

SYNTHESIS AND CHARACTERIZATION OF GRAPHITE BISULFATE



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INTRODUCTION

Graphite and various graphite compounds are widely used in the fields of science, technology, and industry. Particular attention is paid to graphite intercalation compounds (GICs) that are formed by the insertion of different atomic, molecular or ionic species between the layers in a graphite material [1]. These compounds have found wide application as anode materials, heterogeneous catalysts, in production of superconductors, and in pyrophoric stabilization of reactants. Currently, GICs are also used in the preparation of graphite nanoplatelets, expandable graphite, and, most importantly, single-layer graphene [2, 3]. Graphite bisulfate is a GIC with sulfate ions interposed between the graphite layers. This intercalate expands strongly when heated, which weakens the π - π interaction and then graphite layers can be easily separated. Depending on the degree of intercalation, graphite nanocrystals of different thicknesses can be obtained. To obtain a monolayer of graphene, the highest possible level of intercalation is required, which depends on the type of oxidant and the experimental conditions [3].

OBJECTIVE

The aim of this work was to investigate and compare graphite bisulfate compounds obtained by using different oxidizing agents.

EXPERIMENTAL

For the graphite bisulfate (GBS) synthesis 1.6 g of oxidizer ($(\text{NH}_4)_2\text{S}_2\text{O}_8$, $\text{K}_2\text{S}_2\text{O}_8$, CrO_3) was firstly dissolved in 10 ml of H_2SO_4 (98 %). After 5 min 0.25 g graphite was added. The flask was sealed, and the solution was mixed for 2 h. Synthesis were repeated with the addition of P_2O_5 as a water-binding agent.

All obtained products were placed in ceramic vessels, which were inserted in a quartz glass tube. The tube was sealed and placed in an oven. Samples were heated at 800 °C for 15 min in argon atmosphere. Annealed samples were stored in a desiccator.

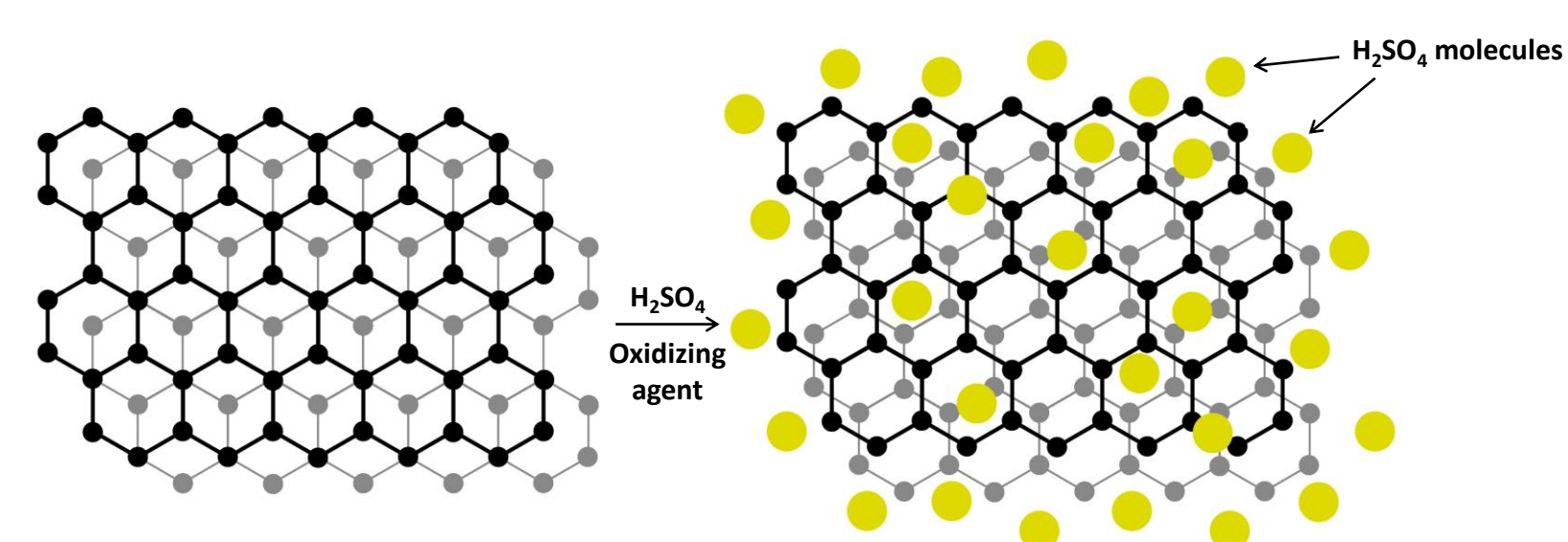
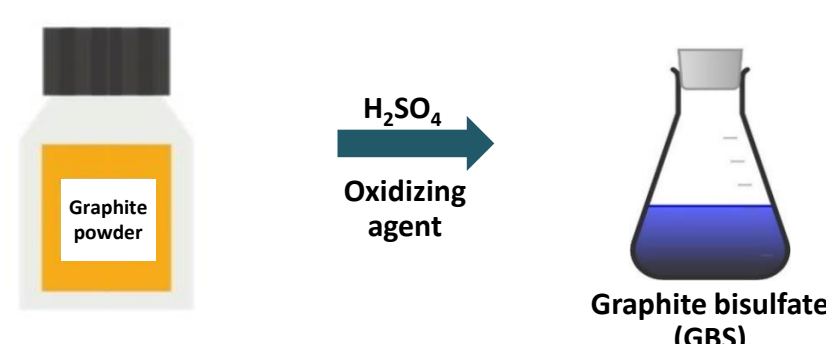


Fig. 1. Schematic illustration of graphite bisulfate (GBS) synthesis [4]

RESULTS

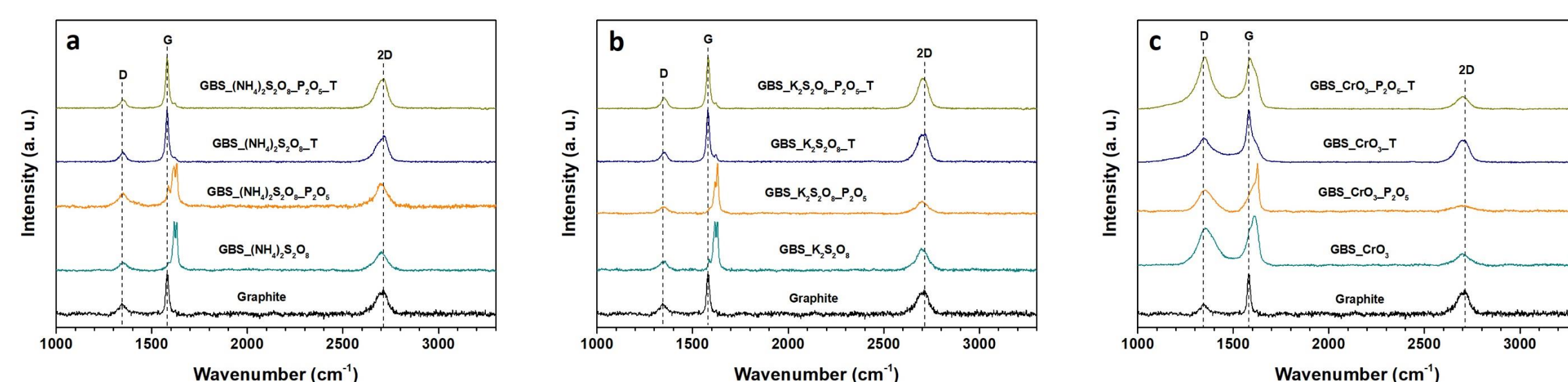


Fig. 2. Raman spectra of graphite and GBS ($(\text{NH}_4)_2\text{S}_2\text{O}_8$) products (a), graphite and GBS ($\text{K}_2\text{S}_2\text{O}_8$) products (b), graphite and GBS (CrO_3) products (c)

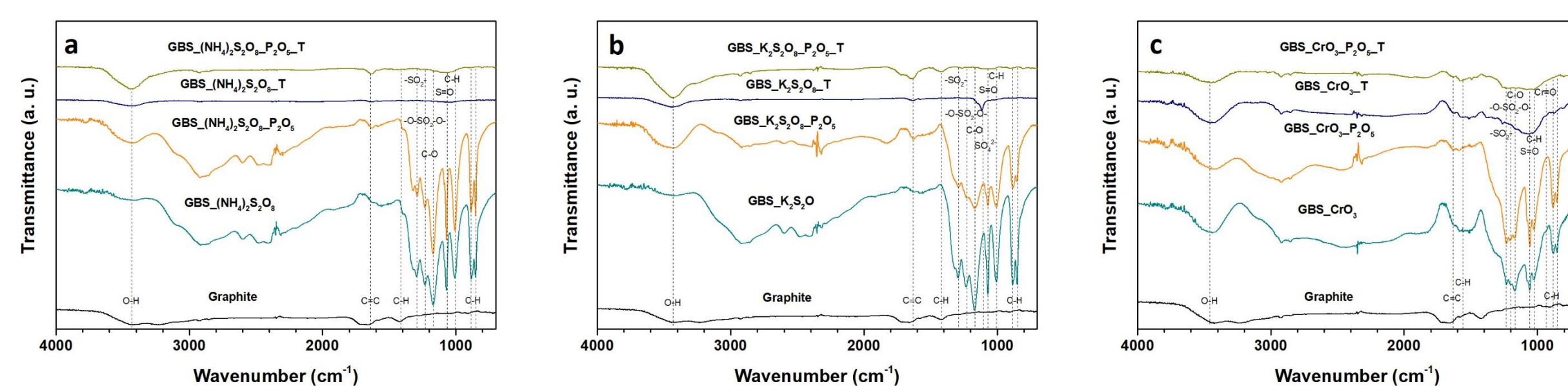


Fig. 4. FTIR spectra of graphite and GBS ($(\text{NH}_4)_2\text{S}_2\text{O}_8$) products (a), graphite and GBS ($\text{K}_2\text{S}_2\text{O}_8$) products (b), graphite and GBS (CrO_3) products (c)

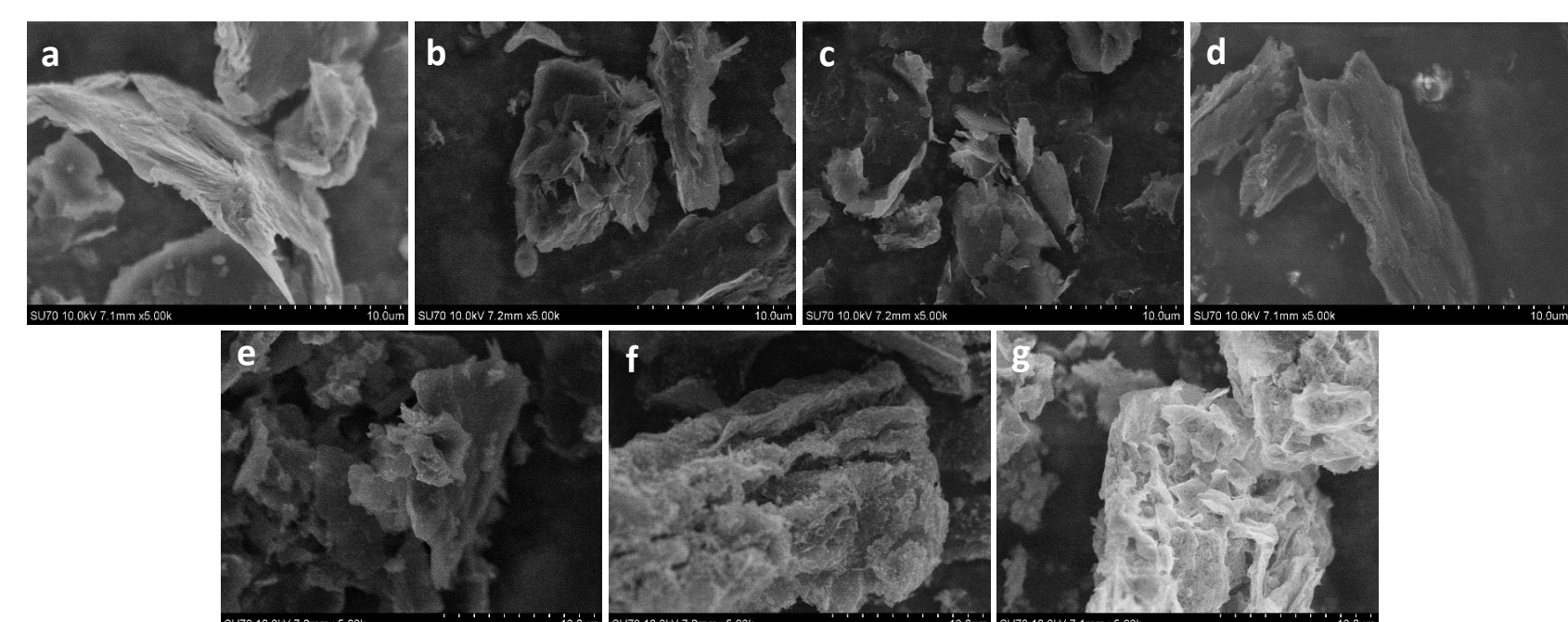


Fig. 3. SEM pictures of graphite (a), GBS_ $(\text{NH}_4)_2\text{S}_2\text{O}_8$ -T (b), GBS_ $(\text{NH}_4)_2\text{S}_2\text{O}_8$ - P_2O_5 -T (c), GBS_ $\text{K}_2\text{S}_2\text{O}_8$ -T (d), GBS_ $\text{K}_2\text{S}_2\text{O}_8$ - P_2O_5 -T (e), GBS_ CrO_3 -T (f), GBS_ CrO_3 - P_2O_5 -T (g)

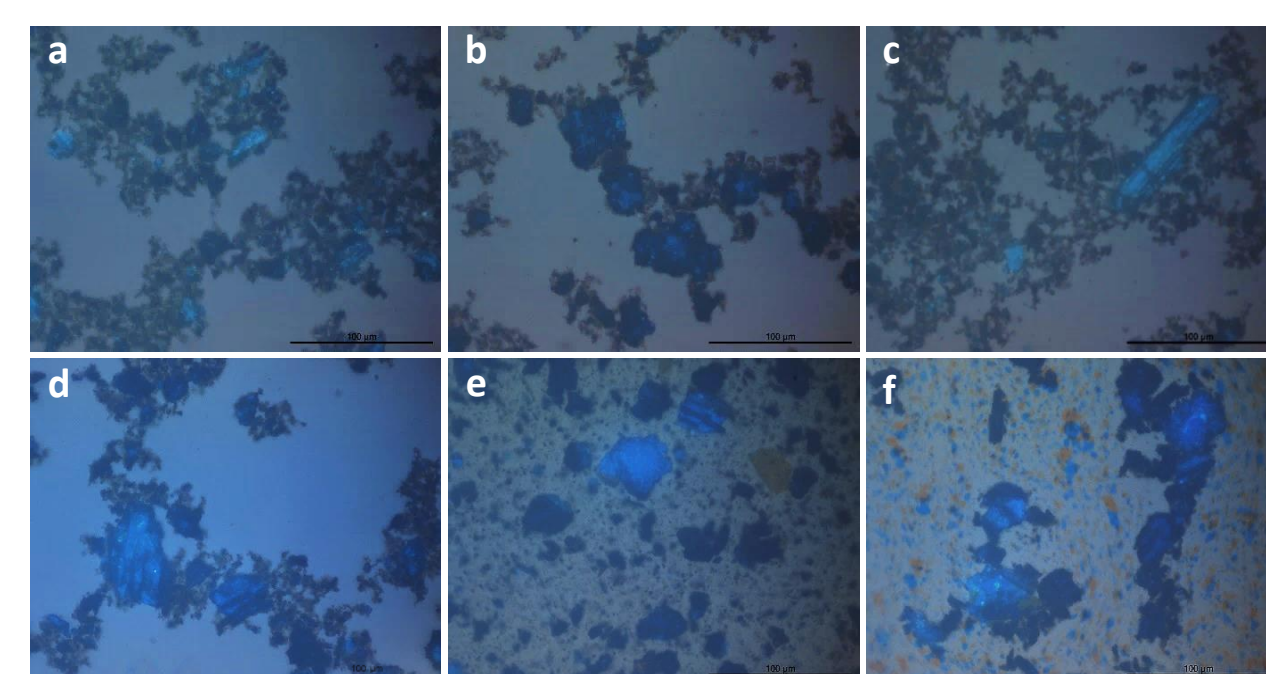


Fig. 5. Optical microscopy pictures of GBS_ $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (a), GBS_ $(\text{NH}_4)_2\text{S}_2\text{O}_8$ - P_2O_5 (b), GBS_ $\text{K}_2\text{S}_2\text{O}_8$ (c), GBS_ $\text{K}_2\text{S}_2\text{O}_8$ - P_2O_5 (d), GBS_ CrO_3 (e), GBS_ CrO_3 - P_2O_5 (f)

CONCLUSIONS

- Raman spectroscopy showed that higher stage of intercalation is obtained using $(\text{NH}_4)_2\text{S}_2\text{O}_8$ and $\text{K}_2\text{S}_2\text{O}_8$ as oxidizing agent. Using CrO_3 as an oxidizing agent gives lower stage of intercalation and more defective structure. Analysis also revealed that the structure of the compounds obtained by heating the samples at 800 °C coincides with that of graphite.
- FTIR analysis revealed that the functional groups of the GBS products correspond to the functional groups of the reagents used. During heating, most of the functional groups were removed, but intercalated S=O groups remained.
- SEM pictures showed that the largest surface changes compared to graphite are characteristic to GBS (CrO_3) samples.
- Photographs taken with an optical microscope show blue GBS particles, what confirms that a graphite intercalation compound was synthesized.

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