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Research Article

Large-scale fabrication of polyethylene glycol-poly(methyl methacrylate) blend with nanosponge-porous structure for high performance bio-triboelectric nanogenerator

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ABSTRACT

Renewable energy has gained increasing attention from researchers all around the world to deal with shortcomings of fossil fuels, environmental pollution and energy crisis. Triboelectric nanogenerators (TENGs) has been considered as a promising way to change to sustainable green energy. In this work, blends of polyethylene glycol (PEG) and poly(methyl methacrylate) (PMMA) with nanosponge-like porous structure are fabricated by a solution phase separation method, subsequently the nanosponge-porous blends are also applied for the development of a high performance TENG. The features of the porous structure including porosity and uniformity are controlled by varying polymer concentrations and PEG/PMMA ratios. Copper nanowires mesh (Cu-mesh) is employed as a mechanical supporter for enhancing the stability of the PEG-PMMA nanosponge. In addition, a convex-micropatterned poly(dimethyl siloxane) (*c*-PDMS), which plays as a counter tribo-surface of a TENG, is also fabricated by the micromolding method in which a highly ordered honeycomb-concave polymer film is exploited as a master mold. A TENG device made of porous PEG-PMMA blend and *c*-PDMS can generate an instantaneous electrical power of 3.2 mW. The present developed TENG can not only serve as a supply power to light several light emitting diodes (LEDs) on but also demonstrate high potential applications as self-powered monitoring healthcare sensors.

Key words: Phase separation, biocompatible, sponge-like porous structure, triboelectric nanogenerator

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INTRODUCTION

With the rapid depletion of fossil fuels and serious pollution issues drive great challenges to sustainable development of a country. To address these issues, the replacement of fossil fuels by green and renewable energy sources has received increasing attention and research in all over the world [1]. In addition, the characteristic features of the next generation wearable electronics including the mobility [2], flexibility [3], breathability and biocompatibility [4], and low-power consumption requires suitable distributed electric power supplies which definitely do not involve conventional power sources such as electrical grids, batteries, or supercapacitors [5,6]. Triboelectric generators (TENGs) have become one of the most promising technologies to harvest random vibration energy to generate electricity. Thanks to their merits such as broad biocompatible material choices, compact and flexibility, cost-effectiveness, and high energy-conversion efficiency even at low operation frequency, TENGs have found vast potential applications [7–9]. For practical applications, the electrical output performance of the TENG is crucially important. So far, enlarging the frictional surfaces through patterning tribo- surfaces or forming porous structures and selecting suitable tribo-material pairs are common ways to achieve high output performance of TENGs [10–12].

Previously, we reported that PMMA could form large-scale three-dimensional sponge-like porous structure on a copper nanowires mesh (NP@Cu-mesh) by a simple, rapid and one-step solution phase separation method. The TENG using the NP@Cu-mesh demonstrated superior output power density of 21 W/m^2 together with promising flexible, breathable and water-resistant properties [13]. However, PMMA is obviously not to be considered as a biocompatible polymer, moreover it has an inherently poor electrification effectiveness, as mentioned in triboelectric series [14]. These characteristics of PMMA limit its practical applications as a potential tribomaterial for a TENG in wearable devices. Based on this context, introducing a hydrophilic biocompatible polymer with excellent electrification behaviour like PEG to the PMMA

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sponge seem to be a promising approach to achieve a multi-functional TENG with breathability, biocompatibility and high performance which is favourable for its further application in wearable devices like healthcare monitoring sensors.

Pursuant to our continued research work, herein, we proposed a novel preparation of PEG-PMMA blend with sponge-like porous structure and its application for a high-performance bio-TENG. A quinary solution of PEG, PMMA, THF, MeOH and water was for the first time exploited to induce the spinodal decomposition through solvent evaporation, thereby spontaneously forming a sponge-like porous structure of the PEG-PMMA blend. The porous structure of PEG-PMMA blends was controlled by varying polymer concentrations and PEG contents. The PEG-PMMA nanosponge on a copper nanowires mesh which plays dual roles as a structural supporter and an effectively structured electrode was applied in a TENG. The electrical generation ability of the PEG-PMMA nanosponge hybrid-based TENG devices was characterized and compared with the one using the common aluminum surface. Moreover, to demonstrate the ability of the TENG for powering microelectronics, the new developed TENG was used to charge commercial capacitors and light on the several LEDs by simple hand pressing.

EXPERIMENTAL METHODS

Materials

Poly(methyl methacrylate) (PMMA EG920) in the form of pellets was provided by LG Chem Co., (Korea). Polyethylene glycol (Mw = 1000), tetrahydrofuran (THF, 99.0 %) and methanol (99.8 %) were purchased from Sigma-Aldrich (USA). Poly(dimethylsiloxane) (PDMS) and its curing agent (Sylgard 184) were received from Dow Corning (USA). Copper mesh (mesh number of 300, average pore size of 37 μm) was bought from Bolin Metal Wire Mesh Co., Ltd (China). Distilled water was made in the laboratory.

Preparation of nanosponge PEG-PMMA/Copper nanowires mesh hybrid

The procedure for preparing porous PEG-PMMA on the copper nonwoven nanowires mesh was provided in Figure 1. As shown in Figure 1, PMMA and PEG were firstly dissolved in the THF solvent. Then water and methanol were added dropwise into the as-prepared solution under intensive stirring to obtain a homogeneous quinary solution of PEG, PMMA, THF, methanol, and water. A large-scale hybrid was made by simple dip-coating copper mesh into the quinary solution followed by drying in the normal air conditions. The nanosponge PEG-PMMA was spontaneously formed and covered the copper mesh as a result of phase separation during the evaporation of solvents.

Preparation of microdome-patterned PDMS

PDMS film with a microdome-patterned surface was produced by the micromolding method where the honeycomb polystyrene was used as a mother mold, as shown in Figure 2. The polystyrene film with a highly ordered honeycomb pattern (*hc*-PS) on copper substrate was prepared firstly using the procedure reported on our previous article[15]. Liquid PDMS base and its crosslinker (base/crosslinker ratio of 10/1) were carefully mixed. Then the liquid was poured on the *hc*-PS followed by degassing in low vacuum oven. Subsequently, the sample was annealed at 70 °C for 4 h, and finally the PDMS replica was detached from the mold to obtain microdome patterned PDMS (*md*-PDMS).

Assembly of nanogenerator

In this study, the TENG device was designed and assembled in a typical contact-separation mode. Correspondingly, the nanosponge PEG-PMMA/copper mesh hybrid was used as a positive tribo-surface while the *md*-PDMS was exploited as a negative surface for the fabrication of a TENG. The nanosponge hybrid was attached to a poly(methyl methacrylate) (PMMA) plate by the double-sided adhesive tape, which served as the mechanical supporting plate of TENG. Besides, the patterned PDMS on another PMMA plate with an aluminum foil sandwiched between them was used as a counter positively charged tribo-surface of the TENG. All the TENG devices with frictional surfaces of 2.5 cm $\times$ 2.5 cm were used for the characterizations of electrical output performance. The distances between the two tribo-surfaces were fixed at about 5 mm by adjusting the mechanical shaker. Finally, the electric wires were connected with both the top and the bottom electrodes.

Characterization methods

The morphology of the surface of samples was characterized using a field-emission scanning electron microscopy (FE-SEM, S-4800, Hitachi, Japan). We used a homemade mechanical shaker with the feasibility

Figure 1: 3D sketch illustrating the fabrication of the nanosponge PEG-PMMA/Copper nanowires mesh hybrid.

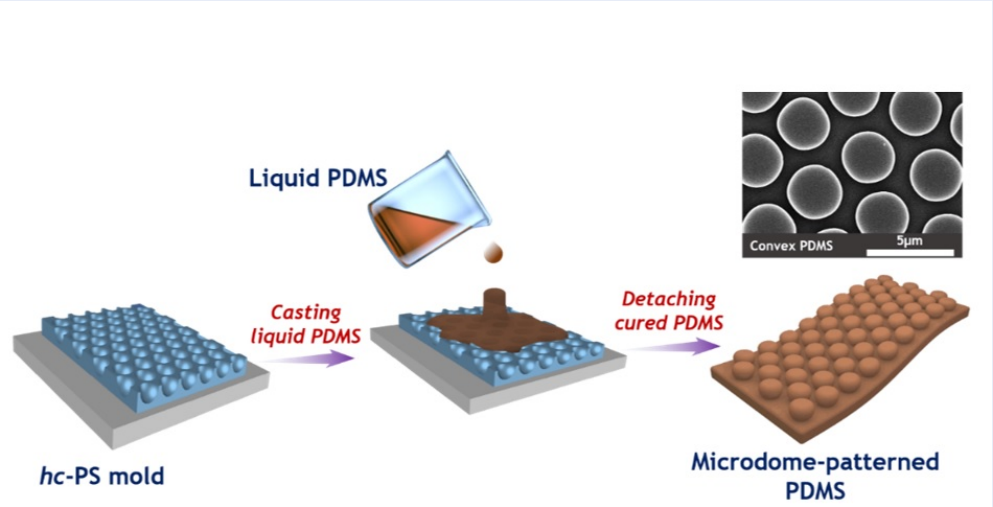

Figure 2: 3D sketch representing the fabrication of the *md*-PDMS via the micromolding method and SEM image of the *md*-PDMS replica.

to control ambient humidity and temperature to apply vertical impulse vibration to the TENG. The electrical output of TENG was characterized by using a digital oscilloscope (Model No. SDS1202X-E, Siglent, China) and a homemade circuit as a current preamplifier [16].

RESULTS AND DISCUSSION

Effect of polymer concentrations

It was reported that non-solvent induced phase separation of polymer solution depends on the polymer concentration and non-solvent content. Figure 3 presents the effect of the polymer concentration on the porous structure of the blends. With low polymer concentration (10 mg/ml), the porous structure was not observed on the copper mesh. This is because the solution with low polymer concentrations has corresponding low viscosity, as a result the amount of solution adhering to the copper mesh in the dip-coating process is too small [16], this dilute solution may not enough for inducing phase separation to form the porous structure. In addition, at a high polymer

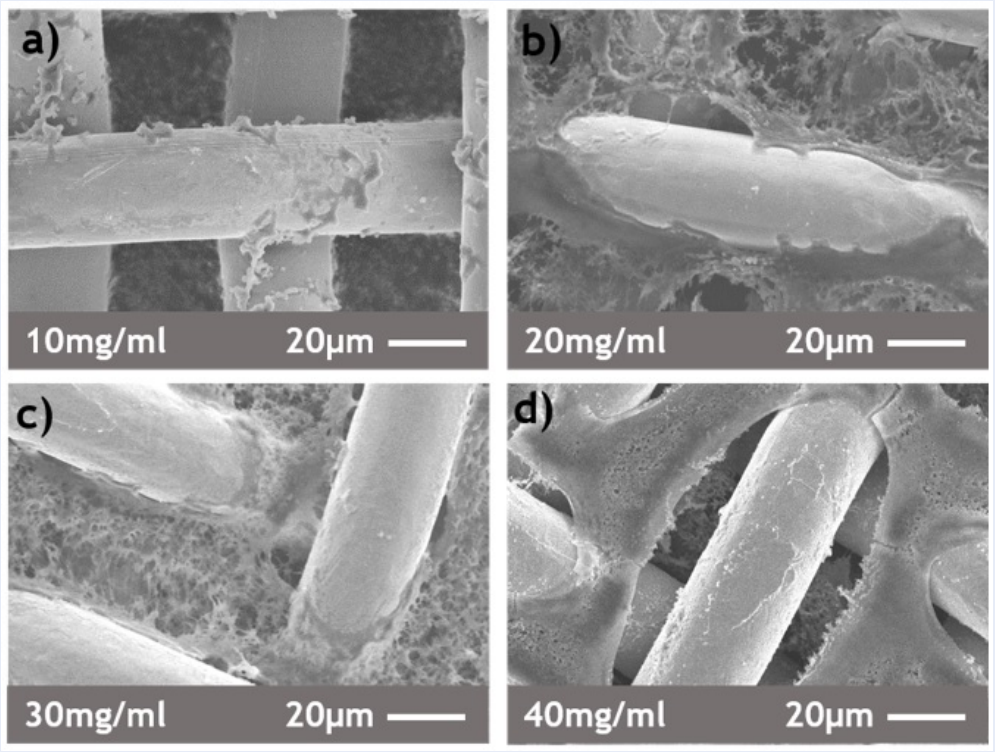

Figure 3: Effect of polymer concentrations on surface morphology of the PEG-PMMA blends. SEM images of the PEG-PMMA nanosponges prepared with various polymer concentrations: (a-d) 10, 20, 30, 40 mg/ml, respectively. All these samples were prepared with PEG/PMMA ratio of 90/10.

concentration of 40 mg/ml, a non-uniformly porous structure with low porosity was formed. The high viscosity of the concentrated solutions caused the reduction of solvent evaporation rate. Consequently, the phase separation was delayed, thereby the low porosity of nanosponge was achieved. The porous structure was uniformly created on the copper mesh with polymer concentration in a range of 20 ÷ 30 mg/ml. The results indicated that the porosity decreased with increasing polymer concentration and the range of polymer concentration from 20 to 30 mg/ml is suitable for achieving uniform structure.

Effect of PEG/PMMA ratio

Figure 4 demonstrates the effect of PEG/PMMA ratios on the porous structure. It can be seen that a uniform structure was formed without using PEG, while the uniformity became lower with successively adding PEG. In addition, the porosity increased with increasing the PEG content. The results can be explained that the hydrophilic PEG with low molecular weight of 1000 can easily dissolve in water, therefore it plays a role as a non-solvent or a pore inducer in the phase separation method.

TENG performance and application

Figure 5 illustrates the output performance of the TENG device which was fabricated in a contact-separation working mode. Accordingly, the PEG-PMMA nanosponges served as a positively charged tribo-materials, while the convex PDMS played a role as a negatively charged counterpart. For comparison, a TENG assembled with the flat aluminum and the same convex PDMS was also characterized. The results indicated that TENG using PEG5 exhibited the best output performance with V_{OC} of ~300 V and I_{SC} of ~85 μA (Figure 5a and Figure 5b), corresponding to the highest power of ~3.2 mW at matching external resistance of 5×10^6 Ω (Figure 5e). These obtained values are much higher than the ones of TENG with flat aluminum (Figure 5b and Figure 5c). The highest output performance of PEG5-based TENG is attributed to the combination of its highly structural uniformity and excellent electrification effectiveness of the PEG. In addition, the TENG's output under different frequencies were investigated, as expected, the V_{OC} remained the same figures with a decrease of the frequency from 5 to 1 Hz (Figure 5d).

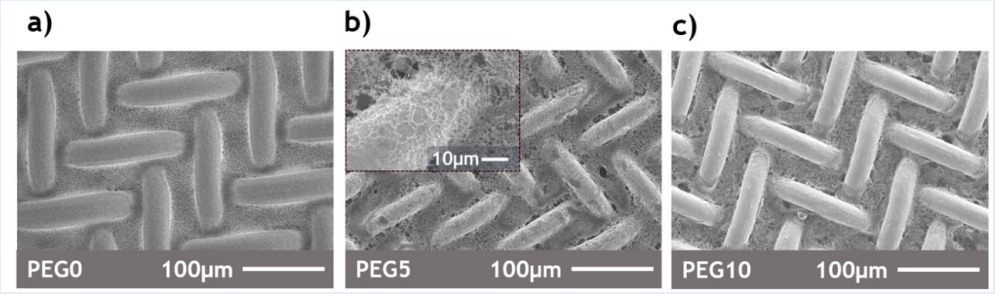

Figure 4: Effect of PEG/PMMA ratios on surface morphology of the PEG-PMMA nanosponges. SEM images of PEG-PMMA nanosponges prepared with various PEG/PMMA ratios: (a) 0/100; (b) 5/95; (c) 10/90. All these samples were prepared with the polymer concentration of 30 mg/ml.

To demonstrate the ability of the TENG for harvesting mechanical energy to generate electricity, the PEG-PMMA nanosponge-based TENG was used to charge a commercial capacitor. As shown in Figure 6a, the 2.2 μF-capacitor could be fully charged to 12,5 V within 3 minutes. Moreover, the developed TENG device could instantaneously light 100 LEDs by simple hand pressing (See Video S1 in Supporting Information.).

CONCLUSION

In this work, PEG-PMMA blends with the sponge-like porous structure were successfully prepared by a rapid and scalable solution-phase separation method based on the spinodal decomposition of a quinary solution of polyethylene glycol, poly(methyl methacrylate), tetrahydrofuran, methanol, and water. The results indicated that the porosity of the nanosponge increased, while their uniformity decreased with increasing PEG contents. The solution with a polymer concentration of 30 mg/ml and a PEG content of 5 % was found to be a suitable condition for preparing the uniform nanosponge hybrid. The TENG using the PEG-PMMA nanosponge exhibited an outstanding output performance of 3 mW which is much higher than the one using a normal aluminum surface. To demonstrate the ability of the TENG to generate electricity for powering microelectronics, the nanosponge-based TENG could fully charge a 2.2 μF-capacitor within 3 minutes, and it also could directly light 100 LEDs by simple hand pressing. We believe that the PEG-PMMA nanosponge hybrid prepared by the present method could not only apply for development of the large-scale high-performance multifunctional TENG with flexible, breathable, biocompatible features but also exhibits potential applications in tissue engineering or filtration.

SUPPORTING INFORMATION

Video S1. A TENG device harvesting mechanical energy to generate electricity to directly lit 100 LEDs on.

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CONFLICT OF INTEREST

The authors declare no competing financial interest.

AUTHORS' CONTRIBUTIONS

Yen Linh Nguyen: Conceptualization, Investigation, Discussion; Gia Huy Nguyen Hoang: Conceptualization, Investigation, Discussion; Pham Anh Quoc: Investigation; Nguyen Pham Cao Quan: Investigation; Thi Le Thanh Nguyen: Discussion; Tran Van Khai: Discussion; Pham Trung Kien: Discussion; **Thu Ha Le**: Writing – Reviewing, Investigation, and Editing; **Van-Tien Bui**: Supervision, Funding acquisition, Resources, Writing –Reviewing and Editing.

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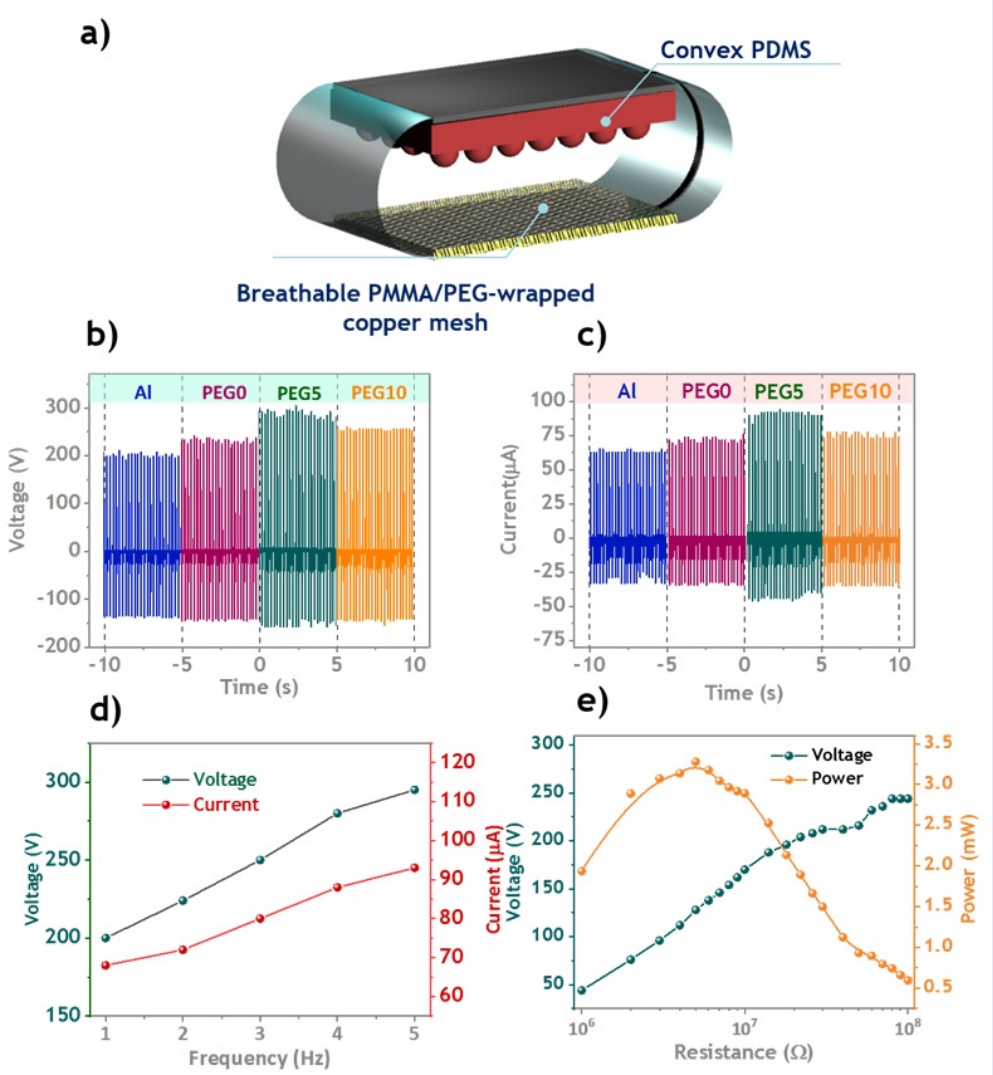

Figure 5: Characterization of electrical output performance of TENG devices. (a) 3D sketch of the TENG device; (b) Open-circuit voltage (V_{OC}); (c) Instantaneous output powers at different external resistances; (d) Effect of triggering frequency on TENG output; (e) Variation of power density of the PEG5-based TENG on resistance of external loads.

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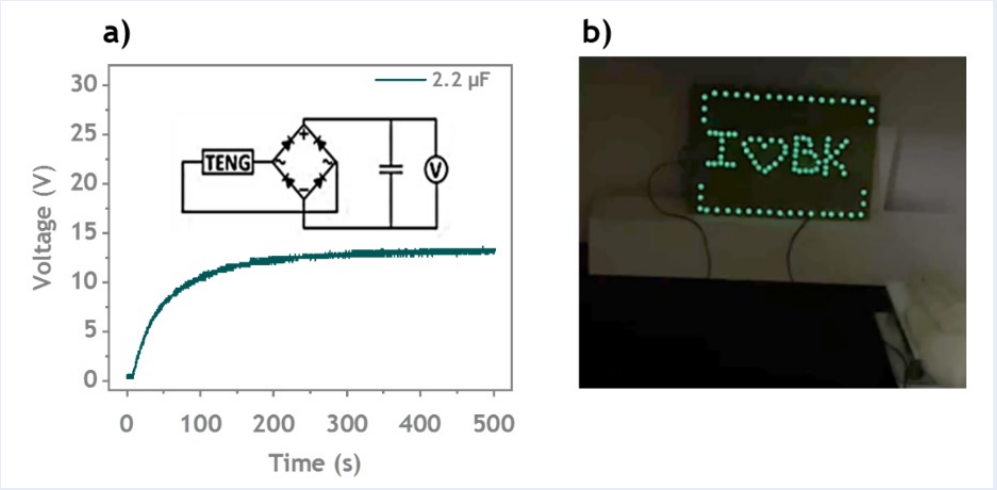

Figure 6: Demonstration of PEG-PMMA nanosponge-based TENG for harvesting mechanical energy and powering electronic devices: (a) Charging curves of a commercial capacitor, (b) TENG device used to instantly turn on 100 LEDs under simple hand pressing.

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Bài nghiên cứu

Chế tạo màng polyethylene glycol-poly(methyl methacrylate) blend cấu trúc xốp liên tục ứng dụng cho máy phát điện nano ma sát

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TÓM TẮT

Năng lượng tái tạo đang ngày càng được quan tâm trên toàn thế giới do những lo ngại về việc cạn kiệt nguồn nhiên liệu hóa thạch, ô nhiễm môi trường, và khủng hoảng năng lượng. Máy phát điện nano ma sát (TENG) được biết đến như là một trong những công nghệ tiềm năng để chuyển đổi sang năng lượng xanh bền vững. Trong nghiên cứu này, hỗn hợp của polyethylene glycol (PEG) và poly(methyl methacrylate) (PMMA) với cấu trúc xốp được chế tạo bằng phương pháp phân pha dung dịch, sau đó màng xốp chế tạo được sử dụng để phát triển máy phát điện nano ma sát hiệu quả cao. Các thông số của cấu trúc xốp bao gồm độ xốp và độ đồng đều cấu trúc được điều khiển bằng cách thay đổi nồng độ dung dịch polyme và tỷ lệ giữa PEG/PMMA. Lưới đồng (Cu-mesh) được sử dụng làm giá đỡ gia cường tăng sự ổn định cho cấu trúc xốp. Ngoài ra, poly(dimethyl siloxane) (c-PDMS) với bề mặt có cấu trúc lồi vi mô trật tự cao, đóng vai trò là bề mặt ma sát tích điện âm, cũng được chế tạo bằng phương pháp đúc vi mô, trong đó màng polyme cấu trúc xốp tổ ong trật tự cao được sử dụng làm khuôn. Thiết bị TENG chế tạo từ xốp PEG-PMMA và c-PDMS cho hiệu quả phát điện đạ 3.2 mW. Thiết bị TENG được phát triển không những có thể được sử dụng để thu năng lượng cơ học cấp nguồn cho các đèn LED mà nó còn thể hiện tiềm năng ứng dụng trong cảm biến theo dõi sức khỏe tự cấp nguồn.

Từ khoá: Phân pha, tương thích sinh học, cấu trúc xốp, máy phát điện nano ma sát

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