ЕҢБЕК ҚЫЗЫЛ ТУ ОРДЕНДІ
«Ә. Б. БЕКТҰРОВ АТЫНДАҒЫ
ХИМИЯ ҒЫЛЫМДАРЫ ИНСТИТУТЫ»
АКЦИОНЕРЛІК ҚОҒАМЫ

ҚАЗАҚСТАННЫҢ ХИМИЯ ЖУРНАЛЫ

ХИМИЧЕСКИЙ ЖУРНАЛ КАЗАХСТАНА

CHEMICAL JOURNAL of KAZAKHSTAN

АКЦИОНЕРНОЕ ОБЩЕСТВО
ОРДЕНА ТРУДОВОГО КРАСНОГО ЗНАМЕНИ
«ИНСТИТУТ ХИМИЧЕСКИХ НАУК
им. А. Б. БЕКТУРОВА»

4 (68)

ОКТЯБРЬ – ДЕКАБРЬ 2019 г.

ИЗДАЕТСЯ С ОКТЯБРЯ 2003 ГОДА

ВЫХОДИТ 4 РАЗА В ГОД

АЛМАТЫ
2019

UDC544.33; 544.34

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OPTIMIZATION CHROMATOGRAPHY METHOD TO IDENTIFY THE COMPOUNDS ANTHRACENE OIL OF COKE CHEMICAL PRODUCTION

Abstract. Anthracene oil is obtained by vacuum fractionation from coal tar from corporation "ArcelorMittal Temirtau". The product was purified from resinous components by column chromatography. The physicochemical characteristics of anthracene oil from corporation "ArcelorMittal Temirtau" are investigated. Using the methods CMS and GLC analysis components of the hydrogenation products were determined . It is found that use the catalyst of iron-containing composite the carrier of carbon allows to increase the degree of conversion of hydrocarbon derivatives.

Key words: hydrogenation, anthracene oil, temperature, hydrogen pressure, catalyst.

Today, in many countries of the world, research and pilot works are continuing on the improvement and improvement of some indicators. These are separate stages in the processing of solid and heavy hydrocarbon feedstock and liquefaction products. This can significantly improve the efficiency of the method as a whole.

In industry, anthracene oil is obtained from coal tar. Coal tar is a complex mixture of organic compounds. The main components are aromatic hydrocarbons and heterocyclic, sulfur, oxygen and nitrogen compounds. By chemical properties, all compounds of the resin are divided into three groups: neutral, acidic and basic.

Anthracene oil is a fraction of a greenish-yellow color obtained during the distillation of coal tar, boiling away within 280-360°C (up to 400°C), its density is 1.09-1.10 g/cm^3. It is a complex mixture of high-boiling, mainly aromatic, compounds, the main of which are anthracene (5% content), phenanthrene - anthracene isomer (20%) and carbazole (6%). When cooled anthracene oil stands out soft mass, the main part of which is anthracene. Anthracene oil is used to isolate anthracene, phenanthrene, to produce carbon black. Anthracene oil is one of the best wood preservatives, so it is used for preparing sleeper impregnation oil.

The feedstock was used coal tar received in the company of "ArselorMittal Temirtau" selected distillation. The tar yield in the production of coke is 6-8% of dry coal. Consequently, the released resin is very similar in structure and appearance to functional groups and structural fragments with the organic mass of the original coal.

An analysis method has been developed for the chromatograph used, including the selection of a column, the testing of parameters (temperature regimes for the injector, detector and thermostat, pressure and gas flow rates). GLC analysis was carried out on a Crystallux 4000M chromatograph with a PID detector on a ZB-5 30m x 0.32mm x 0.25μm column. with thermostat temperature programming with a temperature of 60-28°C at a temperature rise rate of 60°C/min. Data pro-cessing was provided by the program «NetChromv. 2.1". In compiling the data-base of component composition, we used the results of the analysis of standard samples contained in the obtained resin fraction (tetralin, naphthalene, diphenyl, phenanthrene, anthracene), and a similar CMS analysis The CMS study was car-ried out on an HP 5890/5972 MSD instrument manufactured by Agilent (USA), on a DB-5ms capillary column 30m x 0.250mm x 0.50μm, in the temperature range 60-300°C. Substance identification was performed using the NIST98 mass spectral database.

To work out the GLC method for the analysis of coal tar by company "ArcelorMittal Temirtau", vacuum fraction was used to extract a fraction with a boiling point of up to 2100°C/10mmHg. Tar mpurities greatly interfere with the analysis. Since they are deposited in the chromatograph injector and in a capillary column, in which the analyzed object is divided into separate components. Therefore, part of the obtained fraction was purified by the method of column chromatography on silica gel of the brand KSMG/0.15-0.3mm., eluent benzene-hexane 3:1. The resulting solution is evaporated under reduced pressure on a rotary evaporator to the required concentration. Optimal conditions have been established for this object by selection, under which the mixture is divided into individual components (figure 1) on the Crystallux 4000M chromatograph.

It is known that phenols and polyaromatic compounds are present in the sample. Therefore, mixed samples of the starting product with available phenol,

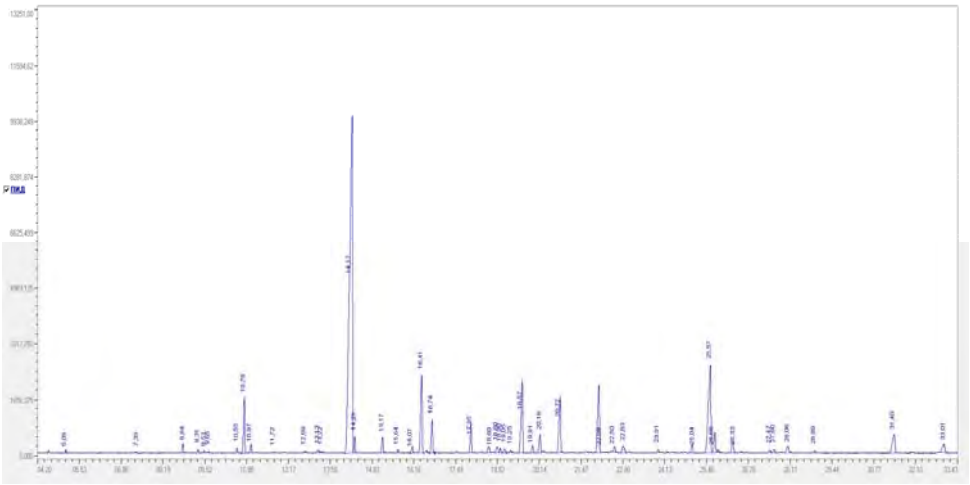

Figure 1 – Chromatogram of coal tar fraction (GLC analysis)

naphthalene derivatives, hydrogenated and individual polyaromatic hydrocarbons were prepared and analyzed. As a result, the main part of the composition of the fraction was determined.

The remaining components were determined using chromato-mass spectrometric analysis in close temperature and gas parameters on a DB-5ms column, an analogue of ZB-5 (figure 2) of the instrument HP 5890/5972 "Agilent".

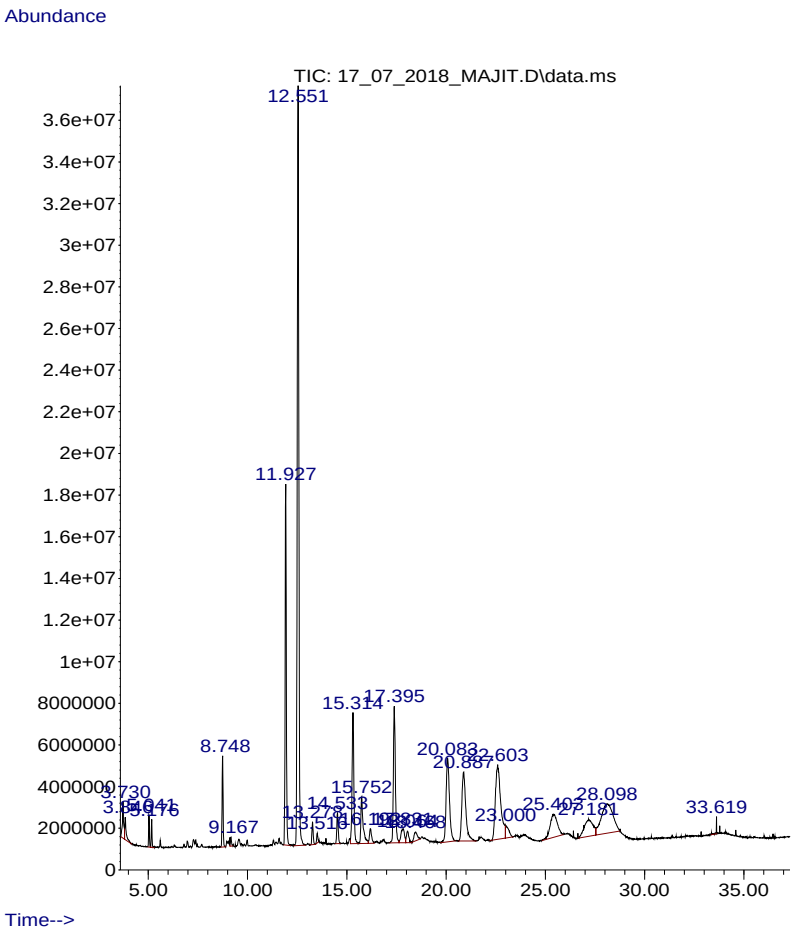

Figure 2 – Chromatogram of coal tar fraction (CMS analysis)

The study found that coal tar from company "ArcelorMittal Temirtau" is polyazeotropic-polyutectic system. Its composition contains resinous substances and pitch (up to 50%). The physicochemical characteristics of anthracene oil of company “ArcelorMittal Temirtau” were studied.

The study of the analysis results allowed to determine that the individual chemical composition of the anthracene fraction of coal tar consists of naphthalene and naphthalene derivatives, polyaromatic hydrocarbons (figures 3, 4).

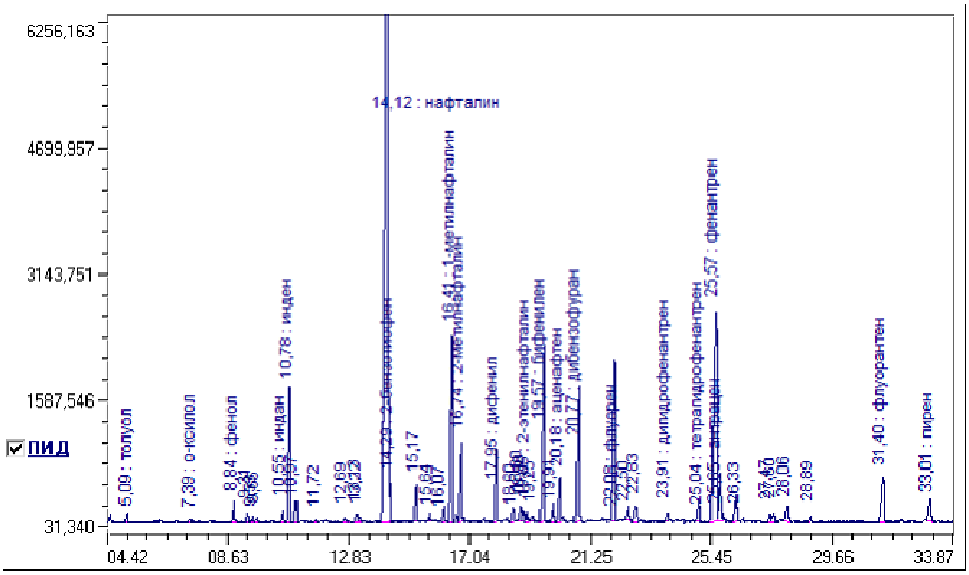

Figure 3 – Chromatogram of anthracene oil fraction

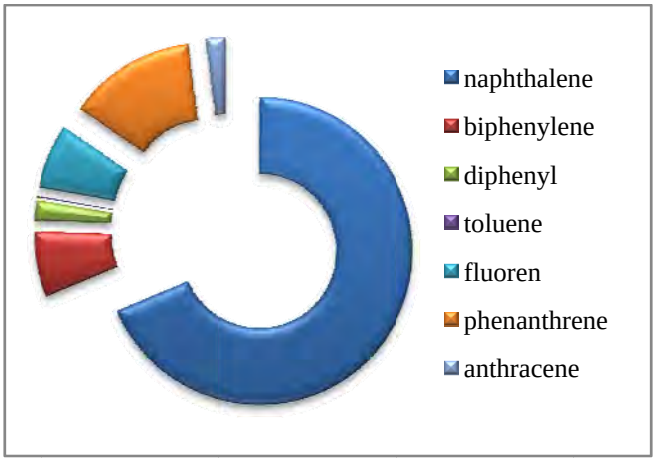

Figure 4 – Diagram of the main components
of the hydrogenated oil fraction of anthracene oil

During the GLC analysis of the anthracene fraction of coal tar, the component composition was set: phenanthrene 9.77%, anthracene 1.54%, naphthalene derivatives > 50%, aromatics > 3%, phenol derivatives> 1%, biphenyline < 6%. The composition of the object under investigation is presented in table.

Mass component identified components is nearly 92% of the total weight of the tar. Unidentified compounds, which account for 8% of the total mass, are of no practical interest at this stage, since each of them accounts for less than 0.01%.

Component composition of the anthracene oil fraction

Component	Time, min	Concentration, %
phenol	8,84	0,42
indan	10,55	0,26
inden	10,78	3,02
naphthalene	14,12	46,58
2-benzothiophene	14,29	0,67
1 methylnaphthalene	16,41	5,07
2-methylnaphthalene	16,74	1,95
diphenyl	17,95	1,67
2-Etenylnaphthalene	19,05	0,30
biphenylene	19,57	5,44
acenaften	20,18	1,25
dibenzofuran	20,77	4,28
fluoren	22,08	5,35
dihydrofenanthrene	23,91	0,20
tetrahydrofenanthrene	25,04	0,58
phenanthrene	25,57	9,77
anthracene	25,65	1,54
fluoranthene	31,40	2,29
pyrene	33,01	0,92
Total	91,77	

Thus, the following main components are present in the high-boiling anthracene fraction of coal tar with a content of 1% and higher: acenaphthene, biphenylene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene. The first three components are concentrated in the absorption fraction and have a significant effect on the formation temperature. When changing the place of selection in the direction of decreasing temperature, the quality of the absorption oil will improve due to the relative increase in the content of methylnaphthalenes. The intermediate fraction 270–310^{0}C, in which acenaphten, diphenylene and fluoren is concentrated, can be used as raw material for further processing to individual products.

Financing source of research *was performed in the framework of the program-target research (№BR05236438) with the financial support of the Committee of science of the ministry of education and science of the Republic of Kazakhstan.*

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Резюме

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КОКСОХИМИЯ ӨНДІРІСІНДЕ АНТРАЦЕН МАЙЫНЫҢ КОМПОНЕНТТІК ҚҰРАМЫН АНЫҚТАУДА ХРОМАТОГРАФИЯЛЫҚ ӘДІСТЕМЕНІ ОҢТАЙЛАНДЫРУ

«АрселорМиталл Теміртау» АҚ тас көмір шайырынан вакуумды бөлу арқылы антрацен майы алынды. Өнімді шайырлы заттардан тазарту бағаналы хроматографиямен жүргізілді. «АрселорМиталл Теміртау» АҚ антрацен майының физико-химиялық көрсеткіштерді зерттелінді.

ГСХ мен ХМС әдістемелерімен гидрлеу өнімінің компонентті құрамы дәйектенді. Теміркұрамды композитті катализаторды пайдалану көмірсутегі туындарының конверсия дәрежесінің жоғарлауына мүмкіндік беретіні анықталды.

Түйін сөздер: гидрогенизация, антрацен майы,температура, сутегі қысымы, катализатор.

Резюме

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М. Г. Мейрамов, Г. Н. Мусина, Ж. Қ. Богжанова

ОПТИМИЗАЦИЯ ХРОМАТОГРАФИЧЕСКОГО МЕТОДА ИДЕНТИФИКАЦИИ КОМПОНЕНТНОГО СОСТАВА АНТРАЦЕНОВОГО МАСЛА КОКСОХИМИЧЕСКОГО ПРОИЗВОДСТВА

Вакуумным фракционированием из каменноугольной смолы АО «АрселорМиттал Темиртау» получено антраценовое масло. Проведена очистка продукта от смолистых компонентов колоночной хроматографией. Исследованы физико-химические характеристики антраценового масла АО «АрселорМиттал Темиртау».

Методами ХМС и ГЖХ анализа установлен компонентный состав продуктов гидрирования. Установлено, что применение железосодержащих композитных катализаторов на углеродном носителе позволяет повысить степень конверсии углеводородных производных.

Ключевые слова: гидрогенизация, антраценовое масло,температура, давление водорода, катализатор.