

Catalytic hydrogenation of coal of the Kazakhstan fields in presence of polymers

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Objects of investigation are the coals of the central region of Kazakhstan and solid organic wastes in the form of polymers. In this work process of hydrogenation of brown coal of the Kazakhstan fields is studied. The mechanochemical activation of ore catalyst affects not only the degree of carbon conversion but also on the composition of the liquid products. Processing increases activity of the iron-containing catalyst in reactions of conversion of the high-molecular asfaltens into maltens possessing smaller molecular weight i.e. increases cracking properties of the catalyst. The presence of the polymer in the coal leads to release of the volatile and soluble low molecular weight products, which are products of the destructive conversion of coal and polymers. Their yield, composition and structure depend on the condition of the hydrogenation, the stage of coalification of coal, the medium, type and mode of mechanical action. The advantage of the proposed method for producing liquid products from coal is the high yield of hydrocarbon products boiling up to 200°C and the exception of the stages of selection and hydrotreating processes of the pastes, possibility of recycling of industrial and household wastes of polymeric materials.

Key words: coal, catalysts, hydrogenation, polymers, mechanochemical activation.

INTRODUCTION

On the background of the current situation of fuel deficiency in Kazakhstan and reduction of the amount of oil produced, as well as decreasing of oil prices especially actual is the question of finding of alternative fuels, in particular from coal. Complex use of the fossil opens up new perspectives in the area of coal-chemical synthesis. The coal reserves prevail over other types of organic materials, and the problem of disposing of used tires and polyethylene from year to year is increasing worldwide in geometric progression. So the co-processing of solid organic wastes and coal, today - is one of the most promising source for production of liquid, gaseous and solid fuels. In addition, due to such reactions there is a possibility of the synthesis of the most important products for further use in the petrochemical synthesis [1]. Production of liquid fuel and various chemical products by hydrogenation of solid organic materials - the most perspective direction in the fuel and power industry. Scientists of the leading countries of the world carry out the intensive researches of processes of production of synthetic liquid fuels.

Various ways to use catalysts in processes of transformation of coals are so far offered [2-6].

Synthesis of liquid fuel can be carried out by means of direct (destructive) hydrogenation of the initial coal or by hydrogenation of gas, which is previously synthesized in the process of gasification

of solid fuels. The best contact is reached at introduction of the catalyst to coal by methods of chemical linking with reactive groups (for example, -COON, -CH₂OH) on the surface of coal, introduction into the volume of coal substance, and also when using catalysts in the dissolved, melted or volatile states. Implementation of catalytic processes for coal processing by the mechanism of indirect catalysis greatly simplifies their technology. In these cases it is possible to achieve a high efficiency of the process by applying of the mechanical mixture of solid catalyst and carbon which is particulate or suspended in a liquid medium, and a fixed or fluidized bed of catalyst particles. The greatest practical interest is represented by cheap catalysts on the basis of iron (for example, iron salts, iron-containing ores and concentrates). Their use allows to refuse from expensive step of catalyst removal from the solid sludge the process of coal hydrogenation [7, 8]. However the analysis of literary data demonstrates that process of a hydrogenation of coal is carried out at high temperature and pressure in the presence of the active molybdenum-containing catalysts and disposable catalytic additives on the basis of sulfides and sulfates of iron.

It's known [9-12] that mechanochemical processing of solvent and coal is one of the perspective directions in the field of producing of liquid products from coal. As a result of mechanical treatment of coal there is its dispersing leading to increase of a specific surface. This fact gives higher solubility of mechanically activated coal in organic solvents and increase of their reactionary ability in

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further chemical reactions. For transformation of the organic mass of coal and solid organic waste in the form of crumb rubber and polyethylene, into liquid products it is necessary to carry out a destruction of coal macromolecules to liquid products and to increase concentration in them of hydrogen that it is usually reached by coal heating under pressure of molecular hydrogen among the pastes-polymers having hydrogen and donor properties. It is known that such polymers as polyethylene contain about 14 wt. % of hydrogen, in worn-out tires the content of hydrogen reaches 9-10%. In the course of the joint thermo-transformation with coal they can act as a potential source of hydrogen necessary for transformation of coal into the easily boiling hydrocarbon fractions. In the course of their joint thermal conversion with coal there is an interaction of radical fragments of thermal destruction of coal with the radicals of polymer that prevents course of secondary reactions of polymerization and polyfunctional condensation of the coal products resulting in the formation of coke.

The aim of the work was to determine the optimal conditions (technological mode of reaction, catalysts) of paste forming to intensify the yield of liquid products from coal and organic wastes. Study of influence of iron-containing catalysts entered into the reactionary mix in the high-disperse form on indicators of process of hydrogenation of brown coal of the Kazakhstan fields.

EXPERIMENTAL

The objects of study were coals of the fields "Karazhira" and "Ekibastuzskii" (Kazakhstan). As the catalysts were used the samples of concentrate of the Balkhash field (BC), Kazakhstan, which are presented the mixture of pyrites and chalcopyrites, in which there are, particularly, Fe-24%, Cu - 30%, Al - 12%, S - 12%. Before carrying out of the experiment the mixtures of solid polymer residues is subjected to heat treatment and then was carried out mechano-processing, joint with coal, to the size of particles more than 200 microns. The resulting mixtures were stored in compliance with the precautions to eliminate the possibility of reaction of oxidation or pollution. As the solvents were used toluene, benzene, alcohols, naphthalene, and acetone.

In the work for the preparation of a necessary additive as a polymer was used a plastic bottle which is warmed and then converted into a powdery state. Polyethylene terephthalate ($C_{10}H_8O_4$)_n is the most widespread representative of the class of polyesters, condensation product of ethylene glycol with terephthalic acid (fig.1). Polyethylene terephthalate belongs to the group of aliphatic-aromatic

polyesters. In the conditions of the mechanoactivation (which is carried out in this work) in which local temperatures reach 1,000°C there is a polymer destruction to producing monomers or oligomers that is easily reached and polymer can be as additional source of hydrogen, leaving at the same time carbon in a catalyst matrix. In the work described in this manuscript, this substance must be as a source of hydrogen, as it has a low decomposition temperature - 350°C.

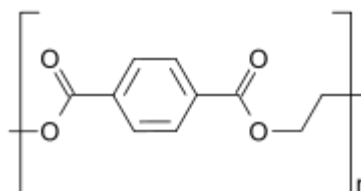


Fig.1. Polyethylene terephthalate ($C_{10}H_8O_4$)_n

IR absorption spectra of the samples were measured with use of the IR spectrometer Perkin Elmer Spectrum 65 in the field 700-3,600 cm^{-1} and UV-750U spectrophotometer in the range of 400-4,000 cm^{-1} in the dismantable cuvettes (liquid samples) and in potassium bromide tablets (solid samples). Definition of micropores, data on textural and structural characteristics of the prepared carbon materials are received with use of the analyzer of a specific surface, and also from calculation of the isotherms of adsorption of nitrogen taken at 350°C in the range of relative pressures P/P_0 from 0.005 до 0.995 on the device "Sorbometr-M". X-ray fluorescent researches and the element analysis of the solid samples were carried out on the FOCUS-M2M microanalyzer with use of Fe-radiation in the range from 2.0 to 37.0 V. The intensity of the diffraction peaks was evaluated by analytical method in the tetragonal system. For samples of initial raw materials (solid polymeric products and coal) the thermogravimetric analysis was carried out. Loss of mass of a sample at the given temperature was determined according to curve of thermogravimetric measurements. The mass loss rate was determined by differential thermogravimetric curve and temperature of maxima of endo - or exo - effects - on the basis of differential thermogravimetric adsorption curve, in accordance to standard procedures. In this work loss of mass is identical to the yield of volatile products, and the rate of loss of mass is equivalent to the rate of allocation of volatile products when heating. Changes of a surface and structure are revealed by means of the scanning electronic microscope NtegraTherma with the lighting modes - "on a gleam" and "on reflection". Aromatics content in the coal hydrogenation products was determined by gas chromatography-

mass spectrometry using Agilent 7890A chromatograph equipped with a mass selective detector Agilent 7000ATripleQuad at registration of the total ion current.

For experiments on coal hydrogenation a rotating 0.5-liter autoclave was used. Reaction was carried out with use of method developed in the laboratory [13-17].

Iron containing catalysts were prepared and introduced into the reaction mixture by following two ways:

1. A powder BC was subjected to mechanochemical treatment in a mixture with water in the centrifugal planetary mill - activator. The activated catalyst was added to a mixture of coal and water (2:10 wt. parts), at the rate of 10% by weight of organic mass of coal (OMC) and was dispersed for 20 min. The mixture was then dried at 105°C to a moisture of <1 wt. %;

2. The powdery concentrate BC was subjected to mechanochemical processing in mixture with water in a mill-activator, was added to tar accordingly to calculation from 5.0 to 45.0 % to OMC and was dispersed with the subsequent removal of water from mixture at 34°C in a drying box.

RESULTS AND DISCUSSIONS

According to X-ray fluorescence analysis of BC, the main crystalline phase in selected samples of ore after mechanical activation of catalysts are hematite and chalcopyrite transforming into pyrite. At the coal hydrogenation hematite is reduced into magnetite, pyrites is decomposed into pyrrhotine.

The element and phase analysis of the ore catalyst before mechano activation is presented in Table 1. Mechanochemical activation of ore catalysts has impact not only on degree of conversion of coal, but also on the composition of liquid products. Processing increases activity of BC of the catalyst in reactions of transformation of the high-molecular asphaltenes to maltens possessing smaller molecular weight, i.e. increases cracking properties of the catalyst.

IR data of initial coal show the presence of amorphous compounds (fig.2), in particular, the organic component of coals represents mixture of various X-ray amorphous components, presence and quantity of which varies among metamorphism row.

Physico-chemical characteristics of the obtained coal with a polymer, in particular Raman spectrum showed that the main product consists, mainly, of carbon (not less than 90.0%) and to 10.0% of the mineral impurity entering to a basis of

polymeric material (fig.3). At the content of polymeric material in mixture less than 10.0 wt. % degree of conversion of mixture into the liquid and gaseous products is low that interferes to the effective implementation of process.

The degree of coal conversion and yield of the low-boiling products grow at increase in amount of synthetic polymeric material in mixture to 70.0% per mixture weight. Data of IR - spectroscopy (fig.4, 5) of initial coal and of coal together with polymer after hydrogenation showed that after mechanical treatment of coals the number of saturated CH-bonds in the form of the methylene and methyl groups is decreased (fig.5) In the liquids extracted from coals after hydrogenation according to IR spectra was observed destruction of carboxyl groups, ketones and esters and decrease in number of the methylene structures. On IR spectra intensity of absorption bands of CH-aromatic groups is increased (in the range of 537-1,017 cm^{-1}); the absorption caused by stretching of vibrations of C-H (the methylene and the methyl) groups in the area of 2,922-3,418 cm^{-1} is decreased. The absorption intensity corresponding to carbonyl groups in aldehydes, lactones, esters (1,720 cm^{-1}) is changed. Data of IR - spectra (fig.5) show that in the reaction products the intensity of absorption bands of aliphatic hydrogen at 2,922 cm^{-1} , of the methyl groups at 1,342 – 1,410 cm^{-1} is increased. Presumably, the growth of the hydroxyl groups associated with the break of ester bonds, an increase of methyl groups is a consequence of the adherence of ethylene from polyethylene and carbonyls growth, is probably the result of acylation of aromatic carbon fragments.

Further increase of the polymeric material in the mixture does not lead to significant changes in these parameters of the process. The proposed process for producing of liquid products from coal can increase the yield of low-boiling hydrocarbon fraction from 12.0 to 60.0 wt.%. An advantage of the proposed method for producing liquid products from coal is the high yield of hydrocarbon products boiling up to 200°C, exception of the processes of allocation and hydrotreating stages of polymers from the technological scheme and possibility of recycling of industrial and household wastes of polymeric materials. In the presence of polymers as a part of mixture coal-polymer the content of high-molecular asphalt and resinous components - asphaltenes and benzene pitches is decreased in the field 360–443°C. As a result of thermal influence in bitumen the role of components with smaller molecular weight - benzene pitches and oils is

Table 1. The composition of the ore samples before and after mechanical activation

The samples		Composition, %				
Elemental analysis	Fe	Zn	Al	Cu	Cl	S
MC	41.4	-	-	36.5	7.3	-
BC	32.8	6.4	21.11	22.58	-	15.5
The main crystalline phase						
Phase analysis	The initial sample			After mechanical activation		
MC	CuO, FeCl ₃			Fe ₂ O ₃ , CuO, Cu		
BC	CuFeS ₂ , Fe ₂ O ₃ , FeS ₂ ,			Fe ₃ O ₄ , FeS, CuO, Cu		

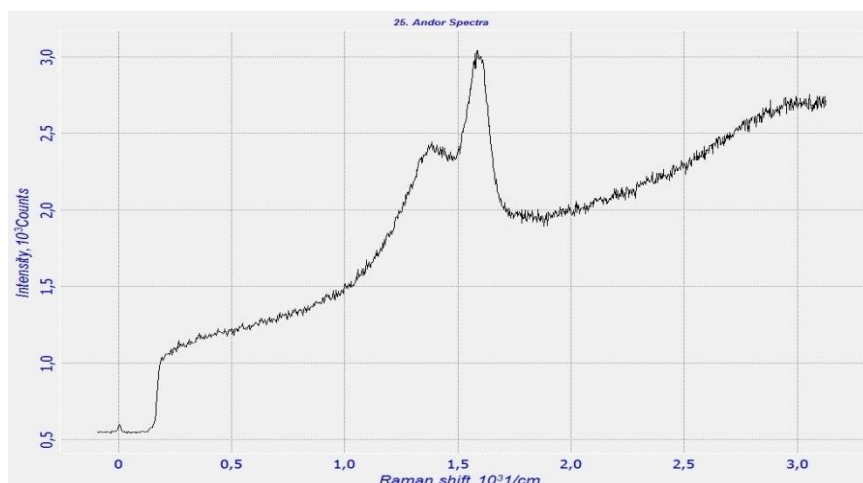


Fig. 2. Raman spectrum of initial carbon materials

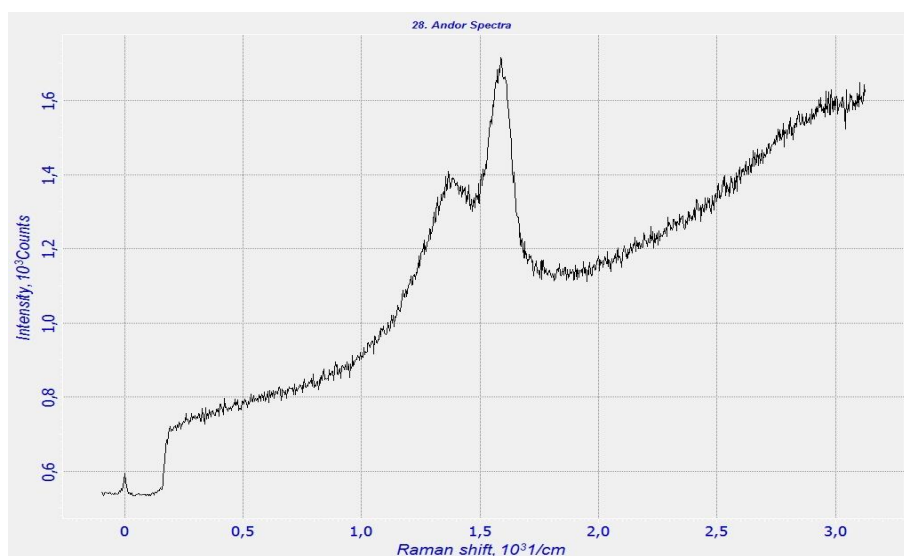


Fig. 3. Raman spectrum of coal with the polymer

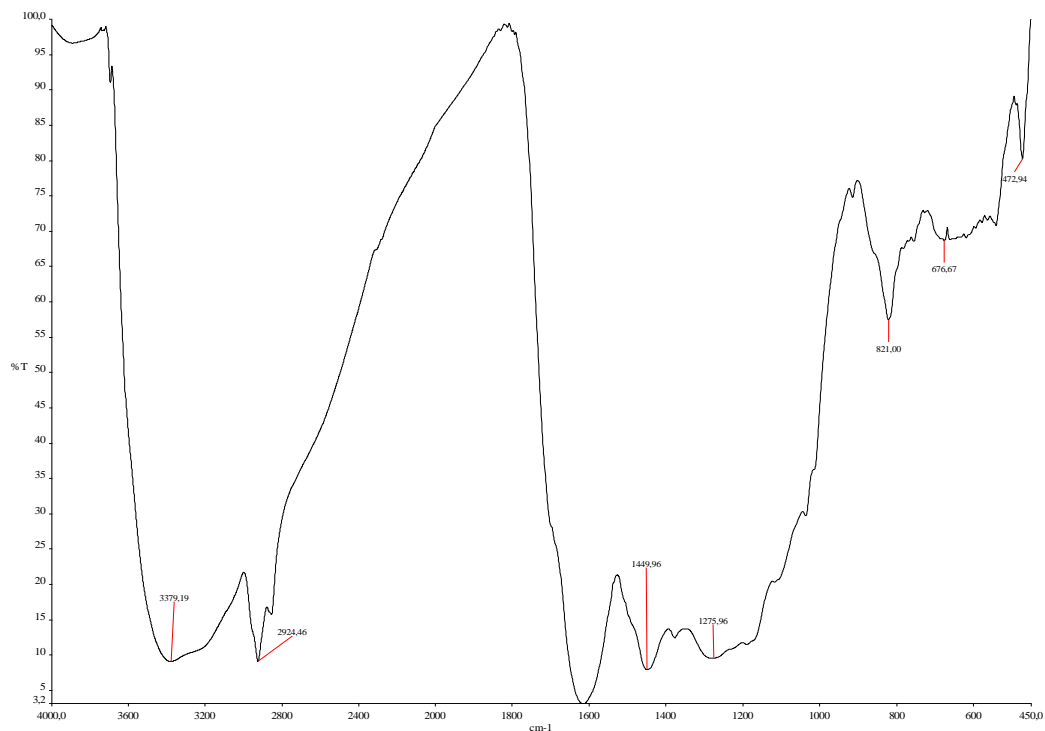


Fig. 4. IR-spectra of the initial coal

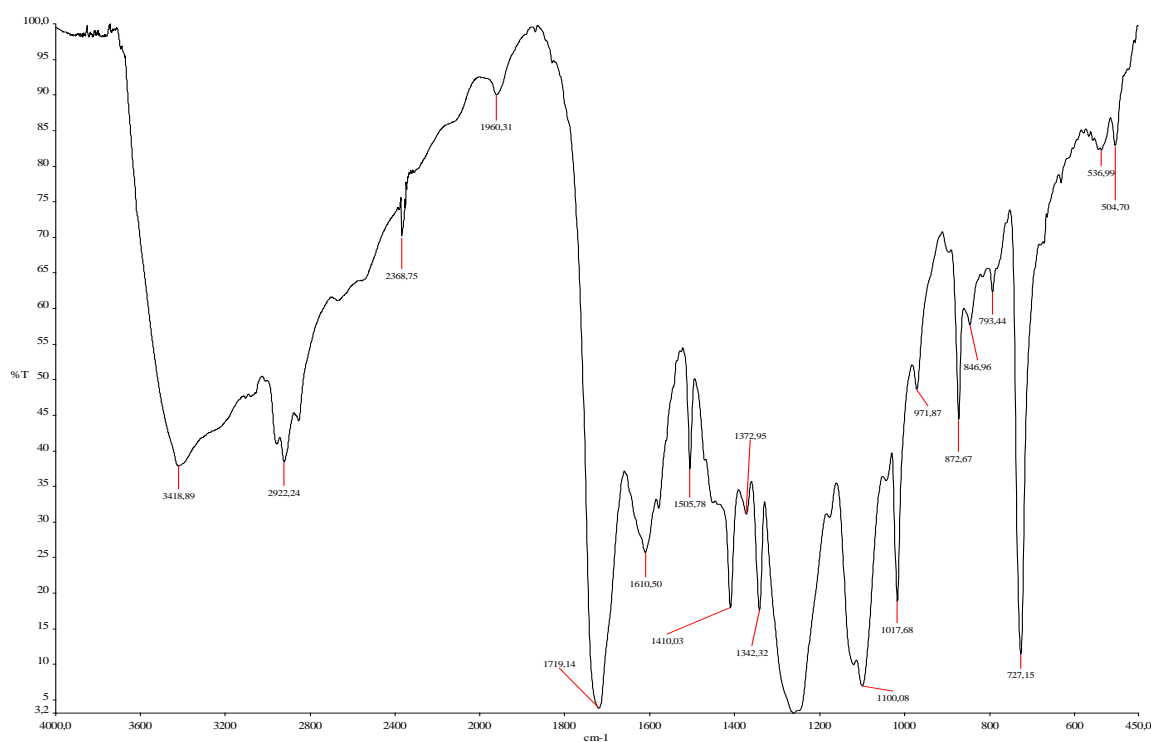


Fig. 5. IR -spectra of coal with a polymer after the hydrogenation reaction

increased. As a part of the oil fraction is increased the content of light petroleum-ether resins and aromatic hydrocarbons in the field 443-527°C. At hydrogenation of coal in tar possessing the hydrogen-donating properties, the researched catalysts considerably increased OMC.

In the course of a coal hydrogenation in the tar possessing hydrogen-donors properties, the studied catalysts considerably increase degree of conversion of the organic mass of coal (OMC) (Tab.2)

Table 2. Hydrogenation of coal in presence of polymers

Composition (coal, tar, concentrate of Balkhash field), wt. %	Degree of OMC conversion, wt. %	Yield of gaseous products, wt. %	Yield of solid products, wt. %	Hydrogen consumption, vol. %
Coal/tar:50/50	68.1	42.1	5.8	0.5
Coal/tar/concentrate: 45/45/10	65.1	43.5	5.7	1.5
Mechanical activated coal/tar/concentrate: 45/45/10	72.1	50.4	5.4	1.8
Mechanical activated coal/polyethylene/concentrate: 80/10/10	70.2	44.2	6.8	1.9
Mechanical activated coal/rubber crumb/concentrate: 80/10/10	69.2	45.2	6.1	2.0

Table 3. Chromatographic analysis of hydrogenation products

Compound	Initial	In the presence of polymer
	The liquid phase, vol. %	
C ₂ H ₅ OH	3.3	2.6
C ₆ H ₆	8.4	9.4
C ₆ H ₅ CH ₃	10.4	10.9
p- xylene	7.0	6.4
ethyltoluene	8.1	6.0
ethylbenzene	4.6	6.6
	Gaseous phase, vol. %	
H ₂	18.1	24.3
CO	3.6	5.1
CO ₂	13.2	19.1
CH ₄	7.5	6.7
C ₂ H ₆	2.1	1.8
C ₂ H ₄	-	1.0
C ₃ H ₈	1.2	0.9
C ₄ H ₁₀	1.3	1.0

The highest exponents of coal conversion (72.1 wt.%) and the yield of liquid products (50.4 wt.%) were observed in the composition of mechano-activated coal/tar/concentrate 80/10/10. For catalyst supported in the highly dispersed form on the surface of brown coal in the presence of polyethylene and crumb rubber the relatively low coal conversion rates were obtained. It can be explained by the fact that the activated iron is in the form of the complex aggregates in structure of polymers of polyethylene and a rubber crumb, which can create diffusion limitations for certain coal thermal degradation products, which can create certain diffusion limitations for the products of coal thermal destruction. In the conditions of process of a hydrogenation tar is dehydrogenated, transforming into liquid hydrocarbons. Apparently, dehydrogenation of tar in presence of coal occurs as result of its interaction with OMC or as the products of its thermal destruction. The studied catalysts

accelerate coal hydrogenation by molecular hydrogen of tar with formation of liquid products that leads to increase in its content in reactionary mixture. Results of a gas chromatography show that gaseous products of a hydrogenation of coal are presented generally by carbon oxides, the content of hydrocarbonic gases C₁-C₄ doesn't exceed 20 wt.%. In the course of a hydrogenation there is a consumption of molecular hydrogen by coal. It can be concluded (Tab.3), that sufficiently high yield of hydrocarbon gases takes place in the presence of polymers, i.e. in cases where the free hydrogen is abundant. Allocation of CO₂ after hydrogenation is can be explained by processes of decarboxylation of polymers, emergence of hydrocarbonic gases promotes to the deep destructive processes of organic synthesis of a liquid phase and substance of coal with the immediate filling of uncompensated bounds with the allocated hydrogen.

Table 4. Influence of polyethylene and composition of the mixture on yield of liquid products of hydrogenation

Composition	Quantity of polymer in the mix, wt. %	Degree of conversion, wt. %	Yield of the products of liquid fraction, wt. %	
			T _{boiling} <200°C	T _{boiling} >200°C
Mechanical activated coal/polyethylene /concentrate: 80/10/10	10.0	70.2	5.2	44.2
Mechanical activated coal/polyethylene /concentrate: 70/20/10	20.0	71.6	10.3	45.4
Mechanical activated coal/polyethylene /concentrate: 50/40/10	40.0	75.2	23.7	37.5
Mechanical activated coal/polyethylene /concentrate: 30/60/10	60.0	77.0	32.5	40.5
Mechanical activated coal/polyethylene /concentrate: 15/75/10	75.0	78.0	30.7	48.0
Mechanical activated coal/tar/concentrate/polyethylene: 10/10/10/70	70.0	73.1	52.4	10.2
Mechanical activated coal/tar/concentrate/polyethylene: 10/5/10/75	75.0	72.1	60	8.3

Type of added polymers has a significant effect on the degree of conversion of OMC and the yield of products (Tab.4). In medium polyethylene, which has the ability to dissolve the coal hydrogenation products, but does not have a hydrogen-donating properties, the degree of OMC conversion much lower than those of obtained in the tar medium. It was revealed that in the selected conditions polyethylene dehydrogenation does not proceed.

In the course of a coal hydrogenation in tar and polyethylene iron-containing catalysts accelerate as reaction of direct hydrogenation of organic mass of coal and products of its destruction by molecular hydrogen, as hydrogenation (reduction of hydrogen and donor properties) of the dehydrogenated hydrogen and donor solvent – tar in the presence of polyethylene. At the content of polymeric material in mixture less than 10 wt.% conversion degree of mixture into liquid and gaseous products is low that interferes with efficient implementation of process. Conversion degree of coal and the yield of the light-boiling products increase at increase in amount of synthetic polymeric material in mixture to 70% per the weight of mixture. The proposed process for producing of liquid products from coal can increase the yield of low-boiling hydrocarbon fraction from 12 to 60 wt. %.

CONCLUSION

Thus, in the work hydrogenation of brown coal of the Kazakhstan fields was studied. Objects of investigation were the coals of the central region of Kazakhstan and solid organic wastes in the form of polymers. Mechano-chemical activation of the iron-containing catalyst affects not only the degree of carbon conversion but also on the composition of liquid products. Processing increases activity of the iron-containing catalyst in reactions of conversion of the high-molecular asphaltenes into maltens possessing smaller molecular weight i.e. increases the cracking properties of the catalyst. It was shown that addition of polymer leads to allocation of the volatile and soluble low molecular weight products, which are the products of the destructive conversions of coal and polymers. The yield, composition and structure of this type of products depends on the conditions of hydrogenation, the stage of coalification of coal, medium, type and mode of mechanical action.

The difference in indicators of non-catalytic transformation of coal in the environment of tar and polyethylene characterizes contribution of hydrogen-donor properties of the paste forming in the total process. In the presence of polymers as a part of mixture coal-polymer is decreased the content of high-molecular asphalt and resinous components asphaltenes and benzene pitches in field 360 – 443°C. As a result of thermal influence in

bitumen the role of components with smaller molecular weight - benzene pitches and oils is increased. As a part of the oil fraction is increased the content of light petroleum-ether resins and aromatic hydrocarbons in the field 443-527°C. At hydrogenation of coal in tar possessing the hydrogen-donating properties, the researched catalysts considerably increased OMC. Conversion degree of coal and the yield of the light-boiling products increase at increase in amount of synthetic polymeric material in mixture to 70% per the weight of mixture. The proposed process for producing of liquid products from coal can increase the yield of low-boiling hydrocarbon fraction from 12 to 60 wt. %. The advantage of the offered method for producing liquid products from coal is the high yield of hydrocarbon products boiling up to 200°C, the exception from the technological scheme of the stages of selection and hydrotreating processes of the pastes, and the possibility of recycling of industrial and household wastes of polymeric materials.

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КАТАЛИТИЧНО ХИДРИРАНЕ НА ВЪГЛИЩА ОТ КАЗАХСТАНСКИ НАХОДИЩА В ПРИСЪСТВИЕ НА ПОЛИМЕРИ

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(Резюме)

Обект на изследването са въглища от централния район на Казахстан и твърди органични полимерни отпадъци. В тази работа е изследван процесът хидриране на кафяви въглища от казахстански находища. Механо-химичното активиране на катализатора влияе не само на степента на превръщане, но и състава на течните продукти. Процесът повишава активността на желязо-съдържащия катализатор на реакцията на превръщане на високомолекулните асфалтови вещества в малтени с по-малка молекулна маса, т.е. повишава се крекиращата способност на катализатора. Наличието на полимери води до отделянето на летливи вещества, продукти на деструктивното превръщане на въглищата и полимерите. Техният добив, състав и структура зависят от условията на хидриране, степента на овъгляване на въглищата, средата, типа и способа на механично въздействие. Предимство на предложения метод за получаването на течни продукти от въглища е високият добив на въглеводородни продукти, кипящи до 200°C и изключването на етапите на селекция и хидро-третиране на твърдия остатък, както и възможността за рециклиране на промишлени и битови отпадъци от полимери.