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Development of expanded graphite from vein graphite via electrochemical exfoliation with sodium sulfate as an electrolyte

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Vein graphite is a promising anode material for rechargeable lithium-ion batteries. Since lithium ions are scarce and expensive, research and development are focused on sodium and potassium rechargeable batteries. However, graphite structure should be modified through expanding the interlayer spacing to facilitate intercalation/de-intercalation of the bigger ions related to these future rechargeable batteries. Among the various methods, the electrochemical exfoliation process has been identified as a promising method to structural modification of the graphite to produce expanded and exfoliated graphite. Electrochemical exfoliation can be performed at room temperature within a shorter period with better efficiency. Hence, it is a more cost-effective and environment-friendly method that consumes less energy compared to mechanical and thermal exfoliation methods. However, detailed information on the investigations of electrochemical exfoliation of vein graphite are limited. Therefore, this study aims to investigate the possibility of producing expanded graphite from Sri Lankan vein graphite using electrochemical exfoliation. Electrochemical exfoliation of graphite rod (1 cm x 10 cm), cut from the vein graphite was carried out using 1 mol dm⁻³ Na_2SO_4 as an electrolyte and a Pt rod as a reference electrode under 10V DC voltage, for 30 minutes. The developed materials were characterized by X-ray diffractometer (Rigaku Ultima IV, Cu K_{α} radiation), Raman spectroscopy (Renishaw Invia, 514 nm laser), particle size analyzer (Horiba Nanopartica SZ-100), and Fouriertransform infrared spectroscopy (Thermoscientific Nicolet is 50, KBr pellet method). Crystallographic characterization using X-ray diffractometry revealed that the interlayer spacing of graphite had increased from 0.33859 nm to 0.33986 nm after the electrochemical exfoliation process. The ratio of the intensity of the D peak and G peak (I_D/I_G) of Raman spectroscopy was used to estimate the average defect density on the graphite surface after the electrochemical exfoliation. I_D/I_G of the edge plane of the graphite increased from 0.48 to 1.27 after the exfoliation. Similarly, the I_D/I_G of the basal plane of the graphite increased from 0.17 to 0.96. This reveals that the average defect density on the graphite edge and basal surface increased after the electrochemical exfoliation. Particle size analysis of expanded graphite was calculated by using the laser diffraction method. A median particle size of 1139.4 nm and polydispersity index value of 0.941 were reported for the exfoliated sample. Fouriertransform infrared spectroscopy analysis confirmed the oxidation of the graphite due to electrochemical exfoliation. Therefore, this study reveals the potential of producing expanded graphite by electrochemical exfoliation of vein graphite using Na₂SO₄ as an electrolyte. Further, material characterization and optimization of parameters such as electrolyte concentration and DC voltage, are currently undergoing to obtain expanded graphite for the investigations in intended rechargeable battery applications.

Keywords: Vein graphite, Electrochemical exfoliation, Expanded graphite, Sodium sulfate, Rechargeable batteries

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