

TERNARY INTERCALATION COMPOUNDS IN THE GRAPHITE – H₂SO₄ – C₂H₅COOH SYSTEM

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Introduction

Graphite intercalation compounds (GIC) with sulfuric acid are called graphite bisulfate (GB). They are used as a raw material in production of exfoliated graphite – material with low density. Modification of intercalated layer and use of ternary GIC (TGIC) allows directional variation of exfoliated graphite's properties. The purposes of the present work are synthesis and investigation of compounds, obtained in the graphite – H₂SO₄ – C₂H₅COOH system.

Results and discussion

Synthesis of ternary GIC, contained both molecules of sulfuric and propionic acids, was carried out by anodic oxidation of HOPG in complex electrolyte H₂SO₄ – C₂H₅COOH. The general results are listed in Table 1.

Table 1. Results of electrochemical synthesis in the graphite – H₂SO₄ – C₂H₅COOH system.

N*	C _{H2} SO ₄ , %	Q, A·s/g	E _{Hg/Hg2SO4} , V	Composition
I	70	350	1.49	C ₈ (H ₂ SO ₄) _{0.77} (C ₂ H ₅ COOH) _{0.23}
		700	1.49	C _{7.6} H ₂ SO ₄
II	30 – 70	170	1.15 – 1.69	C ₁₅ (H ₂ SO ₄) _{0.67} (C ₂ H ₅ COOH) _{0.33}
		800	1.69	C ₁₄ H ₂ SO ₄
III	> 10	120	1.18	C ₂₃ (H ₂ SO ₄) _{0.60} (C ₂ H ₅ COOH) _{0.40}

N* - stage number

Increasing of GIC's stage number, smoothing of potential curves and their displacement to higher potential area are observed via decreasing of H₂SO₄ concentration. Diluting of sulfuric acid in the electrolyte leads to necessity of harder conditions to obtain the same stage.

The formation of I, II, and III stages TGIC is observed when Q is equal to 350, 170 and 120 A·s/g accordingly. Further polarization leads to replacement of C₂H₅COOH by H₂SO₄

molecules, which results GB formation (Table 1). Ternary intercalation compounds are characterized by lower overweight, sulfur content and thickness of intercalated layer (d_i = 7.94 Å) than for graphite bisulfate [1].

Simultaneous thermal analysis (DSC and TG), coupled with analysis of evolved gases by IR – spectroscopy, shown that decomposition of TGIC is endothermal process, which takes place in two steps and is accompanied by mass loss. Deintercalation of propionic acid starts at 120 °C, it was confirmed by data of IR – spectroscopy (Fig.1). The full acid deintercalation ends at 350 °C and the final product is defected graphite (d₀ = 3.38Å).

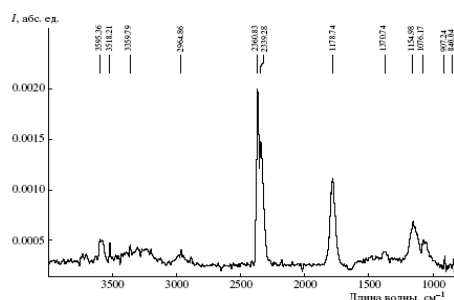


Fig.1. IR – spectrum of gaseous phase at 120 °C.

Conclusions

Application of ternary GIC – H₂SO₄ – C₂H₅COOH as a raw material to produce exfoliated graphite allows decreasing onset exfoliation temperature down to 120 °C and obtaining material with large expansion degree.

References

1. Leshin V.S., Sorokina N.E., Avdeev V.V. Graphite intercalation in H₂SO₄ – CH₃COOH electrolytes. Inorganic Materials 2003;39(8):826-832.