ХИМИЯ

УДК 544.723
doi: 10.18101/2306-2363-2017-1-3-9

ACTIVATED CARBON
OF NARIIN SUKHAIT HIGH RANK BITUMINOUS COAL

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Mongolian high rank coal was used as raw material to prepare activated carbon by physical activation method. The proximate, ultimate, and thermogravimetric analysis (TGA), scanning electron microscopy, surface area, and pore size distribution analysis were used to characterize the surface properties of activated carbons and initial raw coal of Nariin Sukhait. The effect of coal grade on the adsorption properties of the carbons was studied. The carbonization experiment was performed using TGA instrument in argon, at a temperature range of 25-1120°C, with heating rate of 40°C/min in case of carbonization. The mass loss for Nariin Sukhait coal was 30.6%. Activated carbon obtained from Nariin Sukhait coal had highest yield (66.1%), lowest ash content (7.1%),

high iodine adsorption (420 mg/g), surface area (402 m^2) and highest total pore volume (0.54 ml/g).
Keywords: bituminous coal, mass loss, mass loss rate, carbonization, activation, iodine adsorption, coal

Mongolia is one of the top ten countries with large coal resources. Coal is the major energy source among fossil fuels in the country because of its wide distribution and easy availability. Total geological resources of coal are 173 billion tones and the proven coal resource is 9.3 billion tones [1]. In 2010 Mongolian coal production and hard coal export increased to 23 million tones and 17 million tones respectively. Mongolian coal is composed mostly of subbituminous and bituminous grade coal.

Activated carbon which is a versatile adsorbent because of its good adsorption properties can be produced from a variety of raw materials. Among them, coal is the most commonly used precursor due to its low cost and large supply [2].

The presence of contaminant heavy metal ions like cadmium, mercury and lead in the industrial area of capital city Ulaanbaatar is a growing problem due to their high toxicities. Direct use of activated carbon [3] or some of their modified forms such as oxidized carbons [4] for the removal of the toxic metal ionic pollutants present in waste water is of considerable importance.

The objective of the present work was to study the possibility to obtain adsorbent materials from Mongolian coals by conventional physical method.

Experimental section

High-volatile C bituminous Nariin Sukhait (NS) coal was used as precursor in the present study. Proximate and ultimate analysis data of the coal are given in Table 1.

Table 1

Proximate and ultimate analysis of coal (proximate, %)

	W^a	A^d	V^{daf}	S_t^d	Q^{daf}, kcal/kg
Nariin Sukhait	1.65	3.69	32.9	0.78	8138
Ultimate daf, %					
	C	H	N	O (diff.)	H/C ratio
Nariin Sukhait	84.85	4.43	0.51	10.21	0.67

The precursor was ground and sieved to particle sizes 3.0-1.5 mm and subjected to carbonisation in a vertical steel reactor (ID = 50 mm) prior to activation. About 100 g sample was heated (3.3°C/min) to the final temperature 800°C in the absence of air and held for 30 min to ensure removal of all the volatile matters. The yields of products are presented in Table 2.

Table 2

Coal carbonisation data (wt, %)

	Char	Liquid	Gas + loss
Nariin Sukhait	70.1	13.57	16.33

The coke was ground and sieved to particle sizes 1.5-0.6 mm and subjected to activation in a vertical quartz reactor (ID = 20 mm). About 10 g sample was heated (5^{o}C/min) to the final temperature 800^{o}C in the saturated steam flow and held for 180 min. Activated carbon of Nariin Sukhait coal was named as NSAC. The yield of activated carbon was NSAC=66.1%. Weight loss in this series of experiment was calculated on the dried coke basis.

Adsorption of N_2 at -196^{o}C using JW-K equipment was performed to evaluate the surface area and pore size distribution of the activated carbon. The value of surface area were obtained by reference comparison method, and the values of pore volume were obtained by multi-point BET test.

Scanning electron microscope SEMTRAC mini SM-3000 was used for analysis of surface morphology of the activated carbon.

Results and Discussion

During the temperature programmed pyrolysis process, the TG and DTG curves are shown in fig. 1. It can be seen that there are great difference pyrolysis process at heating temperature range of 400-800°C. Coal pyrolysis process in 100% Ar environment has two stages: moisture release and devolatilization. As temperature is lower than 400°C, it is mainly the drying process of coal sample, and moisture removing that made a vital contribution to the weight loss. When temperature is higher than 400°C, the weak bond in the original coal sample commenced breaking, and formed gas product evolved out they are mainly aliphatics (mainly methane, CH_4) and water. With temperature increasing further the organic functional groups were broken and reunited quickly, and big peak was shown in the DTG curve. It might be taken as the primary pyrolysis of coal samples. As displayed in the DTG curve, the peaks of mass loss rate ranged at 400-700°C. In this stage, the main gas products contained CO_2, CO, light aliphatics, CH_4, H_2O, etc., as evidenced by FTIR measurements [5]. After that, with temperatures increased further (>700°C), another weight loss peak was displayed (800°C), which might be attributed to the secondary pyrolysis of condensed carbon matrix, with the evolution of CO and CO_2. It is consistent with a previous report however, during the secondary pyrolysis stage, there was also a large amount of H_2 releasing out with CO [6].

Some properties of the carbonized coal and activated carbon are shown in Table 3. Activated carbons had higher contents of ash and volatile matters compared with carbonized coal. With increasing of activation time, the contents of the mineral matter in carbons were increased due to their partial oxidation and decomposition of organic matter. The data also showed that the yield of active carbons were decreased with increasing time of activation, indicating the oxidation and decomposition degrees of organic matter are different for carbonized coal.

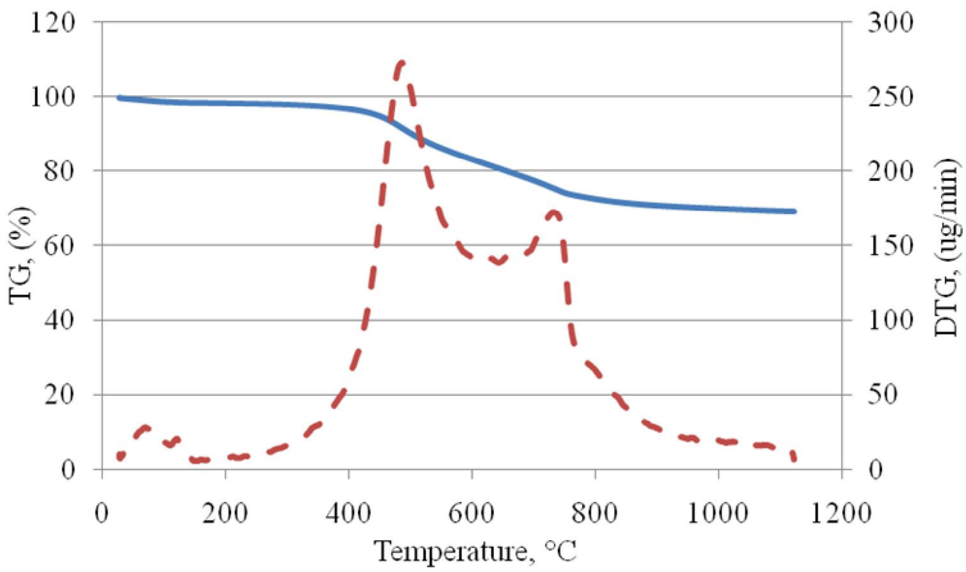


Fig. 1. TG and DTG curves of Nariin Sukhait coal pyrolysis in argon environment

Table 3

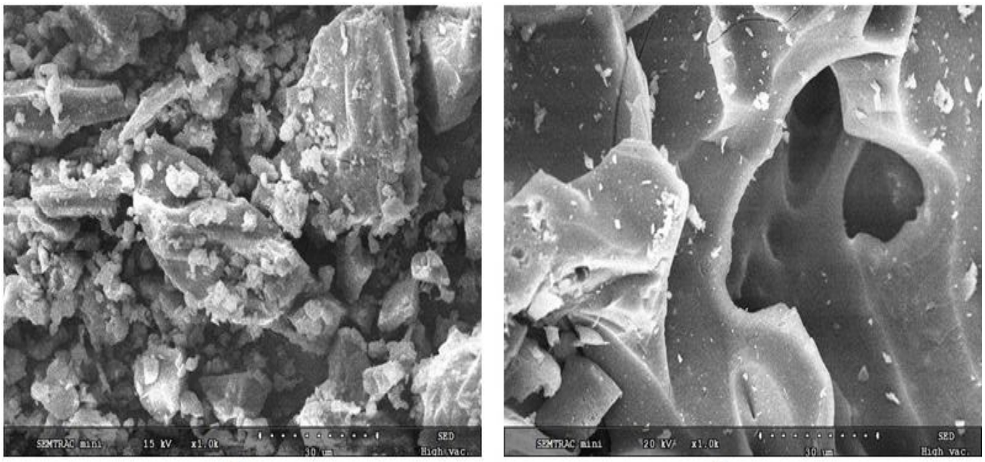
Properties of the active carbons obtained at temperature 800°C

Code	Sample	Time, min	Yield, %	Iodine adsorption, mg/g	Iodine number, %	Surface area, (N_2, 77K), (m^2/g)	Ash, A^d, %	Volatile matter, V^{daf}, %
NSC	Nariin Sukhait coal-carbonized	-	-	4	1.3	-	4.6	1.1
NSAC60	Nariin Sukhait coal-activated	60	98.6	5	1.6	18	6.0	1.6
NSAC140	Nariin Sukhait coal-activated	140	85.5	86	27.2	133	5.1	1.6
NSAC180	Nariin Sukhait coal-activated	180	66.1	420	60.7	402	7.1	1.9

The iodine adsorption capacity of activated carbons increased along with the activation time increased from 60 to 180 min. Sharp reduction (almost 2 times) in the iodine adsorption capacity was observed in case of Nariin Sukhait carbonized coal, during its activation at 180 min. Activated carbon obtained from Nariin Sukhait coal by physical activation had highest iodine adsorption capacity (60.7%) and lowest ash content (7.1%) compared with the other carbons. These values were also comparable with commercial activated carbons obtained from wood (BAU-A carbons data used in Alcoholic and Wine Industries according to Russian standard GOST 6217-74; iodine adsorption capacity (60%) and ash content (6%)). Most suitable condition for the physical activation was determined — temperature 800°C, time 180 min.

Surface area and total pore volume of Nariin Sukhait activated carbons were 402 m^2 and 0.54 ml/g, respectively. The surface area values decreased with the increase of coalification degrees of feed coal. The thermally stable high rank coal has lower degree of burn-off during activation, resulting in relatively higher values of surface area.

SEM microphotographs of Nariin Sukhait coal and activated carbon are presented in fig. 2.



A B

Fig. 2. SEM microphotographs of activated carbon
(Nariin Sukhait coal — A, Nariin Sukhait activated carbon — B)

The SEM photographic images show that Nariin Sukhait initial coal has also some micro poresand after carbonization and activation treatment it was well fused and swelled during carbonization and activation process and became a high developed porous material with macro pores mainly.

Conclusion

1. On the basis of proximate and ultimate analysis it was confirmed that Nariin Sukhait coal is a high rank G mark bituminous coal and it is suitable for thermal processing including carbonization and activation.
2. The results of carbonization experiment of Nariin Sukhait coal show that 70.1% of coal organic mass remained as a hard residue with micro porous structure after carbonization. The yield of all liquid and gas products is 29.9 which shows in g that there was an intensive thermal decomposition of the coal organic mass with higher degree of conversion.
3. Carbon yields of Nariin Sukhait coal are decreasing with activation time while the surface areas of the carbons are increasing.

4. For the Nariin Sukhait coal most suitable condition for the carbonized coal activation was found as 800°C temperature and 180 min.
5. Activated carbon obtained from Nariin Sukhait bituminous coal had relatively higher yield and lower ash contents compared with other chars and might be suitable precursor for production of activated carbons.
6. Pore structure of Nariin Sukhait activated carbons consists of mainly meso- and macro pores and has total pore volume 0.54 ml/g.

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АКТИВИРОВАННЫЙ УГЛЕРОД ИЗ ВЫСОКОКАЧЕСТВЕННОГО БИТУМНОГО УГЛЯ НАРИИН СУХАЙТ

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Монгольский уголь высокого качества используется в качестве сырья для получения активированного угля методом физической активации. Для характеристики поверхностных свойств, площади поверхности и анализа распределения по размерам пор активированных углей и исходного необработанного угля месторождения Нариин Сухайт использовался приближенный, конечный и термогравиметрический анализ (ТГА), сканирующая электронная микроскопия. Изучено влияние угля на адсорбционные свойства углеродов. Эксперимент карбонизации был выполнен с использованием инструментальных методов ТГА в аргоне при температуре 25–1120°C, с температурой нагрева 40°C/мин. Массовая потеря угля Нариин Сухайт в случае карбонизации составила 30,6%. Активированный уголь, полученный из угля Нариин Сухайт имел самый высокий выход (66,1%), низкое содержание золы (7,1%), высокую степень адсорбции йода (420 мг/г), площадь поверхности (402 м^2) и общий объем пор (0,54 мл/г)
Ключевые слова: битуминогель; потеря массы; массообмен; карбонизация; активация; адсорбция йода; уголь.