

Graphite Intercalation Compounds as Precursors of Graphene Nanoplatelets

Alexander Melezhyk, Evgeny Galunin, Nariman Memetov, Alexey Tkachev

Tambov State Technical University

106, Sovetskaya St., Tambov, 392000, Russia

melezhyk2013@yandex.ru; evgeny.galunin@gmail.com; mnr979@gmail.com; nanotam@yandex.ru

Extended Abstract

Exfoliation of graphite materials (especially, surfactant-assisted ultrasonic exfoliation of well-ordered crystalline graphite in water, organic solvents or supercritical liquids) is one of the most promising routes for the mass production of graphene nanoplatelets (GNPs) (Soldano et al., 2010). However, direct exfoliation of pristine graphite is a slow process, and only a small fraction of original graphite is transformed into few-layer GNPs. In this connection, among approaches proposed to improve the efficiency of the graphite exfoliation into GNPs, those ones based on using intercalated graphite (e.g., different forms of thermally expanded graphite synthesized through thermal decomposition of intercalation compounds, potassium-intercalated graphite, oleum-treated graphite, and electrochemically intercalated graphite) appear to be more effective and successful for fabricating GNPs (Inagaki et al., 2014). Previously, we have proposed a new method for the GNPs production based on cold expansion of peroxodisulfuric acid-intercalated graphite, which consists in spontaneous pre-exfoliation with the formation of an expanded graphite intercalation compound (EGIC) having an apparent volume of 300-320 cm³g⁻¹ of the starting material (Melezhyk and Tkachev, 2014).

Considering the aforementioned, the present work aims to compare the exfoliation ability of different graphite materials and study the properties of GNPs obtained dependently on intercalation/deintercalation conditions and reagents. It was found that among those graphite materials, the EGIC possesses maximal ability for the ultrasonic exfoliation. The exfoliation efficiency strongly depends on the content of water in sulfuric acid and is maximal for the EGIC prepared in a sulphuric acid medium containing 1-2% of free SO₃. This may be explained by increased acidity of the medium and, correspondingly, redox potential of the peroxosulfate compounds. This is also related to increased amounts of oxygen groups in the GNPs obtained from the EGIC synthesized in 100% sulfuric acid and diluted oleum.

To deintercalate the EGIC, we used various reagents and found that the nature of surface groups on the GNPs strongly depends on the nature of a deintercalating reagent. Thus, the treatment of the EGIC with different nucleophilic molecules, such as water, ammonia, carbamide, hexamethylenetetramine, organic amines, etc., can yield GNPs with various surface groups. Our experiments showed that the interaction between the EGIC and nucleophilic molecules does not only include the substitution of sulphate groups, but also redox reactions with participation of graphene layers. Depending on the nature of the nucleophile used, those reactions can lead to the formation of different groups (in case of water - oxide groups) attached to the graphene surface. When using ammonia, GNPs with almost pure surface were obtained.

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