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Research on the thermal decomposition of Mongolian Baganuur lignite and Naryn sukhait bituminous coal

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Abstract: The technical characteristics, elemental composition of the organic and mineral matters, ash melting behaviors and carbonization and gasification reactivities of coals from Baganuur and Naryn sukhait deposits were investigated. The results of proximate and ultimate analysis confirmed that the coal from Baganuur deposit can be graded as a low rank lignite B2 mark coal and Naryn sukhait coal is a bituminous G mark one. The carbonization and gasification experiments were performed using TGA apparatus and fixed bed quartz reactor. The data obtained with two experimental reactors showed that Baganuur lignite had lower thermal stability and much higher CO_2 gasification reactivity at 950°C as compared to those for Naryn sukhait bituminous coal.

Keywords: *lignite, bituminous, carbonization, gasification, mass loss, mass loss rate*

INTRODUCTION

Coal has been believed to be the major energy source among fossil fuels in the coming century because of its easy availability. Mongolia is a country with rich coal resources. Total geological resources of coal are 163 billion tones and a proven coal reserve is 9.3 billion tones. In 2010 Mongolian coal production has been increased to 23 million tones, from which 17 million tones were exported to China. Coal is composed mostly of lignite, subbituminous and bituminous coals. The presence of contaminant heavy metal ions like cadmium, mercury and lead in the industrial area of Ulaanbaatar city is a big problem due to their high toxicities. The use of activated carbon for the removal of the toxic metal ionic pollutants in the waste water is of considerable importance. To resolve the problems of environmental pollutions we need to investigate the possibility of obtaining activated carbons with certain adsorbing and filtering ability from different kind of coals, which are the cheapest raw materials in Mongolia. In principle, the processes for the preparation of active carbons can be divided into two categories physical and chemical methods. The physical method consists of carbonization of the precursor followed by partial gasification of the resulting char by steam or carbon dioxide. First step is referred to as pyrolysis or carbonization which may be accompanied by drastic changes in the textural and molecular structure of the coal [1-6]. Second is relatively slow and is usually the rate determining step [7]. Coal is a very complex and heterogeneous material consisting of organic and inorganic materials and there

is no doubt that studies of the reactivity of carbons in gasification are extremely complex. It is generally accepted that the reactivity of coal chars in addition to coal origin, depends on accessibility of active sites in the char for reacting gases. For hetergenous systems, surface area or porosity, besides chemical factors, may play an important role in gasification reactivities [8-17]. Carbon dioxide is not used industrially as much as steam in the gasification or activation processes but it is the preferred agent used at laboratory level and the carbon CO_2 reaction is of great importance. The slower reaction rate at temperatures around 1000 K allows a better control of the gasification process and the analysis of the different variables, which may be modified [6]. Thermogravimetric analysis (TGA) has been found useful to explore various parameters that effect reactivity and has the added advantage of readily showing the changes in reaction rate as gasification of the char proceeds. Numerous investigations on the pyrolysis and gasification of coal with carbon dioxide and steam have been conducted in thermogravimetric balances [17-21]. Such studies may result into better understanding of factors influencing reactivities of coal chars and the may allow estimation of kinetic variables which may be used in predictive models for the pyrolysis and reactivity of chars. The main aim of this study was to investigate possible relationships between the fundamental physical and chemical properties of two different coal samples. Therefore the carbonization and gasification reactivities should be determined by thermogravimetric measurements for Baganuur lignite and Naryn sukhait bituminous coals. The Baganuur lignite deposit is located 110 km east of Ulaanbaatar, in the Bayandelger

village of the Tuv Province, and has been mined since 1978, proven reserves 713.1 million tones. The Naryn sukhait coal deposit is located 849 km of Ulaanbaatar, in the Gurvantes village of the South Gobi, proven reserves 125 million tones [22].

EXPERIMENTAL
Sample preparation, proximate and ultimate analysis of coals from the Baganuur and Naryn sukhait deposits were performed according to Germany Standards DIN 66165 (sieving analysis), DIN 51705 (bulk density), DIN51718 (moisture content), DIN51719 (ash yield), DIN51720 (volatile matter yield), DIN 51734 (fixed carbon) DIN 51900 (gross and lower heating value), DIN 51732 (carbon, hydrogen and nitrogen content), DIN 51724-3 (sulphur content), DIN 51727 (combustible sulfur and chlorine) and DIN 51729-10 (main and minor inorganic elements in fuel and ash for X-ray fluorescence analysis).

Ash melting behaviour: Prior to experimental determination of ash melting behaviour, the coal samples with particle size 0.2 mm were combusted at 815°C using a muffle oven. Tests of ash melting behavior were performed on the basis of the DIN 51730 standard. Coal ashes were compacted to a cylindrical body with a diameter and height of 10 mm each. A high temperature kiln was then used to analyze characteristic shapes of the ash while permanent heating of the kiln under oxidizing conditions. The ash was heated at rate 10°C/min starting from 550°C. Softening temperature (A), spherical temperature (B), hemisphere temperature (C) and flow temperature (D) were measured with an accuracy of ±25°C by visual observation.

TGA-measurements: The carbonization and gasification experiments were performed using TGA instrument, (Mettler Toledo TGA/DSC 1) which is schematically shown in Figure 1. The experiments started with temperature programmed carbonization and ended by gasification of the carbonization residue. For the experiments 20-30 mg of each sample was used and 2 times repeated. Baganuur lignite and Naryn sukhait bituminous coal samples with a particle size 0.2 mm were carbonized in TGA at temperature 950°C, with heating rate 30 K/min, and a N_2 flow of 120 ml/min, for 45 min. Carbonized coals were gasified in TGA at

temperature 950°C, with a N_2 flow of 20 ml/min, CO_2 flow of 100 ml/min and for 50-200 min. After completing a experiment the furnace was cooled at temperature from 950°C to 35°C, with a N_2 flow of 120 ml/min and a rate of 100°C/min, for 10 min. The carbonization conditions were at temperature 950°C, with a flow of N_2 at 120 ml/min and for 200 min. The gasification conditions at temperature 950°C, with a flow of CO_2 at 120 ml/min and for 50-200 min.

The TGA method can be summarized as follows:
Devolatalization/carbonization and gasification conditions:
1. Drying: ϑ=35-110°C; β=10 K/min; V_{N_2}=120 ml/min
2. Devolatalization/Carbonization: ϑ=110-950°C; β=30 K/min; V_{N_2}=120 ml/min, ϑ=950°C; t=10 min; V_{N_2}=120 ml/min
3. Gasification: ϑ=950°C; t=50, 100, 150, 200 min; V_{N_2}=20 ml/min; V_{CO_2}=100 ml/min
4. Cooling: ϑ=950°C-35°C; β=100 K/min; V_{N_2}=120 ml/min

Devolatalization/Carbonization conditions:
5. Drying: ϑ=35-110°C; β=10 K/min; 99.9% N_2, V_{N_2}=120 ml/min
6. Devolatalization/Carbonization: ϑ=110-950°C; β=30 K/min; V_{N_2}=120 ml/min ϑ=950°C; t=50, 100, 150, 200 min; V_{N_2}=120 ml/min
7. Cooling: ϑ=950°C-35°C; β=100 K/min; V_{N_2}=120 ml/min.

Gasification conditions:
8. Drying: ϑ=35-110°C; β=10 K/min; 10% CO_2, V_{CO_2}=120 ml/min
9. Devolatalization/Carbonization: ϑ=110-950°C; β=30 K/min; V_{CO_2}=120 ml/min, ϑ=950°C; t=50, 100, 150, 200 min; V_{CO_2}=120 ml/min,
10. Cooling: ϑ=950°C-35°C; β=100 K/min; V_{N_2}=120 ml/min.

Carbonization and gasification experiments in fixed bed reactor: The carbonization and gasification experiments were carried out in quartz crucibles. Prior to the treatment coal samples were sieved to a particle size < 0.08 mm. Then 54 g coal samples were sealed in the quartz crucibles and heated in a fixed bed at temperature 950°C, without air, with a heating rate of 31.7°C/min and for 30 min. After completion of each experiment, the crucibles were cooled to room
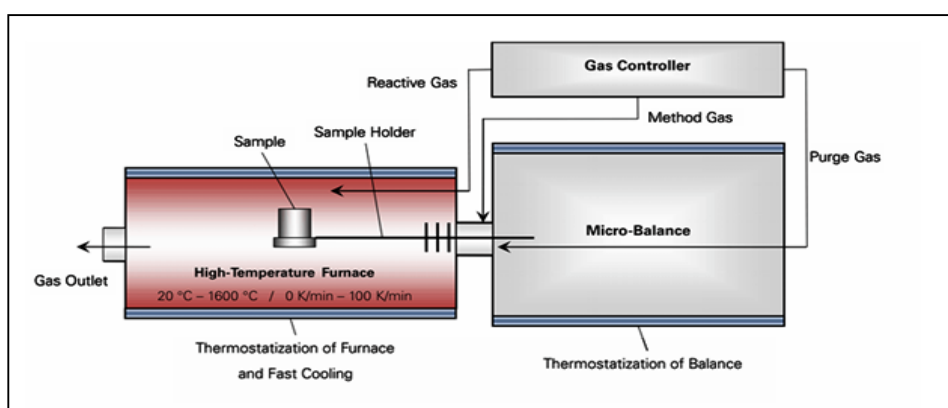

Fig. 1. Schematic diagram of the TGA - thermogravimetric analyzer.

temperature and carbonized coals were weighed. After that, 10 g carbonized coals were heated at temperature 950°C, with a flow rate of 450 ml/min, under N_2 atmosphere and for 20 min. Finally, carbonized coals were gasified at temperature 950°C, with a flow rate of 450 ml/min, under CO_2 atmosphere and for 30 min. After completion of each experiment, the hard residues were cooled to room temperatureand under N_2 atmosphere. The yields of products including solid residue (coal char) determined by weighing, and the weight loss by difference. The degree of coal organic matter (COM) conversion was determined by using following (1) formula;

$$COM \ conversion \% = \frac{Weight \ loss}{100 - A^a} * 100 \qquad (1)$$

RESULTS AND DISCUSSION
The results of sieving analysis (Figure 2) show that particle size distributions of coal samples are different from each other. Baganuur lignite coal sample consists of particles with very different sizes from fine coal powder to lump coals. Naryn sukhait coal sample had large yield of fraction between 2 mm to 10 mm sizes and relative little amount of fractions larger than 10 mm size. Therefore, it can be proposed that the hardness of the Baganuur lignite is higher than that of Naryn sukhait coal. The results of proximate analysis of 2 selected samples showed that Naryn sukhait coal has lower contents

Table 1. Properties of the coals samples

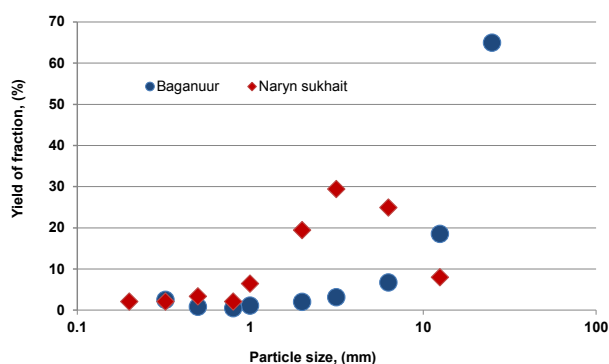| Proximate analysis of coal, (%)	Baganuur	Naryn sukhait
$W^{a\delta}$	7.6	3.3
V^{daf}	47.4	33.8
FC	35.9	53.0
$A^{\delta\beta}$	9.1	9.9
HHV ^{daf}, kcal/kg	6714	7734
LHV ^{daf} kcal/kg	6470	7490
Bulk density (g cm^{-3})	0.55	0.74
Ultimate analysis of coal, (%, daf)		
C	71.6	80.6
H	4.7	4.8
N	1.2	1.3
S_t	0.3	1.4
O^a	22.2	11.9
$S_{combustible}$	0.3	1.2
Cl_t	0.03	0.03

^{a} By difference.

Fig. 2. Particle size distribution of coal samples

of moisture, volatile matter and higher calorific value and bulk density compared with Baganuur lignite. Ash contents of the samples are similar. The results of ultimate analysis showed that Naryn sukhait coal has higher content of carbon, lower content

of oxygen compared with Baganuur lignite. These results confirm the higher coalification rank of Naryn sukhait coal. Comparison of organic mass composition and some properties of the samples with different fuels are shown in Figure 3. From the figures it can be concluded that Baganuur coal belongs to lignite A type and Naryn sukhait coal belongs to high volatile bituminous B type coals by ASTM D388 standard. The results of analysis of ash melting behaviour (Fig. 4) show that all temperatures (shrinkage-, deformation-, hemisphere- and flow-) are higher in case of Naryn sukhait coal. Especially, hemisphere- and flow-temperatures of Naryn sukhait coal ash are 120-140°C higher than that of Baganuur lignite ash. This different behavior might be caused by mineral composition of coal ashes. Naryn sukhait coal ash may contain more minerals with higher melting temperatures [23]. The chemical composition of ash prepared at 1088 K showed in Table 2. Contents of main inorganic elements show the relative higher contents of silicates and lower contents of calcium and magnesium oxides in ash from Naryn sukhait coal. These results are consistent with results of analysis of ash melting behavior. Naryn sukhait coal ash can be classified as bituminous type, while the Baganuur lignite ash is a lignite type [23]. The sum of CaO and MgO (CaO+MgO=8.0 > Fe_2O_3) and the ratio (Fe_2O_3+CaO+MgO+Na_2O+K_2O)/

Table 2. Main inorganic elements in Baganuur and Naryn sukhait coals and their ashes (%, db)

Element in coal	BN	NS	Element in ash	BN	NS	Oxide in ash	BN	NS
Si	2.4	3.1	Si	10.2	12.2	SiO_2	21.8	26.0
Al	1.3	2.4	Al	5.3	10.6	Al_2O_3	10.0	20.0
Fe	0.79	1.6	Fe	2.1	5.3	Fe_2O_3	3.0	7.5
Ca	3.7	1.5	Ca	12.4	4.7	CaO	17.4	6.5
Mg	0.17	0.21	Mg	0.86	0.92	MgO	1.4	1.5
Na	-	-	S	2.9	2.6	SO_3	7.3	6.4
K	0.14	0.24	Na	-	0.87	Na_2O	-	1.2
			K	0.52	0.94	K_2O	0.63	1.1

BN - Baganuur ; NS - Naryn sukhait ; - not detectable
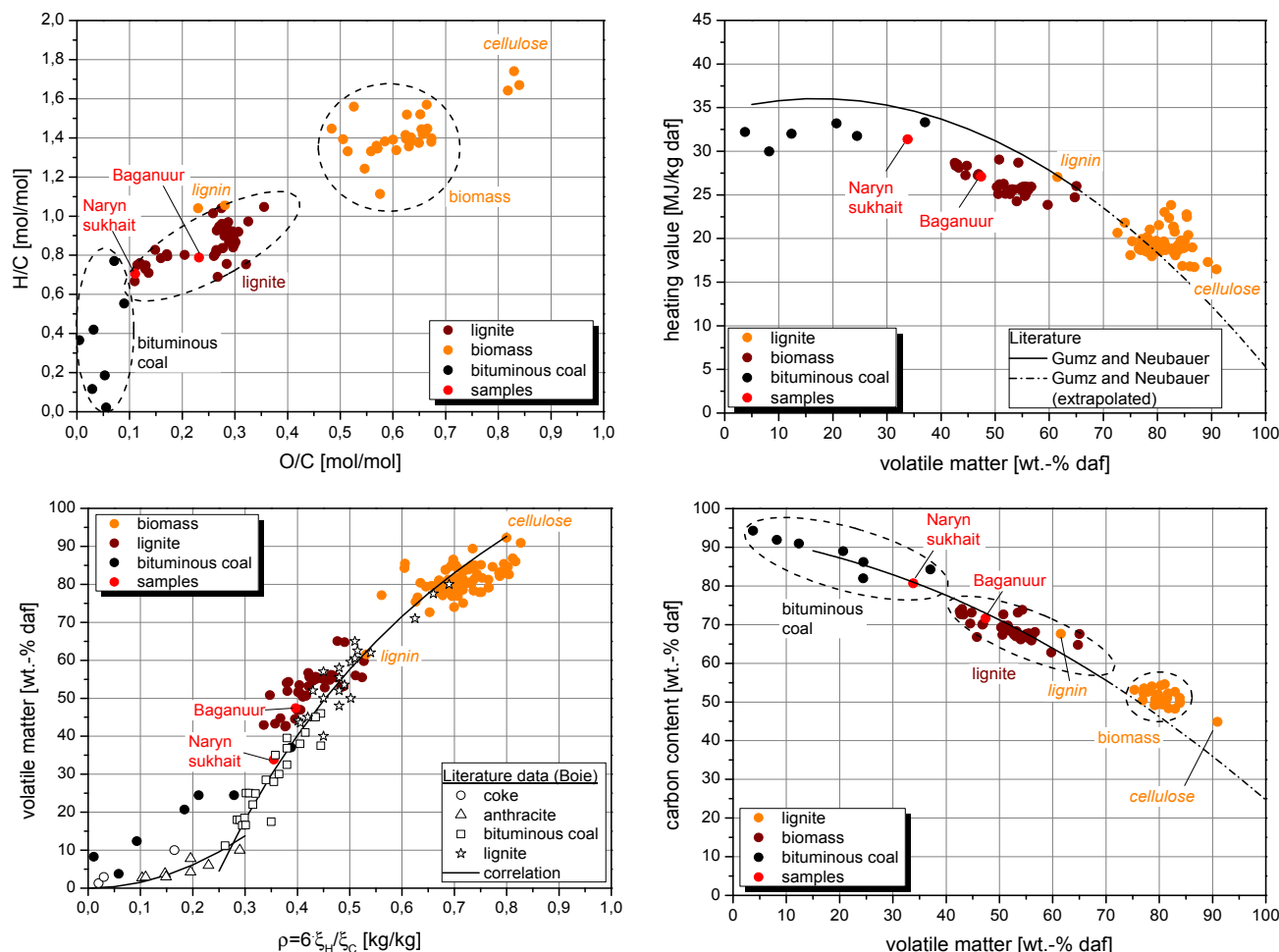
Fig. 3. Comparison of organic mass composition of the samples in relation to different fuels

Baganuur lignite
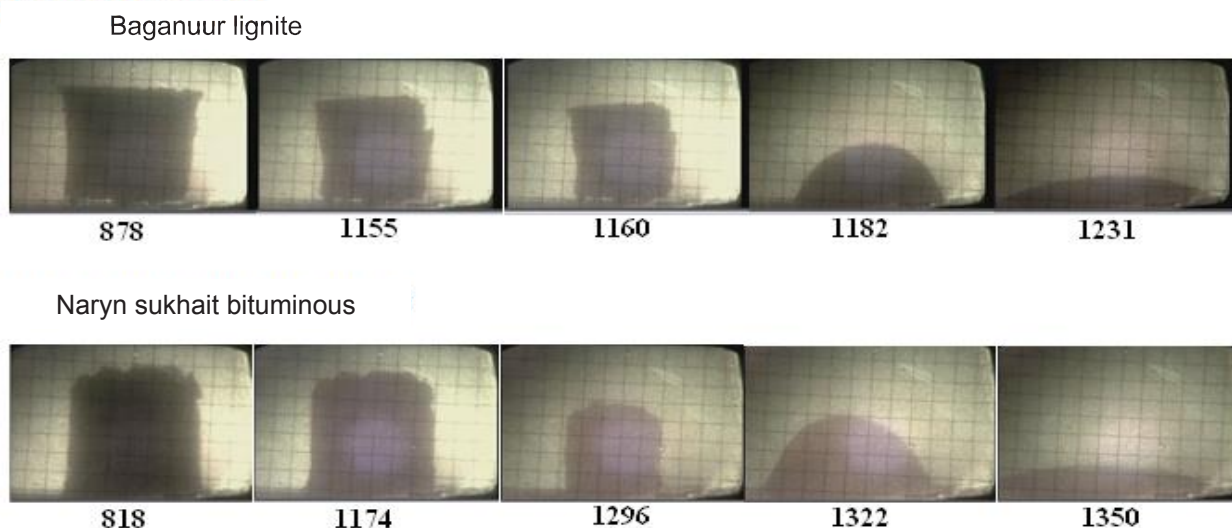

Naryn sukhait bituminous

Fig. 4. Ash melting behaviour of different coals: initial state, shrinkage-temperature, deformation-temperature, hemisphere-temperature and flow-temperature.

$(SiO_2+Al_2O_3+TiO_2) < 1$ show that the Naryn sukhait coal is a bituminous and it is ash has an acidic character. The value of modules of coal ashes quality index are shown in Table 3. The Naryn sukhait coal ash has higher silicate and melting modules has lower medium, hydraulic and pyroxene modules compared with that of Baganuur, which confirm higher melting temperature of Naryn sukhait coal. The coal ash can be used as hydraulic mixture for the construction materials. But

due to low value of the pyroxene module, it can not be used in casting factories.

Contents of minor inorganic elements show in Table 4 that the contents of highly hazardous elements such as As, Hg, Pb, Cd are relatively low in both coal ashes.

Comparison of TGA analysis of samples: The TGA and DTG curves of Baganuur and Naryn sukhait coals carbonization and gasification are shown in Figure 5-8. From Figure 5 and 6 the TGA and DTG curves of

Table 3. Value of modules of coal ashes quality index

| Sample | Modules | | | | Medium |
	$M_{silicate}$	$M_{hydraulic}$	$M_{pyroxene}$	$M_{melting}$	
Baganuur	1.7	0.61	1.06	1.55	0.71
Naryn sukhait	0.95	0.22	0.72	2.15	0.39

Baganuur coal can be observed that the carbonization of coal samples was taken place at 355-950°C. As a temperature is lower than 355°C, it is mainly the drying and dehydration process of coal and moisture removing made to the weight loss. When temperature is higher than 355°C, the weak bond in original coal sample commenced breaking and formed gas product evolved out. With temperature increasing further, the organic functional groups were broken and reunited quickly and a big peak was shown in the DTG curve. As displayed in the DTG curves, the peaks of mass loss ranged at 355-550°C. In this stage, gas products contained mainly CO_2, CO, CH_4, H_2O, light aliphatics from Baganuur coal carbonization at 455.6°C. After that, with temperature increased further (>550°C),

Table 4. Minor inorganic elements in Baganuur and Naryn sukhait coals and their ashes (%, db)

Element in coal	BN	NS	Element in ash	BN	NS
Sb	0.067	0.018	Sb	0.22	0.06
As	0.00096	0.00099	As	0.003	0.0036
Pb	0.0002	0.0006	Pb	0.00035	0.002
Cd	-	-	Cd	-	-
Cr	0.0016	0.002	Cr	0.005	0.008
Co	0.0039	0.0087	Co	0.01	0.03
Cu	0.0022	0.0059	Cu	0.0056	0.017
Mn	0.013	0.037	Mn	0.037	0.12
Ni	0.0010	0.0028	Ni	0.003	0.008
P	0.084	0.079	P	0.086	0.13
Hg	0.00004	0.00002	Hg	0.00003	0.00001
Se	0.00005	0.00022	Se	0.00008	-
Te	-	-	Te	-	-
Tl	0.00002	-	Tl	-	-
Ti	0.11	0.12	Ti	0.31	0.4
V	0.0017	0.005	V	0.0053	0.017
Zn	0.00047	0.0018	Zn	0.0013	0.0061
Sn	0.0066	-	Sn	0.044	0.014

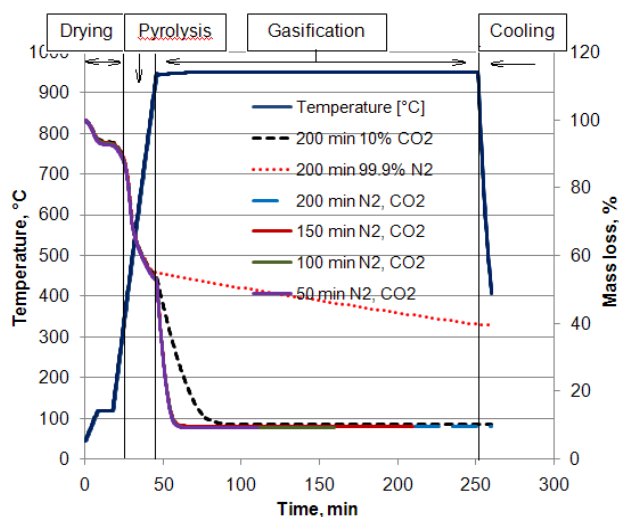BN - Baganuur; NS - Naryn sukhait; - not detectable.

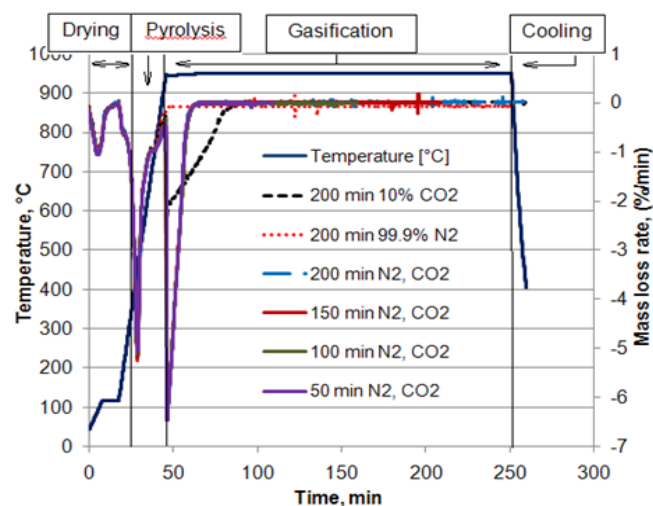Fig. 5. TGA curves of Baganuur coal carbonization and gasification in different time at 950°C.

Fig. 6. DTG curves of Baganuur coal carbonization and gasification in different time at 950°C
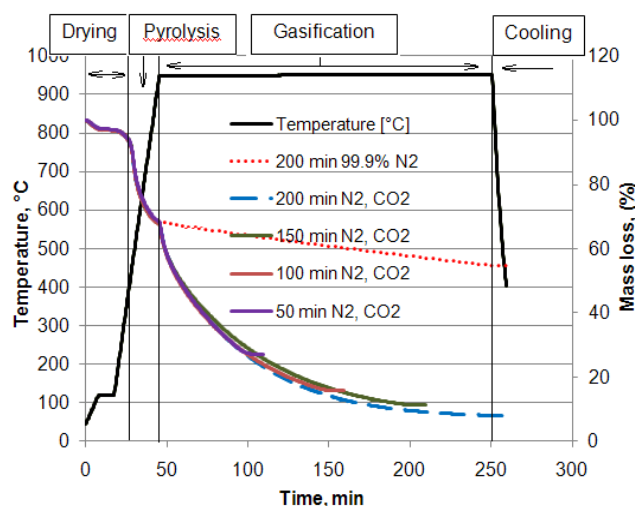

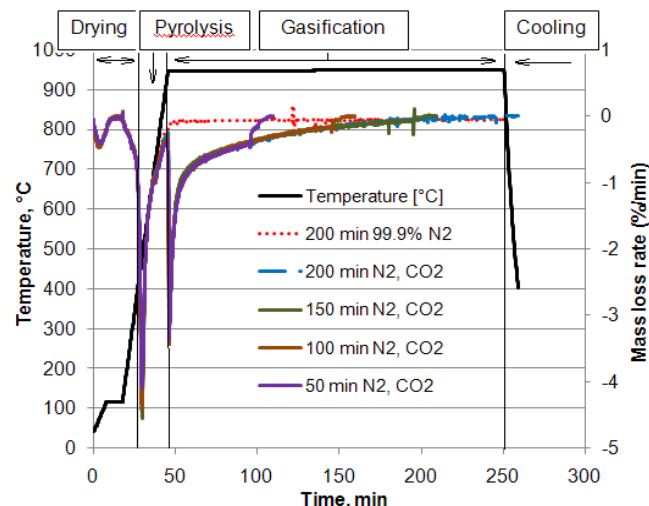Fig. 7. TGA curves of Naryn sukhait coal carbonization and gasification in different time at 950°C

Fig. 8. DTG curves of Naryn sukhait coal carbonization and gasification in different time at 950°C

another weight lost peak was displayed (>550°C), which might be attributed to the secondary pyrolysis of condensed carbon matrix, with the evolution of CO [24-26]. As temperature was lower than 950°C, which was the carbonization stage, there is just some volatile residue removing. At the temperature from 35°C to 950°C, for time period 45 min of carbonization mass loss was 43.1-43.7%. N_2 was switched to CO_2, and the gasification of solid charcoal reacted with CO_2 quickly. From the gasification curves, it can be observed that the gasification reaction was completed in 10 min (from 50 to 60 min) with some ash residue, and after that, no obvious mass loss was detected with time extending further. From the gasification curves, it can be observed that at the temperature 950°C, about 60 min the contents of the mineral matter in carbons were increased due to their partial oxidation and decomposition of organic matter. Baganuur lignite has higher the yield of mass loss after gasification compared with that of after carbonization, which confirm that solid charcoal reacted with CO_2 intensively. From Figures 7 and 8 the TGA and DTG curves of Naryn sukhait coal can be observed that the carbonization of coal samples was taken place at 400-950°C. As a temperature is lower than 400°C, it is mainly the drying and dehydration process of coal and moisture removing made to the weight loss. Peaks in DTG curves located 400°C corresponded to it. When temperature is higher than 400°C, the weak bond in original coal sample commenced breaking and formed gas and volatile product evolved out. With temperature

increasing further, the organic functional groups were broken and reunited quickly and a big peak was shown in the DTG curve. As displayed in the DTG curves, the peaks of mass loss ranged at 400-700°C. In this stage, gas products contained mainly CO_2, CO, CH_4, H_2O, light aliphatics from Naryn sukhait coal carbonization at 500°C. After that, with temperature increased further (>700°C), another weight lost peak was displayed (950°C), which might be attributed to the secondary pyrolysis of condensed carbon matrix, with the evolution of CO [24-26]. As temperature was lower than 950°C, which was the carbonization stage, there is just some volatile residue removing. At the temperature from 35°C to 950°C, for time period 45 min of carbonization mass loss was 29.7-30.7%. N_2 was switched to CO_2 and the gasification of solid char started. With retention time extending, solid charcoal reacted with CO_2 intensively. From the gasification curves, it can be observed that with increasing gasification time, the contents of the mineral matter in carbons were increased due to their partial oxidation and decomposition of organic matter. The dates (Figure 9) also showed that the mass losses were decreased with increasing time of gasification indicating the oxidation and decomposition degrees of organic matter are different for the different types of chars. The results (Table 5) confirm that Baganuur lignite has low thermal stability its decomposition occurs at lower temperature compared with that of Naryn sukhait coal. Starting temperature of devolatalization for Baganuur lignite is approximately 60°C lower than that of Naryn sukhait coal. Therefore the values of

Table 5. Results of the TG and DTG obtained at temperature 950°C

| Sample	Time, min	Mass loss[db], (%)			Starting temperature of devolatalization, °C	Drying		Carbonization		Gasification	
		Carbonization	Gasification	Residue of gasification		Temperature, °C	Value of maximum mass loss rate, (%/min)	Temperature, °C	Value of maximum mass loss rate, (%/min)	Temperature, °C	Value of maximum mass loss rate, (%/min)
Baganuur	50	43.3	46.4	10.3	355.7	91.7	1.02	454.7	5.08	944.4	6.48
	100	43.2	46.9	9.9	356.1	93.3	1.0	454.9	5.13	944.0	6.20
	150	43.1	46.3	10.6	355.8	95.5	0.97	454.1	5.15	944.5	6.26
	200	43.7	46.0	10.3	357.5	91.9	0.99	455.6	5.20	943.7	6.39
Naryn sukhait	50	29.7	40.0	30.2	404.0	79.2	0.38	479.2	4.2	946.8	3.25
	100	30.7	52.8	16.6	402.4	71.3	0.47	479.1	4.44	945.6	3.46
	150	30.4	58.1	11.5	411.9	75.7	0.45	479.1	4.51	945.7	3.45
	200	29.8	60.7	9.6	410.3	80.5	0.38	478.8	4.34	946.4	2.98
Carbonization under N_2, at 200 min											
BN	200	57.8	-	42.2	356	92.6	0.97	454.2	5.29	-	-
NS	200	42.7	-	57.3	409.5	73.8	0.42	479.4	4.53	-	-

BN - Baganuur, NS - Naryn sukhait
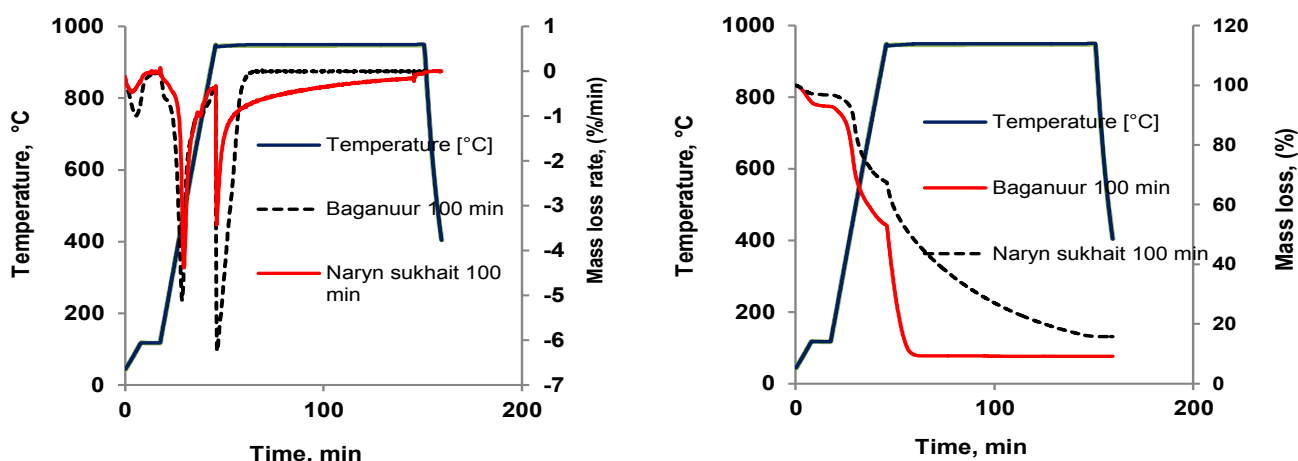
Fig. 9. Comparison of TGA and DTG curves for Baganuur and Naryn sukhait
coals carbonization and gasification in 100 min, at 950°C.

maximum mass loss rate is for Baganuur lignite are much higher than that of Naryn sukhait coal in all stages of processes drying, carbonization and gasification.

Reactivity of char derived from Baganuur lignite is so high, its gasification completed only in 10 minutes at 950°C. A complete gasification of char derived from Naryn sukhait coal at 950°C requires approximately 150 minutes.

Comparison of fixed bed gasification and TGA tests: The results of Baganuur and Naryn sukhait coals carbonization and gasification are shown in Table 6.

Table 6. Yield of the carbonization and gasification products at 950°C, for 30 min

Sample	Atmosphere	Carbonization (%)		
		Char	Weight loss	Conversion
BN	Without air	51.1	48.9	53.3
NS	Without air	65.9	34.1	37.6
Sample	Atmosphere	Gasification (%)		
		Hard residue	Weight loss	Conversion
BN	CO_2	21.3	78.7	85.9
NS	CO_2	42.7	57.3	63.1

BN - Baganuur, NS - Naryn sukhait

The Naryn sukhait bituminous coal has higher yield of char compared with that of Baganuur lignite, which confirm higher thermal stability of Naryn sukhait coal. The results show that 53.3% of organic mass of Baganuur lignite converted to gaseous and volatile compounds during carbonization, which is showing intensive thermal decomposition. It is known that lignite and sub-bituminous coals are more suitable for gasification and liquefaction due to their lower thermal stability. If compare the results, marked difference in product yield is found among gasification under CO_2 at the 950ºC. The yield of Baganuur hard residue is decreasing intensively and the yield of gasification products are increasing intensively compared with that of Naryn sukhait. At this condition 85.9% of organic mass of Baganuur lignite removed, which is showing that there was also an intensive gasification of the coal organic mass with higher degree of conversion.

CONCLUSIONS

1. Coalification degree of the samples by Russian classification could be ordered as following Baganuur (B2)<Naryn sukhait (G). This result shows that Baganuur coal belongs to lignite A type and Naryn sukhait coal belongs to high volatile bituminous B type coals by ASTM D388 standard.

2. The Naryn sukhait coal ash has higher silicate and melting modules and therefore it has an acidic medium, lower hydraulic and pyroxene modules compared with that of Baganuur coal, related to the higher melting temperature of Naryn sukhait coal ash.

3. The results of thermogravimetry analysis confirm that Baganuur lignite has low thermal stability and its thermal decomposition occurs at lower temperature compared with that of Naryn sukhait coal.

4. The Naryn sukhait bituminous coal has higher yield of char compared with that of Baganuur lignite, which confirm higher thermal stability of Naryn sukhait coal.

5. The results show that 53.3% of organic mass of Baganuur lignite converted to gaseous and volatile compounds during carbonization, which is showing intensive thermal decomposition.

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