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FLOTATION OF CARBONACEOUS MATERIAL WITH REAGENTS BASED ON ACETYLENE ALCOHOLS*

One of the key indexes of enhanced efficiency of carbonaceous material flotation process is the search for new reagents and the analysis of their adsorption and flotation capacities towards improvement of flotation performance at reduced power consumption per unit end product owing to increased recovery of valuable components in purposive concentrates.

The article describes test data on using reagents of DMIPEK, which is dimethyl(isopropenylethynyl)carbinol, and DK-80 with a view of enhancing flotation of coal slurry and nano-carbon material. Structural features of carbonaceous material and mechanism of its interaction with acetylene alcohols are discussed. Reagents used in coal flotation and issues of flotation of finely dispersed coal are considered. The flotation tests on different grade coal from the Kuznetsk Coal Basin show an increase up to 12% in recovery and yield of valuable components in concentrate as compared with the common reagents. Moreover, the flotation rate becomes 2 times higher.

With regard to bonding between DMIPEK and DK-80 reagents and carbonaceous material molecules, the interaction between the reagents, carbonaceous matter and froth bubble is modeled. DMIPEK and DK-80 reagents based on acetylene alcohols have proved the universality and efficiency in flotation of carbonaceous material. Their feature is conditioned by the presence of acetylene and ethylene bonds in the structure, which allows maintaining strong ϖ-bonds.

Also, the article illustrates feasibility of flotation in separation of carbon nanotubes from ferriferous manganese catalysts using aliphatic and acetylene alcohols. The mechanisms of flotation to separate carbon nanotubes from natural ferriferous manganese ore used as a catalyzing composite with carbon nanotubes grown on its surface are discussed. The most efficient collecting agent and frother in flotation of carbon nanotubes is DK-80 reagent, enabling recovery of 85–92% of carbon nanotubes in froth product.

Key words: *carbonaceous material, flotation mechanism, acetylene alcohols, coal slurry, coal flotation, carbon nanotubes, carbon-catalyzing composite, carbon concentrate.*
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Introduction

Flotation is a universal process suitable for processing of various minerals, separation of different products and substances or for handling environmental issues [1]. Flotation is widely used in preparation of coal slurry and for extraction of carbon lost in fly ash [2, 3]. This study proposes to use flotation to extract carbon nano-materials from carbon-catalyzing composites.

One of the main indexes of higher effectiveness of carbonaceous material flotation is the research and pursuance of new reagents and the analyses of their sorptive and flotation capacities with a view to improvement of flotation performance and to reduction in power consumption per unit end product owing to growth in recovery and yield of valuable components in purposive concentrates.

The search for special flotation reagents for carbonaceous material remains of concern as current prices of oil and oil products are highly volatile.

Features of carbonaceous material flotation

Carbonaceous material is coal slurry, fly ash, carbon-catalyzing composites, etc.

Coal flotation is efficient for coal particles less than 0.5 mm, which makes 10–20% of one ton of coking coal under preparation [4]. So, this process allows saving deficient grades of coking coal, which is important for fuel and energy economy [2, 5].

Numerous models of coal structure are based on a set of benzene rings connected with various functional groups, aliphatic and hydroaromatic compounds integrated in random structure molecules by cross-link bridges. Van Krevelen's model of coal structure (**Fig. 1a**) includes the largest number of physical, physicochemical and chemical properties of coal and their elemental composition; the "aromatic" part of the structure is shaded in the figure. Molecule is not planar, and its structural cells are not accurately similar as in regular structure polymers [6]. A carbon nanotube has a consimilar structure (**Fig. 1b**) [7].

The phenomenon of peptization of coal slurry by alcohols used as flotation reagents is discovered by Klassen and Nevskaya [8]. The researchers highlight that peptization by alcohols is better with coal slurry than with coal particles. The peptizing effect of alcohols is an explanation of the successful flotation of coal slurry with high content of clayey particles [8, 9].

More than 120 chemical compounds being various production waste are used as surface active substances in flota-

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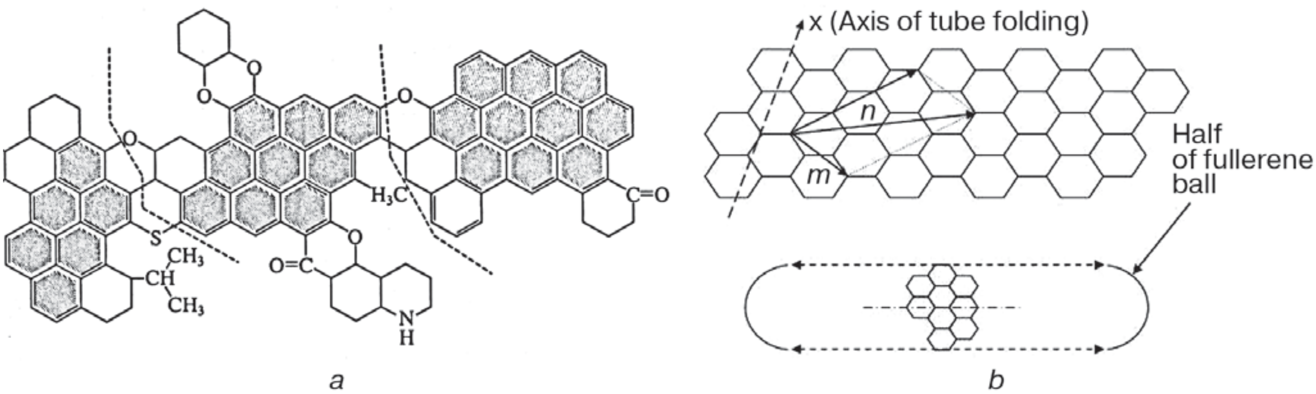

Fig. 1. Coal model by van Krevelen (a) and carbon nanotube structure (b)

tion of coal. These are heteropolar compounds such as monoatomic alcohols with regular and isomeric structure, mono- and polyatomic acids, phenols, phenates, amines, compounds with fused rings, heterocyclic compounds and other substances.

It is assumed that the most efficient for coal flotation are aliphatic alcohols that contain 6–8 atoms of carbon in hydrocarbonic radical [10]. At the same time, it is admitted that waste and secondary products are not a stable resource base for coal flotation as any change in a production technology of base minerals entails unpredictable variation in composition of waste or even leads to waste termination [4].

Coal flotation reagents are selected based on test data as there is yet no a firm theoretical background to identify beforehand which flotation reagent is required [4].

The feature of carbonaceous material flotation is associated with the process of secondary concentration when interaction between reagents, mineral particle and air bubbles in slurry differs from the process in froth bed [11]. The significance of the secondary concentration during flotation consists in the feasibility to adjust the process in the froth bed using mutual influence of reagents and minerals and changing their quantitative characteristics.

In a detailed description of froth dewatering [12], Goden says water flow in interlayers between bubbles captures particles of gangue and valuable minerals. Particles adhered to bubble surface approach one the other, and a filtration-like phenomenon occurs. Such bubble filter easily captures large particles of gangue, but attachment force of gangue particles is much weaker than attachment force of minerals. This phenomenon is typical of bubbles of froth generated by surfactant reagents free of multiple bonds peculiar to acetylene compounds.

It is known that compounds with acetylene bonds are used as collecting agents in flotation of coal [4, 13], sulfide ores of nonferrous metals, precious metals — gold, silver, bismuth, rhenium, potassium ore [14]. A cardinal distinction of the proposed acetylene-containing reagents from classical collectors is the absence of ligands — donor–acceptor atoms — in their functional groups. In this case, reactive centers are ethylene and acetylene bonds, and metal–reagent interaction product is molecular ϖ-complexes rather than ionic compounds.

The backbone of Rebinder's idea is that the three-phase contact line (TPC) is subject to the action of adhesion, surface tension and the third force — similar to friction. That is the reason why meniscus in close proximity to TPC is given mechanical properties of a solid. It has experimentally been proved that friction is effective in movement in perpendicular to TPC, whereas friction is zero in movement along TPC [15].

This conclusion is an explanation of weak attachment of gangue particles and stronger attachment of coal particles in flotation with agents without multiple bonds.

The research laboratory of Innovation Resource Ltd company, with regard to regulations of molecular chemistry and based on the experimental research undertaken, has structured a chemical formula of a compound to a best degree conformable with the requirements imposed on flotation agent — DMIPEK. The synthesis of this reagent was implemented concurrently with the research of its flotation abilities [16]. The resultant substance possesses surface activity, is capable of generation of stable specific froth and exhibits adsorption ability toward floated minerals. DK-80 is an individual compound resultant from direct interaction of acetylene and acetone with subsequent dehydration at the stage of the end product distillation [17].

The presence of the triple and double bonds in DMIPEK molecule and the presence of singular triple bond in DK-80 allows a supposition that between the cores of condensed aromatic structure of coal crystals (**Fig. 2**) and the double and triple bonds of the reagent, complexes of the type of ϖ-bonds are formed, and between carbon of coal particle and hydrogen of methyl group of reagents, C–H bond is formed. These bonds condition the distinctive feature of interaction between molecules of DMIPEK and DK-80 and coal particles (**Fig. 2**).

Such bonds enable coal particles to attach and seat on the surface of bubble generated with DMIPEK or DK-80 stronger than with common frother.

It is noteworthy that coal macromolecules contain no recurrent fragments as most of high-molecular substances. In composition of coal macromolecules, it is impossible to find a structural unit to make a judgment on the properties of the whole structure. It is only possible to speak on ratio of carbon atoms and heteroatoms in aromatic, naphthenic or aliphatic and functional groups. For this reason, it is assumed that for such carbonic nanostructures as carbon nanotubes having a structure similar to that of coal (refer to **Fig. 1b**), the mechanism of interaction with the reagents will be the same.

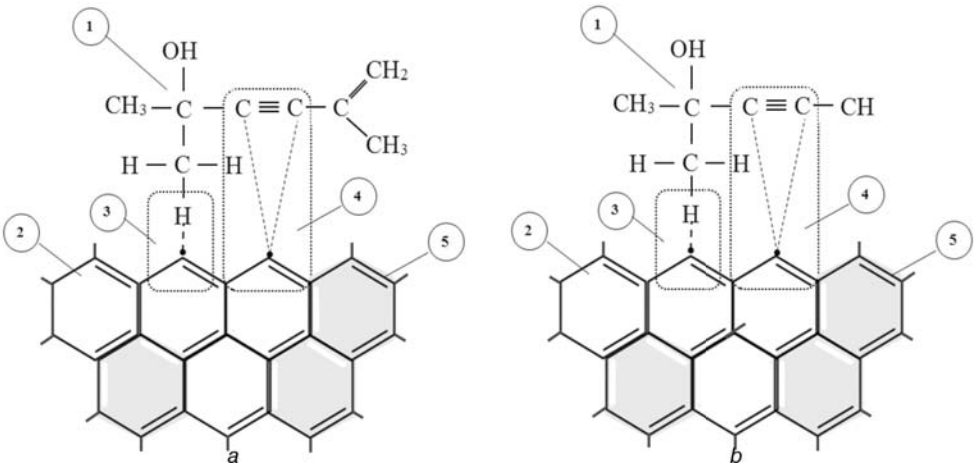

Fig. 2. Model of structure of C–H- and π-bonds between coal particles and molecules of the reagents:
(a) DMIPEK; (b) DK-80; *1* — molecule of DMIPEK (DK-80); *2* — conventional model of coal; *3* — C–H-bond; *4* — π-bond; *5* — aliphatic and aromatic fragments of coal

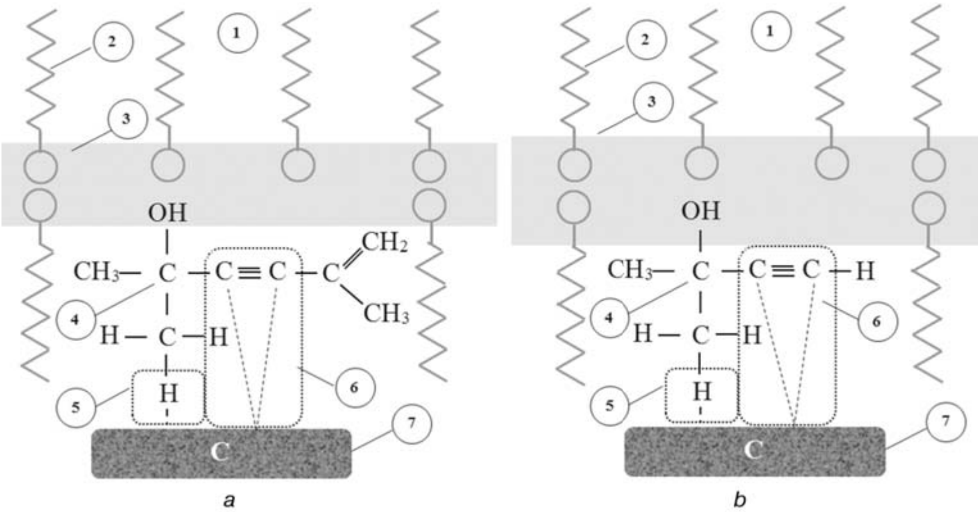

Fig. 3. Models of structures of C–H- and π-bonds between coal-bearing substance, reagents and froth bubble:
(a) DMIPEK; (b) DK-80; *1* — air; *2* — frother molecules; *3* — froth bubble shell; *4* — DMIPEK molecule; *5* — C–H-bond; *6* — π-bond; *7* — coal-bearing substance particle

Considering bonds that are generated during interaction of DMIPEK or DK-80 with carbonaceous material molecules, the models of interaction between the reagents, carbonaceous material and froth bubbles are shown in **Fig. 3**.

Evidently, in the structure of the proposed models, the reagents maintain the concurrent bond with the surface of coal-bearing substance particles and the shells of froth bubbles. It is necessary to take into account that: (1) surface of coal particles is naturally hydrophobic; (2) DMIPEK and DK-80 molecules are directed on coal surface by hydrophobic methyl groups; (3) hydroxyl function group-OH enhances hydrophilic property of carbonaceous substance.

Production and application of DMIPEK and DK-80 allow for economically efficient technology of flotation to be developed [16].

Coal flotation

Coal preparation plants mostly use various combinations of frothers and collecting agents. Frothers may be thermo-gasoil, VPP-86, still bottoms of butyl alcohols; collecting agents are furnace oil, KETGOL, jet fuel, industrial oil and their mixtures.

Flotation activity of DMIPEK was assessed using coals of the Kuznetsk Coal Basin, considering coal grades. Flotation tests were performed in laboratory of Belovo Central Preparation Plant (CPP). The composition of the flotation charge was accepted in accordance with the technology used by the plant: 20% of fat coal, 40% of gas–fat coal and 40% low-caking coal. Presoak time was 20 min in all tests. Flotation cell volume was 1 l. Flotation feed density was 150 g/t.

The test results (see the **Table**) show that, as against check mixture no. 1, reagent mixture composition no. 3 enables higher yield of the concentrate and ash content of the tailings by 5 and 16.1%, respectively, at the shorter duration of flotation by 1.57 times. The concentrate quality decrease by 0.5% is conditioned by the excess consumption of the collecting agent in this flotation mode, which is also specific for reagent mixtures nos. 4 and 7.

Introduction of DMIPEK, individually, in amount of 150 g/t allows higher extraction of coal in the concentrate — to 88.66% (mixture no. 6), which is improved by 4.66% at the higher ash content of tailings by 15.4%.

The tests show that this flotation agent acts as a frother with the collecting abilities and is consumed in amount of 50–100 g/t in combination with a collecting agent. When used individually, DMIPEK is consumed in amount of 120–500 g/t. Increment in coal extraction is from 3 to 10.7% as compared with the check mixtures.

Flotation of carbon-catalyzing composite

A carbon-catalyzing composite is natural ferriferous manganese ore with carbon nanostructures originated on the ore surface in the form of nanotubes. This composite material is a product of pyrolysis of carbonaceous gases from manganese ore of Porozhinskoe deposit under the temperature of 850 °C [18, 19]. According to the X-ray spectrum analysis, carbon content of the product is 42 mass percent. Transmission electron microscopy using Technai-12 microscope shows that the test specimen contains both one-layer and multi-layer carbon nanotubes (CNT) and chains of intercon-

Data of flotation testing of coal from Kuznetsk Basin*

Mixture no.	Flotation mixture	Yield, g	Recovery, %	Ash content, %	Flotation test duration, min
1	*Check* Gasoil — 3000 g/t; KETGOL — 48 g/t	C** — 136.5; T** — 17.5	C — 84.0; T — 11.66	C — 7.0; T — 60,2	14.20
2	Gasoil — 3000 g/t; DMIPEK — 24 g/t	C — 137.5; T — 17.5	C — 85.0; T — 11.66	C — 7.5; T — 57.59	12.00
3	Gasoil — 3000 g/t; DMIPEK — 48 g/t	C — 143.5; T — 11.8	C — 89.0; T — 7.8	C — 7.5; T — 76.3	9.00
4	Gasoil — 2000 g/t; DMIPEK — 72 g/t	C — 144.5; T — 11.8	C — 89.6; T — 7.8	C — 8.5; T — 75.0	8.35
5	Gasoil — 1500 g/t; DMIPEK — 72 g/t	C — 143.8; T — 12.5	C — 88.8; T — 8.3	C — 7,6; T — 74,2	9.10
6	DMIPEK-150 г/т	C — 143.0; T — 12.5	C — 88.66; T — 8.3	C — 7,4; T — 75.6	14.20
7	Gasoil — 2000 g/t; DMIPEK — 96 g/t	C — 143.0; T — 12.5	C — 88.66; T — 8,3	C — 7.8; T — 72.1	12.0
8	*Check* Gasoil — 3000 g/t; KETGOL — 48 g/t	C — 137.5; T — 17.5	C — 85,0; T — 11,66	C — 7,0; T — 59.9	16.2

*The research was carried out with the participation of A. Ya. Mamontov, Head of the Flotation Laboratory, CPP, and B.B. Valeev, Engineer, CCP. The Chief Engineer of CPP, V. I. Basargin took part in the discussion of the research findings.
**C — concentrate; T — tailings.

nected nanotubes. The specimen contains a high-quality carbon nano-structured material, mostly as CNT 500–6000 nm long with a diameter of 100–300 nm (**Fig. 4**).

To create CNT on the surface of a catalyst, growth centers of nanotubes are initiated to be epitaxially connected with the catalyst; i.e. a carbon–catalyst composite is made so that the catalyst and nanotubes are the organic whole. Then, CNT are separated from the catalyst as an individual product with the minimized admixtures.

Industrial-scale separation of CNT, graphite and amorphous carbon includes grinding, high-temperature oxidation (owing to different oxidabilities of allotropic forms of carbon) and concentration based on difference in size and density of particles, for instance, centrifugal separation. The known methods to separate CNT and graphite particles are very complicated and need much expenditure [19, 20].

The analyses of coal preparation mechanism have shown that coal and CNT have the same structures. Based on that, it is proposed to separate CNT and ferriferous manganese catalyst by flotation using acetylene reagents DMIPEK and DK-80. For the comparison, flotation test with aliphatic alcohol C_7–C_{12} is performed.

Treatment of carbon-catalyzing composite has such stages as (**Fig. 5**):

• *preparation* — attrition grinding to 100% content of size grade –44 µm. Then, ultrasound treatment of the specimen in a liquid medium at the frequency of 18.5 kHz during 8–10 min for efficient disintegration of carbon nanotubes and ferriferous manganese catalyst;

• *basic stage* — non-frothing flotation of the treated slurry in Halimond tube with conventional aliphatic alcohols C_7–C_{12} and acetylene alcohol reagents DMIPEK and DK-80.

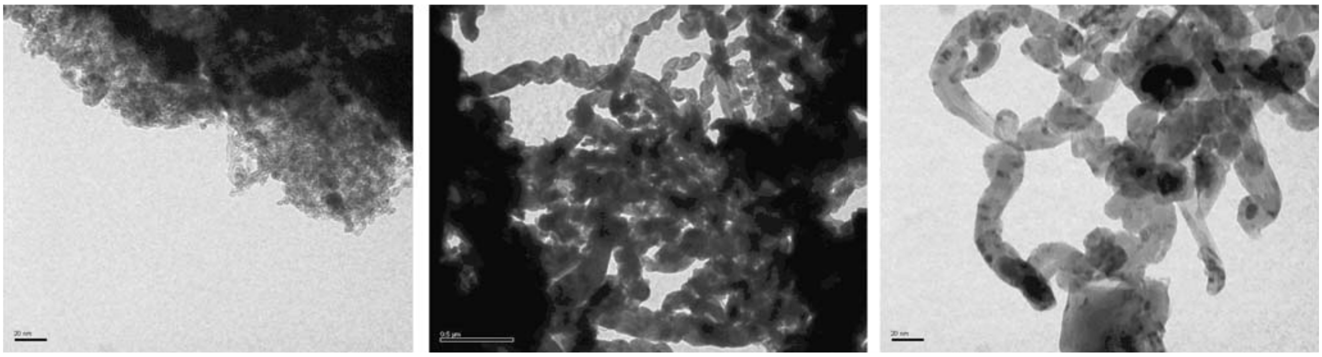

Fig. 4. Electron microscopical shots of carbon structures

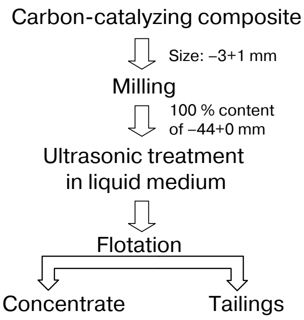

Fig. 5. Circuit of flotation-based separation of carbon and catalyzing composite

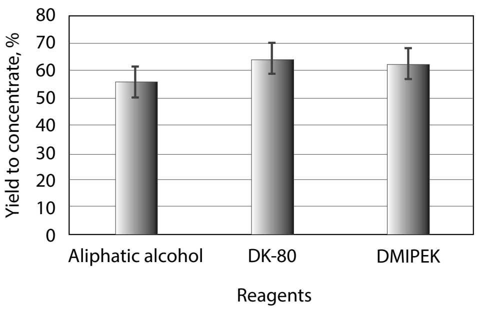

Fig. 6. Bar chart of yield of carbon nanotubes in froth product versus reagent regime

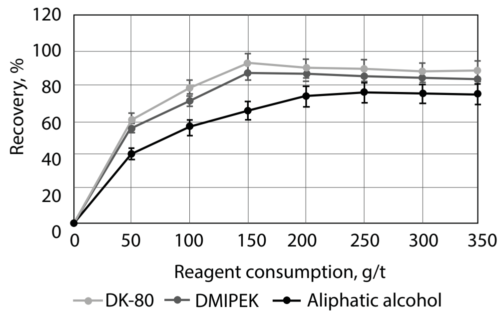

Fig. 7. Recover–reagent consumption curve

Flotation duration is not longer than 6 min, mechanical stirring at 1000 rpm;

• *closing stage* — drying of products (concentrate and tailings) in a drying cell at 105 °C; CNT concentrate production for on-target use.

The analysis of the test data shows that in the tests with the equal consumption of the test reagents, higher extraction of CNT in the concentrate is obtained with DK-80 (**Fig. 6**).

Based on the data on recovery of concentrates versus different consumption of the reagents shows that the optimal concentration of DK-80 is 85–92%. As against conventional reagents of the type of aliphatic alcohols, DMIPEK and DK-80 raise the yield of the concentrate by 5%. The decrease in the concentrate quality by 0.5% is caused by the excess amount of a collecting agent in the chosen flotation regime.

The relationship of the recovery and reagent consumption (**Fig. 7**) illustrates the efficiency of DK-80 as a collecting agent for naturally hydrophobic CNT and as a flotation frother: CNT froth recovery makes 92% which is higher than with alcohols C_7–C_{12} and reagent DMIPEK. The optimal concentration of DK-80 is 120–150 g/t nominally.

Conclusions

1. The flotation agents based on acetylene alcohols — DMIPEK and DK-80 — have proved their universality and efficiency in flotation of carbonaceous material. Their activity is conditioned by the presence of acetylene and ethylene bonds in their structure, which enables maintenance of strong ϖ-bonds.

2. The flotation tests on different rank coals and carbon-catalyzing composite show higher recovery by 1.5–12% and higher yield of valuable components in concentrates as compared with the conventional reagents. The flotation rate is increased by 2 times.

3. The authors have found basic regularities in flotation-based separation of carbon nanotubes and carbon-catalyzing composite represented by natural ferriferous manganese ore with the carbon nanotubes grown on its surface. It is shown that the most efficient collector–frother in flotation of carbon nanotubes is DK-80 that allows recovery of 85–92 carbon nanotubes in froth product.

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DEEP CONVERSION AND METAL CONTENT OF RUSSIAN COALS

In today's fuel, energy and metallurgical complex coal is used as an energy source, as well as coke for steel products.During unstable economic situation, the coal becomes the main source of organic raw materials to many industries, especially in the reduction of production and reserves of oil and gas.Coal processing using an integrated approach opens up new opportunities for the coal industry, and new technologies require significant development, technical and economic assessment and broad implementation. In this paper, we discuss the problem with the deep coal processing of Russian industry, solid fuel gasification and producing of synthetic fuel. The article describes the features of the modern coke production, as well as problems of extraction of rare earth and noble metals from waste coal and coal products.Since the 60s the world of deep coal processing technology continues to evolve and improve. Plants of industrial processing of coal were built and successfully work in South Africa, New Zealand, USA, China. With regard to Russia, despite the huge reserves of coal, chemical processing technology has not yet received the same development as in industrialized countries, primarily because of the lack of proper project financing.

The paper shows that there are all the technological and economic preconditions for improving the quality of extracted coal, including in the production and deep processing of low-grade and off-grade solid fuels for disposal of solid waste from coal mining and coal processing with simultaneous extraction of some metals.

According to some experts, in the coming years, the demand for coal is significantly larger view and possibly coal will be the driving force of the world economy.In particular, European experts believe that the global electricity market is on the verge of transition from gas to coal as the preferred fuel for power plants.This trend is reflected in the Energy Strategy of Russia. Emphasis is placed on the need to harmonize the fuel and energy balance of the country through active development of the coal industry to ensure energy security of the country.

Key words: *coal, ash,metal content, deep coal processing,benefication, coke,gasification, rare metals, synthetic fuels*
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Introduction

Huge resources and growth prospects in coal mining call for a science-based approach to coal utilization in various branches of industry [1]. At the present time, the fuel and energy industry of Russia uses coal mostly as a universal energy carrier at heat power stations and as a feedstock for production of blast-furnace coke and coking chemicals. Deep conversion of coal toward manufacture of heating and chemical products is carried out on a limited scale at Russian plants; at the same time, given the permanent fluctuation of free market prices of oil and oil products and the depletion of oil reserves, production of liquid hydrocarbons from coal becomes of concern. Russia holds the largest coal reserves, and the researches in the area of chemical technologies of coal liquefaction are promising, first of all, in terms of the substitution of import oil products by the new domestic-manufacture energy products of the same quality.

Many countries (USA, China, Japan and others), lacking oil and natural gas reserves, in order to ensure energy safety and for energy market price dumping, develop and implement pilot-scale testing of innovation technologies for production of synthetic motor fuel and synthetic crude for organic synthesis using coal products [2]. For instance, after the slump in oil on the world market, the technologies of hydrocarbon gasification liquefaction (the “U.S. Shale Revolution”) and lignite drainage (pilot scale plants in Germany) now enjoy widespread development [3, 4].

The key problem generated by coal combustion at heat power stations using traditional technologies is dumping of millions of tons of ash with, as a rule, high commercial-level