Experimental study of the dynamics of microwave pyrolysis of peat

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Abstract. The work is devoted to the study of the effect of high-power microwave radiation on sphagnum peat. To implement the microwave pyrolysis process, a laboratory unit based on a coaxial resonator has been created. An industrial magnetron with a frequency of 2.45 GHz was used as a source of microwave radiation. Samples of gas, liquid and solid phases were obtained and analyzed. Studies of soft microwave pyrolysis in conditions of constant removal of gaseous reaction products were carried out. A comparative analysis of the products obtained in microwave pyrolysis and pyrolysis with thermal heating was performed. The aim of the research is to create highly efficient environmentally friendly technologies for processing biofuels with a high yield of combustible gases, suitable for further use in power plants, as well as the production of resinous fraction and carbon residue.

1 Introduction: goals and objectives of the study

Currently, scientific research in the field of physical and chemical processing of organic raw materials is becoming increasingly important. Pyrolysis of organic substances and materials: oil, coal, peat, wood, agricultural and industrial waste, household waste is considered as one of the promising industrial applications of high-power microwave radiation [1-7]. One of the materials, which has a wide variety of its practical applications, including processing by pyrolytic destruction, is peat. Peat is a renewable natural bioresource, which is formed as a result of natural dying and incomplete decay of marsh plants under the influence of biological processes in conditions of high humidity and lack of oxygen [8].

Pyrolysis - thermal degradation of organic compounds on hydrocarbons with a lower molecular weight when exposed to high temperature without oxygen. Depending on the chemical composition of the raw material and the physico-chemical conditions of the reaction (temperature, pressure, humidity, the degree of presence of oxygen, etc.), the pyrolysis process has a specific character with the release of various solid, liquid and gaseous substances [9]. One of the most important parameters affecting the pyrolysis process is the temperature in the reactor. Depending on this, there are three types of

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pyrolysis: soft pyrolysis occurs at temperatures up to 500°C, normal (average) – 500-800°C and hard pyrolysis-800-1100°C. the temperature determines the degree of destruction of the starting substances and the composition of the pyrolysis products. With increasing temperature, the ratio between the three phases of the decomposition products changes: the yield of liquid and solid (semi-coke in the case of soft pyrolysis, coke in the other two cases) residues becomes less and the yield of gases with less and less heat of combustion increases.

The most promising from an environmental point of view is currently the extraction of organic components as a result of the so-called microwave pyrolysis, since the process of heating the microwave radiation does not carry any pollution either in the processed material or in the environment. The most important advantage of microwave exposure is also that electromagnetic waves penetrate to a considerable depth into the processed material, and when absorbed, they create a volumetric distribution of heat sources, thus directly converting microwave energy into heat in materials with low thermal conductivity. Thus, the advantage of microwave pyrolysis is the ability to quickly adjust the rate of pyrolytic reactions and the composition of the products at the output. In the case of a traditional thermal pyrolysis scheme, the thermal conductivity of materials is essential and affects the rate of heat supply to the reaction region.

The paper presents the results of studies on the destruction of peat in the process of microwave pyrolysis. Laboratory setup, which uses a radiation source operating at industrial frequencies of 2.45 GHz / 1 kW, a magnetron with an adjustable level of microwave output power. Studies of the microwave pyrolysis in conditions of constant removal of gaseous reaction products were carried out. To perform a comparative analysis of the products obtained in microwave pyrolysis and pyrolysis with thermal heating.

2 Laboratory setup and experimental technique

To study the process of microwave pyrolysis, a laboratory setup based on a coaxial resonator-reactor was constructed in IAP RAS, which allows to conduct experiments on RF peat pyrolysis at a relatively low level of the microwave power. The choice of such an experimental scheme is associated with a significant simplification of the interpretation of the experimental results for the construction of a mathematical model of microwave pyrolysis and its subsequent verification.

The laboratory setup is shown in Fig.1 [9]. The source of microwave radiation is an industrial 2.45 GHz magnetron (1) with a high-voltage power supply (2), which allows to set a predetermined level of output microwave power. Transmission of radiation from the magnetron to the reactor is carried out by means of a flexible coaxial cable (3). The reactor is a coaxial resonator (4) filled with peat. To reduce the reflection coefficient of radiation from the loaded resonator-reactor at the operating frequency of 2.45 GHz, the initial setting of the optimal resonator length is performed. To control the reflected signal during the process of the microwave pyrolysis, a directional coupler (5) is included in the coaxial path. The temperature was measured at the outer boundary of the peat using a thermocouple (6).

Reactor loading with sphagnum peat at 50% humidity averaged 100 grams. In this series of experiments, the power of microwave radiation was set to about 100 watts. This limitation is due to the thermal heating of the coaxial input of the resonator. The reflection coefficient from the resonator during pyrolysis did not exceed -10 dB. Pyrolysis products were removed from the opposite (relative to the microwave input) side of the resonator through the pumping port (7), liquid and oily fractions were deposited in the sump, and pyrolysis gas was deposited and fixed in the cryogenic trap. A pressure gauge (8) and a gas meter (9) were used to monitor the gas pumping process.

The advantage of this laboratory design (with a small amount of fuel) is the ability to quickly reach the temperature necessary for the soft pyrolysis reaction at a relatively low power level specified above. Heating on the external peat circuit to a temperature of 250°C was carried out for 10-15 minutes.it should be noted that the feature of the experimental setup used is a significant inhomogeneity of the microwave field in the reactor volume, which leads to inhomogeneity of fuel heating inside the reactor volume.



Fig. 1. Experimental setup of microwave pyrolysis of peat with coaxial resonator: (1) microwave source - industrial 2.45 GHz magnetron, (2) hvps, (3) the input device of microwave power on the basis of coaxial cables, (4) laboratory of microwave-rector on the basis of the coaxial resonator, (5) directional coupler, (6) thermocouple, (7) evacuation data analysis port with cooling-the duration of the exhaust gas fractions, (8) manometer, (9) gas meter.

3 Experimental result

The analysis of the gas and liquid fractions obtained during the reaction was performed on a chromatography-mass spectrometer GCMS QP2010 Ultra (Shimadzu USA). The chemical composition was determined by the peaks of molecular and characteristic fragmentation ions.

The results of chromatography-mass spectrometry analysis were processed using Shimadzu proprietary software (GCMS Solution ver. 4.11), the identity of the impurities was using the database 11 NIST (The National Institute of Standards and Technology, U.S. Department of Commerce) [9]. The results of the analysis of the gas sample are presented in table 1.

Table 2 presents the results of the carbon residue after microwave treatment in comparison with conventional thermal heating. The percentage yield of carbon in microwave heating is higher than in conventional heat, which indicates the prospects of the proposed approach. At the same time, according to the measurements, there are no heavy poisonous gases in the process of microwave pyrolysis experiments. At the same time, the

yield of the gaseous residue of the reaction is slightly increased. Thus, combustible gases can increase the efficiency of the installation by subsequent use of the energy obtained in the combustion process of these gases [9].

The oily fraction was also studied using a chromato-mass spectrometer. The results of the analysis are presented in table 2. Water was excluded from the total mixture and 100% of the mass of the substance was recalculated.

Process	CO2+N2+ect.	Propene	Butene	Furan	Cyclopentadiene	Cyclopentene	Isobutanol	1-hexene	Hexane	2-methylfuran	Isopentane's	benzol	Heptane	n-butyl vinyl ether	Methane	Ethane	Propane	2-methylpropanal
Thermal heating	68,9	15, 5	8,5	3,7	0,9	0,1	0,1	0,5	0,1	0,8	0,1	0,6	0,1	0,2	3,7	0,2	2,6	-
Microwave heating	69,9	-	-	7,7	0,2	-	-	-	0,3	0,3	5,0	0,3	0,9	0,2	-	3,3	0,1	1,0

Table 1. Chromatography-mass spectrometry analysis of pyrolytic gases.

 Table 2. Chromatography-mass spectrometry analysis of oily fraction.

Process	acetaldehyde	methanol	acetal	acetone	Acetic acid	5-methyl- 2-fiuraldehvde	furaldehyde	Propionic 2014	2-formyl- 5 mathulfuron	phenol	cresol	Formic	furfural
Thermal heating	7,33	8,67	8,67	18,67	16,67	10,00	30,00	-	-	-	-	-	-
Microwave heating	1,18	5,69	4,12	23,04	19,02	-	-	7,55	8,43	3,82	0,88	2,25	24,02

4 Conclusion

When using the developed laboratory setup based on a coaxial resonator using an industrial 2.45 Hz magnetron as a power source (the level of input microwave power is about 100 W), experiments were carried out to implement the process of soft pyrolysis of peat at a temperature of about 250 °C in conditions of constant removal of gaseous reaction products. Peat pyrolysis products were obtained and analyzed: valuable carbon residue, oily fraction and pyrolysis gas. The advantages of microwave pyrolysis in comparison with the "traditional" thermal analog are demonstrated. In particular, in the case of microwave pyrolysis, the fraction content is characterized by a much larger set of organic compounds.

The experiments demonstrated high potential of using microwave heating in pyrolysis reactions. As a result of the microwave treatment of peat, a valuable carbon residue, an oil fraction, a pyrolysis gas were obtained and the industrial application of these pyrolysis products was considered. Thus, pyrolysis gas containing methane can be used as a fuel or as a starting material for the production of new products, liquid oily fraction - as a starting material for the separate components, carbon residue (carbon content more than 60%) - in various fields of production, including the production of electrodes for the aluminum industry and grounding conductors, new allotropic modifications of carbon

(nanotubes, fullerenes), carbon fiber. According to the chemical analysis, there are no heavy toxic gases in the composition of the gases released during microwave pyrolysis, which in many ways accompany the process of traditional thermal pyrolysis and complicate (from an environmental point of view) its industrial application.

As a further development of works on microwave pyrolysis, it is also interesting to increase the efficiency of gas release from fossil fuels and, thus, the efficiency of the installation. At the next stage of the work it is planned to modify the reactor design with an increase in the power of microwave heating and the mass of the loaded material, as well as to raise the temperature in the reactor.

The work was performed using equipment of the center "New materials and resource-saving technologies" with the support of the grant "UMNIK-2017" No. 13188/2018.

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