International Journal of Mechanical and Production Engineering Research and Development (IJMPERD)
ISSN(P): 2249–6890; ISSN(E): 2249–8001
Vol. 10, Issue 1, Feb 2020, 305–314

SYNTHESIS OF MODIFIED NANOCARBON MATERIALS AND DETERMINATION OF THEIR ADSORPTION CAPACITY

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ABSTRACT

In this work, thermally expanded graphite and carbonized rice husk were obtained by heat treatment at different temperature conditions and different ratios. The samples were purified and characterized by physico-chemical methods. The synthesized samples were tested as oil sorbents in water. The results of determination of sorption capacity of thermally expanded graphite oil showed higher values (5-6%) compared to carbonized rice husks (3.1-3.7%). It should be noted that all samples of thermally expanded graphite have an adsorption capacity at least three times greater than that of natural graphite. The process of carbonization of rice husks was carried out at 600, 700, 800 and 900°C. It was found for samples of carbonized rice husks that the adsorption capacity was maintained in the range of 3.1-3.7% and no special changes were detected which depends upon activation temperature. The results of electron microscopic examination showed that the process of carbonization does not significantly change the macroscopic morphology of rice husks. In general, it is concluded that the carbonization process leads to an increase in the adsorption capacity of samples.

***KEYWORDS:** Oil Spills, Oily Wastewater, Carbonized Rice Husk, Thermally Expanded Graphite, Sorption Capacity & Oil Removal Efficiency*

Received: Oct 18, 2019; **Accepted:** Nov 08, 2019; **Published:** Jan 09, 2020; **Paper Id.:** IJMPERDFEB202026

Original Article

INTRODUCTION

The use of Oil determines the level of economic development and life of modern man. Absolutely, at all stages of oil use, starting from oil exploration and production and ending with the disposal of its waste, there is pollution of the environment, a negative impact on the people's health (Akhmedzhanov et al., 2012; Kamenev et al., 2003; Golubev et al., 2014; Kudaibergenov et al., 2014; Sassykova et al., 2019). Most of the pollution is due to oil spills, emissions of harmful substances into the atmosphere, water bodies and on land (Kudaibergenov et al., 2018; Kudaybergenov et al., 2010). It is important to emphasize that no stage of oil use is waste-free and the greater the amount of work performed, the more intense are formed at these stages of oil and gas flows, and the more significant their negative impact on the environment. Emergencies at the same time only strengthen and concentrate it influence. Impacts of the oil industry on the environment are manifested in the strengthening of the greenhouse effect, the appearance of acid rain, water quality decline, groundwater pollution, loss of biodiversity (Sassykova et al., 2019; Kudaibergenov et al., 2018; Kudaybergenov et al., 2010; Beisembayeva et al., 2017; Tashmukhambetova et al., 2018; Zhou et al., 2019; Ongarbayev et al., 2015). In recent years, serious concern has been caused by the pollution of the oceans by oil as a result of the crash of tankers and oil emissions in boreholes located in the high seas (Chen et al., 2012; Katsuki & Komarneni, 2009; Baiseitov et al., 2015; Tashmukhambetova et al., 2017; Liu & Xing, 2011). By estimates of experts from various sources as a result of activity of people from 5 to 10 million ton of oil get to the ocean. As 1 g of oil, spreading on the surface of the ocean, occupies the space of 12 km, the World Ocean probably is covered for a long time with a thin superficial film of hydrocarbons (Baiseitov et al., 2016). In recent years, due to the increase in oil production at offshore fields, the contribution to pollution of the World Ocean due to offshore drilling platforms has been increasing. The foregoing indicates that not only during accidents at oil facilities, but also during their normal operation, there are often large-scale spills of oil and oil products, which lead to large economic losses, environmental degradation, and, in some cases, environmental disasters (Ongarbayev et al., 2015; Baiseitov et al., 2016; Baiseitov et al., 2017; Sugashini & Begum, 2013; Sugashini & Begum, 2015; Wei et al., 2005).

A specific feature of oil pollution is the ability to capture and concentrate other contaminants, for example, heavy metals and pesticides. When the oil spreads over a large area, the probability of various reactions greatly increases, since substances soluble in oil participate in a variety of chemical processes (Ibrahim et al., 2010; Gabdrashova et al., 2018; Tuktin et al., 2019; Omarova et al., 2019; Kudaybergenov et al., 2012).

In light fractions of oil products of gasoline and kerosene, the surface tension at the boundary with water is higher and the spreading rate on the surface is lower than for petroleum products containing heavy fractions, such as fuel oil and oil. In this regard, petroleum products from light fractions (with the same amount) spread on a smaller surface area of the water (Rengasamy et al., 2011; Lin & Juang, 2009; Sassykova, 2017).

Removing oil and oil products from wastewater using solid sorbents is one of the possible options for water treatment (Angelova et al., 2011; Alvarez et al., 2014; Imagawa et al., 2000; Prabhahar et al., 2018; Kudaibergenov et al., 2014).

The aim of this study was synthesis of solid carbon sorbents by heat treatment of natural graphite and rice husk and their characterization. The obtained sorbents were used for the efficient sorption of oil and oil products. The novelty of this work is the use of heat treatment for the synthesis of carbon sorbents based on natural graphite from the Zavaliyevsky deposit (Ukraine) and rice husk.

EXPERIMENTAL

Natural flake graphite (NG) of Zavaliyevsky deposit (Ukraine), consisting of 92.8 wt% of carbon, was used in the work. Rice husk (RH) was extracted from Kyzylorda field (Kazakhstan). Thermally expanded graphite (TEG) was produced in the laboratory of the Al-Farabi University (Almaty, Kazakhstan) by heat treatment method. This is a very simple method (Ongarbayev et al., 2015; Baiseitov et al., 2016; Baiseitov et al., 2017; Kudaibergenov et al., 2018; Kudaybergenov et al., 2010) in which a mechanical mixture of natural graphite and crystalline zinc nitrate hydrate ($Zn(NO_3)_2 \times 6H_2O$) is heated in a muffle furnace for 20 minutes at different temperatures. The experimental conditions are shown in the table 1.

Table 1: Experimental Conditions for Expanded Graphite (EG) Synthesis

Samples Labels	Percentage of Components, wt. %		Final Temperature, °C
EG350 (80/20)	20.0% NG	80% $Zn(NO_3)_2$ x $6H_2O$	350
EG 400 (80/20)	20.0% NG	80% $Zn(NO_3)_2$ x $6H_2O$	400
EG 600 (80/20)	20.0% NG	80% $Zn(NO_3)_2$ x $6H_2O$	600
EG 800 (80/20)	20.0% NG	80% $Zn(NO_3)_2$ x $6H_2O$	800

Table 1: Contd.,

EG 500 (70/30)	30.0% NG	70% $Zn(NO_3)_2 \times 6H_2O$	500
EG 600 (70/30)	30.0% NG	70% $Zn(NO_3)_2 \times 6H_2O$	600
EG 1000 (80/20)	20.0% NG	80% $FeCl_3 \times 6H_2O$	1,000

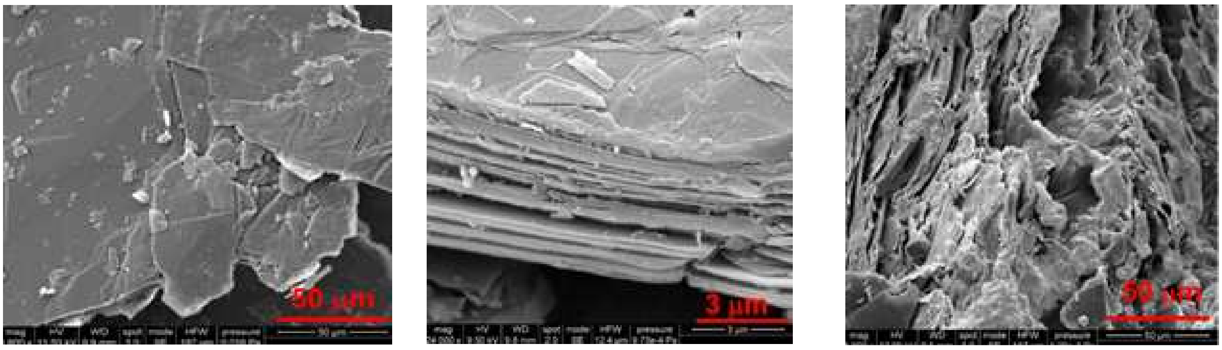
The rice husk carbonization has been performed at the Institute of Combustion Problems (Almaty, Kazakhstan). 15 g of RH were carbonized in a reactor designed in the laboratory under propane atmosphere at different temperatures for 1 hour (Table 2).

Table 2: Experimental Condition for Synthesis of Carbonized Rise Husk (CRH)

Samples Labels	Carbonization Temperature, °C	Carbonization Time, Hour
CRH 600	600	1.0
CRH 700	700	1.0
CRH 800	800	1.0
CRH 900	900	1.0

RESULTS AND DISCUSSIONS

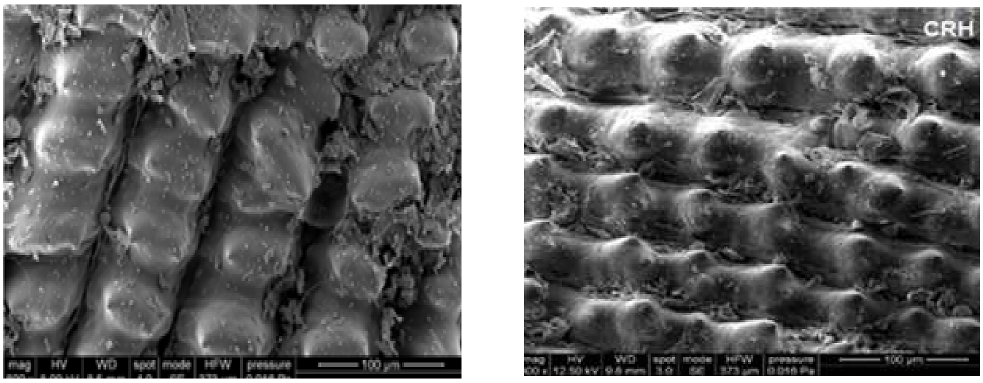
SEM-images (Figure 1) show a typical structure of the NG and TEG samples obtained at a temperature of 500ºC (TEG500 (70/30)). As a result of thermal expansion, graphite increases in volume due to intercalation of salts, which leads to the formation of multilayer thermally expanded graphite. As can be seen from the SEM-photo of natural graphite, the samples in the original form are very dense and do not contain pores on their surface. According to the data of electron microscopic investigation, the structure of the original zavalevsky graphite consists of thin plates with transverse dimensions of about 0.51 microns, a thickness of several to tens of nm. The plates are superimposed on each other and form crystalline polytypes due to errors in their crystallographic superimposition. The SEM images of the TEG obtained from a mechanical mixture of graphite and salt (Figure 1c) show areas of significantly deformed layers, as well as areas with a relatively perfect structure of graphite layers. Violations of the near and far order associated with this phenomenon, and cause just defective Raman scattering of light.



a b c

Figure 1: SEM Images at various Magnifications of Natural Graphite (a, b) and Expanded Graphite (EG) obtained at the Temperature of 500°C (c).

SEM images of raw rice husk and carbonized rice husk at 600°C (CRH600) are shown in the figure 2. It can be seen that the carbonization process does not significantly change the macroscopic morphology of rice husk.



a b

Figure 2: SEM-Images of Rice Husk (RH) (a) and Carbonized Rice Husk (CRH600) (b).

Results of the elemental analysis demonstrate that as expected the carbon content decreases in all the samples of thermally expanded graphite while that of other components increases (Figure 3). The content of carbon, hydrogen and other components (expressed as the sum of nitrogen, oxygen and inorganic substances) in all samples of carbonized rice husk is shown along with samples of the original rice husk in figure 4.

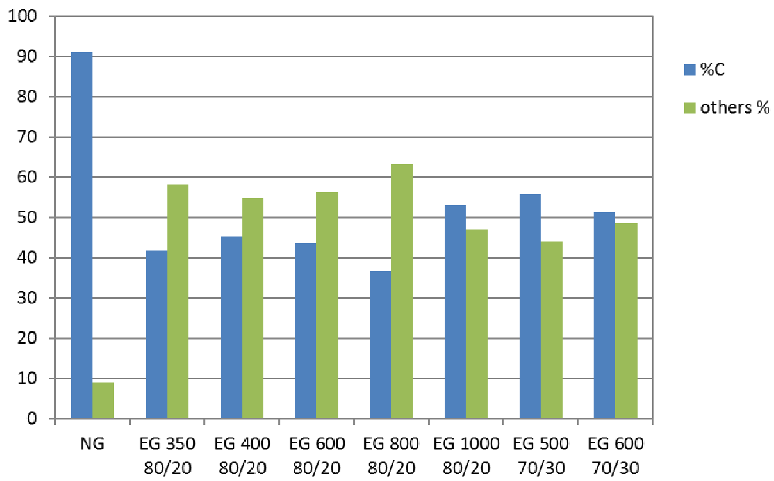


Figure 3: The Content of Carbon and other Components (Sum of Hydrogen, Nitrogen, Oxygen and Inorganic Species) of all EG Samples.

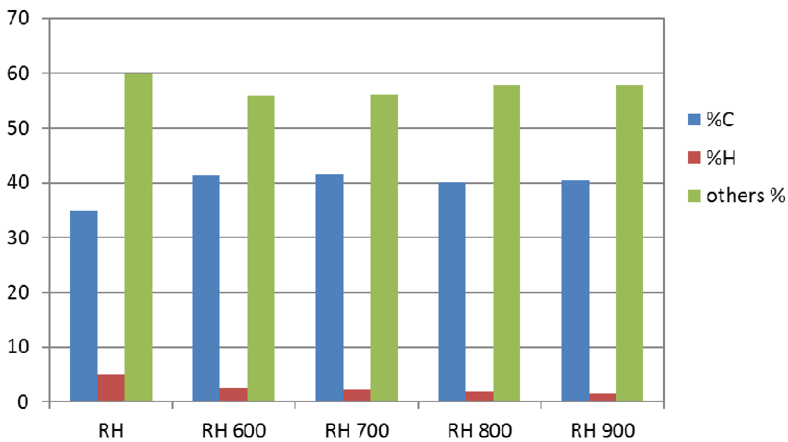


Figure 4: Elemental analysis of Rice Husk and Carbonized Rice Husk.

As expected, the heat treatment causes an increase in the carbon content compared to the original, as well as other components, at the same time also seen a decrease in the hydrogen content. In particular, with an increase in the activation temperature, a noticeable decrease in the hydrogen content is detected.

The data of determination of oil sorption capacity are shown in the Table 3. Results of tests showed that EG samples are better adsorbents compared to carbonized rice husks. It should be noted that, as a general trend, all EG samples have an adsorption capacity of at least three times greater that of natural graphite. As for the CRH samples, the adsorption capacities are similar, and no particular changes depending on the activation temperature were found. As a general result, the carbonization process led to an increase in adsorption capacity.

Table 3: Oil Sorption by the Synthesized Nanocarbon-containing Sorbents

Sorbent	S_{St}, Mass of the Spent Sorbent (Sorbent+Oil), g	S_0, Mass of a Sorbent, g	Oil sorption Capacity, g/g
NG	0.42	0.10	1.8
EG 500(70/30)	0.99	0.10	6.0
EG 600(70/30)	1.1	0.10	5.6
RH	0.35	0.10	1.2
CRH 600	0.73	0.10	3.1
CRH 700	0.67	0.10	3.3
CRH 800	0.69	0.10	3.7
CRH 900	0.66	0.10	3.2

CONCLUSIONS

- The expanded graphite and carbonized rice husk (CRH) were produced at different temperatures and by using different activating conditions.
- Results of the elemental analysis demonstrate that the carbon content decreases in all the samples of thermally expanded graphite while that of other components (the sum of nitrogen, oxygen and inorganic substances) increases.
- The heat treatment causes an increase in the carbon content in all samples of carbonized rice husk compared to the original rice husk, at the same time also seen a decrease in the hydrogen content. With an increase in the activation temperature, a noticeable decrease in the hydrogen content is detected.
- The samples prepared were tested as the sorbents of oil in water. Results of oil adsorption showed that all samples of expanded graphite have an adsorption capacity of at least three times greater than that of natural graphite.
- Adsorption capacity for the samples of carbonized rice husk is similar, and was equal to: CRH600-3.1%; CRH700-3.3%; CRH 800-3.7%; CRH900-3.2%.
- It was shown that the carbonization process has led to an increase in adsorption capacity.

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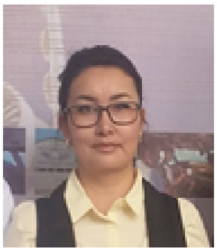

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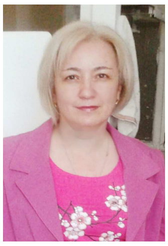

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