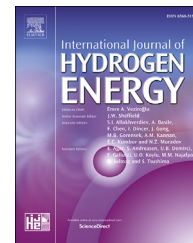




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Enhanced electrocatalytic activity of NiO nanoparticles supported on graphite planes towards urea electro-oxidation in NaOH solution

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ABSTRACT

Nickel oxide nanoparticles are fabricated onto graphite planes [NiO/Gt] by chemical precipitation of Ni(OH)₂ particles with consecutive calcination at 400 °C. The formed electrocatalysts are characterized using X-ray diffraction (XRD) and Transmission electron microscopy (TEM). TEM images demonstrate the deposition of NiO nanoparticles on graphite surface through their crystallite lattice fringes with spacing values of 2.45 Å (111), 2.10 Å (200) and 1.48 Å (220). The electrocatalytic activity of NiO/Gt electrocatalyst is examined towards urea electro-oxidation in NaOH solution using cyclic voltammetry, chronoamperometry and electrochemical impedance spectroscopy. Urea oxidation peak current density is observed at NiO/Gt electrocatalyst containing 15 wt% NiO [NiO/Gt–15] at a potential value of +640 mV (Ag/AgCl) with a current density value of 17.63 mA cm⁻². The loading amount of NiO in the prepared electrocatalyst significantly affects its electrocatalytic performance. NiO/Gt–15 exhibits the highest urea oxidation current density with the desired stability. The lower Tafel slope, charge transfer resistance and the higher exchange current density and diffusion coefficient values of urea molecules at NiO/Gt–15 surface elect its application as a promising electrocatalyst material during urea oxidation reaction in fuel cells.

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Introduction

Urea as a stable, cheap, non-toxic and non-flammable solid at room temperature has many applications. It could be used as a fertilizer to release nitrogen in plants and as an additive in animals' food. It also acts as a hydrogen carrier for long-term sustainable energy supply [1]. During the industrial fabrication of urea, large amount of wastewater containing urea would be formed. Toxic ammonia would be released to the atmosphere when urea was decomposed using urease

enzyme [2,3]. This would raise air pollution level as well as the immobilization and deactivation of this enzyme would also affect the long-term and large-scale decomposition of urea. Therefore, it is necessary to adopt efficient methods to decompose urea during wastewater treatment. For this purpose, hydrolysis, adsorption, biological decomposition and chemical oxidation were employed [4]. However, high energy may be consumed or sophisticated equipments may be utilized through these investigated methods. Electrochemical way to decompose urea is a straightforward method that

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purifies urea-rich wastewater resulting in the evolution of non-toxic gases as CO₂ and N₂ as well as valuable H₂ gas [5–9].

Many noble metal electrocatalysts were examined for urea electrolysis [10–13]. Recently, low-cost nickel-based electrocatalysts act as the most active components for urea electro-oxidation in alkaline medium in relation to Pt, Ir, Rh and Ru [5]. Nickel hydroxide electrode, formed on stainless steel foil by cathodic electrodeposition through a monolayer of polystyrene spheres as a template, demonstrated increased urea oxidation current density value as cup-like pore arrays when compared to the film and hollow-sphere structure. This was attributed to the large number of wide channels that easily transport electrolyte ions and gases in addition to the network structure with interconnected walls for electron conduction [14]. Nano-sheet Ni(OH)₂/Ni foam electrode, synthesized using a template-free growth method, displayed urea oxidation current density value of 337 mA cm⁻² at 450 mV (Ag/AgCl) in (0.6 M urea + 5 M KOH) solution [15]. Open-ended α-Ni(OH)₂ nanotubes grown on three-dimensional Ni framework that were prepared by hydrolysis of nickel chloride in the presence of hexagonal ZnO nanorods exhibited much higher current density value during urea electrolysis than that with attached Ni(OH)₂ film [16]. Ye et al. [17] have prepared highly porous nickel@carbon sponge electrode through sponge carbonization procedure with nickel electrodeposition. It showed lower onset potential with much increased current density values for urea oxidation reaction when compared to Ni/Ti flat electrode fabricated by similar method. This enhanced electrocatalytic activity was rationalized to the increased intrinsic electronic conductivity, unique porous structure and rich surface with nickel active sites that could boost the charge transfer rate during urea oxidation reaction. Nickel hexacyanoferrate could form carbon-encapsulated nickel-iron nanoparticles through a pyrolysis process. An ultra-thin layer of these nanoparticles could be deposited on three-dimensional macroporous nickel foam using electrophoresis technique. Lowered onset potential and increased urea oxidation current density values were gained at this modified electrode in relation to that at bare nickel foam [18]. Nickel-cobalt nanowire arrays, synthesized by one-step galvanostatic electrodeposition with a polycarbonate membrane as the template, showed the lowest onset potential value for urea oxidation reaction when Co proportion in Ni and Co bath solution was adjusted into 10% [19]. NiCo₂O₄ nanosheet electrode demonstrated an increased urea oxidation current density by 28 times at a potential value of 500 mV (Hg/HgO/1.0 M NaOH) when compared to that of bulk nickel hydroxide electrode [20]. NiMoO₄ nanosheet arrays, formed on Ni foam by a hydrothermal process, exhibited a specific current density of 830 mA cm⁻² mg⁻¹ for urea oxidation reaction at a potential value of 500 mV (SCE) as 4.2 times higher than that at nickel hydroxide nanosheet arrays on Ni foam [21]. NiMn nanoparticles-decorated activated carbon containing 10 wt% Mn showed an onset potential value of -85 mV (Ag/AgCl) with urea oxidation current density value of 130 mA cm⁻² [22]. Ni-WC/MWCNTs electrocatalyst showed an increased urea oxidation current density value by over 3 and 15 times than those at Ni-WC/C and Ni/C, respectively [23]. NiO as a metal oxide is a promising candidate for electrocatalytic purposes due to its advantageous features including high

electrocatalytic efficiency, oxygen ion conductivity, high chemical stability, nontoxicity and high electron transfer properties [24,25]. Fe₂O₃/NiO microflowers, formed by facile cyanometallic framework-templated strategy, maintained high lithium storage capacity of 1652.6 mAh g⁻¹ at 100 mA g⁻¹ and good cycling stability of 1210.4 mAh g⁻¹ capacity retention at 50th cycle [26]. Wang et al. [27] have rationalized the excellent lithium storage performance of NiO nano octahedron aggregates to the synergistic effect of nano-sized and exposed (111) surface of NiO octahedron that improved the kinetics of charge transportation and Li ion diffusion. NiO nanoparticle-nanographitic flake composite was synthesized through a direct current electrophoretic deposition process. It showed high electrocatalytic performance for ethanol oxidation due to the selective deposition of low-weight NiO nanoparticles on nanographitic flakes, thus reducing the formation of agglomerated NiO nanocomposites [28]. Wang et al. [29] have studied the effect of nitrogen doping, calcination temperature and content of NiO nanoparticles in NiO/nitrogen-doped carbon nanotubes on their electrocatalytic activity for methanol oxidation in alkaline medium. Fast charge-transfer process, excellent accessibility and stability were achieved during alcohol oxidation reaction.

Nanoscience development has offered efficient challenges in the field of energy and environmental pollution [30–32]. The large surface area and controlled dimensional structures of nanocarbon supports such as carbon nanotubes and graphene sheets enable them to effectively catalyze the electro-oxidation of many compounds reducing their overpotential and increasing the corresponding current density value [33–35]. Graphite displayed an ordered structure as arrays with less lattice defects. Enhanced electrical conductivity with increased current output could be considered as advantageous characteristics of graphite when compared to amorphous carbon [36,37]. To convert amorphous carbon to graphite, an elevated temperature value is required [2200–3000 °C] that would consume an increased amount of energy and requires the application of a specific equipment. On the other hand, lower temperature values (<1000 °C) are needed to fabricate graphite that could save highly consumed energy and sophisticated instruments [38–40]. Graphite-based nanocomposites were applied in many fields including nanoelectronics, batteries and sensors. In this present work, NiO nanoparticles supported on graphite were fabricated in different metal oxide loading values in chemical precipitation process coupled with subsequent calcination at 400 °C. They are inexpensive materials and stable in alkaline solution with increased electrical conductivity. For the best of our knowledge, this is the first time that NiO/Gt nanocomposites were employed as electrocatalysts for urea oxidation reaction in alkaline solution. A systematic study was carried out including the effect of scan rate, urea concentration and NiO weight percentage value on the activity of formed electrocatalysts. Some kinetic parameters including Tafel plots and diffusion coefficient of urea molecules during the electro-oxidation reaction were calculated. NiO/Gt electrocatalyst demonstrated enhanced electrocatalytic activity, stability and tolerance during urea oxidation reaction. This could be attributed to the increased nickel oxide active sites on the special planar sheets of graphite support that could enable the

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