Strontium-Modified Bacterial Cellulose and a Polyvinylidene Fluoride Composite as an Electroactive Material

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Abstract

Sr^{2+} was chemically grafted onto the hydroxyl position of bacterial cellulose. Fourier transform infrared and UV-Vis spectroscopy were used to evaluate the chemical bonding of the modified bacterial cellulose structure. Then a chemical bond between Sr-O was formed. The presence of strontium was confirmed using X-ray fluorescence. A significant enhancement in the dielectric properties was observed. Next, a small amount of modified bacterial cellulose was prepared as a binary blend composite. The ratio between polyvinylidene fluoride and the modified bacterial cellulose was investigated. They had superior thermal stability and mechanical properties compared with the neat modified bacterial cellulose. The degradation temperature was higher than 300°C. The outstanding property of the composite was its flexibility. The excellent properties of the composite of modified bacterial cellulose and polyvinylidene fluoride make it a good electroactive biocomposite candidate.

There has been rapid development in bio-based materials. Numerous approaches have been applied to many types of bio-based material for a range of engineering purposes. The engineering role of bio-based materials has been studied in both academic and industrial research (Faruk et al. 2012; Ummartyotin and Manuspiya 2015a, 2015b). Their applications include infrastructure, automotive parts, medical, pharmaceutical products, and electronic devices. In the field of electronic devices, our research group focuses on bio-based materials for electronic substrates (Nogi and Yano 2008, Ummartyotin et al. 2012a, O-Rak et al. 2014, Ummartyotin and Sain 2014). A key achievement has been the organic light-emitting diode. From a fundamental viewpoint, in the organic light-emitting diode, the emissive electroluminescent layer is a film composed of an organic compound that emits light in response to an electric field. This organic semiconductor layer is placed between two electrodes, at least one of which is commonly transparent (Shinar and Shinar 2011, Thejokalyani and Dhoble 2012). This achievement required the development of transparent substrates and conductive electrodes.

In contrast, from an industrial commercialization viewpoint, glass and conductive ceramic electrodes are used as substrates. Although the conductive ceramic electrode provides good energy conversion to weight, its stiffness and brittleness limit its use when notably small displacements are required. Moreover, the processing of ceramic-based materials often requires high temperatures, cost-effectiveness, and safety. Therefore, the development of transparent polymers has been encouraged to provide a substrate for use in electronic devices. Their flexibility and ease of processing make them attractive for integration with microelectromechanical systems. However, in recent years, the use of polymeric-based materials has been viewed less favorably. Concern over the shortage of petrochemical materials and the ever-growing issue of global waste, which is driven by rising oil prices and the pending exhaustion of landfills, has created a demand for eco-friendly materials. Numerous types of bio-based materials, such as cellulose, chitin-chitosan, and polylactic acid, have been investigated as replacements for

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petrochemical materials (Ravi Kumar 2000, Averous 2004, Bhatnager and Sillanpaa 2009, Balan and Verestiuc 2014). One of the most effective bio-based materials is cellulose. Remarkably, cellulose can be prepared from two different approaches. In one approach, cellulose from forestry is prepared as feedstock in the top-up route. Cellulose from forestry is prepared in small dimensions, and chemical modification is used to purify cellulose from any impurities. In the other approach, cellulose can be prepared from the bacterial species *Acetobacter xylinum,* which is one of the most effective species for bacterial cellulose preparation and has high percent yield and uniform size and shape. Both cellulose preparation routes are promising for nanoscale networks in terms of diameter because of the high flexibility and transparency.

Our research groups have successfully developed a bacterial cellulose–based composite for electronic substrates (Ummartyotin et al. 2012a; O-Rak et al. 2013, 2014; Ummartyotin and Sain 2014). The performance of this bacterial cellulose–based composite was designed with additional features of flexibility and transparency. The surface of the bacterial cellulose–based composite and its water vapor absorption ability were improved using ferrofluid abrasion and Si-O film formation, respectively (Ummartyotin et al. 2011, 2012b). To make the use of the bacterial cellulose–based composite more attractive, the electroactive properties of the bacterial cellulose–based composite were improved. A multiwall carbon nanotube was successfully grafted onto the hydroxyl position of the bacterial cellulose unit, and the bacterial cellulose was successfully blended with an electroactive polymer. More recently, bacterial cellulose and polyvinylidene fluoride were successfully prepared as a miscible blend composite. The bacterial cellulose–based composite satisfied all criteria for use as an electroactive material. Until now, electroactive materials were used in various sectors of industrial research, such as touch-screen applications in electronic devices. The correlation between the electric and mechanical properties of a material can be manipulated. Furthermore, the composite was used in medical and pharmaceutical research in artificial muscles because of its ability to emulate the operation of biological muscles with fracture toughness, large actuation strain, and inherent vibration damping. The composite has a high potential for use in the design of microfluidics, such as drug delivery systems, microfluidic devices, and lab-on-a-chip systems. From a fundamental viewpoint, a microfluidic platform technology is based on stimuli-responsive gels. To avoid the electrolysis of water and microfluidic devices, the use of an electroactive material is significantly appreciated.

The objective of this study was to develop a new type of composite of strontium-grafted bacterial cellulose and polyvinylidene fluoride. The grafting of strontium onto bacterial cellulose significantly enhanced the polarity of the composite. The structural, morphological, and thermal properties of the modified composite were investigated. A preliminary experiment on the dielectric properties was conducted.

Materials and Methods

Materials

Bacterial cellulose was successfully extracted from the nata de coco product (Chaokoh coconut gel in syrup; Ampol Food Processing Ltd., Nakornpathom, Thailand). This is an indigenous dessert whose main component is bacterial cellulose. Bacterial cellulose extracted from nata de coco was characterized in a previous work (Ummartyotin et al. 2012a). Its characteristics matched those of bacterial cellulose extracted from a culture of *A. xylinum*. Strontium chloride with a molecular weight of 266 g/mole was purchased from Ajax Fine-chemi Co. Ltd. (Thailand). Polyvinylidene fluoride with a molecular weight of 20,000 g/mole was purchased from Sigma Aldrich Co. Ltd. (Thailand). Analytical-grade dimethylformamide was purchased from Sigma Aldrich. All chemical reagents were used as received without further purification.

Methods

Bacterial cellulose extraction and purification.—Bacterial cellulose was extracted from the nata de coco gel, which was rinsed with distilled water to remove excess sugar and blended in a laboratory blender to obtain nata de coco pellicles. The nata de coco pellicles were treated with 0.1 M NaOH at 80°C for 1 hour to remove any remaining microorganisms, medium components, and soluble polysaccharides. Bacterial cellulose was extracted and modified as in a previous study (Ummartyotin et al. 2012a, O-Rak et al. 2014).

Preparation of bacterial cellulose–Sr^{2+} complex.—Chemical modification of bacterial cellulose was performed in a chemical grafting process with strontium ions. First, 1, 3, 5, and 10 wt% of strontium chloride was poured into a bacterial cellulose–based water suspension with pH 7. The reaction time and temperature were 3 hours and 80°C, respectively. The structure of the modified bacterial cellulose was investigated using Fourier transform infrared (FTIR) and UV-Vis spectroscopy. Chemical modification of bacterial cellulose by grafting with Sr^{2+} ions is shown in Figure 1. Prior to the structural investigation, the bacterial cellulose–Sr^{2+} complex was stored in a refrigerator at a controlled temperature of 4°C.

Preparation of a Sr^{2+}-grafted bacterial cellulose and polyvinylidene fluoride composite.—Sr^{2+}-grafted bacterial cellulose was embedded into the polyvinylidene fluoride composite. First, 1 g of Sr^{2+}-grafted bacterial cellulose was prepared in a water-based suspension. In parallel, 1 g of polyvinylidene fluoride was dissolved in dimethylformamide. The time and temperature were set to 2 hours and 80°C, respectively. Then 1 g of the Sr^{2+}-grafted bacterial cellulose suspension was added to the polyvinylidene fluoride solution, and the mixture was continuously stirred for 3 hours. To prepare the composite, the solvent was removed from the suspension by filtration through a Buchner funnel, which was fitted with a polytetrafluoroethylene membrane filter (0.1-μm mesh, 90-mm diameter) and connected to a Buchner flask and vacuum pump. Then the composite was dried between two polytetrafluoroethylene membranes under an applied pressure of 58 psi following standard SCAN C 26:76 (Laboratory Sheet Press, Pulp Testing Instrument).

Thermogravimetric analysis, X-ray fluorescence (XRF) spectroscopy, scanning electron microscopy (SEM), and a universal testing machine were used to investigate the thermal decomposition, elemental analysis, morphological properties, and mechanical properties, respectively. To investigate the potential application of the composite as an electroactive material, an experiment on the dielectric properties was conducted.
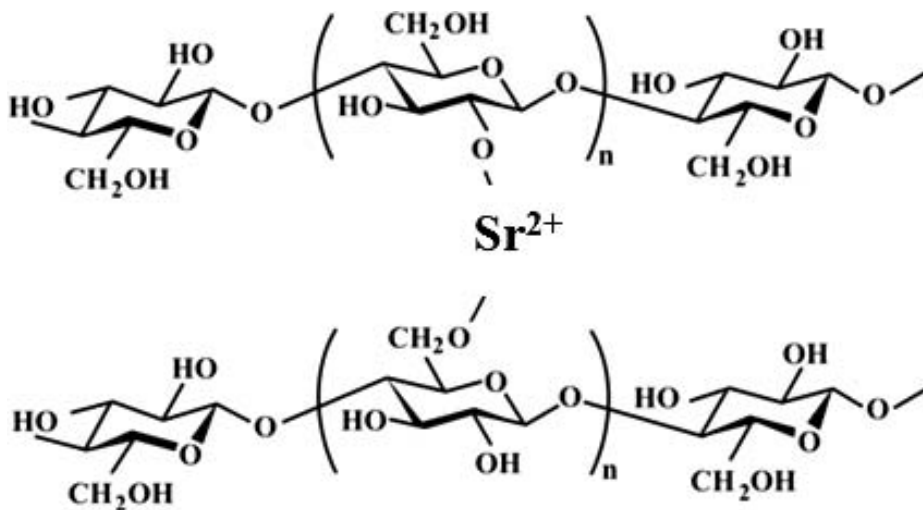
Figure 1.—Chemical modification of bacterial cellulose with Sr^{2+}.

Characterization technique

Fourier transform infrared.—FTIR was performed with a Bruker Vector 22 mid-IR spectroscope (Bruker, Germany). All FTIR absorption spectra were recorded in the 4,500- to 500-cm^{-1} wavenumber region at a resolution of 8 cm^{-1} with 1,024 scans using a deuterated triglycine sulfate detector. A straight line between the two lowest points in the respective spectra region was selected as the baseline.

UV-Vis spectroscopy.—A Varian Cary 5000 UV-Vis NIR spectrophotometer (Agilent Technologies, USA), which was equipped with a transmittance accessory, was used to record the electronic spectrum of the samples over wavelengths of 200 to 700 nm. This record enabled the absorbance spectra of the samples to be studied. The accessory was composed of a 110-nm-diameter integrating sphere and an in-built high-performance photomultiplier. Each sample was placed in a sample cell that was specifically designed for this instrument. A baseline was recorded and calibrated using a polytetrafluoroethylene reference cell.

X-ray fluorescence.—A Phillips 1404 XRF Wavelength Disperse Spectrometer was used to determine the trace elements of the sample. It was equipped with an array of five analyzing crystals and fitted with an Rh X-ray tube target. A vacuum medium was used in the analyses to avoid the interaction of X-rays with air particles. One gram of sample was mixed with 6 g of H_3BO_3 and compressed under 10 tons of force.

Field emission SEM.—The morphological properties of the bacterial cellulose–based hydrogel were investigated with a field emission SEM (S-4800; Hitachi) at an acceleration voltage of 2 kV. Prior to the investigation, the samples were stored in desiccators to reduce the humidity. Each sample was placed on a carbon tape and sputtered with gold particles before analysis.

Thermogravimetric analysis.—The thermal degradation characteristics of the bacterial cellulose–based hydrogel were investigated using thermogravimetric analysis (TGA; Perkin Elmer). Each sample (10 mg) was heated at a heating rate of 10°C/min in a nitrogen atmosphere from room temperature to 500°C. The TGA balance flow meter was set at 20 psi for N_2, whereas the purge flow meter was adjusted to 20 psi for synthetic air.

Determination of mechanical properties.—Tensile tests were conducted in accordance with ISO 527:1996 (International Organization for Standardization 1996). The sample was cut with a Zwick cutter into a dog-bone specimen type 5A. The samples had an overall length and gauge length of 75 and 20 mm, respectively. The initial distance between grips was 50 mm, and the width at the grip end was 12.5 mm. The width at the narrowest part was 4 mm, and the thickness was 0.3 to 0.5 mm. The testing speed was 1 mm/min. The specimens were preconditioned at 20°C in 54 percent relative humidity in a desiccator that contained a saturated solution of $Mg(NO_3)_2$ for at least 48 hours prior to testing. The tensile tests were conducted using a universal material testing machine (Instron 4502; Instron Corporation, USA) with a 1-kN load cell. At least five specimens were tested per sample to obtain a statistical average.

Dielectric spectroscopy.—The dielectric constant and dielectric loss were measured by a precision LCR meter (Agilent E4980A) at room temperature and various frequencies of 10^3 to 10^6 Hz. Prior to measurement, the sample was sputtered with a Pt coat as an electrode on both sides.

Results and Discussion

Chemical modification of bacterial cellulose by a Sr^{2+} graft

Modified bacterial cellulose was successfully prepared with grafted Sr^{2+} at the hydroxyl position. The features of the modified bacterial cellulose remained similar to those of the untreated cellulose sheet. The role of Sr^{2+} was to change the polarization under an applied electric field from free ions. Figure 2 shows the FTIR pattern of Sr^{2+}-grafted bacterial cellulose, and the pattern of neat bacterial cellulose is provided for comparison. The reaction between the hydroxyl group positions of bacterial cellulose was chemically bonded with the strontium ion. From the structural viewpoint, there are three positions of the hydroxyl group in the glucose unit of bacterial cellulose. Chemical bonding can occur at all positions of the hydroxyl group.

After Sr^{2+} was successfully grafted at the hydroxyl position of the bacterial cellulose, a peak appeared at 2,250
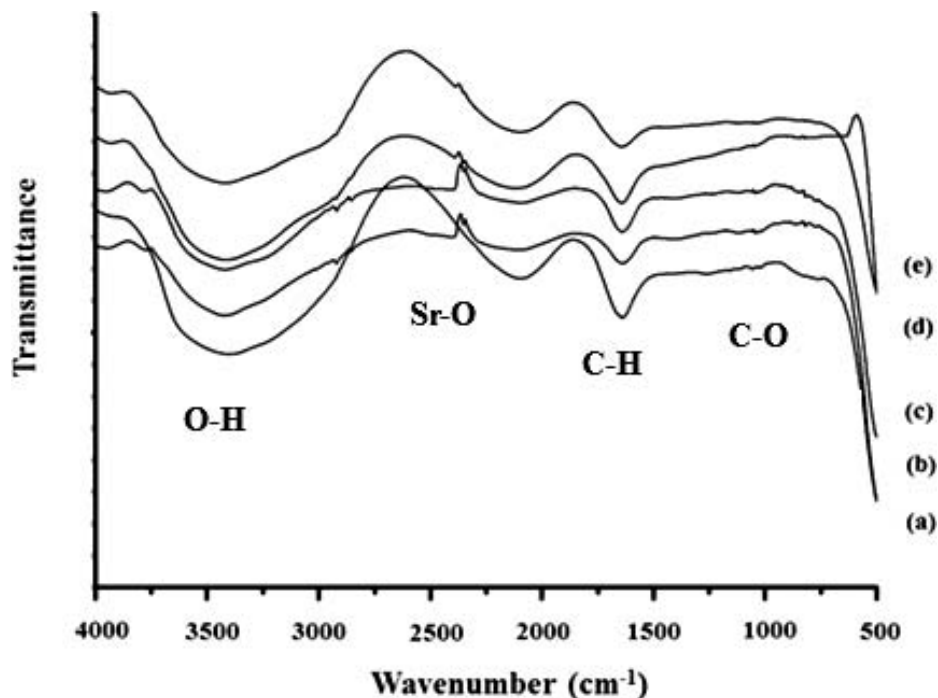
Figure 2.—Fourier transform infrared pattern of Sr^{2+}-modified bacterial cellulose: (a) neat bacterial cellulose, (b) 1 wt% of Sr^{2+}-grafted bacterial cellulose, (c) 3 wt% of Sr^{2+}-grafted bacterial cellulose, (d) 5 wt% of Sr^{2+}-grafted bacterial cellulose, and (e) 10 wt% of Sr^{2+}-grafted bacterial cellulose.

to 2,500 cm^{-1}. This peak is related to the chemical bonding of O-Sr^{2+} and can be ascribed to the cleavage of the primary and secondary hydroxyl groups in the coordination complex formation with Sr^{2+}. The broad peak at 3,000 to 4,000 cm^{-1} is attributed to the hydroxyl position (O-H) from the unreacted bacterial cellulose or any remaining humidity. The bacterial cellulose suspension was prepared by dispersing bacterial cellulose particles in water. The resulting sensitivity to water suggests that highly water-sensitive applications should be avoided. The peak in the region of 1,500 to 1,700 cm^{-1} represents the asymmetrical vibration of C-O.

The UV-Vis absorbance spectra of Sr^{2+}-grafted bacterial cellulose were investigated. Figure 3 shows the UV-Vis spectra of Sr^{2+}-grafted bacterial cellulose. There were two intense absorption bands at 250 and 350 nm in the UV-Vis spectra of the modified bacterial cellulose, which were assigned to the Sr^{2+}-O electronic charge transfer. The UV-
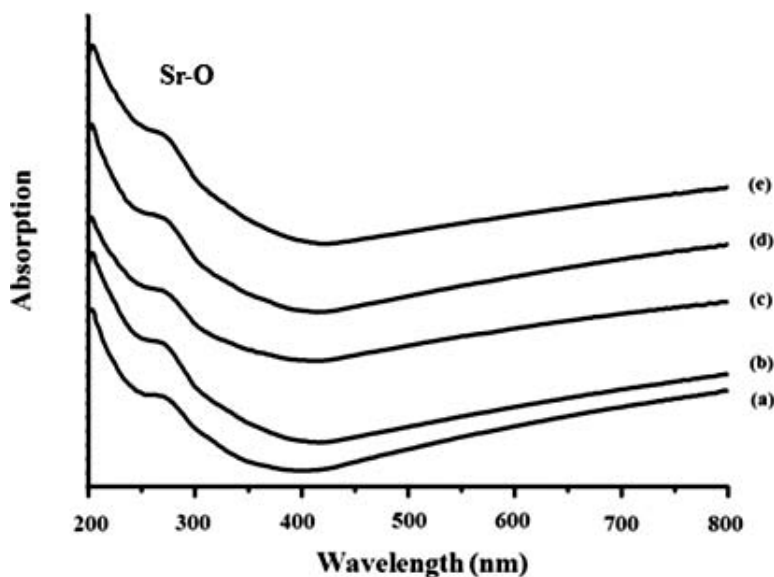

Figure 3.—UV-Vis spectra of Sr^{2+}-modified bacterial cellulose: (a) neat bacterial cellulose, (b) 1 wt% of Sr^{2+}-grafted bacterial cellulose, (c) 3 wt% of Sr^{2+}-grafted bacterial cellulose, (d) 5 wt% of Sr^{2+}-grafted bacterial cellulose, and (e) 10 wt% of Sr^{2+}-grafted bacterial cellulose.

Vis absorbance spectra of bacterial cellulose come mainly from the electronic charge transfer of the modified glucose unit. Thus, the bacterial cellulose structure promotes the probability of an intermolecular bond between Sr^{2+}-O electronic charge transfer with an energetic benefit. This bond formation is consistent with the FTIR spectra.

Investigation on the composite of Sr^{2+}-grafted bacterial cellulose and polyvinylidene fluoride

The thermal degradation behavior can be observed using TGA, as shown in Figure 4. The thermal degradation behavior of Sr^{2+}-modified bacterial cellulose and its binary blend with the polyvinylidene fluoride composite was investigated. Neat bacterial cellulose was provided for comparison. The thermal degradation of neat bacterial cellulose can be divided into three different regions. The first region is from room temperature to 250°C. A small weight loss was observed because of water evaporation from the bacterial cellulose network. A significant weight loss at 300°C to 500°C occurred because of material decomposition. After 500°C, the percentage of weight loss was constant and exhibited as char and residuals. In the case of the Sr^{2+}-modified bacterial cellulose and polyvinylidene fluoride blend, large weight losses were observed at 300°C to 400°C and 400°C to 500°C. The presence of polyvinylidene fluoride inhibited thermal degradation. Polyvinylidene fluoride was prepared as a solution, and it diffused along the bacterial cellulose network. This diffusion created a nonporous, reduced free space along the bacterial cellulose structure. In the Sr^{2+}-modified bacterial cellulose structure, the weight loss was shifted to a higher temperature, depending on the amount of added Sr^{2+}. The characteristics of the weight loss were strongly consistent with the XRF experiments.

The XRF technique was used for qualitative analysis of a composite of 10 wt% Sr^{2+}-modified bacterial cellulose and polyvinylidene fluoride. Figure 5 shows the XRF pattern of the sample. Strontium was observed. Table 1 shows the quantitative analysis of the composite of Sr^{2+}-modified bacterial cellulose and polyvinylidene fluoride and its elemental composition. The major elements were carbon, oxygen, and hydrogen; the minor element was strontium. The strontium amount was similar to that in the preparation method.

SEM was used to determine the morphological properties of the strontium-modified bacterial cellulose and polyvinylidene fluoride composite. Figure 6 shows the morphological properties. The variation in strontium amount was investigated. The as-purified bacterial cellulose had an average diameter of 30 nm, and the morphological properties confirmed the presence of a nanofibril network. The size of bacterial cellulose at the nanoscale level suggests that it could be further developed as a nano-reinforcement in the polyvinylidene fluoride matrix. However, remarkably, polyvinylidene fluoride provided a matrix to form the bacterial cellulose composite. Moreover, compared with the strontium-modified bacterial cellulose, no alteration in the morphological properties was observed. The morphological properties remained, and the physical appearance was identical to that of the modified composite. A small amount of spherical shape was observed, which may represent an incomplete reaction with the strontium-based salt but did not affect the morphological properties.

The mechanical properties of the strontium-modified bacterial cellulose and polyvinylidene fluoride composite were investigated (Table 2). The significant factors were tensile strength, Young's modulus, and elongation at break. The composite of bacterial cellulose and polyvinylidene fluoride and its chemical modification were significantly different. The properties of the neat bacterial cellulose were consistent with those in previous literature (Juntaro et al. 2012; Ummartyotin et al. 2012a; Ummartyotin and Manuspiya 2015a, 2015b). Although polyvinylidene fluo-
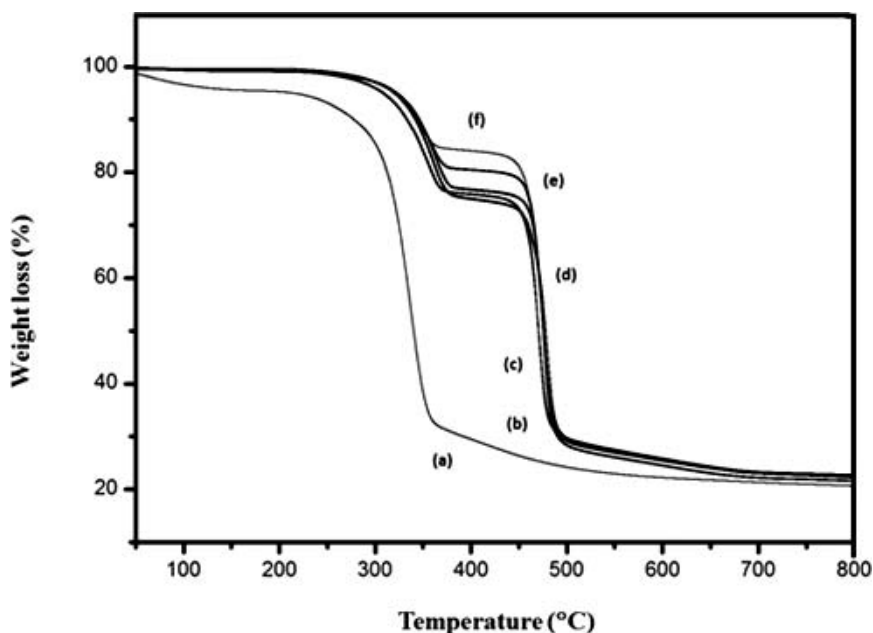

Figure 4.—Thermal degradation behavior of Sr^{2+}-modified bacterial cellulose: (a) neat bacterial cellulose, (b) bacterial cellulose and polyvinylidene fluoride blend, (c) 1 wt% of Sr^{2+}-grafted bacterial cellulose, (d) 3 wt% of Sr^{2+}-grafted bacterial cellulose, (e) 5 wt% of Sr^{2+}-grafted bacterial cellulose, and (f) 10 wt% of Sr^{2+}-grafted bacterial cellulose.
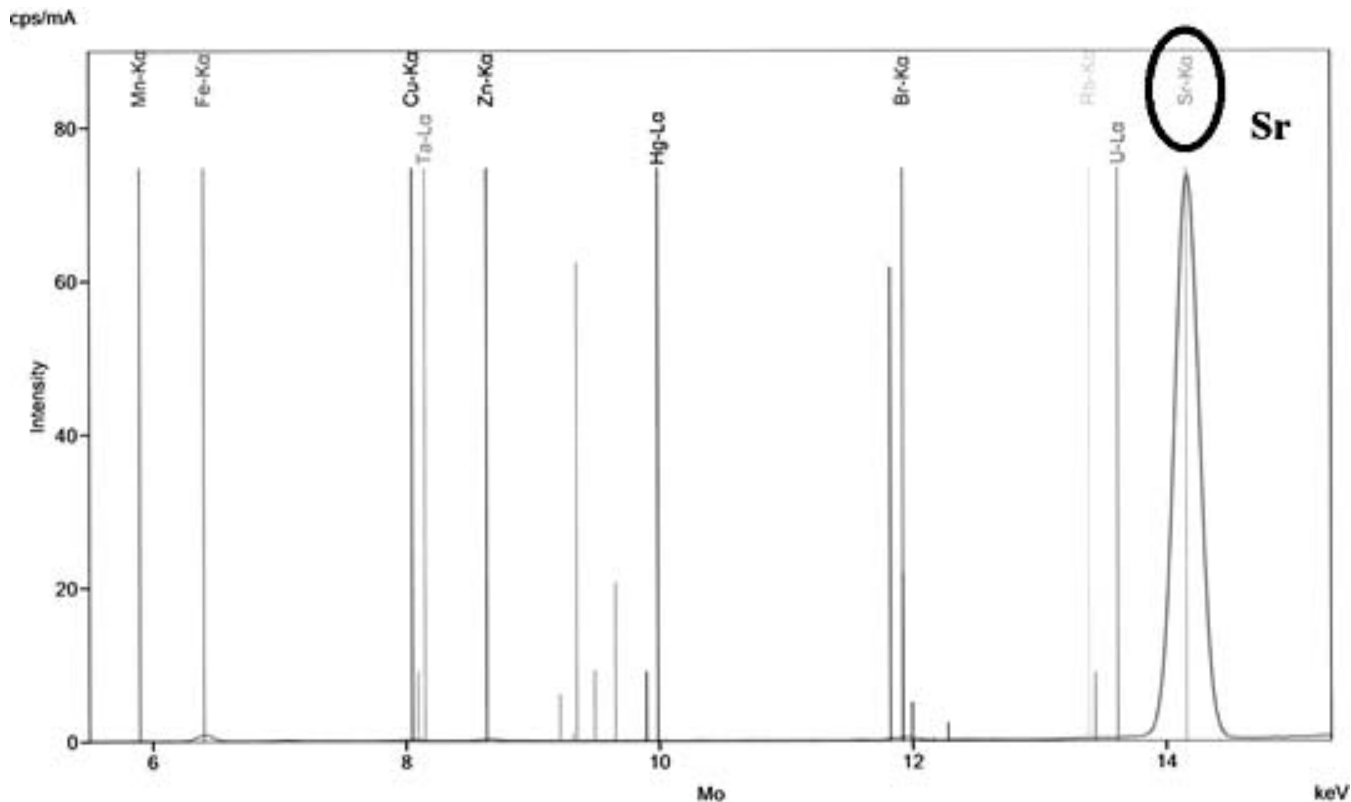
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Figure 5.—X-ray fluorescence qualitative analysis of 10% wt of Sr^{2+}-modified bacterial cellulose.

ride had excellent morphology and thermal stability, its limitation appeared when an external force was applied. To improve the design, the feasibility of adding a compatibilizer should be investigated.

Preliminary investigation of the dielectric properties of Sr^{2+}-grafted bacterial cellulose and polyvinylidene fluoride composite

A preliminary experiment on the dielectric properties was conducted to investigate frequency dependence. The role of frequency dependence on the dielectric properties can be explained using the polarization mechanism. In the investigation on neat bacterial cellulose, little information on the dielectric properties was detected. However, with the strontium modification, a signification enhancement in dielectric properties was observed. The dielectric phenomenon can be explained by the polarization effect of strontium. Figures 7 and 8 show the frequency dependence on the dielectric properties of the composite of Sr^{2+}-grafted bacterial cellulose and polyvinylidene fluoride. Neat bacterial cellulose and the composite of bacterial cellulose and polyvinylidene fluoride are provided for comparison. In the low-frequency region, the dielectric properties were strong and slightly decreased with the increase in applied frequency. This result is attributed to the relaxation behavior. At low frequencies, the material has sufficient time to reflect the polarity and dielectric properties. In contrast, at high frequencies, vibration is induced, and the material does not have sufficient time to respond, which results in low dielectric properties. The structural properties of the composite of Sr^{2+}-grafted bacterial cellulose and polyvinylidene fluoride significantly improved its dielectric properties. We attribute this result to the strontium ion, which acted as a conductive filler and modified the structure of bacterial cellulose. The dielectric properties were enhanced because strontium-grafted bacterial cellulose acted in an island-like manner, which created a polarity. From the structure and morphological viewpoint, strontium-modified bacterial cellulose was a polar cluster that was incorporated into the polyvinylidene fluoride matrix. A concern is the prevention of a conductive pathway from the strontium ion on the bacterial cellulose structure. An appropriate amount of loaded strontium can be applied to optimize the dielectric properties of the composite based on the percolation threshold.

Conclusions

A strontium ion was successfully grafted at the hydroxyl position of bacterial cellulose. A small amount of modified bacterial cellulose was integrated into the polyvinylidene fluoride matrix. The presence of strontium improves the dielectric properties compared with neat bacterial cellulose. There is no significant alteration in the dielectric
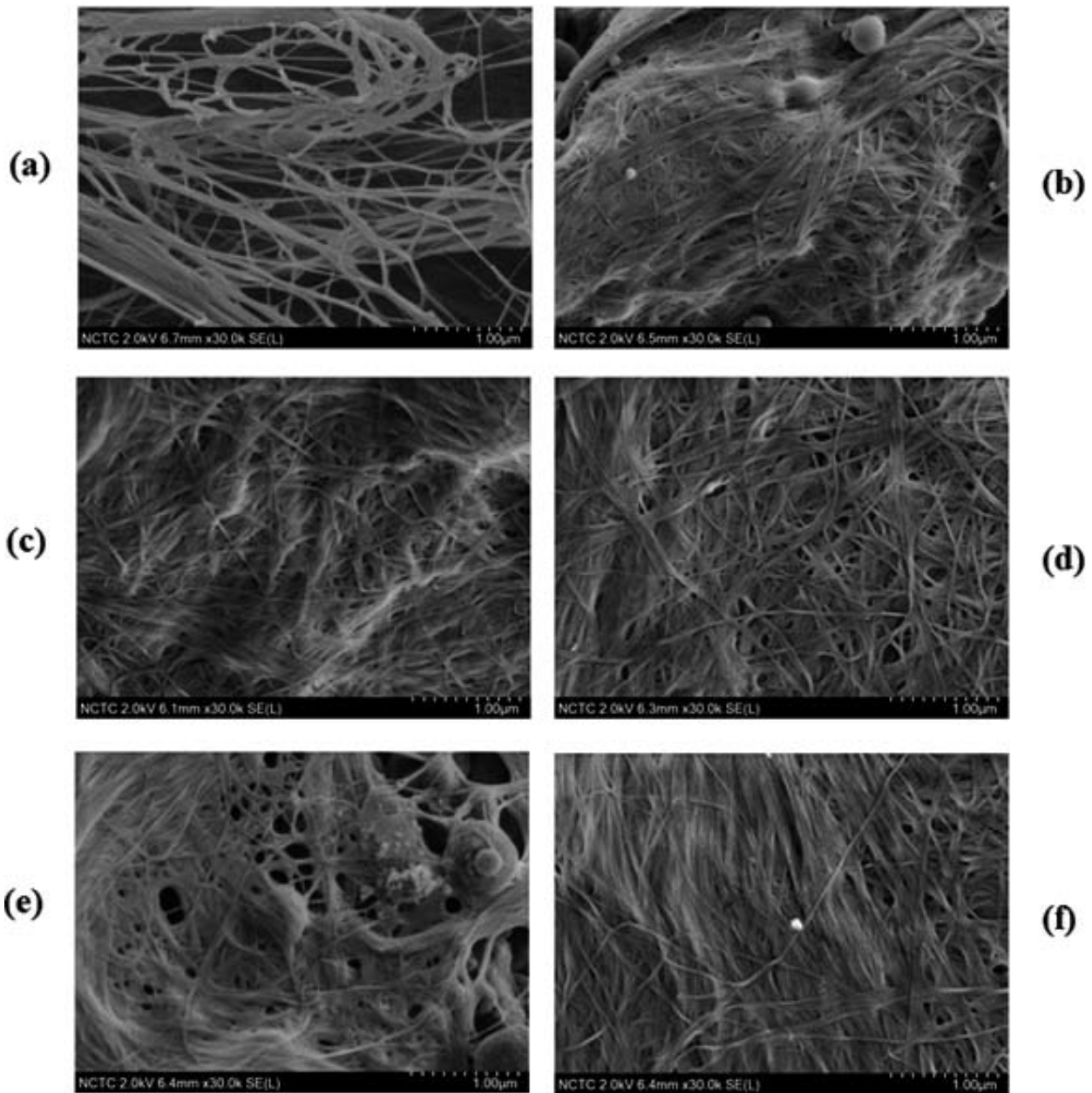
Table 1.—Quantitative analysis of Sr^{2+}-modified bacterial cellulose and polyvinylidene fluoride.

Composite	Amount of strontium (wt%)
1 wt% of Sr^{2+}-modified bacterial cellulose	1.4
3 wt% of Sr^{2+}-modified bacterial cellulose	2.1
5 wt% of Sr^{2+}-modified bacterial cellulose	4.5
10 wt% of Sr^{2+}-modified bacterial cellulose	8.4

Figure 6.—Morphological properties of Sr^{2+}-modified bacterial cellulose: (a) neat bacterial cellulose, (b) bacterial cellulose and polyvinylidene fluoride blend, (c) 1 wt% of Sr^{2+}-grafted bacterial cellulose, (d) 3 wt% of Sr^{2+}-grafted bacterial cellulose, (e) 5 wt% of Sr^{2+}-grafted bacterial cellulose, and (f) 10 wt% of Sr^{2+}-grafted bacterial cellulose.

properties of the composite. Polyvinylidene fluoride was inserted into the modified bacterial cellulose network to make the composite. Therefore, it appeared in the mechanical properties. No phase separation was observed.

A composite of modified bacterial cellulose and polyvinylidene fluoride was successfully prepared. The composite exhibited a strong potential to be a good candidate for electroactive materials.

Table 2.—Mechanical properties of strontium-modified bacterial cellulose and polyvinylidene fluoride composite.

| Sample	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
Bacterial cellulose	102	4,918	2.02
Bacterial cellulose and polyvinylidene fluoride composite	3.39	42	7.81
1 wt% of Sr^{2+}-modified bacterial cellulose	2.03	18	11.57
3 wt% of Sr^{2+}-modified bacterial cellulose	1.92	12	16.30
5 wt% of Sr^{2+}-modified bacterial cellulose	2.66	23	11.79
10 wt% of Sr^{2+}-modified bacterial cellulose	1.76	14	12.78

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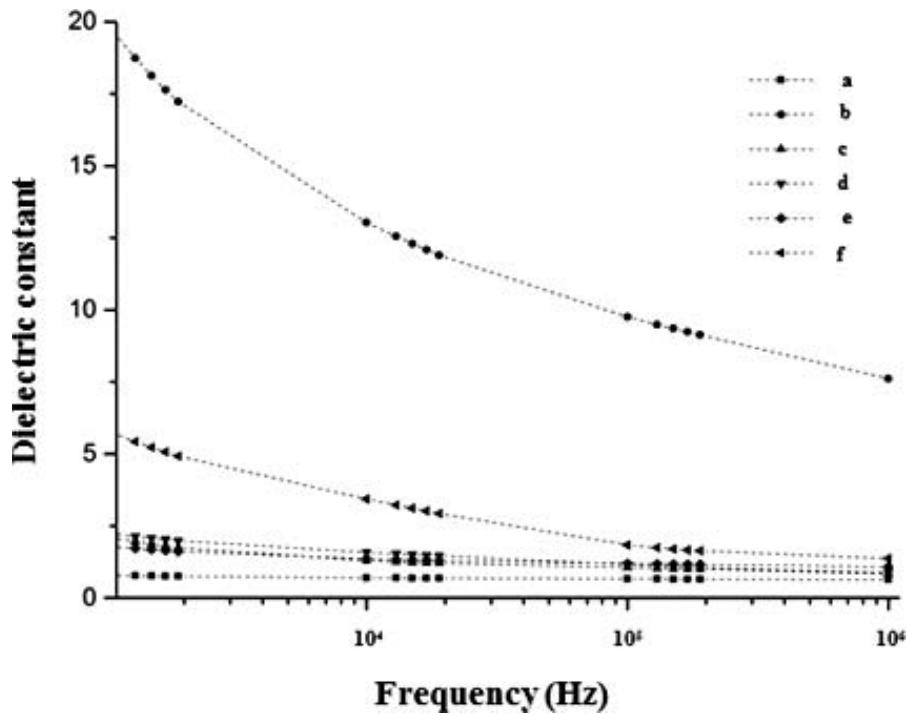
Figure 7.—Frequency dependence on dielectric constant of Sr^{2+}-modified bacterial cellulose: (a) neat bacterial cellulose, (b) bacterial cellulose and polyvinylidene fluoride blend, (c) 1 wt% of Sr^{2+}-grafted bacterial cellulose, (d) 3 wt% of Sr^{2+}-grafted bacterial cellulose, (e) 5 wt% of Sr^{2+}-grafted bacterial cellulose, and (f) 10 wt% of Sr^{2+}-grafted bacterial cellulose.
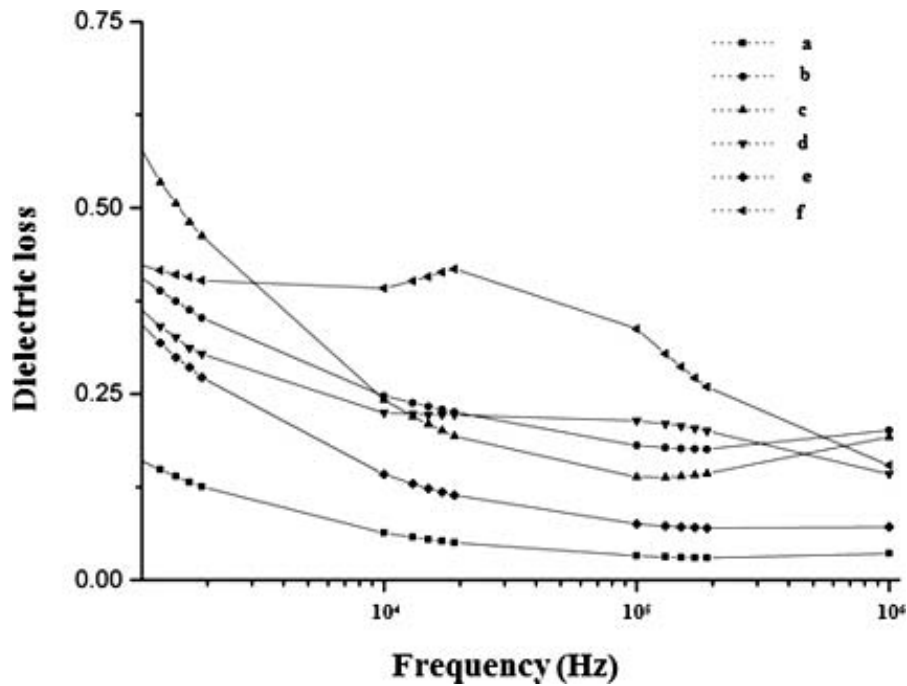

Figure 8.—Frequency dependence on dielectric loss of Sr^{2+}-modified bacterial cellulose: (a) neat bacterial cellulose, (b) bacterial cellulose and polyvinylidene fluoride blend, (c) 1 wt% of Sr^{2+}-grafted bacterial cellulose, (d) 3 wt% of Sr^{2+}-grafted bacterial cellulose, (e) 5 wt% of Sr^{2+}-grafted bacterial cellulose, and (f) 10 wt% of Sr^{2+}-grafted bacterial cellulose.

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