



# Synthesis of Ni/NiO core-shell nanoparticles for wet-coated hole transport layer of the organic solar cell

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## ABSTRACT

Nickel oxide (NiO) nanoparticle is a promising material as a *p*-type semiconductor to replace PEDOT:PSS which is frequently used as a hole transport layer in organic solar cells. In this study, we synthesized Ni/NiO core-shell nanoparticles via direct thermolysis of Ni(acac)<sub>2</sub> in the presence of trioctylphosphine oxide ligand and subsequent air oxidation to increase NiO shell thickness. Transmission electron microscopy analysis revealed the core-shell structure of Ni/NiO with average size of ~10 nm as well as an increased shell thickness by air-oxidation. The oxidized nanoparticles were well dispersed in organic solvent such as toluene, which can directly form a hole transport layer of organic solar cell by wet coating method. By spin coating various Ni/NiO nanoparticles on the ITO surface, organic solar cells with P3HT:PCBM bulk heterojunction as an active layer were fabricated. Solar cells utilizing air-oxidized Ni/NiO core-shell nanoparticle interlayer exhibited a better performance compared to those utilizing the as-synthesized Ni/NiO layer, or with no interlayer in between ITO and active layer.

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## 1. Introduction

As a promising candidate for clean renewable energy sources, organic solar cell (OSC) has gained considerable attention due to its advantageous features such as low processing cost, light weight, design flexibility and etc [1–5]. In OSC, excitons are produced at the active layer upon light exposure, and the dissociated electrons and the holes from the excitons are transported to cathode and anode respectively. For efficient dissociation of excitons, bulk heterojunction (BHJ) nanostructures are preferred as the active layer. To date, a blend of regioregular poly(3-hexylthiophene) (P3HT) and the fullerene derivative [6,6]-phenyl-C61 butyric acid methyl ester (PCBM) has shown the best power conversion efficiency (PCE) due to a large surface area provided by phase separated BHJ structure, and well-matched energy level of active materials with the anode and the cathode to allow facile electron transport. For P3HT:PCBM active layer, the lowest unoccupied molecular orbital (LUMO) of PCBM is matched with the energy state of the cathode (usually Al) to facilitate electron transport while the highest occupied molecular orbital (HOMO) of P3HT is similar to the fermi level of indium tin oxide (ITO) to collect holes from active layer. One very important interface in OSC is that between an active layer and ITO, where there can be physical contact between electron transporting PCBM and ITO to result in the non-geminate recombination at the

interface. To prevent this, an organic thin layer of semiconducting poly(3,4-ethylenedioxythiophene):polystyrene sulfonate (PEDOT:PSS) blend is often employed since it serves as an excellent electron blocking layer (EBL) or in other words hole transport layer (HTL) [6]. However, PEDOT:PSS has many drawbacks such as high acidity, morphological instability, hygroscopic property, and so on [7–10]. Thus, there have been efforts to replace PEDOT:PSS with stable *p*-type inorganic semiconductor nanoparticles to enhance device lifetime, and several metal oxide systems such as V<sub>2</sub>O<sub>5</sub> (Vanadium oxide), [11,12] MO<sub>3</sub> (Molybdenum oxide), [13] and NiO [14–16] were suggested as the candidates for promising HTL materials. Recently Irwin et al. successfully employed vacuum-deposited NiO as anode interfacial layer for OSC. They reported that NiO layers thinner than 10 nm showed superior HTL efficiency than PEDOT:PSS. However, previous studies used vacuum deposition techniques such as pulsed laser deposition (PLD) and RF sputtering to form metal oxide layer. However, recent advances in metal oxide nanoparticle synthesis have enabled us to obtain nanoparticles well-dispersed in organic solvents, which might be applicable to the HTL for OSC [17–19]. In this study, we demonstrate the synthesis of uniform Ni/NiO nanoparticles and direct application of their dispersions as OSC HTL by wet coating method.

## 2. Experimental

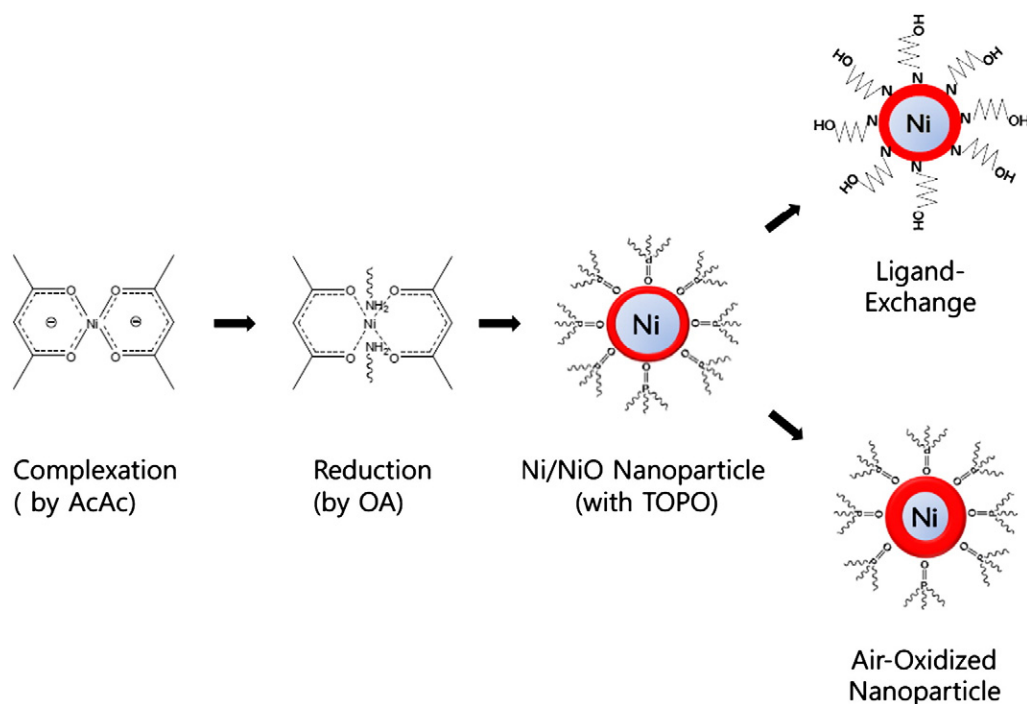
### 2.1. Synthesis of Ni/NiO nanoparticles

Ni/NiO core-shell nanoparticles were synthesized by modifying the procedures reported by Hyeon and coworkers [17,18]. Entire

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**Fig. 1.** Synthetic scheme of Ni/NiO core-shell nanoparticles. As synthesized Ni/NiO nanoparticle was capped with TOPO ligand, and it was either reacted with 5-amino-1-pentanol to enhance dispersibility in alcohol, or air-oxidized in *n*-octylether.

synthetic scheme is illustrated in Fig. 1. Throughout the syntheses, anaerobic condition was maintained by flowing  $N_2$  gas through the reaction vessel. All the chemicals were used as received. First, 1.0 g

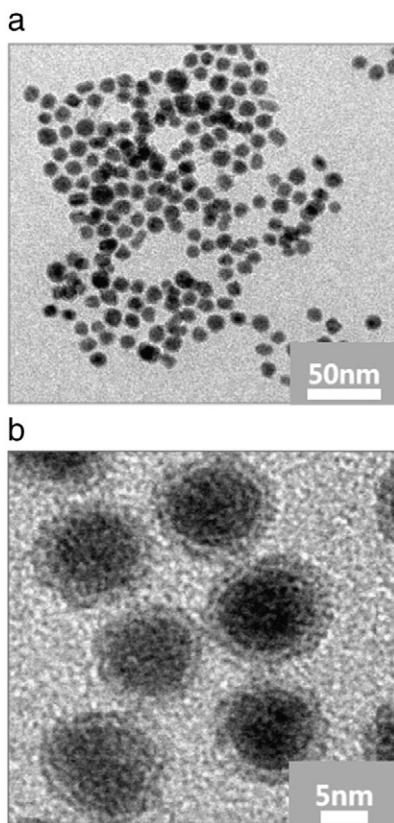
(3.9 mmol) of Nickel acetylacetonate ( $Ni(acac)_2$ , 95%, Aldrich) was added to the flask containing 10.4 g (39.0 mmol = 10 equiv.) of oleylamine (OA, Aldrich), and 1.21 g (3.12 mmol) of Trioctylphosphine oxide (TOPO, 90%, Aldrich). After reacting for 2 h at 100 °C, 0.8 equiv. of Trioctylphosphine (TOP, 90%, Aldrich) was added and the temperature was rapidly raised up to 240 °C. Initially aquamarine color of the reaction mixture turned black upon production of the nanoparticle. After 30 min of aging at 240 °C, reaction flask was cooled to room temperature (r.t.), and the precipitate was obtained by pouring excess amount of ethanol. The final product was well dispersed in nonpolar solvent such as toluene or *n*-hexane. To enhance dispersibility of the nanoparticle in alcohol medium, ligand exchange was carried out. 5-amino-1-pentanol (AP) and Ni/NiO nanoparticle was mixed in chloroform, and the mixture was stirred for 4 h at r.t. The ligand-exchanged product was precipitated with *n*-hexane, and dispersed in ethanol. The dispersion was stable for several days.

## 2.2. Air-oxidation of Ni/NiO nanoparticle

Ni/NiO nanoparticle with TOPO ligand was dispersed in *n*-octylether (Aldrich), and oxidation of nanoparticle was carried out with air-bubbling at an elevated temperature of 250 °C for several hours [19]. The oxidized Ni/NiO core-shell nanoparticle was flocculated and washed with ethanol and redispersed in toluene at ~1 wt.% concentration.

## 2.3. Characterization of nanoparticles

A nanoparticle dispersion in toluene was diluted to  $\sim 10^{-3}$  wt.% and then drop-cast onto carbon-coated TEM grid. After drying in air and subsequently in vacuum, the nanoparticles' shape and morphology were characterized by transmission electron microscopy (TEM) (tecna F20, FEI). The crystal structure of Ni/NiO nanoparticle was characterized by X-ray diffractometer (XRD, Rigaku D/Max-2500) operating with  $Cu-K\alpha$  radiation. Surface roughness of an air-oxidized Ni/NiO nanoparticle film was measured by obtaining atomic force microscope (AFM, Asylum) image of the film on Si wafer.



**Fig. 2.** (a) TEM image of the as-synthesized Ni/NiO nanoparticles. (b) Magnified view of the nanoparticles showing thin NiO shell surrounding dark Ni core. Average particle diameter was measured to be ~13 nm.

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