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Preparation of Expanded Graphite Using a Thermal Method

Nyssanbayeva Gulnura^{1,2,*}, Kudaibergenov Kenes^{1,2}, Ongarbayev Yerdos¹,

Mansurov Zulkhair² and Roberto Di Capua³

¹Al-Farabi Kazakh National University, Almaty, Kazakhstan

²Institute of Combustion Problems, Almaty, Kazakhstan

³University of Naples “Federico II”, Italy

*E-mail: gulnur.83.29@mail.ru

Abstract. This article reports a new way to prepare expanded graphite using a thermal method. Natural graphite was added to crystalline hydrate of metals with mechanical stirring at room temperature, taken in the number of 20-80% of the mass of mix. This was then placed in muffle furnace. All process of activation takes from 10 to 20 minutes. X-ray diffraction patterns were used to analyze the structure and confirm that expanded graphite had indeed been prepared. A scanning electron microscope was utilized to observe the morphologies of the expandable graphite and expanded graphite.

1. Introduction

The liquidation of oil spills on water surfaces aims to reduce the damage to the ecological and socio-economical resources, while reducing the time required for recovery of these resources and providing acceptable standards of cleanup [1]. Different methods can be used for the removal of petroleum from the water surface, for example, thermal, biological, mechanical and physicochemical (using coagulants and adsorbent materials) techniques [2].

Expandable graphite (EG) is a type of graphite intercalation compound (GIC). Its derivatives are functional carbon materials that can be applied in various fields, such as airtight materials, oil absorbents, flame retardants, high-power batteries, electrodes, and military materials [3-6].

V. Sridhar [7] reported that GICs can be obtained only by one-step at room temperature with intercalation by concentrated sulfuric acid and ammonium persulfate. The resultant EG (denoted as RTEG) exhibits an expanded volume (EV) of up to 225 ml/g. Finally, the excessive usage of concentrated H₂SO₄ as the intercalant in the conventional method can also cause serious environmental pollution [8]. Therefore, it is of great value to seek a simpler and less-polluted approach to prepare EG.

2. Experimental part

In this work are used native graphite and crystalline hydrates of zinc. The graphite from the Zavalye Graphite Plant (Ukraine) is a large-scaly natural graphite subjected to chemical desalination under industrial conditions. The result is achieved by mechanical mixing of powder of initial graphite by the making foam agent by crystalline hydrate of zinc for training of porous structure, taken in the number of 20-80% of the mass of mix. First, NG (2 g) was added to crystalline hydrate of zinc (8 g) with mechanical stirring for 1 min at room temperature. This was then placed in muffle furnace. The muffle furnace was sealed and quickly heated to the desired temperature. Where it was maintained for 20 min.



It was allowed to cool to room temperature naturally. The expanded volume was determined by expanding of the EG and then measuring its volume with a graduated cylinder. So, the experiment proceeds in two stages: 1) mixing of graphite with crystalline hydrate of zinc; 2) and heating of components at a temperature of 350-1000°C. The mix heats up 5-10 minutes. All process of activation takes from 10 to 20 minutes. Scheme of obtaining synthesis of thermally expanded graphite are given in figure 1.

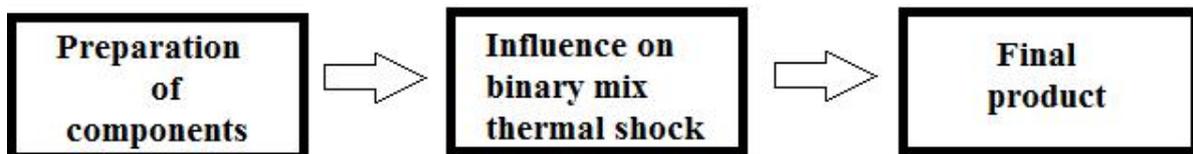


Figure 1. Scheme of the technology of synthesis of the interconnected graphite.

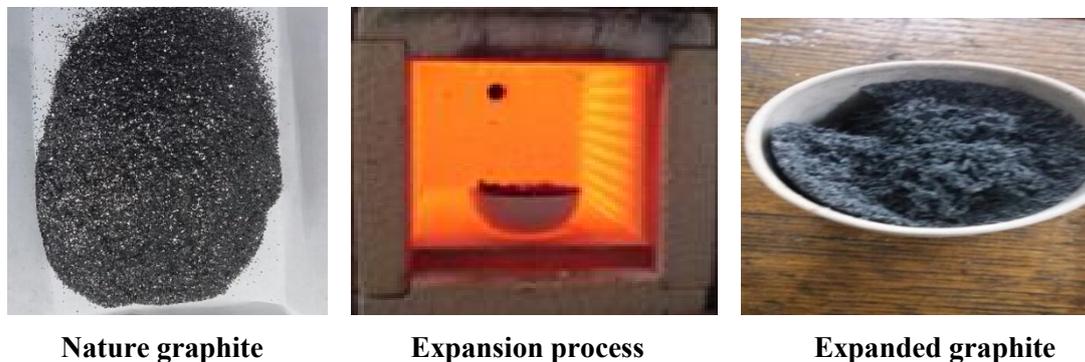


Figure 2. Schematically illustration of formation of EG and the synthesis.

In figure 2. shows, how the production of expanded graphite proceeds. On the picture 1 take mix of natural graphite and metal salts. And next picture show expansion process. It is proceeds in a muffle furnace. After 20 minutes we get the finished product. Finished product is expanded graphite.

3. Results and their discussion

The single most striking observation to emerge from the data comparison was formation of the homogeneous melted bubbling mass. After end of this stage there is a sharp foaming of graphite which is followed by allocation of insignificant amount of brown gas (figure 3).



Figure 3. a) mechanical mix before heat treatment b) obtained porous mix after heat treatment.

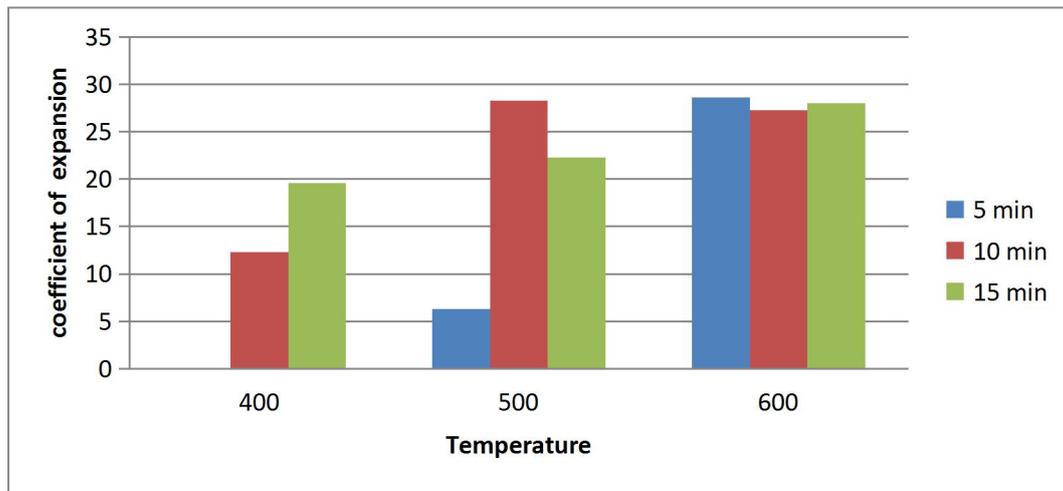


Figure 4. - diagram between coefficient of expansion (C_V), temperature (τ) and time interval.

Figure 4 shows the diagram between coefficient of expansion (C_V), temperature (τ) and time interval. It can be explained with the fact that increase in temperature causes thermal decomposition of crystalline hydrate in reagent structure, therefore, the relative content of oxygen increases. Qualitative analyses of reagent structures are submitted in the figure 4.

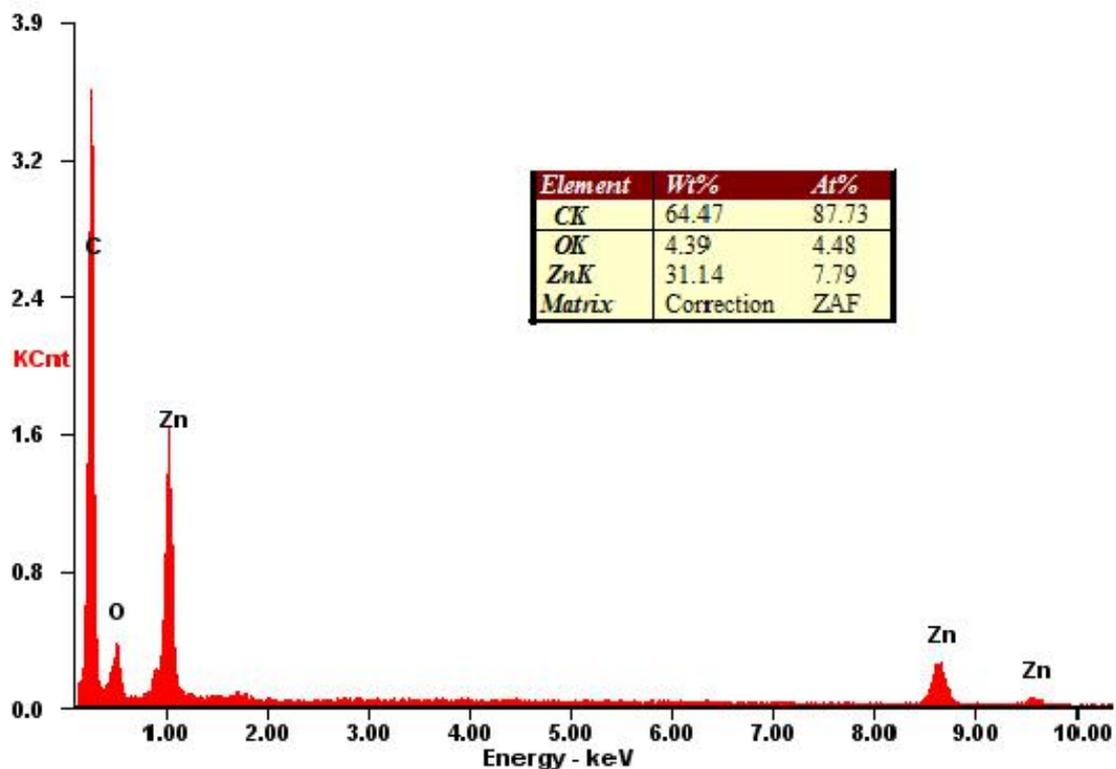


Figure 5. Microanalysis of expanded graphite.

Figures 5 shows the results of microanalyses of expanded graphite using SEM/EDAX. In figures 5 the variation of weight percent of three elements (C, O, Zn) in expanded graphite. Figure 5 also shows that the principal elements in expanded graphite is carbon.

Results microanalysis the thermally expanded graphite obtained from mechanical mix of graphite and crystalline hydrates of metals shown in figure 6.

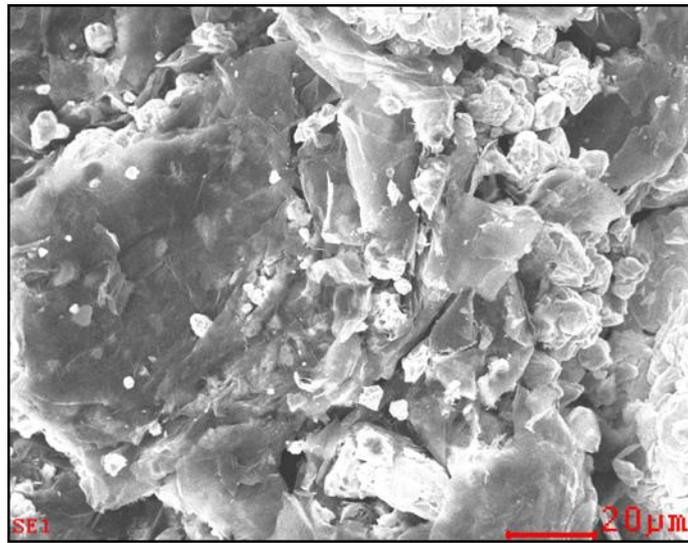


Figure 6. SEM micrograph of expanded graphite.

Figure 6 shows SEM images of the EG samples. The morphology of the EG samples is wormlike and there are a lot of pores that can also be observed on the surface. It is the particular loose and porous structures that would provide EG samples with good adsorption property for the macromolecular compounds.

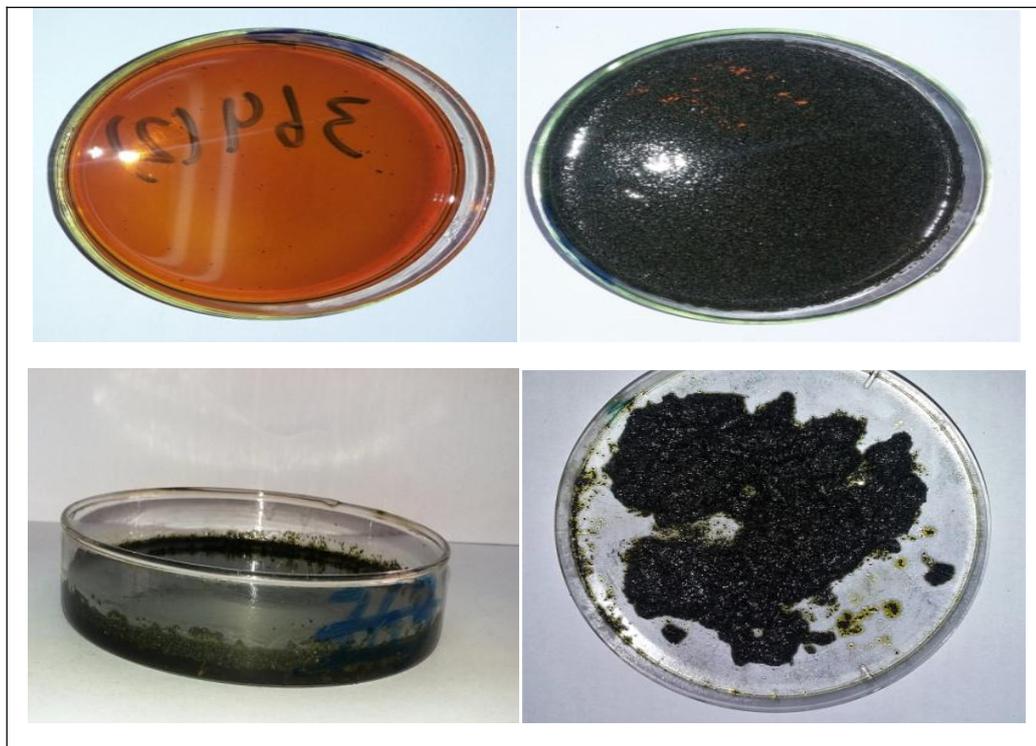


Figure 7. Oil Sorption capacity of exfoliated graphite.

Figure 7 shows the sorption capacity of expanded graphite. As can be seen from the presented data, the maximum sorption of oil is in the first minutes (~ 3-4 minutes).

4. Conclusions

In this work were to obtain expanded graphite by heat treatment. Morphology of expanded graphite was verified by scanning electron microscopy analysis. Expanded graphite shown outstanding adsorption performance for oil.

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