



Fabrication and characterization of a sandpaper-based flexible energy storage



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ABSTRACT

In this paper, graphene and carbon nanotubes dispersed in a pectin solution are examined as a precursor for electrode fabrication for supercapacitor applications. The carbon nanotubes not only prevent the stacking of graphene sheets, but also act as spacers and binders. Dropping the hybrid conductive suspension onto sandpaper is found to form a sandpaper-based electrode that improves the specific capacitance of a subsequently fabricated supercapacitor because of its high surface area. In particular, the large contact surface of the sandpaper allows it to absorb more electrolyte ions and increases the number of ions assembled on the electrode surface. For the supercapacitor fabrication, replacing the liquid or solid electrolyte with a gel electrolyte prevents leakage and contact discontinuity. Therefore, a high-performance supercapacitor can be constructed with one separator coated with a gel electrolyte inserted between two fine-sandpaper-based electrodes, which can be assembled into a sandwich structure by hot pressing. Electrochemical analysis shows excellent cycle stability and flexibility of the fine-sandpaper-based supercapacitor. Because of the simple and low-cost assembly of this flexible and lightweight supercapacitor, it has potential applications in many energy storage fields, including wearable electronics and flexible products.

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1. Introduction

The soft electronics era has recently begun with the extensive study of many lightweight flexible electronic devices such as flexible panel displays, flexible solar cells, and wearable electronics. However, a highly efficient energy storage device is still required for such soft electronic devices [1–4]. Supercapacitors, also known as electrochemical capacitors or ultracapacitors, are very promising alternatives to batteries for these energy storage applications because of their high power density. Furthermore, supercapacitors can provide faster charge/discharge rates, high energy densities, long lifetimes, and low maintenance costs [5,6]. On the basis of their different charge storage mechanisms, supercapacitors can be classified into electrical double-layer capacitors (EDLCs) and pseudocapacitors. On the one hand, EDLCs have drawn considerable attention as highly efficient devices. The capacitance of an EDLC comes from charge accumulation at the electrode/electrolyte interface and thus is highly dependent on the pore structure of the electrode and accessible surface area for the electrolyte [7,8]. On the

other hand, pseudocapacitors use fast and reversible redox reactions at the surfaces of electroactive materials for charge storage. However, the life cycles of pseudocapacitors are shorter than those of EDLCs [9,10]. Therefore, many researchers have prepared flexible supercapacitors with EDLC storage mechanisms [11].

Carbon nanomaterials are ordinarily used as the electrodes in EDLCs, because they can provide high conductivity, electrochemical stability, open porosity, and other attractive features [12]. Along these lines, there is a large amount of interest in the use of graphene in supercapacitors [13,14], because it exhibits a very large theoretical surface area and high electrical conductivity and because it can be produced at low cost [15,16].

However, the major problem with the use of graphene in supercapacitors is that graphene sheets tend to restack, effectively reducing the available surface area and thus the effective capacity of graphene to store energy. To address this, in most studies adhesives have been added to prevent the conductive materials from peeling off and restacking. For example, Shakir et al. added carbon nanotubes (CNTs) to graphene not only to prevent the stacking of graphene sheets, but also to act as a binder between graphene sheets [17]. Similarly, in this study, we have prepared binder-free flexible electrodes consisting of graphene sheets separated and blended with CNTs to form new electrodes for supercapacitors.

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Solid-state electrolytes are often used in flexible energy storage devices because of their good mechanical properties and to prevent electrolyte leakage. However, they often lead to poor contact at the electrode/electrolyte interface, which has restricted the application of solid polymer electrolytes to supercapacitors in particular [18,19]. Many researchers have replaced the liquid or solid electrolyte with a gel electrolyte to prevent both leakage and contact discontinuity. Owing to the strong adhesive force between the gel electrolyte and the electrode material, gel electrolytes can easily penetrate into the micropores of electrode materials, which prevent contact discontinuity during charging and discharging. Therefore, gel electrolytes could improve the specific capacitance in supercapacitors [20,21]. The final main component of a supercapacitor is the separator, a porous membrane placed between electrodes of opposite polarity that is permeable to ionic flow but prevents electric contact between the electrodes [22–26]. A poly(vinyl alcohol) (PVA) porous separator can be fabricated according to our previously reported method [27], which can keep the electrodes apart to prevent electrical short circuits and increase the flexibility in the bent state.

For flexible electrode substrates, the major choices are metal, plastic, textile, and sponge [2–4,12,18]. In addition, Hu et al. synthesized MoS₂ hierarchical nanospheres by the hydrothermal method which resulted in high specific area and porosity [28]. MoS₂ hierarchical nanospheres was used to fabricate solid state flexible supercapacitors which showed excellent electrochemical performance such as high capacitance 368 F/g at a scan rate of 5 mV/s and high power density of 128 W/kg at energy density of 5.42 Wh/kg. On the other hand, polyaniline (PANI) coated hierarchical porous carbon (HPC) composite was applied as the supercapacitor electrode material, which presents a high specific capacitance of 531 F/g at current density of 0.5 A/g and superior cycling stability of 96.1% (after 10,000 charge discharge cycles at current density of 10 A/g) [29].

In our laboratory, we have previously manufactured a paper-based supercapacitor with high electrochemical performance [30]. In this study, this technique was extended to the sandpapers as substrates to increase the contact area between the electrode and gel electrolyte and gain a better understanding of the influence of surface roughness on the energy storage performance of a supercapacitor. More ions could be stored at the interface between carbon sandpaper electrode and gel, since more contact surface areas were provided from the uneven surface of sandpaper, thereby the specific capacitance was enhanced. Furthermore, the capacitance behavior and cycle stability of the sandpaper-based supercapacitor were investigated under different bending conditions.

2. Experimental

2.1. Preparation of sandpaper-based electrodes

First, 0.5 g of graphene (P-ML20, Graphage) was added into 0.5 wt% apple pectin (Apple pectin, Sigma) aqueous solution and then ultrasonically dispersed by a high-power probe sonicator (VCX 750, SONICS) for 30 min. Subsequently, the solution was centrifuged at 4800 rpm for 15 min, and the precipitate was collected. The graphene suspension was prepared by mixing the precipitate with 10 ml of deionized water.

Similarly, the CNTs (VGCFTM-X, Showa Denko K.K.) were dispersed into 0.5 wt% pectin solution [30,31] by a high-power probe sonicator for 30 min. After the optimum dispersion was achieved, the CNT dispersion was centrifuged to remove possible remaining CNT aggregates. Two 15 min centrifugation cycles were performed at 4000 rpm, and the supernatant was collected after each cycle. To fabricate the electrodes, a blended conductive suspension was prepared by mixing the graphene and

CNT suspensions in a ratio of 1:5 (v/v). This suspension was then dropped onto the surfaces of sandpapers with different surface roughness (#600C and #1000C silicon carbide coated on water-proof abrasive paper; Sanlu Co., China). To complete the electrode fabrication by solvent evaporation, the coated sandpapers were dried under a vacuum oven at 60 °C for 4 h. The mass of the carbon coated electrode was $\sim 6.89 \pm 0.02$ mg/cm².

2.2. Preparation of gel electrolyte

The gel electrolyte was prepared by mixing 10 g of PVA (MW: 25,000) with 100 ml of deionized water. This solution was mechanically agitated by magnetic stirring at 85 °C to thoroughly dissolve the PVA in the water. Then, the 20 g of H₃PO₄ (purity 99%, Sigma) was added dropwise to the PVA solution with magnetic stirring. After the mixture was stirred thoroughly, the PVA/H₃PO₄ liquid electrolyte was dried in a vacuum oven at 60 °C for 12 h to make the gel electrolyte.

2.3. Preparation of porous PVA separator

PS spheres were synthesized using the emulsifier-free emulsion polymerization method [27]. A mixture of 90 ml of deionized water, 10 ml styrene (St), and 5 mg *p*-styrenesulfonic acid sodium salt (NaSS) was added to a four-necked flask equipped with a reflux condenser and a mechanical stirrer. After this mixture became homogeneous, a deoxygenated aqueous solution of 0.087 g of KPS was added and the reaction was carried out using the reflux condenser under a nitrogen atmosphere. After 24 h, a PS suspension with a particle size of about 2 μm was obtained. Then, the dilute suspension was obtained by mixing the PS sphere suspension and ethyl alcohol in a ratio of 2:15 (v/v).

The dilute suspension was poured into a Petri dish at room temperature and spin-dried at 30 rpm for 30 min by rotary shaking. Then, it was placed in a vacuum oven at 60 °C for 20 min to remove excess ethyl alcohol, after which a close-packed array of PS spheres was obtained. Later, 10 wt% PVA solution was poured onto the PS array to form the PVA/PS template. After drying vacuum at 60 °C for 2 h, this PVA/PS template was placed in toluene to remove the PS array, and the PVA microporous separator was obtained. A schematic illustration of the preparation of the PVA porous separator is shown in Fig. 1(a).

2.4. Fabrication of supercapacitors

A sandpaper-based supercapacitor was assembled from two fine-sandpaper-based electrodes and a PVA porous separator coated with H₃PO₄/PVA gel electrolyte, then packaged between two PET sheets by hot pressing. A schematic illustration of the structure of the sandpaper-based supercapacitor and its EDLC charge storage mechanism is shown in Fig. 1(b).

2.5. Characterizations

The morphologies and cross sections of the blended graphene and CNT materials, sandpaper, and sandpaper-based electrodes were examined by high resolution scanning electron microscopy (HR-SEM, Hitachi-SU800). Additionally, the morphology of the PVA porous separator was examined by environmental scanning electron microscopy (ESEM, FIE Quanta 400F).

To evaluate the electrochemical properties of the sandpaper-based electrodes and supercapacitors, electrochemical measurements were performed in a three-electrode electrochemical cell with a Pt counter electrode and an Ag/AgCl reference electrode in either gel or liquid electrolytes. All electrochemical characteristics were evaluated by CV, and the galvanostatic charge/discharge

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