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RAPID COMMUNICATION

Preparation and application of iron oxide/graphene based composites for electrochemical energy storage and energy conversion devices: Current status and perspective

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Abstract
This review summarizes the research progresses in the preparation of graphene based iron oxide composites for electrochemical energy storage and conversion devices like lithium ion batteries, supercapacitors and fuel cells. Iron oxides (including Fe_3O_4, α-Fe_2O_3 and γ-Fe_2O_3) are promising materials for these electrochemical devices because of their low cost, nontoxicity, good chemical stability and high theoretical capacity. However, iron oxides suffer from aggregation after reaction, poor capacity retention and low electronic conductivity. The loading of iron oxide on graphene cannot only solve these problems but also minimize the aggregation or restacking of graphene or reduced graphene oxide and enhance the properties of graphene. The high surface area, high conductivity, excellent chemical and thermodynamic stability, unique lightweight characteristic and superior optical, thermal and mechanical properties make graphene exactly excellent for applications in those electrochemical energy storage and conversion devices. The preparation of graphene based iron oxide composite therefore becomes extremely important. Hydrothermal decomposition, thermal decomposition, chemical precipitation and co-precipitation are the most frequently applied methods, which can be used for a production with a large amount of composites. Hydrothermal and thermal decomposition make a better production with higher dispersion and easier quality control. Other preparation methods, like sol-gel method, atomic layer deposition, microwave heating, plasma decomposition, electrochemical approach and hydrolysis, have also been exploited for a further improvement in the size, structure and performance control or for a simple and easy fabrication with use of fewer chemicals. No matter which method is

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applied, the nucleation and crystal growth of iron oxide nanoparticles on the graphene substrate are the key influencing factors but still need to be investigated. The functional groups or structure defects in graphene or reduced graphene oxide play a very important role in the nucleation and crystal growth. Many opportunities and challenges exist with the design and controllable formation of these functional groups or structure defects.
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Introduction

A great effort has been made worldwide towards the development of electrochemical energy storage and conversion devices like lithium ion batteries, supercapacitors and fuel cells. Since its discovery in 2004 [1], the magic single atomic layer graphene has been rapidly exploited for the applications of electrochemical energy storage and conversion devices [2-8]. Graphene has a theoretical surface area of 2630 m^2/g, ~260 times higher than that of graphite and twice than that of carbon nanotubes [9]. It possesses zero energy gap between the highest occupied molecular orbit (HOMO) and the lowest unoccupied molecular orbit (LUMO) with fast electron transport property [10]. The high surface area, high conductivity, excellent chemical and thermodynamic stability, unique lightweight characteristic and superior optical, thermal and mechanical properties make graphene excellent for many promising applications including applications in the electrochemical energy storage and conversion devices [11,12].

On the other hand, iron oxides (including Fe_3O_4, α-Fe_2O_3 and γ-Fe_2O_3) are promising materials too for electrochemical energy storage and conversion devices because of their low cost, nontoxicity, good chemical stability and high theoretical capacity. However, iron oxides suffer from aggregation after reaction, poor capacity retention and low electronic conductivity, which hamper their practical applications. The loading of iron oxide on graphene cannot only solve these problems but also enhance or improve the properties of graphene. The formed iron oxide nanoparticles minimize the aggregation or restacking of graphene or reduced graphene oxide (rGO). The loading and dispersion of iron oxide on graphene or the preparation of graphene based iron oxide composite therefore become extremely important.

Figure 1 shows schematically representatives of graphene. Obviously, the prefect graphene is chemically inert for the loading or nucleation of iron oxide nanoparticles (and other nanoparticles). It is seldom used directly as a starting material due to its poor dispersibility in most solvents resulting from its fewer surface functional groups [13]. Fortunately, graphene oxide (GO), a nice precursor of graphene, possesses abundant oxygen-containing groups that can serve as the sites to bind with metal or metal oxide. Exactly, for those preparations directly using graphene, some trace amount of oxygen-containing groups on graphene plays an important role (together with defects). GO is a two-dimensional material with graphene structure but with oxygen-containing groups like epoxy (C-O-C), hydroxyl (OH) and carboxyl (COOH) on both basal planes and edges. GO in bulk form is also named as graphite oxide, reported firstly by Brodie in 1855 [14]. GO has been considered to be a convenient and flexible precursor for the preparation of graphene and graphene based materials. GO can even serve as an oxide to oxidize Fe^{2+} in $FeCl_2$ or $FeSO_4$, leading to an *in situ* deposition of Fe_3O_4 nanoparticles onto the self-reduced rGO sheets [13,15]. Figure 2 shows schematically representatives of GO and rGO. The latter is also frequently applied for the composite substrate.

GO has been employed as the precursor for synthesis or preparation of graphene based iron oxide composites. During the synthesis, the positive charged Fe ions interact with the functional groups of GO. The attachment of Fe ions to the surface and edges of GO (via chemisorption, physisorption, electrostatic interaction, van der Waals or covalent bonding) [21] leads to the nucleation sites. The further formation of iron oxide composite can be performed with various methods like hydrothermal approaches, thermal decomposition, microwave heating and plasma decomposition.

In this review, we summarize the recent progresses in syntheses of iron oxide/graphene (or GO) composites for applications in electrochemical energy storage and conversion devices (lithium ion batteries, supercapacitors and fuel cells). For those applications with catalyses (photo-assisted water splitting and Fischer-Tropsch synthesis [22,23]), environmental science (detection and degradation of hazardous materials, magnetic separation of pollutants and microwave absorption) [24-29], biomedicine (targeted drug delivery, bio-sensor, magnetic resonance imaging, photothermal therapy, enzyme immobilization and protein adsorption) [30-38], magnetic-controlled switches [39] and others [40,41], the synthetic methods of the composites are the same. The present discussions will be useful for these applications too.

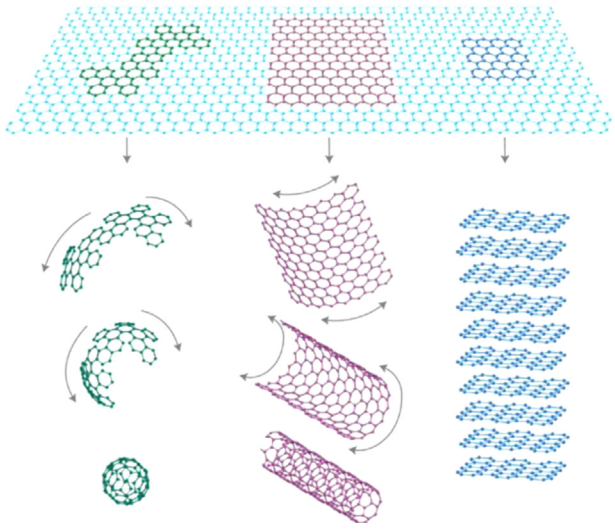

Figure 1 Graphene is a 2D building material for carbon materials of all other dimensionalities. It can be wrapped up into 0D buckyballs, rolled into 1D nanotubes or stacked into 3D graphite. Reproduced with permission from Ref. [16] Copyright 2014, Springer.

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