Heterostructured nanomaterials, such as coaxial nanowires, core–shell nanoparticles, and nanostructured composites, are drawing a tremendous amount of attention in the nanoscience research field because they offer not only extraordinary properties brought about by confining their dimensions but also synergic properties or functionalities from combining different materials.1–11 There are a variety of ways to assemble nanocomponents into heterostructured nanomaterials. For example, layer-by-layer (LBL) assembly4,12,13 has been widely used to fabricate core–shell nanoparticles4 and coaxial nanowires.6,13 They form by coating two or more layers of different materials onto existing nanostructured materials. A straightforward and powerful method for fabricating nanostructured composites is to disperse or assemble inorganic nanoparticles (e.g., metal14 and magnetic nanocrystals17) with small sizes into the matrices, voids, and pores, or surfaces of nanostructured hosting materials18 (e.g., polymer nanowires,8,14 silica nanotubes,17 or TiO2 nanotubes16).

To date, heterostructured nanomaterials with metal oxide and conductive polymer have been rarely reported.19,20 In our previous paper, we reported the synthesis of coaxial MnO2/poly(3,4-ethylenedioxythiophene) (PEDOT) nanowires by a one-step coelectrodeposition method.7,11 Herein, we report the loading of finely dispersed MnO2 nanoparticles into PEDOT nanowires by simply soaking PEDOT nanowires into a potassium permanganate solution. We describe the synthesis of the nanowires and nanoparticles, characterize their chemical constituents and bonding, propose a mechanism of MnO2 formation, and report the electrochemical energy storage properties and volume change of the nanostructures. The pristine MnO2 nanoparticles in these MnO2 nanoparticle enriched PEDOT nanowires show very high specific capacitance (410 F/g) as supercapacitor electrode materials due to their extremely high exposed surface area with nanosizes. By the same token, these nanoparticles almost reach the theoretical limit of Li+ insertion and nanoparticles, characterize their chemical constituents and bonding, propose a mechanism of MnO2 formation, and report the electrochemical energy storage properties and volume change of the nanostructures. The pristine MnO2 nanoparticles in these MnO2 nanoparticle enriched PEDOT nanowires show very high specific capacitance (410 F/g) as supercapacitor electrode materials due to their extremely high exposed surface area with nanosizes. By the same token, these nanoparticles almost reach the theoretical limit of Li+ insertion.

**KEYWORDS:** heterostructured nanomaterials · nanowire · manganese oxide · PEDOT · electrochemical energy storage · supercapacitor · lithium ion battery

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coefficient and charge storage capacity as the potential cathode materials in the Li ion battery.

One advantage of this synthetic method is that no additional steps are needed to prepare MnO₂ nanoparticles before synthesizing the heterostructured material. When the PEDOT nanowires enter the solution, KMnO₄ is reduced via the redox exchange of permanganate ions with the functional group on PEDOT, triggering the formation and simultaneous spread of MnO₂ nanoparticles into the nanoscale pores of the nanowires. Varying the concentrations of KMnO₄ and the reaction time controls the loading amount and size of the MnO₂ nanoparticles.

Compared to the heterostructured MnO₂/PEDOT co-axial nanowires in our previous report,21 MnO₂ and PEDOT are more uniformly mixed in the heterostructured nanowires. As mentioned earlier, these imbedded MnO₂ nanoparticles have ultrahigh surface area, which enhance the energy storage capacity of PEDOT (e.g., to a high specific capacitance of 252 F/g) but without causing excessive volume expansion in the polymer. The highly conductive and porous PEDOT matrix facilitates fast charge/discharge of the MnO₂ nanoparticles and prevents them from agglomerating. These combined properties enable the MnO₂ nanoparticle enriched PEDOT nanowires, when applied as the electrode materials for supercapacitors, to have well-maintained high specific capacitance even at high charge/discharge rates.

RESULTS AND DISCUSSION

Scheme 1 illustrates the synthesis of MnO₂ nanoparticle loaded PEDOT nanowires, which will be abbreviated as MnO₂-NP/PEDOT nanowires. PEDOT nanowires were first electrodeposited in an alumina template and then exposed by template removal. The freestanding PEDOT nanowires were subsequently soaked in potassium permanganate solution for various times. MnO₂ nanoparticles grew in the PEDOT matrix during this soaking process.

The color of the PEDOT nanowires turned from navy blue (Figure 1a) to a green hue (Figure 1b) when the brownish colored MnO₂ NP formed. It is worth noting that the thus-formed MnO₂ nanoparticles can be

- K₃MnO₄ (10 mM)
- H₂SO₄ (20 mM)
- Treated PEDOT Nanowires (10 min)
- Treated MnO₂/PEDOT Composite Nanowires

Figure 1. Digital images of (a) PEDOT nanowires dispersed in ethanol, (b) MnO₂-NP/PEDOT nanowires in ethanol. (c) Sulfuric acid treated MnO₂-NP/PEDOT nanowires, MnO₂ is stripped off (d) overoxidized PEDOT nanowires after soaking PEDOT nanowires in 10 mM KMnO₄ for 30 min.
stripped off by dilute sulfuric acid, which causes the PEDOT nanowires to return to navy blue color (Figure 1c). Soaking the nanowires in higher concentrations of KMnO₄ or at longer times will etch and overoxidize the PEDOT matrix (discussed later), rendering the PEDOT nanowires brownish in color, as shown in Figure 1d.

The SEM image (Figure 2a) of the KMnO₄ treated PEDOT nanowires shows that they aggregated into piles driven by surface tension during solvent evaporation. To prevent aggregation from affecting the electrochemical testing, the electrode never was allowed to completely dry. Figure 2b shows the high resolution SEM image of the same nanowires, which reveals their cigar shapes and rough surfaces. Figure 2c is the TEM image from a segment of KMnO₄ treated PEDOT nanowire. Some dark nanoparticles are vaguely visible but are clearer in a higher resolution TEM image (Figure 2d). These nanoparticles are less than 5 nm in size and finely dispersed in the PEDOT matrix. XRD studies show that the nanoparticles are X-ray amorphous, but TEM studies show that the nanoparticles have weak crystalline structures, which may have formed due to annealing caused by the TEM electron beams. The lattice spaces and electron diffraction ring patterns only partially match the XRD indices of α-MnO₂. (See Supporting Information SI1.) Thus these embedded MnO₂ nanoparticles are probably in the forms of amorphous MnO₂ or weakly crystallized α-MnO₂, which has been studied for its application in the electrode materials of supercapacitors.²²,²³

Additional methods were used to analyze the KMnO₄ treated nanowire and confirm the existence of MnO₂. Figure 3a–d shows the TEM images, Mn K map, S (sulfur from the thiophene ring on PEDOT) K map, and their mixed map on a segment of MnO₂-NP/PEDOT nanowire. Figure 3e shows the EDS spectra from a single MnO₂-NP/PEDOT nanowire. The atomic ratio of Mn to S in the nanowire is 1.42:1, which suggests that a large amount of MnO₂ NP has been loaded into the PEDOT matrix.

Figure 4 shows the XPS spectra obtained from the MnO₂ NP loaded PEDOT nanowires. The peaks of Mn 2p₃/₂ and Mn 2p₁/₂, which are centered at 642 and 653.8 eV, respectively, with a spin energy separation of 11.8 eV, are in good agreement with reported data of Mn 2p₃/₂ and Mn 2p₁/₂ in MnO₂.²⁴

We propose and discuss several plausible mechanisms of MnO₂ formation. On the basis of the Mn/S ratio shown in Figure 3e, we can assume that 1 mol of PEDOT can encapsulate ca. 1.4 mol of MnO₂ NP. This process requires 4.2 mol of electrons to reduce the 1.4 mol of KMnO₄. However, it is difficult to conceive that 1 mol of PEDOT can provide 4.2 mol of electrons because the PEDOT is the already oxidized form of EDOT. The maximum number of electrons it can provide depends on its doping level, which is 0.3.²⁵ This suggests that 1 mol of PEDOT can only provide 0.3 mol of delocalized electrons during the doping (oxidation) process, which is not enough to reduce 1.4 mol of KMnO₄.

By looking at the structure of PEDOT, one would find that there are unsaturated bonds on the PEDOT conjugated rings, which might be broken and oxidized by the KMnO₄. If breakage occurs, the oxidation will destroy the conjugation of PEDOT, and it will lose its conductivity and electroactivity. However, experimental tests ruled out this possibility: After the MnO₂ NPs are loaded in the PEDOT matrix, dilute sulfuric acid was applied to the heterostructured nanowires to strip off the MnO₂. Electrochemical tests on these MnO₂ stripped PEDOT nanowires showed that the electrochemical properties were retained (data not shown) when compared to the electrochemical properties of PEDOT nanowires before MnO₂ NP insertion.

Figure 2. (a,b) SEM and (c,d) TEM images at different magnifications of PEDOT nanowires after being treated with KMnO₄ (10 mM) for 10 min.

Figure 3. (a) TEM image and EDS mapping of (b) Mn, (c) S, and (d) mixture of Mn and S on a segment of MnO₂-NP/PEDOT nanowires.

Figure 4. XPS spectra obtained from the MnO₂ NP loaded PEDOT nanowires.
On the basis of the above analysis, the mechanism of MnO₂ NP formation becomes more ambiguous. Taking a glance at the EDOT monomer structure, however, reveals another possibility: The sulfur on the thiophene ring may serve as a site for KMnO₄ reduction. Indeed, MnO₂ nanoparticles have been synthesized previously by reducing KMnO₄ at the nitrogen sites of aniline monomers.²⁶

Figure 5a shows the IR spectra of PEDOT nanowires treated with different concentration of KMnO₄ for 10 min. An absorbance peak at 1044 cm⁻¹ gradually appears as the concentration of KMnO₄ increases and can be identified clearly when the KMnO₄ concentration reaches 50 mM. The arising peak at 1044 cm⁻¹ can be assigned to the sulfoxide (S=O bond) stretching mode,²⁷ which suggests possible oxidation of the thiophene sulfur into sulfoxide by KMnO₄ (see Scheme 2.) Figure 5b shows that the sulfur peak shifts slightly to the higher binding energy after the PEDOT has been treated by KMnO₄, which suggests that the sulfur has been oxidized. The new peak (168 eV) in XPS analysis alludes to the possible oxidation of thiophene sulfur into the sulfone O=S=O group.²⁸ Similar oxidation on the sulfur sites of PEDOT:PSS by hydrogen peroxide²⁹ or NaOCl³⁰ has been described elsewhere. Oxidation of sulfides to corresponding sulfones by KMnO₄ is also reported.³¹ In order to prevent PEDOT from losing its electroactivity, the concentration of KMnO₄ and the soaking time were kept low and short so that only a small portion of the PEDOT is oxidized. It is worth noting that only partial oxidation of thiophene sulfur sites on PEDOT will not be able to provide enough electrons.
for the KMnO₄ reduction ratio of 1.4:1 MnO₂/PEDOT. Nevertheless, such reduction reactions may form the initial seeds that accelerate the water reduction of KMnO₄ to MnO₂ and further grow the MnO₂ nanoparticles.

We also analyzed the effects of the soaking conditions on the Mn/S ratio in the PEDOT nanowires. By controlling the soaking time and the concentration of the KMnO₄, we can regulate the loading amount of MnO₂. Figure 6a shows the Mn/S ratio in the nanowires as determined by EDS analysis after PEDOT reacted with different concentrations of KMnO₄ for 1 min. It is worth noting that the MnO₂ loading increases more rapidly at higher concentration of KMnO₄, which could be caused by the complete etching away of PEDOT after it is overoxidized into soluble product. Figure 6b shows how the Mn/S ratio responds to the soaking time more linearly.

It is interesting to note that not only can the loading amount of MnO₂ be varied but also the nanoparticle size and structure can be tuned when different concentrations of KMnO₄ or different soaking times are applied (see Supporting Information SI2).

In order to study the electrochemical energy storage benefits of loading MnO₂ into the PEDOT matrix, cyclic voltammetry (CV) (Figure 7a and Supporting Information SI3) and galvanostatic charge/discharge (Figure 7b and Supporting Information SI4) tests were performed on the PEDOT nanowires before and after KMnO₄ treatment. As we can see from Figure 7a, CV shapes of PEDOT nanowires before and after MnO₂ NP enrichment all look like a rectangular envelope, which suggests that both PEDOT nanowires and MnO₂-NP/PEDOT nanowires have good capacitive properties at this scan rate. The inclusion of MnO₂ nanoparticles has resulted in the capacitive current increasing 4-fold. Both techniques are used to calculate the specific capacitances of MnO₂-NP/PEDOT nanowires at various current densities (see Supporting Information SI5). Thus, the calculated specific capacitance of MnO₂-NP/PEDOT nanowires is 250 F/g, which is higher than our previously reported MnO₂/PEDOT coaxial nanowires. Eighty percent of the maximum specific capacitance is maintained, while the current density is boosted over 25 mA/cm² (see Supporting Information SI6). The well-maintained capacitance value even at high current density or high charge/discharge rate is due to small dimensions of the PEDOT nanowires and MnO₂ NPs as well as the high conductivity of PEDOT. Due to the large specific surface area of MnO₂ NPs, the pristine specific capacitance of MnO₂ NPs can be calculated as 410 F/g, which is higher than other synthesized nanostructured MnO₂ supercapacitor electrodes at the same charge/discharge rate (see Supporting Information SI5). Figure 7b also demonstrates that the MnO₂-NP/PEDOT nanowires can serve as potential cathode materials for lithium ion battery. It shows that the charge capacity significantly increased at least 4 times after loading of MnO₂ nanoparticles. Indeed, nanostructured MnO₂ or PEDOT has been reported for applications in lithium ion batteries. The pristine charge storage capacity of MnO₂ can be estimated as 300 mAh/g, which is very close to the theoretical charge storage capacity of MnO₂ (308 mAh/g). The corresponding lithium insertion coefficient (x value in the LiₓMnO₂) can be calculated as 0.97, which is also very close to the maximum value of 1. Such high insertion coefficient is due to complete lithium intercalation into the small nanosized MnO₂ nanoparticles (see Supporting Information SI5).

The electrochemical stability test of the MnO₂ NP enriched PEDOT nanowires was performed using cycled galvanostatic charge/discharge methods. As shown in Figure 8, the MnO₂-NP/PEDOT nanowires displayed satisfactory electrochemical stability. On the basis of the charge/discharge time, the charge storage capacity loss can be calculated as 10% loss after 500 cycles and 2% loss after 100 cycles. In addition, the charge/discharge...
curve maintained symmetrical shapes after 100 and 500 cycle tests.

It is worth noting that, although the inclusion of MnO2 NPs significantly increases the charge capacity of PEDOT nanowires, the volume of the PEDOT did not expand significantly. Figure 9 shows the TEM images of PEDOT nanowires (grown on Au substrate) before and after the KMnO4 treatment. The \( d_{\text{PEDOT}} \) and \( d_{\text{MnO2/PEDOT}} \) represent the diameters of the PEDOT nanowire before and after KMnO4 treatment, and \( d_{\text{Au/PEDOT}} \) and \( d_{\text{Au-MnO2/PEDOT}} \) represent the diameters of the their corresponding gold bottom. The diameter expansion of the MnO2-NP/PEDOT nanowires is less than 8%, which is easily accommodated by the available space between the PEDOT nanowires after template removal.

Assuming that the diameter of the bottom gold is not affected by the KMnO4, and the PEDOT nanowire swelling is isotropic, the volume expansion rate is given by

\[
\frac{V_2 - V_1}{V_1} = \left( \frac{d_{\text{MnO2/PEDOT}}/d_{\text{Au-MnO2/PEDOT}}}{d_{\text{PEDOT}}/d_{\text{Au/PEDOT}}} - 1 \right)^3 \quad (1)
\]

where \( d_{\text{PEDOT}} \) and \( d_{\text{MnO2/PEDOT}} \) represent the diameters of the PEDOT nanowire before and after KMnO4 treatment and \( d_{\text{Au/PEDOT}} \) and \( d_{\text{Au-MnO2/PEDOT}} \) represent diameters of the their corresponding gold bottom. The calculated volume expansion is 25%.

Volume expansion of the PEDOT nanowire can also be theoretically estimated by the equation

\[
\frac{V_2 - V_1}{V_1} = \frac{n_2 d_2 M_2}{n_1 d_1 M_1} \quad (2)
\]

where \( n_2/n_1 \) is the mole ratio of the MnO2 to PEDOT (herein 1.42), \( d_2 \) and \( d_1 \) are the densities of MnO2 and PEDOT,40 and \( M_2 \) and \( M_1 \) are the molecular weights of MnO2 and PEDOT. On the basis of eq 2, the estimate of the volume expansion is 28%, which well matches the experimentally obtained volume expansion. The small volume expansion is mainly due to the low volumetric mass density and high porosity of the PEDOT matrix, which provides spaces to include a large amount of MnO2.

In our previous paper,21 for the MnO2/PEDOT co-axial nanowires synthesized at 0.75 V, the core MnO2 takes 25% of the total volume, and the mole ratio of Mn to S is approximately 1:1. In this paper, for the MnO2-NP/PEDOT nanowires, the MnO2 nanoparticles take 20% of the total volume while maintaining the mole ratio of Mn to S at 1.42:1. Such high loading capacity is probably because the chemically formed MnO2 nanoparticles are denser than the electrodeposited nanostructures. Thus, incorporated MnO2 nanoparticles utilize less space while providing more energy capac-
ity, which further increases the energy density and power density per volume.

CONCLUSION

In summary, we described the fabrication of MnO2 nanoparticle loaded PEDOT nanowires by simply soaking the PEDOT nanowires in KMnO4 solution. On the basis of the spectroscopic evidence from IR and XPS for the S=O group formation in the PEDOT, the formation of MnO2 nanoparticles is most likely triggered by the redox exchange of KMnO4 with the sulfur sites on the PEDOT structures. The nanosized MnO2 nanoparticles can efficiently store the lithium ions and boost the energy storage capacity by 4 times. The hosting material, highly conductive and porous PEDOT, facilitates the fast charge/discharge rate of the MnO2 nanoparticles and prevents them from aggregating. These synergic properties enable the MnO2 NP enriched PEDOT nanowires to be promising electrode materials with high capacitance at high charge/discharge rates for supercapacitors and lithium ion batteries. The present work may pave a new pathway of controlled synthesis of other inorganic/organic multifunctional heterostructured nano-materials via chemical reaction of oxidative inorganic precursor and polymer materials with a reductive functional group.

METHODS

Chemicals and Materials. 3,4-Ethylendioxythiophene (EDOT) was obtained from Aldrich (Milwaukee, WI). Lithium perchlorate and potassium permanganate were obtained from Fisher Scientific (Fair Lawn, NJ). Acetonitrile and propylene carbonate were obtained from Sigma Aldrich. Gold electroplating solution (Orotemp 24) was purchased from Technic (Cranston, RI). Deionized water (ca. 18 MΩ·cm resistivity) was obtained by using a Milli-Q water purification system from Millipore (Dubuque, IA). Alumina membranes, with a pore diameter of 200 nm and thickness of 60 μm, are commercially available from Whatman (Clifton, NJ).

Synthesis. PEDOT nanowires were synthesized potentiostatically (1.2 V) in the acetonitrile solution of 100 mM EDOT and 100 mM LiClO4. All potentials were measured relative to a Ag/AgCl reference electrode using a Pt foil as a counter electrode, if not specified otherwise. Alumina template was subsequently removed by 3 M NaOH solution to expose the PEDOT nanowires. MnO2 nanoparticles were loaded into the PEDOT nanowires by soaking the above exposed PEDOT nanowires in potassium permanganate solution with different concentrations (5 – 50 mM), depending on the desired amount of MnO2 nanoparticles to be loaded. KMnO4 (especially under acidic environment) is a very powerful oxidizing reagent, which may overoxidize the PEDOT. To prevent this overoxidization, we use neutral MnO2 solution with low concentration (typically 10 mM) and short treating time (typical 10 min). Gold bottoms for the volume expansion studies were electrochemically deposited at the current density of ~1 mA/cm2 for 30 min in the Orotemp 24 gold plating solution. The preparation of a working electrode is as follows: First, a thin layer of gold (ca. 500 nm) was sputtered onto the branched side of an alumina membrane with a Denton Vacuum Desktop III sputtering system. The Au-coated membrane was connected to an electrical circuit using a copper tape (3M). Defining and sealing an electroactive window (30.5 cm² in nominal area) was performed using silicone rubber or parafilm. Considering the porosity of the membrane (60%), the corrected surface area of the electroactive window was 0.2 cm². The mass and the length (thickness in film) of the resulting PEDOT nanowires were controlled by fixing the total charges passed during the electropoly- sition. Typically, coaxial nanowires with lengths of 10 μm can be obtained after the charge passed about 200 μC. Diameters of these nanowires correspond to the pore diameters of the alumina template (ca. 300 nm).

Characterization. The MnO2-NP/PEDOT nanowires were investigated using a field emission scanning electron microscope (Hitachi SU-70 SEM, operated at an acceleration voltage of 10 keV) and transmission electron microscope (JEOL, JEM 2100 field emission transmission electron microscope (FE-TEM), 200 keV). The sampling methods for SEM and TEM analysis are briefly described: gold-coated side of a small piece of an alumina template was tightly attached onto an SEM specimen holder by using a carbon tape. The template was dissolved to expose the nanowires by using 3 M NaOH. After rinsing it with deionized wa- ter repeatedly, the sample was dried in air before observation. For TEM sampling, sonication is applied to the MnO2-NP/PEDOT to disperse them in ethanol. The released nanowires were repeatedly rinsed with deionized water and ethanol. Then, 6 μL of the nanowire solution was dropped and dried on a TEM grid. FT-IR spectra were obtained using a Thermo Nicolet Nexus 670 FT-IR instrument equipped with a photoelastic modulation module. The spectra were collected using a DTGS KBr detector. The samples were prepared by soaking electrodedeposited PEDOT films in various concentrations of KMnO4 (0, 10, 20, and 50 mM) for 10 min. The PEDOT and resulting MnO2/PEDOT films were then ground with KBr and pressed into homogeneous pellets. Each pellet including a blank for the background was scanned 32 times from 4000 to 400 cm⁻¹ using the FT-IR instrument with a resolution of 4 cm⁻¹. Background subtraction was done by Omnic software in auto gain mode. XPS analysis was done on Kratos AXIS 165 spectrometer. C 1s (for hydrocarbon or hydrocarbon groups) = 284. Eight electronvolts was used as the calibrant.

The electrochemical studies of MnO2-NP/PEDOT nanowires are performed in the standard three-electrode system: Ag/AgCl is used as the reference electrode, and Pt foil is used as the counter electrode. In order to calculate specific capacitance, cyclic voltammetry at different scan rates (100–500 mV/s) and galvanostatic charge/discharge tests at different current densities (10–50 mA/cm²) were performed by cycling potential from 0 to 1 V in 1 M LiClO4 in water. Aqueous system is chosen because the charge/discharge speed of MnO2-NP/PEDOT will be faster in the water system, which is critical in the supercapacitor application. Galvanostatic charge/discharge for the lithium ion insertion study is done by cycling the potential from –0.7 to 0.9 V at current density of 1.25 mA/cm² (corresponds to about 10 C charge/discharge rate for MnO2-NP/PEDOT nanowires) in the solution of 1 M LiClO4 in the propylene carbonate. Organic solvent (PC) is chosen to achieve the broader range of electrochemical windows and prevent the electrolyte from drying. All of the above electrochemical experiments were performed using a bipotentiostat (Bi-STAT; Princeton Applied Research).

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Supporting Information Available: High-resolution TEM and electron diffraction patterns of the MnO2 nanoparticles. TEM images of MnO2-NP/PEDOT nanowires with less KMN0.4 treating time. Cyclic voltammograms of MnO2-NP/PEDOT nanowires at different scan rate. Galvanostatic charge/discharge curves of MnO2-NP/PEDOT nanowires. Detailed calculations of specific capacitance, lithium storage capacity of the MnO2-NP/PEDOT nanowires, and pristine MnO2 nanoparticles. Specific capacitance of MnO2-NP/PEDOT nanowires at different charge/discharge current densities. This material is available free of charge via the Internet at http://pubs.acs.org.

REFERENCES AND NOTES


