Investigation of the Laser Radiation Effects on Lignite with the Products Analysis by Mass Spectrometry

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Abstract

In this paper, one studied the effect of pulsed laser radiation (1064 nm, 120 μs) on lignite. The method of mass spectrometry was used to analyze the formed gaseous reaction products after the exposing of coal samples to the laser radiation. The pressed out of the pre-milled coal particles tablets were used as samples. In the mass spectra of the reaction products of laser radiation of the samples, the peaks with a mass to ion charge ratio m/z = 2, 18, 44 were detected, that corresponds to the main peaks of H₂, H₂O, and CO₂ molecules. The pattern of formation of the end products CO₂, H₂, H₂O when heating a sample of coal by laser radiation is proposed. The energy release factor is calculated, which is defined as the ratio of the calorific value, released during combustion of the sample to the dose of energy absorbed by the sample during irradiation.

1. Introduction

The coal particles ignition is a combination of physicochemical processes occurring simultaneously, viz. moisture evaporation, heat transfer and mass transfer, thermal decomposition of coal particles, chemical interaction of pyrolysis products with water vapor, oxidation of gaseous combustible components on the coal particles surface, etc. [1].

The laser radiation is one of the promising tools for initiating the coal particles’ ignition process [2‒6]. The coal combustion is a multi-step process consisting of chemical transformations in the condensed and gas phases. The complexity of studying the mechanism of this process is due to the high rates of chemical reactions occurring simultaneously in the solid and gaseous phase and the interfacial space, as in the case of high-energy materials combustion [7].

As a result of laser radiation on the coal substance, the fast-flowing thermochemical processes are initiated in it, leading to the release and ignition of volatile matters followed by the carbon skeleton (backbone) combustion. The electro-optical methods were used to record this complex process of igniting coal samples [8‒16].

Three stages of the kinetics of laser ignition of coal with a wide dispersion of 0.2 to 100 μm were established in [12‒16]. Each stage has a pronounced threshold character. For each stage, the ignition thresholds H_{cr} (critical energy density corresponding to 50% of the ignition probability) are determined. Figure 1 shows the dependences of the ignition probability P on the laser energy density H.

The first stage manifests itself (appears) during the laser pulse duration of 120 μs with a threshold energy density H_{cr}^{(1)} = 0.6 J/cm². The second stage involves the release and ignition of volatile matters in the time interval of 0.5–4 ms at a threshold energy density of H_{cr}^{(2)} = 1.9 J/cm². At the third stage, a vertical flame with a height of up to 10 cm is observed above the coal sample with a threshold energy density of H_{cr}^{(3)} = 2.8 J/cm². The glow duration reaches 100 ms. The flame occurrence is associated with the rapid heating of the sample (the first stage) that results in emission and ignition of the volatiles (the second stage) with the subsequent formation and combustion (burning) of the coke residue of the coal particle.

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Thus, [12‒16] show that the process of ignition and combustion of a coal particle has three stages: the heating of a coal particle as a result of the influence of laser radiation and the subsequent development of the thermochemical reactions in a sample, the emission and ignition of the volatiles, the combustion of the coke residue of a coal particle. When exposed to the powerful laser radiation on coal particles, several physical and chemical processes are superimposed, viz. heating the coal skeleton, diffusing and desorbing hydrocarbons, the combustion of hydrocarbon molecules on the surface, desorbing the light hydrocarbons and their combustion in the gas phase, the combustion of coke residue.

The study of the mechanism of reactions occurring during the coal fuel combustion is a complex task. It includes a detailed analysis of the combustion products in the gas phase as one of the important areas of research. In this study, the molecular composition of the gaseous reaction products produced by the exposure of the laser radiation on the lignite samples was studied by mass spectrometry.

2. Object and methods

The brown coal of the Kaychaksky field (Kuznetsk coal basin, the Russian Federation) was used in the work with the following characteristics: the moisture content was $W_a = 11.1\%$, the ash content was $A_d = 9.5\%$, the volatile matters content was $V_{daf} = 51.4\%$, the carbon content was $C_{daf} = 61.4\%$, and the hydrogen content was $H_{daf} = 5\%$.

For the preparation of samples, the coal was pre-ground in a ball mill, then it was sieved through a sieve with a mesh size of 100 microns. Further, using the method of pressing in a special mold, the coal samples were obtained in the form of tablets with a diameter of 4.2 mm, thickness of 4.7 mm and weight of 65 mg.

As a source of laser radiation, YAG:Nd$^{3+}$-laser (SOLAR Laser Systems LQ929, Minsk) was used, operating in free-running mode at a wavelength of $\lambda = 1064$ nm. The pulse duration was 120 $\mu$s. The energy density of the laser pulse in the experiments was 1.5 J/cm$^2$. The laser radiation energy was determined with the use of PE50BF-C pyroelectric energy meter (Ophir® Photonics). The diameter of laser beam was 4.2 mm. It coincides with the sample diameter. The instability of the laser pulse energy did not exceed 3%.

The experimental unit is shown in Fig. 2. The laser radiation (L) is attenuated by the neutral light filters (1) then it is focused on the sample (7) with the help of a deflecting mirror (3) and a lens (4). By the beam splitter plate (2), a part of the laser radiation energy was diverted to a calibrated photodiode (P) to control the laser pulse energy.

The recording of gaseous products was carried out via a gas analyzer (MS) SRS QMS 300. The products were formed in the reaction of the laser radiation action on the samples. A feature of the analyzer is the measurement becomes possible under the pressure of the analyzed gases in the experimental chamber in the range of $10^3$–$10^5$ Pa. Therefore, the experiments were carried out in a hermetic chamber with a reduced initial pressure of $0.9\cdot10^5$ Pa in air. The ionization of gases in the analyzer is carried out using an electron impact ionization.
with an energy of 70 eV. The ionizer converts the investigated gaseous products into ionized gas. The gas is collected into a quadrupole mass filter by electric fields. The ions passing through the filters are measured by a detector. The detector is a Faraday cup or a secondary electron multiplier. The mass spectra were measured in the range from 1 to 300 AMU.

The mass spectra of vapors of chemically-pure benzene, hexane and toluene were studied as a control experiment. Figure 3 shows the mass spectra of vapors of chemically pure benzene, hexane and toluene in comparison with the mass spectra taken from the NIST database.

The obtained mass spectra correlate well with the NIST data. The intensity ratio of the benzene base peak (m/z = 78) to the intensity of the mass peaks of the daughter ions (m/z = 51, 52, 50) is: 78/51 = 4.59, 78/52 = 5.13, 78/50 = 5.40, which is close to the NIST data: 78/51 = 4.54, 78/52 = 5.02, 78/50 = 5.24. The intensity ratio of the hexane base peak (m/z = 57) to the intensity of the mass peaks of the daughter ions (m/z = 43, 41, 29) is: 57/43 = 1.23, 57/41 = 1.27, 57/29 = 1.59, according to NIST: 57/43 = 1.29, 57/41 = 1.30, 57/29 = 1.64. The intensity ratio of the toluene mass peak (m/z = 91) to the intensity of the mass peaks of the daughter ions (m/z = 39, 65) is: 91/39 = 5.20, 91/65 = 6.43, according to NIST: 91/92 = 1.45, 91/39 = 5.01, 91/65 = 6.68. The intensity ratio of the toluene base peak (m/z = 91) to the intensity of the mass peak with m/z = 92 is: 91/92 = 1.41, according to NIST: 91/92 = 1.45. Thus, one can conclude that the obtained spectra are in accordance with the NIST catalog.

The sample was placed in a hermetic experimental chamber (position 6, Fig. 2) for the experiment. The initial pressure of 0.9·10^5 Pa was created in the chamber.

The pulse repetition rate of the laser radiation varied for each series of experiments. The laser pulse was characterized by the following parameters: the pulse energy was 210 mJ, the energy density was 1.5 J/cm^2, the pulse duration was 120 μs, the pulse power was 1.75 kW, the pulse power density was 12.5 kW/cm^2.

In the first series of experiments, the pulse repetition rate of laser radiation was 3 Hz. The total exposure time of laser pulses and the analysis time in the accumulation mode of the reaction products of exposure was 20 min. During this time, the sample was exposed to 3600 laser pulses. The total dose of laser energy acting on the sample was 0.75 kJ (the total energy density was 5.4 kJ/cm^2).

Fig. 3. The mass spectra of vapors of chemically pure benzene, hexane and toluene in comparison with the mass spectra taken from the NIST database.
Under similar conditions, a series of experiments were conducted at the laser pulse repetition rates of 6 and 9 Hz. For experiments with a pulse repetition rate of 6 Hz, the total dose of laser energy acting on the sample was 1.5 kJ (the total energy density was 10.8 kJ/cm²). For experiments with a pulse repetition rate of 9 Hz, the total dose of laser energy acting on the sample was 2.3 kJ (the total energy density was 16.2 kJ/cm²). After the cessation of exposure of laser radiation, the sample was kept up in the chamber within 5 min. For preparation for the next experiment, the experimental chamber was depressurized and purged within 20 min for cleaning the products of the previous experiment.

The gas sampling from the experimental chamber was carried out through a capillary tube (position 8, Fig. 2) with an inner diameter of 0.125 mm and a length of 1.2 m. The capillary was connected with aperture adapters to a mass spectrometer (MS, Fig. 2). Its working chamber created a pressure of 10⁻³ Pa using a system of pumps and valves. The delivery flow rate of gaseous products into the working chamber of the mass spectrometer was 1 ml/min. The gas sample was taken continuously. The analysis was initially carried out in the range of AMU from 1 to 300. The high-molecular peaks were not detected in the recorded mass spectra of the reaction products of the laser radiation exposure on coal samples. The further analysis was performed in the range of AMU from 1 to 100. The required time was 12 s for one analysis. Data of the analysis were transferred to a computer where were registered by means of the special software provided with the device.

3. Result and discussion

Before conducting the experiments, the background mass spectrum of gaseous materials was recorded in the experimental chamber. The technique of measuring the background mass spectrum is as follows: the sample was placed in an experimental chamber, the experimental chamber was sealed, the mass spectrum of gaseous materials was recorded in the chamber without any exposure to laser radiation.

Figure 4 illustrates the example of mass spectra of the main reaction products when exposed to laser radiation with a pulse repetition rate of 6 Hz at time 1000 s after the start of exposure.

![CO₂ mass spectrum](image)

![H₂ mass spectrum](image)

![H₂O mass spectrum](image)

Fig. 4. The example of mass spectra of the main reaction products when exposed to laser radiation with a pulse repetition rate of 6 Hz at time 1000 s after the start of exposure: (a) – CO₂; (b) – H₂; (c) – H₂O. In the inserts for clarity, peaks with low intensity are shown enlarged in scale by 85 (a), 280 (c) times.
The peaks with mass numbers m/z = 44 (CO₂⁺), 28 (CO), 16 (O), 12 (C), 22 (CO₂⁺) were registered in the mass spectra of the gas phase above the coal sample. The intensity values of similar peaks taken from the background mass spectrum were subtracted from the intensity values of these peaks (Fig. 4a) for further studies. A comparison of the relative intensities of these peaks with catalog values let conclude that these peaks correspond to carbon dioxide CO₂ with the base peak m/z = 44.

The fairly high values of peaks’ intensities with the mass numbers m/z = 2 (H₂) and 1 (H) were simultaneously recorded in the experiments. The values indicated the formation of hydrogen in the reaction mixture (Fig. 4b).

The high intensities of peaks with the mass numbers m/z = 18 (H₂O), 17 (OH⁻), 16 (O) were also recorded (Fig. 4c). These peaks correspond to the water vapors with the base peak m/z = 18. The high peaks’ intensities corresponding to water vapors indicate the following processes: the moisture evaporation from the sample and the initiation of combustion of hydrocarbon molecules desorbed from the sample with the formation of carbon dioxide and water.

The registered products comprised the increased content of hydrogen and carbon dioxide that can be attributed to the possible course of reactions of the conversion of water vapor at the transmission over the red-hot carbon [17].

\[
\begin{align*}
C + H₂O & \rightarrow CO + H₂ + 131.3 \text{ kJ/mol} \quad (1) \\
CO + H₂O & \rightarrow CO₂ + H₂ - 41.2 \text{ kJ/mol} \quad (2)
\end{align*}
\]

According to [17], the reactions (1, 2) can proceed at a temperature exceeding 1000 °C. The hydrogen formation was detected during the coal pyrolysis in several works, e.g. by passing a mixture of nitrogen and water vapor through a coke sample in the temperature range 800–1400 °C [18], during laser pyrolysis in vacuum (10⁻³ Pa) in the temperature range 240–800 °C [19].

The work [20] showed that the heating temperature of surface a carbon sample could reach 1050–1150 °C due to the continuous exposure to the laser radiation of CO₂. The heating temperature of surface a carbon sample can reached higher values under the pulsed exposure. Thus, the surface temperature of the carbon sample during the laser pulse exposure was ~ 2700 °C in [15]. It was determined by spectral pyrometry method.

Figure 5 shows the dependence of the base peaks’ intensity of the gaseous reaction products on the time of exposure to the laser radiation at the various frequencies of pulse repetition.

Let us offer the pattern of formation of the end products CO₂, H₂, H₂O when heating a sample of coal by laser radiation.

The coal material consists of a carbonic hydrocarbon skeleton (C) filled with a mixture of individual hydrocarbons (CₙHₘ). Therefore, the main macro chemical reactions of the components of oxidation (combustion) can be written as follows:

\[
\begin{align*}
2C + O₂ & \rightarrow 2CO - 110.5 \text{ kJ/mol} \quad (3) \\
C + O₂ & \rightarrow CO₂ - 393.5 \text{ kJ/mol} \quad (4) \\
CₙHₘ + \left( n + \frac{m}{2} \right) O₂ & \rightarrow nCO₂ + \left( \frac{m}{2} \right) H₂O \quad (5) \\
C + H₂O & \rightarrow CO + H₂ + 131.3 \text{ kJ/mol} \quad (6) \\
CO + H₂O & \rightarrow CO₂ + H₂ - 41.2 \text{ kJ/mol} \quad (7) \\
C + CO₂ & \rightarrow 2CO + 172.5 \text{ kJ/mol} \quad (8)
\end{align*}
\]

The interaction of carbon and oxygen can occur along with the formation of CO and CO₂ according to the reactions (3) and (4). If an excess of carbon appears in the system, oxygen will be completely expended and the gas phase will consist of CO and CO₂. The composition of the equilibrium gas will be determined by the course of the reaction (8) under these conditions.

The heating of carbon skeleton leads to the release of individual hydrocarbons. Their interacting with atmospheric oxygen lead to combustion with the formation of carbon dioxide and water molecules (reaction (5)).

When water vapor interacts with red-hot coal, carbon monoxide and molecular hydrogen can form (reaction (6)). In this case, the molecular hydrogen is the end product of reaction (6).

In turn, carbon monoxide of reaction (6) can react with water vapor of reaction (5) to form the end products of carbon dioxide and hydrogen (reaction (7)).

Thus, the example of carbon and a mixture of hydrocarbons prove the mechanism of oxidation (combustion) of a multicomponent material as a complex multi-stage process that involves parallel or sequential endo- and exothermic reactions. This is fair even at the elementary reactions.

The mechanism of oxidation reactions of coal material under laser radiation exposure is definitely more complex process than we describe through reactions 3–7. The series of experimental and thermodynamic studies are in progress.
Probably, the formation of the end products \( \text{H}_2, \text{H}_2\text{O}, \text{CO}_2 \) (they are experimentally registered in this work) requires the courses of several dozen parallel-serial reactions with the formation of intermediate gaseous products with a high-molecular weight.

A study of the effectiveness of laser-induced combustion of coal was conducted. The methodology of the experiment is as follows. 10 samples were sequentially irradiated with laser pulses with the pulse repetition rate of 3, 6 and 9 Hz for 500 s. The sample was weighed after irradiation. After that, the proportion of the reacted sample per unit mass was determined:

where \( m_1 \) and \( m_2 \) are the masses of the sample before and after irradiation, respectively;

The dependence of the mass loss of a coal sample on the pulse repetition rate of laser radiation is shown in Fig. 6.

The Fig. 6 shows that prolonged exposure to laser radiation with the pulse repetition rate of 3 Hz for 500 s leads to the combustion of up to 67% of the mass of the sample. During this time, the sample was exposed to 1500 laser pulses. The total dose of laser energy acting on the sample was 0.32 kJ.

The gross calorific value \( Q'_g \) of the coal used is 15.6 MJ/kg [21]. During the combustion of 67% of the sample mass (43.5 mg), \( Q'_g = 0.68 \) kJ is released. Thus, we can conclude that the energy release factor \( k \), which is defined as the ratio of the calorific value, released during combustion of the sample, to the dose of energy absorbed by the sample during irradiation, could be estimated as 2.13.
Exposure at a pulse repetition rate of 9 Hz for 500 s leads to combustion of up to 90% of the mass of the sample, which corresponds to the ash content in the sample (A\textsubscript{d} = 9.5%). During 500 s, the sample was exposed to 4500 laser pulses. The total dose of laser energy acting on the sample was 0.95 kJ, calorific value \( r_Q \), released during the combustion of the sample is 0.91 kJ, that is \( k \sim 1 \). Such a significant decrease in the energy release factor is probably due to the shielding of the surface of the coal sample, which can be carried out as follows:

1. An increase in the pulse repetition rate leads to the formation of coal aerosol over the sample surface, which leads to its shielding;

2. Shielding of the surface of the sample with ash, formed as a result of coal combustion.

An additional experiment was carried out to establish the effect of the pulse repetition rate of laser radiation on the value of \( m \). The proportion of the reacted sample per unit mass was determined for the same dose of total energy 0.32 kJ at various pulse repetition rates. The exposure time with a pulse repetition rate of 3 Hz was 500 s, 6 Hz – 250 s, 9 Hz – 170 s. Regardless of the pulse repetition rate of laser radiation at total energy of 0.32 kJ, constant values \( m = 0.66–0.71 \) are observed. Therefore, if the effect of shielding of the surface of the sample by particles in the gas phase is realized it is very insignificant. Thus, presumably, shielding of the surface of the sample is carried out by ash, formed as a result of coal combustion.

The experimental data presented are obtained without optimization of the combustion chamber, the oxidizing gas medium, the duration and energy density of laser pulses. This makes it possible to increase the efficiency of laser-induced combustion of coal both in the degree of reacted mass of the sample and in the ratio of the released chemical energy to the deposited energy of the laser pulses.

4. Conclusions

1. The thermochemical processes of conversion of lignite of the Kaychaksky field (Kuznetsk coal basin, Russia) were studied under the pulsed laser radiation. The lignite possessed the moisture content \( W^a = 11.1\% \), the ash content \( A^d = 9.5\% \), the volatile matters’ content \( V^\text{daf} = 51.4\% \), the carbon content \( C^\text{daf} = 61.4\% \), the hydrogen content \( H^\text{daf} = 5\% \). The surface of lignite samples was exposed to pulsed laser radiation (1064 nm, 120 μs) and heated to high temperatures (\( T > 1000 \) °C). At these temperatures, the thermochemical transformation of the coal material occurred along with the desorption of water vapor and high molecular weight hydrocarbons following to the oxidation reactions.

2. The mass spectrometric analysis of the gas phase above a lignite sample with the pulsed laser radiation exposure showed that the main products of the reaction are carbon dioxide and water molecules. A significant content of carbon monoxide and hydrogen molecules is also observed in the reaction products. In general, the formation of reaction products increases up to 500 s exposition and then stays roughly constant in the interval 500–1200 s.

3. The hydrogen formation in the process of laser radiation exposure to lignite samples is a matter of special interest and the main reaction occurrence is proposed as interaction of water vapor with carbon at the temperatures above 1000 °C.

4. The energy release factor \( k \), which is defined as the ratio of the calorific value, released during combustion of the sample, to the dose of energy absorbed by the sample during irradiation, could be estimated as 2.13.

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