CHARACTERISATION OF MONGOLIAN EAST KHOOT COAL AND ITS PYROLYSIS PRODUCTS

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ABSTRACT. Pyrolysis of East Khoot coal was carried out at different heating temperatures and yields of solid, liquid and gaseous products were determined. It was discovered that the optimal heating temperature is 500–550 °C. Raw coal from the East Khoot deposit and pyrolysed coal samples were analysed for ash content and composition. The results of proximate, ultimate and petrographic analysis confirm that the East Khoot coal is a low-grade (B2) brown coal. The porosity of raw coal, char of pyrolysed coal and activated carbon of pyrolysed char was determined by means of scanning electron microscopes. The liquid tar product of pyrolysed East Khoot coal was investigated using Fourier transform infrared spectroscopy and its liquid tar pyrolysis product by means of GC/MS chromatography.

Keywords: coal, petrographic analysis, pyrolysis, activated carbon

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ХАРАКТЕРИСТИКА УГЛЯ МЕСТОРОЖДЕНИЯ «ВОСТОЧНЫЙ ХОТ» МОНГОЛИИ И ПРОДУКТОВ ЕГО ПИРОЛИЗА

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РЕЗЮМЕ. Был проведен пиролиз угля месторождения «Восточный Хот» при различных температурах нагревания и анализ выхода полученных твердых, жидких и газообразных продуктов. Оптимальная температура нагревания – 500–550 °C. Анализу была подвергнута зола проб угля месторождения «Восточный Хот». Результаты технического, химического и петрографического анализа подтверждают, что уголь месторождения «Восточный Хот» – это бурый уголь низкого ранга марки B2. Пористость угля, смолы угля и активированный уголь изучались с использованием электронных микроскопов. Продукты пиролиза угля месторождения Восточный Хот исследовались методом ИК-спектроскопии. Жидкие продукты анализировались методом ГХ/МС хроматографии.

Ключевые слова: уголь, петрографический анализ, пиролиз, активированный уголь

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INTRODUCTION
Coal continues to be one of the world's most important energy sources, especially for power generation, with global coal demand projected to continue to grow over the next few years [1]. Apart from its use as a primary energy fuel, coal represents an important feedstock for the chemical industry, with different products such as metallurgical coke, coal tar and synthetic gases being derived and manufactured from coal. The conversion of coal into oil and gas allows it to be utilised as an alternative to conventional fuels, which has implications for Mongolia's national security and sustainable economic development. Among other operating factors for coal pyrolysis, temperature, pressure and type of catalysts play important roles in the composition of derived products. Typical pyrolysis products of raw coal are generated primarily through the decomposition of aliphatic side chains, oxygen functional groups, and low-molecular-weight compounds [2]. Compared to oil and natural gas, coal resources are more unevenly distributed worldwide and often readily accessible, e.g. by surface mining [1]. While lacking major oil deposits, Mongolia is relatively rich in coal resources, with potential coal reserves estimated at more than 160 billion tonnes. However, the majority of Mongolia's coal resources remain undeveloped due to a lack of infrastructure. For example, the huge Tavan Tolgoi deposit in the South Gobi lies more than 400 km from the nearest railway. Mongolia's coal reserves are primarily located in a large brown coal basin (Jurassic origin) located in the central economic region of Mongolia, which contains the Baganuur, Ovdkoghudag, Aduunchuluun, Tevshiin Govi, Khoot, Tsaidam Nuur and Shivee Ovoo deposits.

Coal samples from the Tavan Tolgoi deposit have been assessed for beneficiation and coke production, while samples from Baganuur, Bayanteeg and Shivee Ovoo deposits have been characterised for pyrolysis, hydrogenation and gasification [3, 4]. Moreover, samples from the Ovdokhudag and Aduunchuluun deposits have been assessed for their liquefaction potential using facilities in Japan [5]. However, the present study represents the first detailed investigation of coal from the Khoot deposits.

An array of products can be made through the fragmentation of the macromolecular coal network [6, 7]. Pyrolysis conditions, e.g., heating rate and final pyrolysis temperature, have significant influences on the characteristics of the resultant gases, liquids and solid residues (char) [8]. Both oils and gases produced by pyrolysis showed relatively high overall heating values, comparable to some conventional fuels, revealing the potential application of these products as fuel [9]. Generally, calcium, magnesium, potassium and silicon minors acted as inert materials, inhibiting the pyrolysis and combustion rates of the samples [10]. For this reason, the coal of Khoot deposit was chosen for the first time for investigating thermal processing, including pyrolysis and thermal dissolution (thermalysis) and the characterisation of obtained solids (hard residues) and liquid products.

EXPERIMENTAL
Since 1973, coal from the East Khoot deposit has been exploited in an open cast mine located near the village of Matad in the province of Dornod around 700 km to the east of Ulaanbaatar in the central economic region of Mongolia.

Samples were prepared for analysis according to ASTM D 2797. Petrographic analysis was performed on polished particulate mounts following recommendations by the International Committee for Coal and Organic Petrology [11]. Vitrinite reflectance was measured using an Axio Imager M2m microscope (Zeiss, Germany) and Fossil software (Hilger, Germany) on 50 individual vitrinite macerals in random mode conducted according to ASTM D 2798. In addition, a modification of ASTM D 2799 using fluorescence microscopy was carried out for rapid qualitative information on maturity and organofacies. Vitrinite particles were characterised as having higher reflectivity than autochthonous vitrinite. Typically, only the vitrinite population with the lowest reflectance values is measured and reported. Maceral group analysis was performed using the same microscope and software by point counting of 500 individual macerals. Petrographic analysis, vitrinite reflectance and maceral group composition are important for microcomponent investigation of the coal organic mass and subsequent determination of the coal type and quality, which are important characteristics for the thermal decomposition of coal organic mass.

The pyrolysis experiments of coal samples were performed in a stainless steel vertical cylindrical retort containing 1 kg of sample. The retort was placed in an electric furnace (model SNOL) with a maximum temperature of 950 °C. A chrom-alumel thermocouple was immersed in the coal bed to measure the actual heating temperature with temperature control (potentiometer). The retort was connected with an air-cooled iron tube and water-cooled laboratory glass condenser and a collection vessel for the liquid product condensate (pitch and pyrolysis water). Following water-cooled condensation, the non-condensable gases were removed from the system via a thin glass tube. The experiments were carried out to a temperature of 500 °C and the heating rate was 20 °C min⁻¹. The yields of solid and liquid residue products (coal char), tar and pyrolysis water were de-
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terminated by weighing, while the yield of gases was determined by calculating the difference.

The pyrolysed coal samples (10–15 g) were placed in quartz tube, streamed with nitrogen to remove the oxygen, heated up to 800 °C and processed (activated) with heated water steam for 120 min.

The Fourier transform infrared spectroscopy (FTIR) spectra of the samples were obtained using the Interspec 200-X series of FTIR spectrometers with PIKE Diffusion IR accessories using a KBr disc containing 1% of finely ground samples. All the spectra were measured in the frequency range of 4000 to 400 cm⁻¹, with each sample being subjected to 32 scans.

The liquid condensed by-product of coal pyrolysis consists of tar and pyrolysis water. These form two non-miscible layers and can thus be easily separated using a glass separating funnel. The upper layer consists of tar (viscous liquid) having a dark-brown colour and a pungent smell. The bottom layer comprises pyrolysis water (non-viscous liquid) also having an unpleasant aroma and a brown colour. For the final cleaning of tar from the pyrolysis water residue, thermally treated CaCl₂ is generally applied when mixing and separating (filtering or centrifuging).

The column chromatography characteristics of East Khoot coal tar pyrolysis are as follows:
- Small glass column – 5,0 ml;
- Sample of tar 0,2 g for each solvent;
- Organic solvents used (pure for chromatography): hexane, benzene and dichloromethane – 20,0 ml from each solvent;
- Sorbent used – activated silica gel 4,0 g.

The column chromatography carried out for obtaining of the soluble in hexane (H), benzene (B) and dichloromethane (M) fractions of the pyrolysis tar. These fractions were used as parent solutions for GC/MS analysis. The organic solvent was evaporated from the obtained fraction for the determination of the yield of each fraction.

The conditions of GC/MS analysis of each fractions are:
- The analytical sample of each fraction – 1 microlitre of each fraction in 1 ml of each solvent;
- GC/MS analysis sample – 1 microlitre from each analytical sample;
- Used apparatus: Agilent 7890A Agilent 5975C GCMS system and capillary column J&W DB-5,30 mx, 0,25 mm I.D. 0,25 µm (122-5032);
- Carrier gas – Helium;
- Mass range – 50–550;
- Starting temperature of furnace – 100 °C;
- Heating temperature and time – 220 °C, 46 min.

RESULTS AND DISCUSSION

The results of ultimate, proximate and organic petrographic analysis of coal samples from the East Khoot deposit are shown in Table 1, Fig. 2 and Table 2. For the characterisation of coal from the East Khoot deposit, the results of FTIR analysis of the samples are shown in Fig. 1.

Results of proximate and ultimate analysis indicate that coal from the East Khoot deposit comprises a low rank B2 (ISO 11760) brown coal, which has a low ash and sulphur content. According to the ASTM D-388 classification, it is brown coal of low rank.

For the characterisation of coal of the East Khoot deposit, the results of FTIR analysis of coal samples are shown in Fig. 1.

Fig. 1. FTIR spectra of coal from the East Khoot deposit

Рис. 1. ИК-спектры проб угля месторождения «Восточный Хот»
In the FTIR spectra, East Khoot coals can be characterised as: 700–900 cm$^{-1}$ for $\text{C}-\text{H}$; 1033–1112 cm$^{-1}$ for vibration of bonds in various oxygen-containing groups; 1160–1269 cm$^{-1}$ for skeletal vibrations of aromatic rings; 1375 cm$^{-1}$ for skeletal vibrations of aromatic rings; 1600 cm$^{-1}$ strongest band for $>\text{C}=\text{O}$ bonds in acids, ketones, aldehydes and quinines; 2854, 2924 cm$^{-1}$ for stretching vibrations of $\text{-CH}_2$ and $\text{-CH}_3$ groups in saturated aliphatic structures; and 3383 cm$^{-1}$ for stretching-associated vibrations of $\text{-OH}$ groups in aromatic rings and aliphatic structures.

Petrographic analysis was carried out on polished coal samples using the same microscope and software by point counting of 500 individual macerals. The black-and-white and coloured petrographic photographs are presented in Fig. 2; the vitrinite, inertinite and liptinite compositions of the maceral group are 88%, 4 and 5%, respectively. The vitrinites consist of grey-coloured fragments with different sizes, while the inertinites are seen as white coloured stripes in the black-and-white photograph (Fig. 2, a).

The liptinites are not observed in the black-and-white photograph (Fig. 2, a) but can be seen in the form of yellow-coloured stripes in the colour photograph (Fig. 2, b). The vitrinite fragments are black-coloured in the colour photograph (Fig. 2, a) and the green-coloured background in colour photograph (Fig. 2, b). The minerals in the polished coal sample are observed as brilliant pieces in the microscope, but are absent in the black-and-white (a) and coloured (b) petrographic photographs.

The degree of vitrinite reflectance (0,34%) was measured using an Axio Imager M2m microscope and Fossil software on 50 individual vitrinite macerals in random mode. This value of 0,34% is characteristic for a low rank brown coal. Therefore, the results of the determined technical characteristics, elemental and maceral group composition, as well as the degree of vitrinite reflectance, show that East Khoot coal is a low rank B2 mark brown coal, which is suitable for thermal processing such as pyrolysis and thermolysis (thermal dissolution).

![Fig. 2. The black-and-white (a) and coloured (b) petrographic photographs of polished coal sample of East Khoot deposit](image)

Рис. 2. Черно-белые (a) и цветные (b) фотографии шлифованных проб угля месторождения «Восточный Хот»

### Table 1

| Moisture, % | Ash, % | Yield of volatile matter, % | Sulphur, % | Caloric value, kcal kg$^{-1}$ | Carbon, % | Hydrogen, % | Oxygen and others (N + O), % |
|------------|--------|----------------------------|------------|-----------------------------|----------|-------------|-----------------------------|
| 11,0       | 8,9    | 46,8                       | 1,1        | 6232,8                      | 64,4     | 5,8         | 29,7                        |

### Table 2

| Coal sample | Vitrinite reflectance | Vitrinite, % | Liptinite, % | Inertinite, % | Minerals, % |
|-------------|-----------------------|--------------|--------------|--------------|-------------|
| East Khoot  | 0,34                  | 81,00        | 4,00         | 4,00         | 11,00       |
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Fig. 3. TG analysis of East Khoot coal

Рис. 3. ТГ-анализ угля месторождения «Восточный Хот»

Table 3

| Mgo | Al_{2}O_{3} | SiO_{2} | P_{2}O_{5} | SO_{3} | K_{2}O | CaO | MnO | Fe_{2}O_{3} | TiO_{2} | SrO |
|-----|-------------|---------|-----------|--------|-------|-----|-----|------------|--------|-----|
| 7.8 | 13.0        | 30.5    | 0.02      | 13.6   | 0.9   | 13.1| 0.1 | 18.2       | 0.6    | 0.2 |

The chemical compositions of East Khoot coal ash are shown in Table 3.

The data in Table 2 show that highest content is made up of SiO_{2} and Fe_{2}O_{3}; the brown colour of ash is due to the high Fe_{2}O_{3} content. The mass ratio between the sum of oxides (Fe_{2}O_{3}+CaO+MgO +Na_{2}O+K_{2}O/SiO_{2}+Al_{2}O_{3}+TiO_{2} =0.95 <1) which indicates the acidic character of East Khoot coal ash.

The thermal decomposition of East Khoot coal in nitrogen atmosphere was characterised by means of TG analysis. The TG analysis of East Khoot coal is shown in Fig. 3.

The heating of the East Khoot coal sample at temperatures in the range of 25–1000 °C in a nitrogen atmosphere shows that the thermal decomposition of coal ends with a 45% weight loss and 55% hard residue at 1000 °C (Fig. 3). The TG curve in Fig. 3 consists of different temperature intervals (steps) such as 25–250, 250–550, 550–850 and 850–1000 °C. In the first step (25–250 °C), the weight loss is due to the release of some absorbed gas and moisture from the coal sample. In the second step (250–550 °C), intensive thermal decomposition of the organic matter of the coal samples starts forming liquid (tar and pyrolysis water) and gas products. In the third step (550–850 °C),
there is a strong decrease in the weight loss, which indicates the end of thermal decomposition and start of carbonisation of the coal sample. In the fourth step (850–1000 °C) the rate of weight loss slightly increases, which is related to the release of gas, e.g. CO₂, H₂, CO from the mineral matter of the coal sample. From the TG curve in Fig. 3, the following thermal stability indices of the East Khoot coal have been determined as T₅% = 83.1 °C; T₁₅% = 314.5 °C; T₂₅% = 434.6 °C. The first minimum peak of DTA at 160 °C shows an endothermic reaction process associated with the release of adsorbed gas and moisture from the coal sample. The large exothermic reaction peak at 400 °C is related to the intensive thermal destruction of the organic mass of the coal sample. From the DTG curve it can be seen that the release of moisture and adsorbed gas at about 100 °C – as well as the thermal destruction reactions of coal organic mass at about 500 °C – are taking place at the maximum rate.

The Khoot coal sample was pyrolysed in a quartz retort at 200 °C to 700 °C for 80 min (shown Fig. 4).

![Graph](image1.png)

**Fig. 5. FTIR spectra of liquid tar product following pyrolysis**

**Рис. 5. ИК-спектры смолы пиролиза на основе преобразования Фурье**

![Graph](image2.png)

**Fig. 6. GC/MS chromatography of soluble in hexane fraction of the pyrolysis tar of East Khoot coal**

**Рис. 6. ГХ/МС хроматография растворимой в гексане фракции смол пиролиза угля Восточного Хота**
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Yields of tar fractions according to silica gel chromatography

Таблица 4
Выход фракций смол с использование хроматографии на силикагеле

| Tar | Hexane soluble fraction (%) | Benzene soluble fraction (%) | Dichloromethane soluble fraction (%) | All separated fractions (%) |
|-----|------------------------------|------------------------------|-------------------------------------|----------------------------|
| East Khoot coal | 3.97 | 29.03 | 16.01 | 49.01 |

The most suitable pyrolysis temperature for Khoot coal was determined to be 500–550 °C. The sample was pyrolysed in a larger retort at 500–550 °C and the yield of pyrolysis products determined as follows: char (57.1%), tar (9.9%), pyrolytic water (14%) and gas (19.5%). The yield of all liquid and gaseous products (43%) shows that there is an intensive thermal decomposition of the coal organic mass with higher degree of conversion. As it is known that the organic mass of lignite and low rank brown coal is characterised with lower thermal stability than bituminous coal and therefore they are more suitable for gasification and liquefaction.

The East Khuut coal of liquid tar product after pyrolysis FTIR analyses are shown in Fig. 5.

In the FTIR spectra of tar product after pyrolysis, several absorption bands were observed for H of aromatic -CH group at, 698, 752 cm$^{-1}$ and for H of aliphatic -CH; -CH$_2$ and -CH$_3$ groups with middle intensity at 1249 cm$^{-1}$ and sharp bands with the highest intensity at 2854–2923 cm$^{-1}$. Strong absorption bands were additionally observed for >C=O groups at 1596 cm$^{-1}$ and for -O- groups at 1454 cm$^{-1}$. A less sharp band for H of -OH and -NH groups was observed at 3387 cm$^{-1}$. Therefore, tar product of East Khoot coal following pyrolysis consists of mainly aliphatic, aromatic and aromatic-aliphatic hydrocarbons.

The pyrolysis tar of East Khoot coal was subjected to silica gel chromatography, separation of soluble in hexane, benzene and dichloromethane fractions and the chemical composition of each fraction determined by GC/MS chromatography (Table 4 and Fig. 6, 7 and 8).

The results of silica gel chromatography of coal tar (Table 4) show that most of the tar is soluble in benzene while a smaller part is soluble in hexane, indicating that the majority of the tar consists of aromatic hydrocarbons.

Fig. 7. GC/MS chromatography of soluble in benzene fraction of the pyrolysis tar of East Khoot coal

Рис. 7. ГХ/МС хроматография растворимой в бензоле фракции смол пиролиза угля Восточного Хота

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Fig. 8. GC/MS chromatography of soluble in dichloromethane fraction of the pyrolysis tar of East Khoot coal

Рис. 8. ГХ/МС хроматография растворимой в дихлорметане фракции смол пиролиза угля Восточного Хота

Fig. 9. SEM photographs of: a – Khoot coal; b – activated carbon of pyrolysed char; c – pyrolysed char

Рис. 9. (СЭМ) изображения: a – пробы угля месторождения «Хот», b – активированного угля; c – кокса

Table 5

| GC/MS compositions of tar | Hexane soluble fraction (H) | Benzene soluble fraction (B) | Dichloromethane soluble fraction (M) |
|---------------------------|-----------------------------|------------------------------|-----------------------------------|
| GC/MS chromatograms       | All registered peaks | All identified peaks | All registered peaks | All identified peaks | All registered peaks | All identified peaks |
| 50                        | 32                          | 100                         | 50                              | 62                     | 30                     |

Registered and identified peaks of GC/MS chromatograms of each fractions

Пикы ГХ/МС хроматограмм
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Table 6

| Sample of East Khoot coal | Moisture, % | Ash, % | Volatile matter, % |
|---------------------------|------------|--------|--------------------|
| Initial coal sample       | 11,0       | 8,9    | 46,7               |
| Hard residue after pyrolysis | 0,8       | 12,8   | 8,8                |

All registered and identified peaks of GC/MS analysis for pyrolysis tar of East Khoot coal soluble in hexane, benzene and dichloromethane fractions are given in Table 5 and the names of all identified peaks are in appendix-1.

As a result of GC/MS analysis of pyrolysed tar of East Khoot coal soluble in hexane, benzene and dichloromethane fractions, 32 (from all registered 50 hexane soluble fractions), 50 (from all registered 100 benzene soluble fractions) and 30 (from all registered 62 dichloromethane soluble fractions) organic substances were determined and identified (Appendix 1).

The next step of our work is to determine the characteristics of the obtained char after pyrolysis to compare with the results of initial coal samples given in Table 6.

The data in Table 6 show that the content of moisture and volatile matter of char following pyrolysis decreased by between 10,0 and 5,3 times than that of initial coal sample, an indication for the intensive thermal decomposition of the coal organic mass.

One of the most important applications of the char after thermal processing is to produce activated carbon. For this reason, the char produced after pyrolysis was activated by preheated water steam at 800 °C for 120 min.

The images of scanning electron microscopes (SEM) of prepared activated carbon from pyrolysed char in comparison with initial coal sample and pyrolysis char are different as presented in Fig. 9. For example, the SEM image of initial coal sample has compact solid pieces. The SEM images of carbonised activated carbon sample show porosity structure while the SEM image of char after pyrolysis shows micro porous structure in comparison with that of activated carbon sample.

For example, the SEM image presented in Fig. 9, a of initial coal sample has compact solid pieces. The SEM images of carbonised and activated carbon sample shows (Fig. 9, b) shows the macro-porous structure, while the SEM image of char after pyrolysis (Fig. 9, c) shows the micro-porous structure in comparison with that of the activated carbon sample.

CONCLUSIONS

1. On the basis of proximate, ultimate, petrographic and FTIR analysis, it can be concluded that East Khoot coal is a low rank (B2) brown coal, which is suitable for pyrolysis.

2. On the basis of thermogravimetric analysis of East Khoot coal, the following thermal stability indices of the East Khoot coal have been determined as \( T_{5\%} = 83,1 \, ^\circ C; \ T_{15\%} = 314,5 \, ^\circ C; \ T_{25\%} = 434,6 \, ^\circ C. \) These thermal stability indices show that East Khoot coal has lower thermal stability and a bid exothermic reaction peak at 400 °C related with intensive thermal destruction of the coal organic mass of the sample.

3. The results of the pyrolysis experiment of East Khoot coal show that 57.1% of the organic mass of the coal remained as a hard residue following pyrolysis. The yield of liquid and gas products is 22.9%, showing that there was an intensive thermal decomposition of the coal organic mass with higher degree of conversion.

4. The results of X-ray fluorescence analysis of East Khoot coal ash shows that the SiO₂ and Fe₂O₃ are the most significant constituent compounds with the brown colour of the ash being due to the high Fe₂O₃ content. The mass ratio between sum of oxides (Fe₂O₃ + CaO + MgO + Na₂O + K₂O/SiO₂ + Al₂O₃ + TiO₂ = 0,95 < 1) which is an indication of acidic character of the ash of East Khoot coal.

5. The FTIR spectra of tar after pyrolysis has several absorption bands for H of aromatic -CH group at 698.752 cm⁻¹ and for H of aliphatic -CH; -CH₂ and -CH₃ groups with middle intensity at 1249 cm⁻¹ and a sharp band with the highest intensity at 2854-2923 cm⁻¹. Strong absorption bands for >C=O groups were also present at 1596 cm⁻¹ – and, for -O- groups, at 1454 cm⁻¹. A less sharp band for H of -OH and -NH groups is observed at 3387 cm⁻¹. Therefore, the tar of East Khoot coal consists of mainly aliphatic, aromatic and aromatic-aliphatic hydrocarbons.

6. As a result of GC/MS analysis of pyrolysed tar of East Khoot coal soluble in hexane, benzene and dichloromethane fractions, 32 (from all registered 50 hexane soluble fractions), 50 (from all registered 100 benzene soluble fractions) and 30 (from all registered 62 dichloromethane soluble fractions) organic substances were determined and identified (Appendix 1).

7. The SEM image of initial coal sample shows compact solid pieces, while the carbonised and activated carbon samples consist of hard material with a highly-developed macro- and micro-porous structure.
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Contribution

Purevsuren B., Bazarova J.G., Bazarov B.G., Batbileg J., Namkhainorov B., Batkhishig D., Battsetseg M., Dorzhieva S.G. carried out the experimental work, on the basis of the results summarized the material and wrote the manuscript. Purevsuren B., Bazarova J.G., Bazarov B.G., Batbileg S., Namkhainorov B., Batkhishig D., Battsetseg M., Dorzhieva S.G. have equal author’s rights and bear equal responsibility for plagiarism.

Conflict of interests

The authors declare no conflict of interests regarding the publication of this article.

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