and low reflection performance of the sheet indicate the usefulness of the composite for microwave technology applications. This sheet could be also used as electromagnetic shields (EMS) for high-frequency devices. The water absorption speed of the CNT/cellulose sheets can be controlled by applying DC voltage. As cellulose is biocompatible and biodegradable, applications of the sheet will be expanded to bioscience.

Competing Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

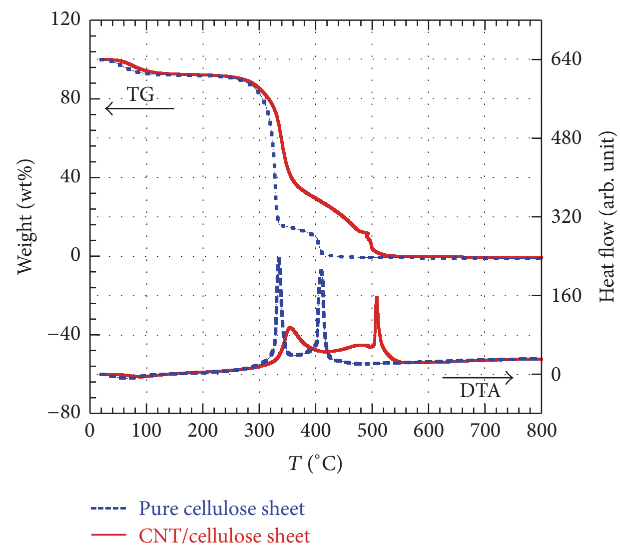

FIGURE 6: TG/DTA curves for a pure cellulose sheet (blue dotted line) and a CNT/cellulose sheet (red solid line).

FIGURE 7: Photographs of flame retardancy test for the pure cellulose sheet (left white sample) and the CNT/cellulose sheet (right black sample). At $t = 0$ s, a fire is added on the bottom of the samples.

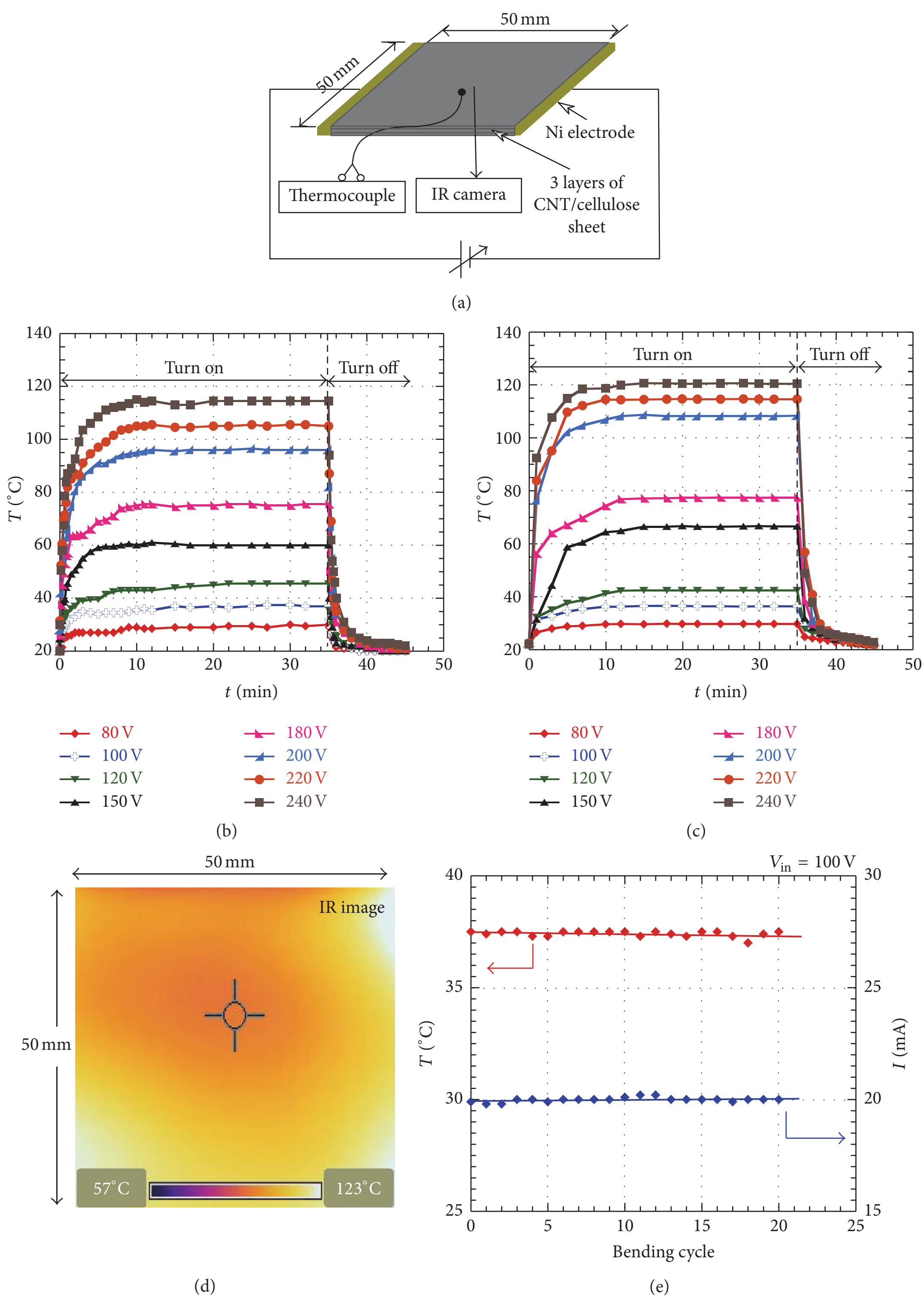

FIGURE 8: (a) Schematic of the heating test measurement of the CNT/cellulose sheet by a thermocouple and an IR camera. Temperature versus time curves during heating and cooling of the CNT/cellulose sheet measured (b) by a thermocouple and (c) by an IR camera. (d) IR image of the sheet at V_{in} = 220 V (I_{in} = 6 mA) showing the uniform surface temperature of the area (50 mm × 50 mm) where the temperature scales are shown at the bottom part. (e) Variation of temperature and current after repeated bending, where the input voltage V_{in} = 100 V (I_{in} = 2 mA).

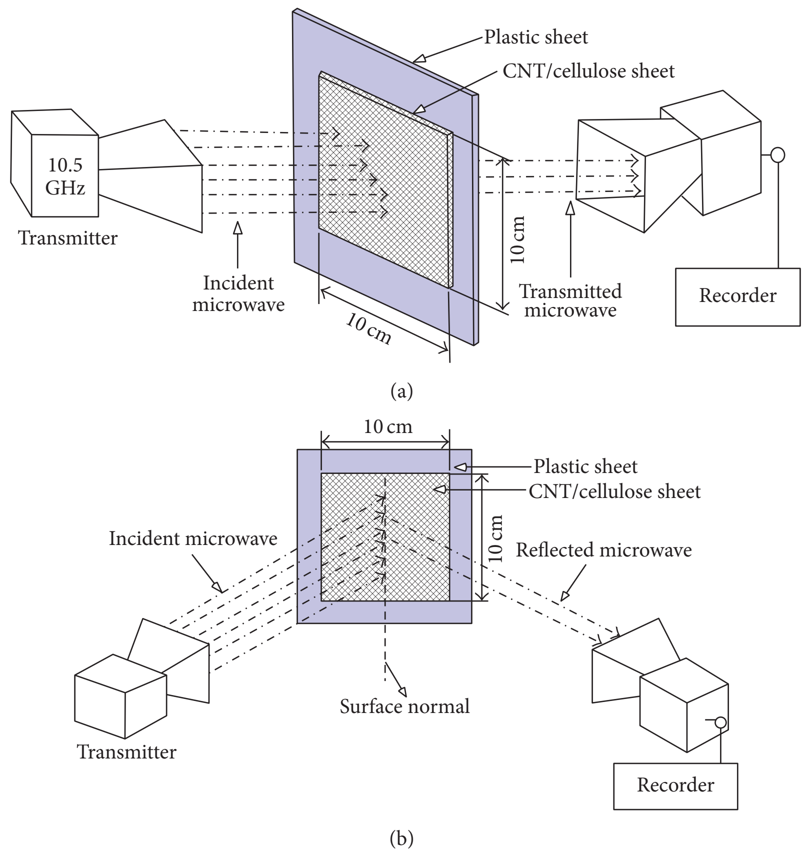

Figure 9: Schematic representation of the (a) microwave absorption measurement and (b) reflection measurement of the CNT/cellulose sheet.

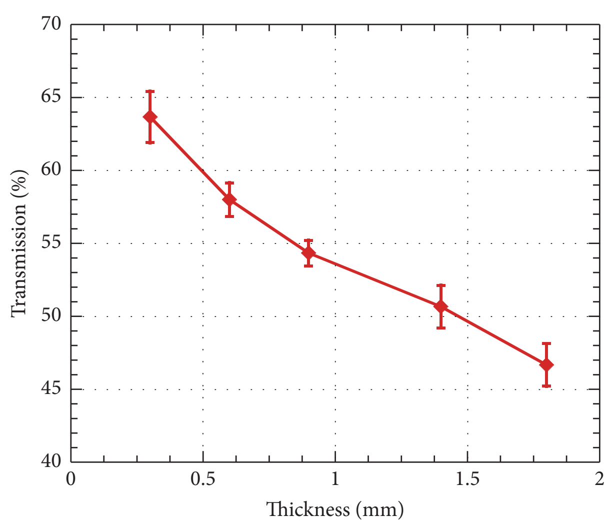

Figure 10: Variation of microwave transmission property of the CNT/cellulose sheet with the increase of thickness, where MWNT content increases with the thickness.

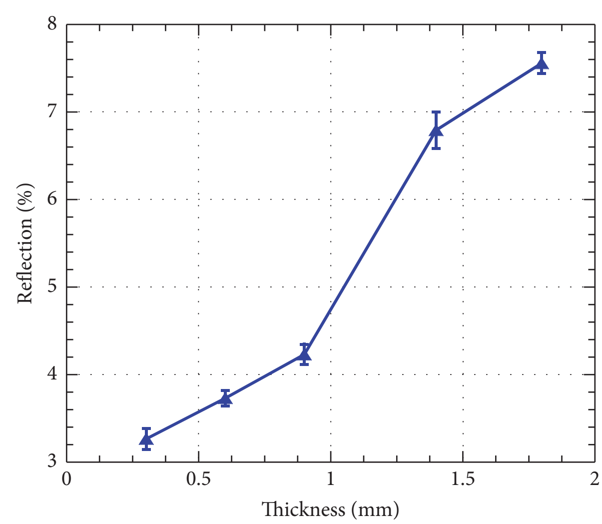

Figure 11: Variation of reflection property of the CNT/cellulose sheet with the increase of thickness, where MWNT content increases with the thickness.

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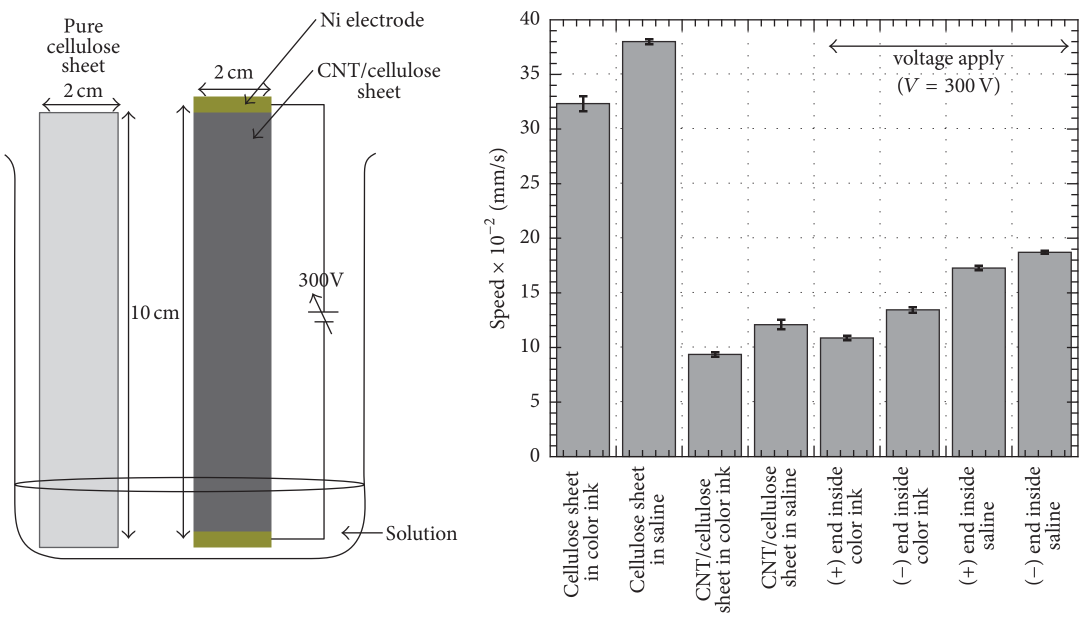
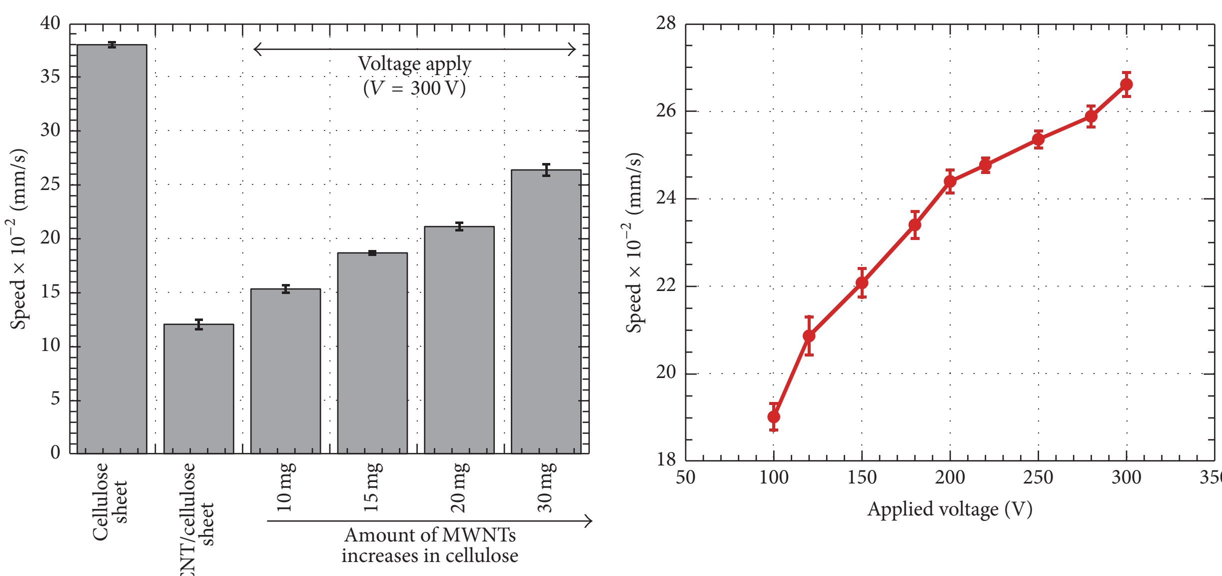


(a) (b)

Figure 12: (a) Schematic of the absorption speed of liquids measurement of the CNT/cellulose sheet and the pure cellulose sheet. (b) Absorption speeds of the cellulose sheet and the CNT/cellulose sheet in different solution conditions.



(a) (b)

Figure 13: (a) Absorption speed of the cellulose sheet and the CNT/cellulose sheet with the increase of MWNT content. (b) Absorption speed of the CNT/cellulose sheet with the increase of applied voltage.

done at Research Institute of Electronics, Shizuoka University.

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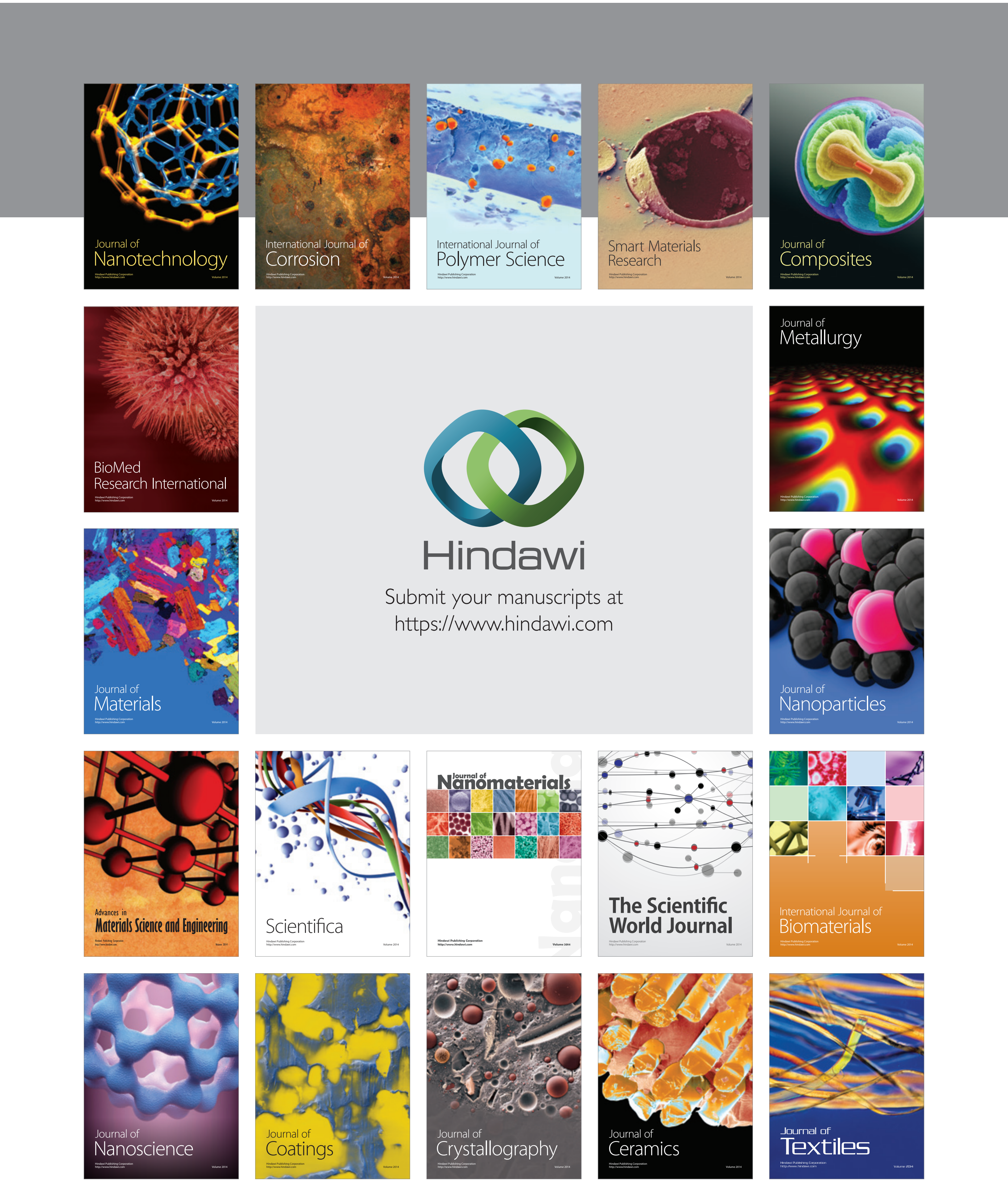
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