Toward a Rapid-Fabricated Triboelectric Device with a 1,3-Phosphorylated Poly(vinyl alcohol) Polymer for Water Turbulence Energy Harvesting

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ABSTRACT: Electricity generation from coal, nuclear reaction, and shale gas has brought environmental, safety, and health concerns. The electricity industry is constantly seeking sustainable, safe, and healthy way of electricity generation. The use of triboelectric device is promising for producing electricity from water energy. In this study, we report on the rapid fabrication of a 1,3-phosphorylated poly(vinyl alcohol) gel-based triboelectric device and direct harvesting of water turbulence energy using this device. The gel was prepared by the reaction of poly(vinyl alcohol) with dipotassium phosphate. The synthesized gel was characterized by mass spectroscopy, thermogravimetric analysis/differential thermogravimetry, Fourier transform infrared spectroscopy, X-ray photoelectron spectroscopy, scanning electron microscope, Raman, and carbon and oxygen K-edges soft X-ray absorption near edge structure spectroscopy. The triboelectric device was used to harvest electricity from water turbulence.

1. INTRODUCTION

Coal is one of the most affordable and largest generated sources of electricity. The burning of coal brings many environmental issues, such as air and water pollution. Also, the access to coal is restricted in a global scale. This issue plus the concern of increased carbon production are driving factors behind the development of new materials and technology for clean energy. Nuclear power that generates electricity through nuclear reaction has been considered as a sustainable and clean energy. However, the well-known nuclear power station accident, Fukushima Daiichi nuclear disaster that happened in Japan in 2011, has brought safety concern about the development of nuclear power. Shale gas, the extraction of natural gas from shale rock formations, has transformed the energy landscape in many countries. The development of shale gas extraction was considered as an effective way to cope with the growing electricity demand while reducing the dependence on coal. However, the extraction of shale gas has brought concerns, such as water pollution, greenhouse gas emission, and detrimental health impacts. It indicates that the electricity industry has to continue to develop safe, sustainable, and healthy way of generating electricity.

Triboelectric devices are novel energy converters that convert mechanical energy into electrical energy. Triboelectric devices hold the merits of low weight, low fabrication cost, self-power, enforced green technology, orientation to clean energy, and resource abundance. The triboelectric devices were invented by Wang and co-worker in January 2012. Since their birth, research interest in seeking simple fabrication and new materials system for triboelectric devices is growing, which shows the great potential for solving the issue of electrical power shortage in developing countries, electrical power-limited regions, and rural areas. Wang and co-workers proposed the use of triboelectric devices to harvest the mechanical energy from ocean. It was called blue energy, including that from tidal, water wave, and ocean current.

Following this trend, polymer-based materials system can be one of the answers to this demand. The polymer is generally dielectric material, which is easily used for building up triboelectric power on the device surface. However, no specific polymer has been proposed as the standard raw material for...
making triboelectric devices so far. Efforts are still required for searching new polymer sample for specific triboelectric application.

Poly(vinyl alcohol) (PVA) is a nontoxic, slowly biodegradable polymer that is widely used in paper industry. Also, it is water-soluble, which makes it much more convenient and cheaper to be processed compared to other polymers only dissolved in organic solvents. We aimed to tailor it by using some nontoxic compound and use PVA-based materials for making triboelectric devices.

Extensive research endeavor over the past 5 years has led to progress and development for triboelectric devices, which have been applied to various sources, including human motion, mechanical vibration, automobile brake, wind, rotation, water waves, and tides. However, new application of triboelectric devices toward large-scale energy harvesting remains an important task.

Turbulence is commonly observed in any flow system, such as reservoirs, rivers, lakes, and oceans. Especially, oceans cover more than 70% of the earth’s surface and are exceedingly rich resources for turbulence. However, current study of triboelectric devices for ocean energy harvesting is focused on water waves/tides energy, referring to the kinetic and potential energy from ocean surface waves. To the best of our knowledge, there are no reports about triboelectric devices for water turbulence harvesting.

Here, we report on rapid fabrication of a 1,3-phosphorylated poly(vinyl alcohol) (PPVA) gel-based triboelectric device and direct harvesting of water turbulence energy using this device. PPVA gel was prepared by the reaction of PVA with dipotassium phosphate, a nontoxic compound widely used in food industry as a food additive. The synthesized PPVA was characterized by a scanning electron microscope (SEM), mass spectroscopy, thermogravimetric analysis (TGA), differential thermogravimetry (DTG), X-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, and soft X-ray absorption near edge structure spectroscopy (XANES) of carbon K-edge, oxygen K-edge. The PPVA gel was mixed with silica powder for making a triboelectric device. We found that the electrical current output of the triboelectric device was dependent on the magnitude of the flow speed of water turbulence. Due to the simple chemistry, facile fabrication, and exceptional performance, the PPVA-based triboelectric device is envisioned to play a significant role in many low-cost and rapidly processed water/blue energy harvesting applications.

2. RESULTS AND DISCUSSION

To synthesize the PPVA gel, dipotassium phosphate (20 g) was first mixed with ultrapure water (200 mL) in a 500 mL beaker and stirred. After a clear solution was obtained, PVA (15 g) was added to the beaker. Heating at 80 °C temperature was applied to the beaker. After stirring the mixture for 15 min, a clear and soft gel was obtained. The gel was washed with ultrapure water 10 times and stored for further characterization. It should be mentioned that in the stirring process, normal magnetic stirrer was not workable given that the gel emerging in the solution from the very beginning was very sticky. To overcome this difficulty, we used a Teflon rod (length = 15 cm) driven by a Worm Gear Direct Current (DC) motor with the rated torque of 21 kg cm to stir the gel. A proposed synthesis scheme for PPVA is shown in Scheme 1. The structure of PPVA is similar to that of Pupkevich’s work.11 The surface morphology of the synthesized gel was characterized by the SEM (Figure 1). Interestingly, the synthesized gel surface shows a nanopillar feature (Figure 1c).

Polymer chemistry of PPVA can be studied by mass spectra (Figure 2a), TGA curve, and DTG curve (Figure 2b). As proposed by Hanton,12 the molecular weight distribution is determined by the number-average molecular weight, \( M_n \), the weight average molecular weight, \( M_w \), and the polydispersity \( M_w/M_n \). The two average molecular weights are the first two moments of the distribution of oligomer molecules.12 Using this method, these parameters can be calculated as: \( M_n = 5487.076 \), \( M_w = 7593.756 \), and polydispersity = 1.384.

The TGA and DTG curves are shown in Figure 2b. The weight loss at 50–150 °C is due to moisture vaporization. The weight loss at 250–350 °C is due to the degradation of the PPVA molecule. The weight loss at 350–450 °C is possibly due to the byproduct generated by PPVA during the TGA thermal degradation process. The DTG curve shows three peaks at 143, 321, and 431 °C.

Soft X-ray absorption spectroscopy, a complementary technique to X-ray photoelectron spectroscopy, unravels the electronic structure of the materials by elucidating the local electronic structure and bonding. We exploited the XANES of carbon and oxygen K-edge absorption measurement to probe the electronic bonding and structure of the PPVA sample. Figure 3 shows the carbon K-edge absorption spectroscopy of the PPVA film formed on the gold-sputtered silicon wafer substrate. For comparison, the carbon K-edge XANES spectrum of graphite and PVA are presented in Figure 3. The graphite spectrum exhibits peaks at 285.4 eV (\( \sigma^* \)), 291.8 eV (\( \sigma^* \)), 292.9 eV (\( \sigma^* \)), 297.9 eV (\( \sigma^* \)), 303.7 eV (\( \sigma^* \)), and 307.8 eV and a shoulder at 319–336 eV. The PVA spectrum presents peaks at 287.5, 292.6, 294.6, and 299.5 eV and a shoulder at 307.2 eV.

For PPVA, the carbon K-edge XANES spectra show peaks at 284.5, 288.8, 293, 297.5, and 300.3 eV. Two distinctive peaks, which are also observed in the absorption spectrum of the graphite, are the resonances due to the transitions from C 1s electron state to \( \pi^* \) and \( \sigma^* \) states. These features have sp2 bonding characteristics and appear at 284.5 eV (\( \pi^* \)) and 293 eV (\( \sigma^* \)) excitation energies for PPVA in Figure 3. The broad peak centred at 293 eV photon energy embeds \( \sigma^* \)-(C=O) resonance as well. The shoulder located at the left side of this peak at 292 eV is attributed to the \( \sigma^* \) resonance of the \((C=O)\) bond.13,14 The strong absorption peak between sp2-derived \( \pi^* \) and \( \sigma^* \) resonances emerges from transitions from C (1s) state to the unoccupied state of carbon that functionalized with oxygen. Concomitant with the XPS results detailed below, it is determined that this prominent peak with 289 eV excitation energy in the PPVA absorption spectrum is a band depicting C (1s) → \( \pi^* \)(C=O) transition. The C 1s XPS spectrum also confirms the formation of C=O bond, shown in Figure 5. Both absorption and excitation energies of this bond indicate that the bond is a part of carboxyl (COOH) functional group. Note that there exists a shoulder at the middle intensity of this peak with a
288 eV excitation energy. We assign this absorption feature to $\sigma^*(C\equiv H)$ resonance. Two sharp absorption peaks residing at higher photon energies around 300 eV in the spectrum are identified as L-edge transition of potassium in the PPVA sample. The peaks at 297.5 and 300.3 eV are transitions from 2p$_{3/2}$ and 2p$_{1/2}$ electronic states of potassium. These absorption energies of the L-edge peaks are higher than those of reported as around 294 and 297 eV.\textsuperscript{15} However, it has been also published that if potassium involves in a strong ionic bonding, it results in an L-edge absorption energies shift as much as 3 eV.\textsuperscript{14,16} Moreover, we assign the peak above the potassium L-edge features, appearing as a broad band around 303 eV as a $\sigma^*(C\equiv O)$ resonance.\textsuperscript{17}

The oxygen K-edge XANES spectra (Figure 4) was also used to investigate the nature of oxygen bonding in the sample. For the PPVA sample, we observe two distinctive features in the O K-edge XANES spectra at 531.4 eV and 537 eV and a shoulder around 550−580 eV. The peak at 531.4 eV is a transition from oxygen 1s electronic state to $\pi^*(C\equiv O)$ unoccupied state. The main peak at 536 eV is attributed to $\sigma^*(C\equiv OH)$ and (C\equiv O−C) bonds, as indicated from the O 1s XPS spectra of the sample (Figure 5c). We think the shoulder residing at 539.7 eV photon energy stems from an oxygen 1s electron transition to $\sigma^*(C\equiv O)$ bond, which is shown in the O 1s XPS spectra (Figure 5c). It...
has been documented that oxygen K-edge absorption spectrum discloses $\sigma^\pi(P-O)$ resonance at 535 eV; we believe that the shoulder at this absorption energy in Figure 6 has a contribution from the (P–O) bond. Compared to the oxygen K-edge absorption spectrum of PPVA, that of PVA showed two peaks at 531.8 and 537.1 eV and a shoulder around 550–580 eV. The spectroscopic meaning of the peaks at 531.8 and 537.1 eV for PVA is similar to the peaks at 531.4 and 537 eV for PPVA. The peak at 531.8 eV for PVA represented oxygen 1s electronic state to $\pi^*$ (C=O) unoccupied state. The peak at 537.1 eV for PVA is assigned to the resonance of $\sigma^*(C-OH)$ and (C–O–C) bonds. We speculated that the shifting of the photon energy and the change in the intensity for the peaks of PPVA were due to the introduction of P–O resonance.

The XPS scans in Figure 5 show the expected C 1s, O 1s, and P 2p peaks. The synthesized XPS profile of C 1s presented three peaks at 284.5, 285.9, and 288.9 eV, which are attributable to the C–H, C−O−C, and carboxylate −COO species. The synthesized XPS profile of O 1s showed three peaks at 531.5, 532.2, and 532.9 eV, which are assigned to phosphate, C=O, and C−O−C/C−OH. The synthesized XPS profile of P 2p showed a peak at 133.6 eV, which is attributed to phosphate.

Figure 6a compared the FTIR spectra of PVA and PPVA. The FTIR spectra of PVA showed peaks at 835.9, 911.1, 1085.3, 1243.1, 1325.0, 1419.4, 1574, 1741, 2912.6, and 3278.5 cm$^{-1}$ and a shoulder around 500–753 cm$^{-1}$. Compared to the FTIR spectra of PVA, that of PPVA showed much sharper peaks at 520, 938, 1080, 1259, 1382, 1426, 1656, 1713, 2941, and 3276 cm$^{-1}$. The peak at 520 cm$^{-1}$ is attributed to the in-plane vibration of the C−C=O group. The weak peak at 938 cm$^{-1}$ is due to the C−C stretching. The peak at 1080 cm$^{-1}$ is assigned to the P−O− groups. The peak at 1259 cm$^{-1}$ is attributed to the P−O= double bonds. The peak at 2941 cm$^{-1}$ is assigned to C−H stretching. The peak at 3276 cm$^{-1}$ is assigned to O−H stretching.

The Raman spectra of PPVA (Figure 6b,c) show the bands at 127, 193, 248, 345, 428, 528, 642, 868, 892, 923, 1086, 1123, 1137, 1607, 1722, 1200, 1238, 1356, 1449, 1500, 2433, 2738, and 2931 cm$^{-1}$ and a shoulder at 1655 cm$^{-1}$. The band at 428 cm$^{-1}$ is due to the P(OH)$_2$ bend. The band at 1086 cm$^{-1}$ is assigned to C−H vibration. The band at 642 cm$^{-1}$ is attributed to O−C=O in-plane bending mode. The band at 923 cm$^{-1}$ is due to the C−C stretching. The band at 2931 cm$^{-1}$ is attributed to C−H vibration. The band at 1449 cm$^{-1}$ is assigned to the mixture of C−H bending and O−H bending.

A triboelectric device was rapidly fabricated by using the synthesized PPVA gel (Figure 7a). First, three different kinds of dielectric materials were made by mixing PPVA gel with varied wt % of silica powder, which were pure PPVA gel, 9 wt % silica-filled PPVA gel, and 15 wt % silica-filled PPVA gel. Then, they were put inside a plastic bag, which was set under a heavy iron cylinder. A man was standing above the cylinder. The weight of his body (165 lbs) would be used to press the PPVA gel into a slide. The thickness of the slides made was around 1 mm. Finally, the triboelectric device was made by the origami of the slides.

As presented in Figure 7a, the light-blue layer represented the PPVA gel filled with 9 wt % silica; the green layer indicated a
pure PPVA gel without filling any silica; the pink layer represented PPVA gel filled with 15 wt % silica. It should be noted that the weight of the silica powder used in the device was carefully chosen and optimized. Filling too much silica powder into the PPVA gel would make it very stiff, causing much difficulty in the continued material processing, such as mixing, stirring and slide-pressing, bending, and origami-construction. Also, introducing too much silica powder into the PPVA gel would lead to the increase in the electrical resistance of the whole device, which made the electrical current undetectable with a microamperemeter. Figure 7b presents the actual image of the triboelectric device made.

The electrical current was expected to be generated when external pressure was applied to this triboelectric device. A commercial bridge rectifier (model: KBP307) was connected to the device for AC-to-DC convention. A microampere meter was wired to monitor the change in the electrical current. A silver paste was used for electric contact. To generate water turbulence, turbine blades were self-developed. First, commercial 1 feet by 2 feet, 22 gauge weldable steel sheet (model: M-D Building Products 56066) were cut into small rectangular steel sheets (dimension: 1.5 cm by 6 cm) and a round steel sheet (diameter: 8.5 cm). Those small rectangular sheets were bent and soldered on the surface of the round steel sheets using an ordinary 60 W electronic soldering iron kit.

A Worm Gear DC motor (rated torque 21 kg cm) with a speed controller was purchased from Amazon. Then, one end of an iron rod (length: 10 cm, diameter: 4 mm) was soldered to the DC motor and anther end of the rod was soldered to the turbine blades. It should be mentioned that all the soldering in the operation was done by the electronic soldering iron kit, which was convenient for laboratory operation. These turbine blades were driven by the DC motor (Figure 8c) and placed inside a tank of water to generate a turbulence flow. A flow sensor was employed to measure the flow speed of the water turbulence. The flow speed of the water turbulence was adjusted through the speed controller of the DC motor. A tank of Milli-Q water (5 gallon) was prepared. The triboelectric device fabricated was placed inside the water tank to harvest the triboelectric current where the water turbulence was constantly hitting the triboelectric device.

As shown in Figure 9a, the current harvested was dependent on the flow speed of the water turbulence. This indicated that the fabricated PPVA-based triboelectric device could be used as a prototype for harvesting water turbulence energy. When two different layers of dielectric materials were in close contact, the electric current would be generated. This triboelectric effect has been well known for decades, which was called as triboelectrification. However, a full-scale debate is still under way on what makes it happen.28 It has been proposed that energy level misalignment, ion transfer, and/or materials species transfer could result in electron charging.28

In the following, we derived a simple mechanism to explain the phenomena that the triboelectric current generation depended on the flow speed of the water turbulence based on the fabricated triboelectric device.

We considered that it was due to the interaction of electrical polarization of the dielectric layers. Figure 9b indicates the formation of electrical polarization on the surface between dielectric layers. When water turbulence hit the triboelectric device, the device was pressed. In this process, the dielectric...
layers were squeezed to generate electric charge, leading to the formation of $P_1$ and $P_2$. Here, $P_1$ was the electrical polarization between the blue and green layers, whereas $P_2$ was the electrical polarization between the green and pink layers. The final polarization for electric current generation was $P_1 - P_2$ or $P_2 - P_1$. When the water turbulence moved away from the triboelectric device, the whole device was released. The electrical polarization disappeared. But when it was hit by water turbulence, electrical polarization was produced again. If the water turbulence hit the device at a higher speed, a bigger electrical polarization could be expected, given that much more areas of the dielectric layers were squeezed and much more electric charges were generated. That is the reason why the higher speed of the water turbulence speed would lead to a higher triboelectric current (Figure 9a).

It should be noted that we used ultrapure water for this proof-of-concept experiment in laboratory with room temperature. However, the performances/stability of the triboelectric devices are probably affected by salinity, pH value, temperature, or external environment (e.g., wind with high speed). For instance, when the triboelectric device is placed inside sea or river or reservoir for practical application, the change in salinity, pH value, temperature, and even speed of wind, may bring some impact on the device. For example, the weight of high-salinity water may hit the triboelectric device heavily and generate a much more triboelectric energy compared to that of the low-salinity water with the same speed; the water with high salinity/high pH value/high temperature may degrade the device, given that the device was mainly made by the polymer; the wind with high speed may bring extra triboelectric energy for the device. The consideration of these impacts is valuable and paves the way for our future endeavor in the practical application of the PPVA-based triboelectric device.

3. CONCLUSIONS

We synthesized PPVA polymer gel using simple chemistry. The gel was characterized by mass spectroscopy, TGA/DTG, FTIR, XPS, Raman, and carbon and oxygen K-edges XANES. A triboelectric device was fabricated by folding different layers of dielectric materials made by PPVA/silica. The triboelectric device was used to harvest water turbulence energy. In future applications, the triboelectric device can be expected to be scaled up in a network for large-scale energy harvesting from the ocean.

4. EXPERIMENTAL SECTION

4.1. Materials. The raw materials used to synthesize phosphorylated poly(vinyl alcohol) include dipotassium phosphate and poly(vinyl alcohol).

4.2. TGA, Mass Spectroscopy, XPS, Raman, FTIR, and XANES Characterization. The prepared gel were studied via TGA, mass spectroscopy, Raman spectroscopy, FTIR spectroscopy, XPS, and soft X-ray absorption spectroscopy of the carbon and oxygen K-edges near edge structure. Thermogravimetric analysis (TGA) was performed via a TA Instruments Q600 SDT under nitrogen atmosphere with a 100...
mL/min flow rate. Dynamic ramps were performed at the rate of 10 °C/min from 50 to 750 °C in N₂.

Matrix-assisted laser desorption/ionization mass spectrometry (Bruker Ultraflx Xtreme MALDI-TOF/TOF) was used to study the polymer. The matrix of cyano-4-hydroxycinnamic acid was used.

Raman spectra were obtained through a Jobin Yvon Horiba LABRAM Integrated Raman spectroscopy system equipped with an HeNe laser (excitation wavelength: 632.81 nm). The Raman spectra were collected with 0.2 cm⁻¹ resolution in the wavenumber range of 100−3000 cm⁻¹.

The XPS analyses were performed via a K-Alpha XPS instrument (Thermo Scientific). The FTIR spectra were collected via a ThermoFisher Nicolet iS 10 Fourier transform infrared (FTIR) spectrometer equipped with a Smart iTR diamond ATR accessory and with deuterated-triglycine sulfate detector. The FTIR spectra collected were average of 32 scans with 4 cm⁻¹ resolution in the wavenumber range of 4000−400 cm⁻¹.

4.3. Soft X-ray Absorption Spectra of Carbon and Oxygen. The K-edges near edge structure spectra were obtained by using a variable-line-space plane-grating-monochromator (VLSPGM) beamline. This beamline facility was constructed by the Center for Advanced Microstructures and Devices, Louisiana State University, Baton Rouge, LA. It provides a resolution better than 0.20 eV at the carbon K-edge. The soft X-ray beamline monochromator that is equipped with two interchangeable gratings can deliver monochromatic photons in the energy range of 180−1000 eV. The total electron yield technique is used for collecting the absorption spectra. The sample’s drain current was measured with an energy step size of 0.20 eV. A gold-sputtered mesh was used to acquire primary beam current for spectra normalization. The base pressure of the vacuum chamber was kept at 2 × 10⁻⁹ Torr. A graphite foil was used as a reference sample to calibrate the energy scale of the grating and identify the carbon-edge absorption of the sample. The sample was dried over night on a gold substrate before measurement.

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