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Synthesis Dynamics of Graphite Oxide

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Highlights

- The graphite oxide synthesis dynamics was investigated by sampling technique.
- The role of H_2O_2 in the increase of GO oxidation degree was found.
- The additions of ice and H_2O_2 induce the formation of surface functional groups.
- The formation of the graphite oxide phase begins after the addition of $KMnO_4$.

Abstract

Graphite oxide synthesis dynamics were investigated using a sampling technique. The synthesis of graphite oxide was carried out by a modified Hummers' method. Small samples of the solid phase (30–50 mg) were collected from the reaction mixture and analyzed by thermogravimetric analysis, differential scanning calorimetry, scanning electron microscopy, X-ray diffraction, Raman spectroscopy, energy dissipative X-ray spectroscopy, and X-ray photoelectron spectroscopy. The strongest oxidation was detected 10 min after the start of the synthesis, i.e., after the addition of $KMnO_4$, when the formation of the graphite oxide phase with intercalated guest molecules begins. The intercalation of graphite started after 30 min of synthesis when the temperature was increased to 35°C. The addition of ice into the reaction mixture leads to the increase in the COOH group concentration, whereas the concentration of C=O groups slightly changes,