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Obtaining expanded graphite by heat treatment

One of the promising materials of the 21st century is expanded graphite or thermally expanded graphite. The preparation of nanostructure and thermally expanded graphite with improved specific surface, bulk density and expansion along the trigonal axis of the graphite matrix is associated with the thermal shock of intercalated graphite The general principle of these methods is the introduction into graphite of either gaseous substances or compounds that, during thermal heating of the intercalated compound of graphite or their derivatives, transform into a gaseous state and there by create an intralayer pressure that expands the graphite particle. In the given work thermally expanded graphite, obtained during the heat treatment crystalline hydrates of metals and nature graphite obtain expanded graphite material. The expanded graphite obtained was used as a sorbent. The performed studies in this work demonstrated the potential use of the sorbents as adsorbents for the removal of thin oil films. In this paper, it can be seen that the optimum mixture ratio of graphite to other contents (graphite : $Zn(NO_3)_2{\times}6H_2O$: graphite : $FeCl_3{\times}6H_2O$) is 2(g) : 8(g) : 2(g) : 8(g), which obtained the best expansion volume. Here, we propose a simple, effective method to prepare expanded graphite in which the intercalation and expansion of graphite are realized by only one step not involving any sophisticated devices.

Keywords: graphite, expanded graphite, intercalated graphite compound, thermally expanded graphite.

Introduction

In the recent decades expanded graphite (EG) has been one of the major interesting research subjects because of nanostructure. Obtained expanded graphite by heat thermal shock is a low-density carbon material, and it used as a base for sealing and fire-retardant materials. Expanded graphite is a promising material for high temperature. Physicochemical properties of expanded graphite depend from a graphite synthesis condition. While thermal characteristics of EG obtained by the thermal shock of convenient graphite intercalation compounds or their hydrolyzates are well studied [1, 2] the characteristics of EG obtained by thermal shock are scarcely investigated. The synthesis of EG describe through the heat treatment of oxidized graphite and the investigation of thermal properties. The general principle consists in introduction natural graphite of compounds of salts at thermal heat [3, 4].

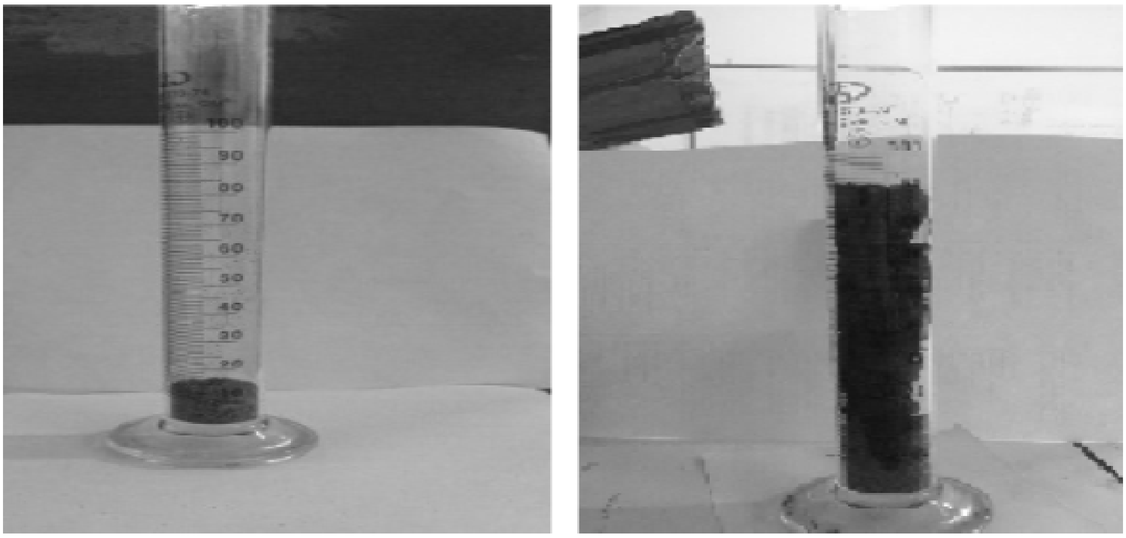
Experimental part

In this work were used native graphite and crystalline hydrates of metals. The graphite from the Zavalye Graphite Plant (Ukraine) is a large-scaly natural graphite subjected to chemical burning under industrial conditions. The result is achieved by mechanical mixing of powder of initial graphite by the making foam agent by salts of metals for training of porous structure, taken in the quantity of 20–80 % of the mass of mix. The experiment proceeds in two stages: 1) mixing of graphite with salts of metals; 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. In Figure 1 shows scheme of obtaining synthesis of thermally expanded graphite.

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

Results and their discussion

The single most striking observation to emerge from the comparison, it 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 (Fig. 2).



a) before heat treatment *b*) after heat treatment

Figure 2. Formation of the homogeneous melted bubbling mass

In Table shown main sorption properties of thermally expanded graphite.

Table

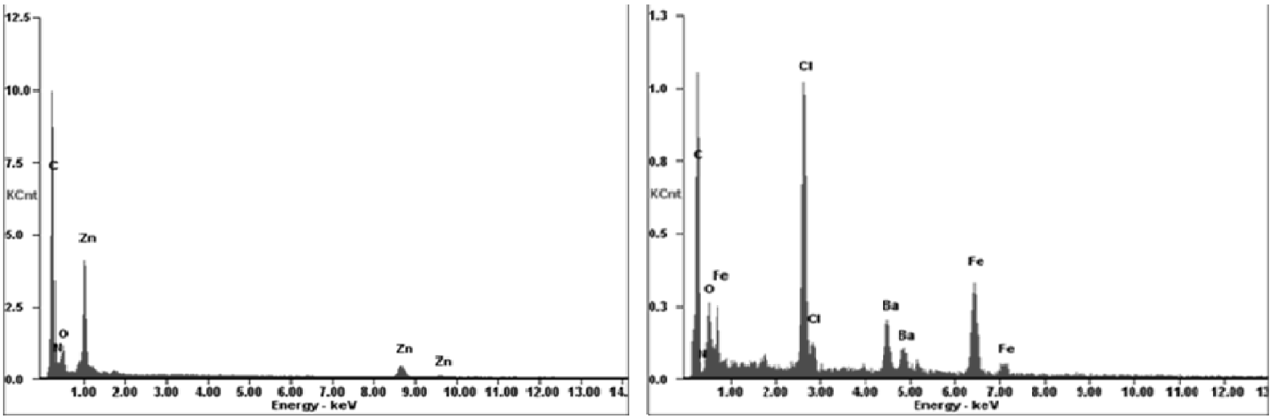
Main sorption properties of structure

System «graphite — salt»	Oil capacity, g/g	Water absorption, g/g	Buoyancy, %
Graphite — $FeCl_3 \times 6H_2O$	20	0,5	80
Graphite — $Zn(NO_3)_2 \times 6H_2O$	25	0,1	99

In Table shown that the structure «graphite — $Zn(NO_3)\ _2 \times 6H_2O$» differs in smaller water absorption, than «graphite — $FeCl_3 \times 6H_2O$». Results of an experiment shown that the reagent structure «graphite — $Zn(NO_3)\ _2 \times 6H_2O$» adsorbs 25 g of heavy oil.

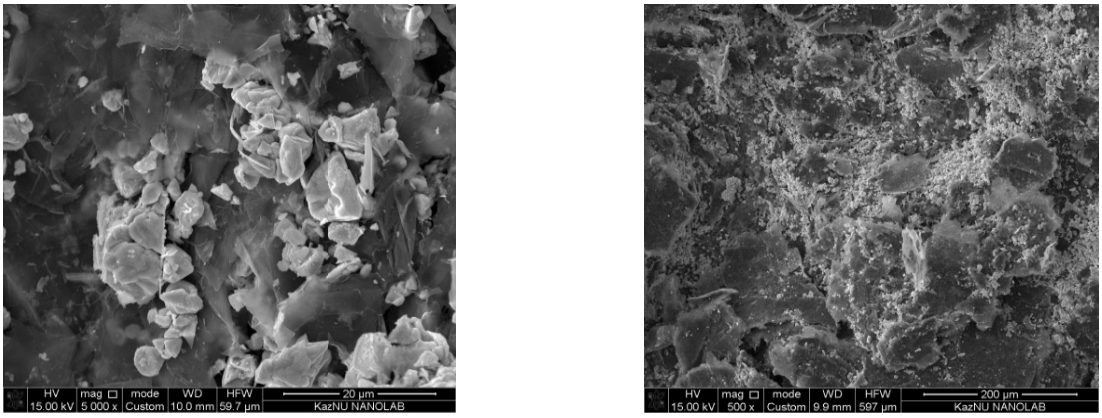
In details, the mass percent of oxygen in natural graphite increased from 5 to 10 %, the content of carbon decreased from 95 to 46 %. It can be explained with the fact that increased of the temperature causes thermal decomposition of crystalline hydrate in reagent structure, therefore, the relative content of oxygen increases. Qualitative analyses of reagent structures are submitted in the Figure 3.

Figure 4 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.

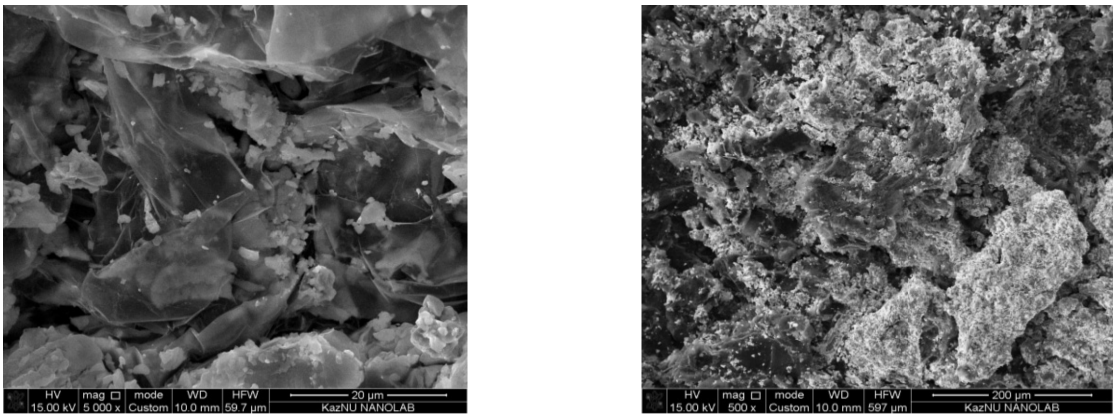


Graphite — $Zn(NO_3)_2 \times 6H_2O$ Graphite — $FeCl_3 \times 6H_2O$

Figure 3. Elemental composition of the thermally expanded graphite using SEM/EDAX



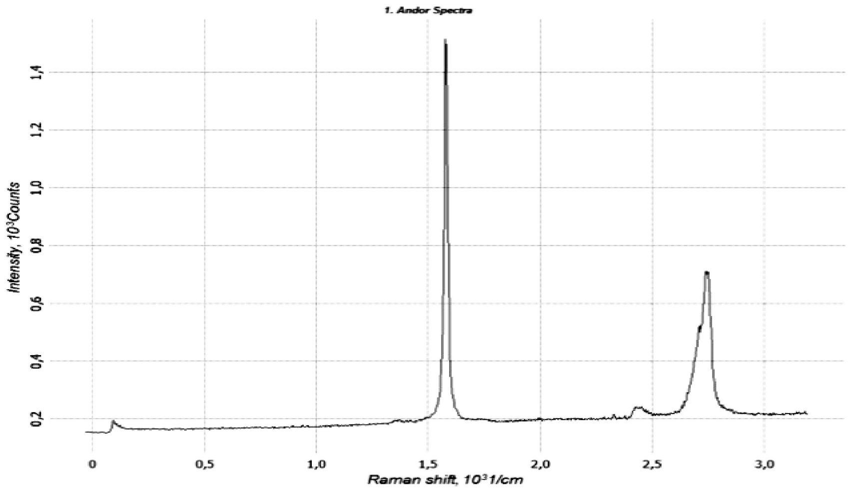
a) graphite — $Zn(NO_3)_2 \times 6H_2O$



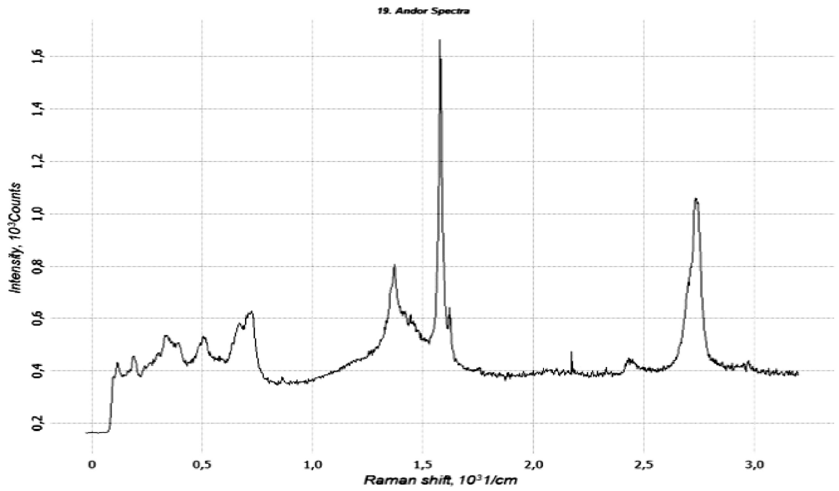
b) graphite — $FeCl_3 \times 6H_2O$

Figure 4. (*a*) and (*b*) show the surface morphology of the expanded graphite

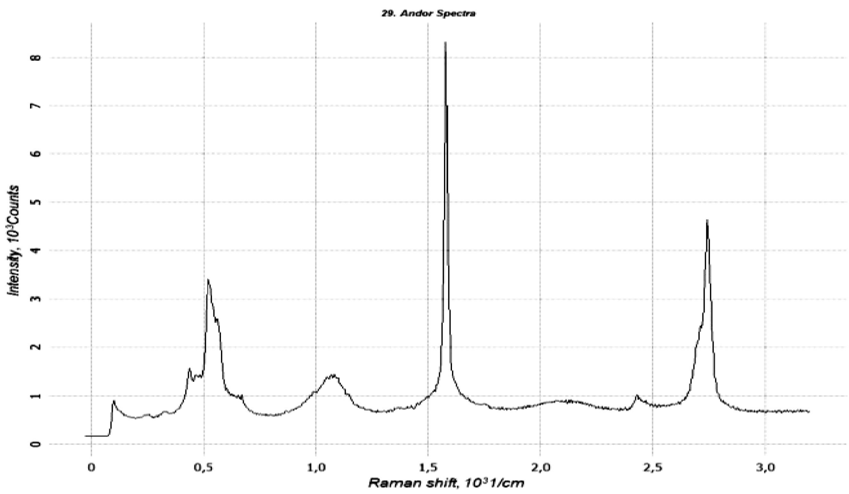
Produced samples were also investigated by Raman spectroscopy. As shown in Figure 5, the spectra that all samples have carbon structure.



a)



b)



c)

Figure 5. Raman spectra of natural graphite (*a*), graphite impregnated with salts (*b*, *c*)

In the Figure 6 it is shown oil sorption of TEG in laboratory conditions.

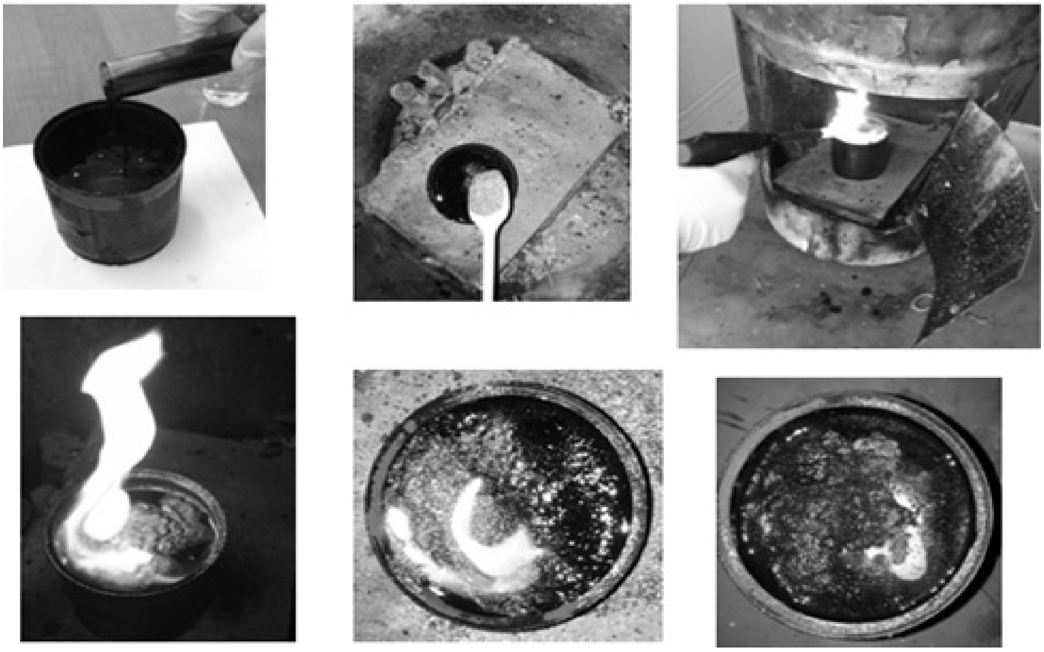


Figure 6. Oil sorption of TEG in laboratory conditions

At the beginning, on the surface of oil a small amount of flammable liquid is sprayed, then fire. During burning of oil we scatter a sorbent on the surface of the poured oil or it is possible to scatter before oil burning, and then to burn out oil. At the combustion of oil porous graphite is formed and in a few minutes there is a full adsorption of oil product.

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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Термиялық өңдеу жолымен кеңейтілген графит алу

XXI ғасырда болашағы бар материалдардың бірі кеңейтілген графит, немесе термиялық кеңейтілген графит, болып табылады. Жоғары көрсеткішті беттік ауданы, графит матрицасындағы тригоналды осіндегі кеңейтілген дәрежедегі және сусымалы тығыздығы жоғары наноқұрылымды термокеңейтілген графит алу, әдісінің жалпы қағидасы графитті термиялық қыздыру барысында газ тәрізді немесе қосылысты материалдарды кеңейтілген графиттің ішкі қабаттарына енгізу болып табылады. Мақалада металдың кристалдық гидраттарын және табиғи графитті термиялық өңдеу барысында термикалық кеңейтілген графит материалын алуға болатындығы айтылған. Алынған кеңейтілген графит сорбент ретінде пайдаланылды. Мұнда графиттің тұздармен қосылысы келесідей (графит : $Zn(NO_3)_2 \times 6H_2O$; графит : $FeCl_3 \times 6H_2O$) 2(г) : 8(г) , 2 (г) : 8 (г), осы қатынастар жақсы кеңейту көлемін алды. Біз кеңейтілген графитті алудың қандай да бір күрделі құрылғыларды қажет етпейтін, бір қадаммен жүзеге асырылатын, қарапайым әрі тиімді әдісін ұсындық.

Кілт сөздер: графит, кеңейтілген графит, интеркалирленген графит, термокеңейтілген графит.

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Получение расширенного графита путем термической обработки

Одним из перспективных материалов XXI века является пенографит, или терморасширенный графит. Общий принцип получения наноструктурированного терморасширенного графита с улучшенными показателями удельной поверхности, насыпной плотности и степени расширения вдоль тригональной оси графитовой матрицы термического удара интеркалированного графита заключается во внедрении в графит газообразных веществ или соединений, которые переходят в газообразное состояние при термическом нагревании интеркалированного графита или его производных и тем самым создают внутрислоевое давление, расширяющее графитовую частицу. В статье термически вспененный графит получен термообработкой кристаллогидратов металлов и природного графита. Полученный расширенный графит использовали в качестве сорбента. Исследования показали возможность потенциального использования сорбента для удаления тонких масляных пленок. Показано, что оптимальным соотношением компонентов, при котором получен наилучший объем расширения, является: графит : $Zn(NO_3)_2 \times 6H_2O$ = 2(г) : 8(г); графит : $FeCl_3 \times 6H_2O$ = 2(г) : 8(г). Авторами предложен простой и эффективный способ получения вспененного графита, при котором расширение графита реализуется в одну стадию.

Ключевые слова: графит, расширенный графит, интеркалированное соединение графита, терморасширенный графит.

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