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INFLUENCE OF THE BINDER TYPE AND QUANTITY ON PHYSICAL, MECHANICAL AND THERMAL PROPERTIES OF SHALE WASTE BRIQUETTES

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Abstract: *This study investigates the influence of different binding agents—petroleum pitch, coal tar pitch, and bitumen BND 50/70—on the physicomechanical and thermotechnical properties of shale briquettes produced from fine shale fractions. The results demonstrate that the nature and amount of the binder play a decisive role in the formation of the structural framework, mechanical strength, thermal stability, and energy efficiency of the briquetted fuel. The increase in binder content from 5 to 15 wt.% leads to a consistent rise in density, compressive strength, and calorific value, alongside a decrease in porosity, water absorption, and ash content. Coal tar pitch was found to be the most effective binder, providing the highest mechanical strength (3.0–5.5 MPa), lowest water absorption (8–16%), reduced abrasiveness, and the maximum lower heating value (11.2–13.4 MJ/kg). Its high aromaticity and tendency toward carbonization contribute to the formation of a dense coke residue, ensuring long-lasting and stable combustion. Petroleum pitch exhibited moderate performance in both mechanical and thermotechnical parameters and can serve as a balanced and economically feasible binding agent. Bitumen BND 50/70 resulted in briquettes with the lowest strength, highest porosity and water absorption, and the least stable combustion behavior due to its high volatile content and limited carbonization capability. The obtained results confirm a strong correlation between the chemical nature of the binder, its thermal decomposition behavior, and the resulting performance characteristics of shale briquettes. These findings allow for the targeted optimization of briquette formulations depending on their intended application—ranging from high-strength, thermally stable industrial fuel to low-cost, rapidly igniting briquettes for domestic use.*

Keywords. *shale briquettes; binder; petroleum pitch; coal tar pitch; bitumen BND 50/70; physicomechanical properties; thermotechnical characteristics; thermal stability; energy efficiency; briquette structure.*

INTRODUCTION

In recent years, against the background of the global energy transition, increasing demand for resources and the need to diversify sources of carbon raw materials, there has been a growing interest in technologies for processing unconventional energy resources,in particular, shale. According to the data of geological exploration carried out by the State Committee for the Use of the Subsoil of Azerbaijan and confirmed by a number of scientific publications, the total reserves of oil shale in the country are estimated at about 18-20 million tons, while the main deposits are concentrated in the areas of Guba, Dzhangychai and Diyally [1-3]. Despite this, industrial development of these resources remains limited, due to both technological and environmental factors.

Oil shale is a type of solid fossil fuel containing a significant amount of organic matter-kerogen, which can be converted into liquid, gaseous and solid hydrocarbon products under thermal influence. Due to its wide distribution, including on the territory

of Azerbaijan, oil shale is considered as an alternative and affordable raw material for the fuel and energy and chemical industries [4-6].

One of the most promising areas is their thermal decomposition (pyrolysis, semi-coking, hydro-pyrolysis, etc.), which results in the formation of liquid hydrocarbons suitable for the production of fuel, bituminous materials and resinous components, as well as gaseous products and solid residues potentially applicable in power engineering and construction.

Along with this, thermal processing of oil shale is accompanied by emissions of harmful substances, including nitrogen oxides, sulfur dioxide, phenols and polycyclic aromatic hydrocarbons. For example, when processing 1 million tons of shale in chamber furnaces, the emission of nitrogen oxides can reach 7 tons per year, and when burning shale in power plants-up to 500 tons [7,8]. In addition, increased concentrations of phenols and other toxic compounds are observed in the wastewater of oil shale processing plants [9].

The legislative framework of Azerbaijan in the field of energy and environmental protection provides for the regulation of activities related to the extraction and processing of energy resources. The Law of the Republic of Azerbaijan "On Energy" defines the basic principles and requirements for energy activities, including the need to comply with environmental standards [10] In this regard, the relevance of the research aimed at developing environmentally friendly technologies for thermal processing of oil shale and efficient use of the resulting products is obvious. This will not only expand the country's raw material base, but also minimize the negative impact on the environment, contributing to the sustainable development of Azerbaijan's energy sector.

The aim of the study was to determine the effect of the type and amount of organic binders (oil pitch, coal pitch, and BND 50/70 bitumen) on the physical, mechanical, and thermal properties of shale fines briquettes.

EXPERIMENTAL PART

Based on the results of previous studies, the object of this research is selected oil shales from the Iyimishly field (Azerbaijan) - typicalallagite shales with a high kerogen content [11,12]. Their shale fines up to 6 mm in size were used for briquetting. Petroleum and coal tar pitch, as well as petroleum road bitumen BND 50/70, were used as binders (table 1) in the amount of 5-15% by weight.

The composition was briquetted on a laboratory hydraulic press PGL-20 at a pressure of 10 MPa without wetting. Determination of the strength characteristics of the obtained shale briquettes was carried out according to the standard method (GOST 21289-75) "Coal briquettes. Methods for determining mechanical strength". The briquette was placed between the cylindrical inserts of the press so that the inserts rested against the centers of its parallel surface, and brought it to destruction. Since oil shales contain up to 50% mineral impurities, which leads to an increase in the strength of briquettes, the number of discharges was increased from 4 (according to GOST 21289-75) to the number leading to complete destruction of briquettes. The thermal stability of shale briquettes (they do not collapse during heat treatment) was determined at a temperature of 800-1000 °C in a muffle furnace.

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Table 1

Physical and mechanical characteristics of binders

Parameters	Petroleum pitch	Coal pitch	Bitumen BND 50/70
Density, kg/ m^3	1054	1264	984
Penetration at 25°C, mm	17	1,8	65
Extensibility at 25°C, cm	6	0	144
Cohesion at 25°C, N/m^2	$(1,62)\cdot10^6$	$(2,3)\cdot10^5$	-
Softening temperature, °C	74	87	48
Meltingpoint, °C	101	108	-
Extremely broken structure, °C	184	186	-
Flashpoint, °C	218	232	320
Viscosity, Pa · s at 100°C	576	343	361
at 200°C	0.9	0.6	0.4

RESULTS AND DISCUSSION

According to literature data, the extraction and processing of oil shale forms from 30 to 40% of shale fines, which, except for burning at thermal power plants, does not find industrial use and accumulates in significant quantities on stacks. At the same time, when producing shale gas in gas generators, up to 12% of heavy high-resinous resin is obtained. In addition, shale processing is accompanied by the formation of ash (up to 50% of the initial mass of oil shale).

The data presented in table 2 allow us to estimate the influence of the binder type (petroleum pitch, coal pitch, BND 50/70 bitumen) and its content (5, 10 and 15 %) on the main physical and mechanical properties of shale briquettes. In all three cases, an increase in the proportion of binder leads to an increase in density, which is associated with an improvement in the compaction of the grain structure and the filling of pores with an organic component. Coal tar pitch is characterized by maximum density values (1180-1260 kg / m^3), which reflects its ability to form the most solid carbonaceous framework. The BND values of 50/70 are minimal (1120-1170 kg / m^3), which is explained by the lower carbonization and more ductile nature of bitumen. Oil pitch occupies an intermediate position-the density is from 1150 to 1220 kg / m^3.

There is an obvious dependence of the crushing strength - it increases with increasing binder content, but the growth pattern differs for each type: coal pitch is the best reinforcing component (strength: 3.0-5.5 MPa), oil pitch - average efficiency (strength: 2.5-4.5 MPa) and the lowest strength in briquettes with HDPE (strength: 2.0–4.2 MPa).

The impact strength index reflects the ability of the briquette to retain mass under mechanical stress. the best results were obtained for briquettes containing coal tar pitch (85-95 %). It provides the most resistant to destruction of the frame. The indicator was especially low at 5 % BND, which indicates the weakness of the plastic bitumen frame.

Table 2

Influence of the binder type and quantity on physical and mechanical parameters of briquettes

Index	Binder type and quantity		
	5 %	10 %	15 %
Petroleum pitch			
Density, kg / m^3	1150	1200	1220
Compressive strength, MPa	2.5	4.0	4.5
Impact strength, % saved weight	80	90	92
Abrasionresistance, % weight loss	18	12	10
Water absorption in 24 hours, %	18	13	10
Type of compression failure	brittle	transition	visco-viscoelastic
Coal pitch			
Density, kg / m^3	1180	1230	1260
Compressive strength, MPa	3.0	4.8	5.5
Impact strength, % retained weight	85	93	95
Abrasionresistance, % mass loss	15	10	8
Water absorption in 24 hours, %	16	11	8
Type of compression failure	brittle	transition	viscoelastic plastic
BND 50/70			
Density, kg / m^3	1120	1150	1170
Compressive strength, MPa	2,0	3,0	3,8
Impact strength, % saved weight	70	82	88
Abrasionresistance, % weight loss	22	16	12
Water absorption in 24 hours, %	20	18	13
Type of compression failure	Brittle	ductile-brittle	ductile

The durability of the briquette was evaluated byits abrasionresistance. Minimal abrasion was observed when using coal tar pitch (8-15%), while for BND - maximum, especially when the binder content is low (12-25 %).

Minimal water absorption was provided by the introduction of coal tar pitch (8-16 %), which is most important for storing briquettes. In our opinion, this is due to high hydrophobicity and carbonation. Unlike pitches, bitumen does not create a solid carbon frame, remains soft and elastic, so it does not completely fill the pores between particles. As a result, the maximumwater absorption of BND is observed (13-22 %).

The structural evolution of the frame as the binder increases for all binders is as follows: brittle → transient → visco-plastic (or ductile). Coal and oil pitch form the most durable and carbonaceous coke frame. While bitumen forms a plastic matrix, it weakly resists destruction.

Thermal engineering indicators are the key characteristics of fuel briquettes, since they determine the energy value, combustion efficiency and the possibility of industrial use of the resulting fuel. Table 3 shows comparative data on the most important heat engineering indicators of briquettes of the studied compositions.

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Table 3

Influence of the binder type and quantity on heat engineering parameters of briquettes

Figure	Type and amount of binder		
	5 %	10 %	15 %
Petroleum pitch			
Lower heat of combustion, MJ/kg	10,5	11,5	12,5
Higher heat of combustion, MJ/kg	11,5	12,6	13,6
operating humidity, %	6	5	4
Total ash, %	52	50	48
Content of volatile substances, %	44	46	50
Flash point, °C	430	420	410
Character of combustion	medium with a tendency to agglomerate	more stable uniform	steady, pronounced coke residue
Coal tar pitch			
Lower heat of combustion, MJ/kg	11,2	12,4	13,4
Higher heat of combustion, MJ/kg	12,2	13,5	14,5
operating humidity, %	6	5	4
Total ash, %	51	49	47
Content of volatile substances, %	39	37	35
Flash point, °C	450	440	430
Character of combustion	dense, moderate; tendency to agglomerate	stable, smooth burning; dense coke residue	long burning; pronounced coke residue
BND 50/70			
Lower heat of combustion, MJ/kg	10,8	11,6	12,2
Higher heat of combustion, MJ/kg	11,8	12,7	13,4
operating humidity, %	7	6	5
Total ash, %	53	51	49
Content of volatile substances, %	46	48	50
Flash point, °C	410	400	390
Character of combustion	quick start, a moderate burning	smoother, with the stabilization of combustion	sustained combustion, coke soft phase

Data analysis shows that an increase in the binder content from 5 to 15 % leads to an increase in both the lowest and highest calorific values in all the systems studied. This is due to an increase in the proportion of the organic component in the briquette structure and a decrease in the relative content of the mineral part. The highest values of calorific value were observed for briquettes on coal-fired pitch (13.4-14.5 MJ / kg), which is explained by the high content of aromatic carbon structures and a high degree of carbonization of pitch during heating. Petroleum pitch shows moderate values of calorific value, while BND 50/70 bitumen provides the lowest values.

The working humidity decreased with increasing binder content, which is explained by an increase in the hydrophobicity of briquettes and a decrease in capillary porosity. However, an important pattern is observed between binders: bitumen briquettes have the highest humidity (7-8 % at 5% of the binder), which is associated with the softness of bitumen and the saturation of the material with open pores, briquettes on coal pitch are characterized by the lowest real availability of moisture, which confirms a high degree of hydrophobization of the structure.

Ash content is one of the key parameters that determine the energy efficiency of shale briquettes and their behavior during combustion. The mineral part, which is part of shale fines and binders, does not participate in the combustion process, so an increase in its share leads to a decrease in the real calorific value of fuel and an increase in the inertial heat balance. In addition, high ash content contributes to the formation of a significant amount of solid residues, which can lead to slagging of furnace devices, reduce heat exchange and increase operating costs. Theobtained data show that as the binder content increases, the relative ash content decreases, which is associated with an increase in the organic phase and better binding of mineral particles. The lowest ash content values are typical for briquettes in coal-fired pitch, which further explains their higher calorific value and combustion stability. Thus, reducing the ash content is an important criterion for improving the quality of briquettes and is directly related to the effectiveness of the selected binder type. Among the samples under consideration, the ash content naturally decreases with an increase in the proportion of the organic part, while the minimum ash content corresponds to coal pitch (47-53 %), the maximum-to bitumen, which, as noted in table 2 is associated with less completecarbonization and a large relative contribution of the mineral part.

The content of volatile substances is fundamentally different by the type of binder and reflects the thermal decomposition and structure of the organic matrix. Rapid ignition, low ignition temperature and a less stable combustion zone are associated with the maximum volatility of bitumen-containing briquettes (46-52 %). Oil pitch has average volatile values (44-50%), which ensures a more uniform combustion process.

The values of the ignition temperature fully correlate with the content of volatile substances: bitumen - minimum temperatures (390-420 °C), which ensures a quick start of combustion, but leads to instability of the process; oil pitch is characterized by moderate values (410-440 °C), which creates balanced ignition conditions and reproducible combustion mode; coal pitch has the highest ignition temperatures (430-465°C), which is a sign of high temperature resistance and the formation of a strong coke residue.

The nature of combustion also fully confirms this pattern: bitumen provides rapid ignition, but a soft and unstable coke phase; oil pitch provides a transition from moderate to stable combustion with an increase in the binder content; coal pitch forms the longest, stable combustion with a developed strong coke frame.

CONCLUSION

The obtained results on the study of physical, mechanical and thermal characteristics of shale briquettes with various types of binders (oil pitch, coal pitch, BND 50/70) allow us to identify patterns that determine the formation of the structure, strength and energy properties of composite fuel. The comparative analysis shows that the nature and amount of binder have a decisive influence on the macro - and

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microstructure of briquettes, their performance and thermal behavior during combustion.

First of all, attention is drawn to the steady increase in density and compressive strength with an increase in the amount of binder in all the systems under study. This is explained by the fact that the organic binder phase acts as a structure-forming component that fills interparticle pores, improving the contact between mineral grains of shale fines and reducing the number of defects in the structure. The most pronounced increase in mechanical strength is typical for briquettes in coal-fired pitch. Its high aromaticity and ability to further polycyclize when heated ensure the formation of a dense carbon frame, which is manifested in the maximum strength values at 10-15 % of the binder. Oil pitch shows intermediate values, while BND 50/70 bitumen forms the most plastic, but less strong structure due to insufficient carbonizing ability and increased residual porosity.

Abrasion and impact strength, which characterize the resistance of briquettes to mechanical influences, also show the advantages of coal pitch. Minimal abrasion values with high impact strength values indicate a strong structural grid and high resistance to destruction. At the same time, bitumen briquettes show the greatest mass loss during abrasion — which is a consequence of the high plasticity of the binder, weak interparticle adhesion and a larger number of open pores. These results confirm a close relationship between the structure of the organic binder, the degree of compaction of the briquette and its resistance to mechanical loads.

Water absorption is an important operational indicator that determines the behavior of fuel during storage and transportation. Analysis of the data shows that briquettes on coal pitches have minimal water absorption, which is due to the high hydrophobicity and low porosity of the carbon frame. Oilfield briquettes show moderate values, while bitumen briquettes show maximum values, which is due to the lower ability of bitumen to compact the structure and the preservation of a significant number of capillary channels. Thus, the water absorption level clearly reflects the structural density and hydrophobicity of the binder, which affect the resistance of briquettes to external moisture.

Thermal engineering indicators also show significant differences depending on the type of binder. In particular, the heat of combustion increases with increasing amount of binder, which is explained by an increase in the proportion of organic phase and a decrease in the relative ash content. Coal tar pitch provides maximum calorific value due to the high concentration of polycyclic aromatic structures that are prone to carbonization, and the formation of a strong coke residue, which contributes to long-term, uniform combustion. Petroleum pitch forms a moderately pronounced coke residue, ensuring stable combustion, while bitumen gives a soft and less stable coke phase, which leads to faster burnout and less stability of the process.

The content of volatile substances plays a key role in the ignition mechanism. Bitumen contains the maximum amount of volatiles (up to 50-52 %), which provides easy ignition, but leads to less thermal stability and rapid burnout. Oil pitch exhibits intermediate values of volatility. The lowest values are typical for coal tar pitch, which corresponds to its high temperature resistance and the need for higher ignition temperatures. Accordingly, the ignition temperature is a reflection of the depth of aromatization of the binder and directly affects the nature of the flame and the thermal mode of combustion.

A combined analysis of thermal engineering and physical and mechanical data allows us to conclude that coal tar pitch is the most effective binder, providing the optimal combination of high strength, low porosity, minimal water absorption and maximum calorific value. Oil pitch forms balanced characteristics and can be considered as a universal option for the production of medium-strength shale briquettes. It is advisable to use BND 50/70 bitumen only if there are no requirements for high mechanical strength and combustion stability, since it forms the most porous, less strong structure and has limited heat resistance.

Thus, the discussion of the results demonstrates a close relationship between the chemical nature of the binder, its quantitative content, and the totality of thermal and mechanical characteristics of briquettes. The obtained regularities can serve as a basis for optimizing the composition of shale briquettes depending on the intended purpose: from высокотермостойкихhighly heat-resistant fuel forms to cheap and easily flammable briquettes for household needs.

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