NAMANGAN DAVLAT UNIVERSITETI
ILMIY AXBOROTNOMASI, [2026-9]
ISSN:2181-1458
ISSN:2181-0427

02.00.00-KIMYO FANLARI
journal.namdu.uz
CHEMICAL SCIENCES

FRACTIONATION OF PYROLYSIS PRODUCTS AND IDENTIFICATION OF TARGET AROMATIC HYDROCARBONS FOR THE VINYLATION PROCESS

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Abstract: *The chemical composition of the pyrolysis distillate and oil produced at the «Uz-Kor Gas Chemical» complex was studied using the GC-MS method, and the composition, relative fractions, and fractional distribution of aromatic hydrocarbons were determined. In the pyrolysis distillate, 97.17 % of the components were identified, and the predominance of arenes was confirmed with a proportion of 67.18 %. As a result of the analysis of the sequentially obtained samples, a decrease in the proportion of light aromatic hydrocarbons and an increase in the proportion of C_9 alkylbenzenes and high-boiling aromatic compounds were noted. The highest relative proportion of m-ethyltoluene was 17.91 %, in the fraction above 200 °C, it was 8.96 % of 1-methylenediene, and the proportion of azulen in some samples was 33.75%. The pyrolysis oil was rectified at a cubic temperature of 220 °C. During the process, four parts of the liquid were returned to the column relative to each part of the separated distillate. As a result, the fraction in the range of 160-180 °C was obtained with a yield of 25-28 %, while the fraction in the range of 180-200 °C was obtained with a yield of 24-26 %. The separated fractions were analyzed using the GC-MS method, and their chemical composition and distribution depending on the boiling point of the aromatic hydrocarbons were determined. It was studied with which compounds m-ethyltoluene, 1-methylinden and azulen are found in pyrolysis products. Based on these results, these three compounds were selected for further chemical synthesis work. It was determined which fractions can be used for their separate isolation and purification, and which impurities should be taken into account. The percentages given in GC-MS analysis represent the relative proportion of peak surfaces on the chromatogram, not the mass content of the substances.*

Keywords: *pyrolysis distillate, pyrolysis oil, fractionation, GC-MS, 1-methylinden, azulen, m-ethyltoluene, aromatic hydrocarbons.*

PIROLIZ MAHSULOTLARINI FRAKSIYALASH VA VINILLASH JARAYONI UCHUN MAQSADLI AROMATIK UGLEVODORODLARNI ANIQLASH

Annotatsiya: *«Uz-Kor Gas Chemical» majmuasida hosil bo‘ ladigan piroliz distillati va moyining kimyoviy tarkibi GC-MS usulida tadqiq etilib, aromatik uglevodorodlarning tarkibi, nisbiy ulushlari va fraksiyalar bo‘ yicha taqsimlanishi aniqlandi. Piroliz distillatida komponentlarning 97,17 % i identifikatsiya qilinib, arenlarning 67,18 % ulush bilan ustunlik qilishi tasdiqlandi. Ketma-ket olingan namunalarning tahlili natijasida yengil aromatik uglevodorodlar ulushining kamayishi, C_9 alkilbenzollar va yuqori haroratda qaynovchi aromatik birikmalar ulushining ortishi qayd etildi. m-Etiltoluolning eng yuqori nisbiy ulushi 17,91 %, 200 °C dan yuqori fraksiyada 1-metilindenniki 8,96 %, azulenning ayrim namunalardagi ulushi esa 33,75 % ekanligi aniqlandi. Piroliz moyi kub harorati 220 °C bo‘ lgan sharoitda rektifikatsiya qilindi. Jarayon davomida har bir qism ajratib olingan distillyatga nisbatan to‘ rt qism suyuqlik kolonnaga qaytarildi. Natijada 160-180 °C oralig‘ idagi fraksiya 25-28 %, 180-200 °C oralig‘ idagi fraksiya esa 24-26 % chiqim bilan olindi. Ajratilgan fraksiyalar GC-MS usulida tahlil qilinib, ularning kimyoviy tarkibi va aromatik uglevodorodlarning qaynash haroratiga qarab taqsimlanishi aniqlandi. Piroliz mahsulotlarida m-etiltoluol, 1-metilinden va azulen qaysi birikmalar bilan birga uchrashi o‘ rganildi. Shu natijalarga ko‘ ra, ushbu uchta birikma keyingi kimyoviy sintez ishlari uchun tanlandi. Ularni alohida ajratib olish va tozalashda qaysi fraksiyalardan foydalanish mumkinligi hamda qanday aralashmalarni hisobga olish kerakligi aniqlandi. GC-MS tahlilida berilgan foizlar moddalarning massaviy miqdorini emas, balki xromatogrammadagi pik yuzalarining nisbiy ulushini bildiradi.*

Kalit so‘ zlar: *piroliz distillati, piroliz moyi, fraksiyalash, GC-MS, 1-metilinden, azulen, m-etiltoluol, aromatik uglevodorodlar.*

ФРАКЦИОНИРОВАНИЕ ПРОДУКТОВ ПИРОЛИЗА И ИДЕНТИФИКАЦИЯ ЦЕЛЕВЫХ АРОМАТИЧЕСКИХ УГЛЕВОДОРОДОВ ДЛЯ ПРОЦЕССА ВИНИЛИРОВАНИЯ

Аннотация: *Химический состав пиролизного дистиллята и пиролизного масла, полученных на комплексе «Uz-Kor Gas Chemical», исследован методом ГХ-МС, определены состав, относительные доли и фракционное распределение ароматических углеводородов. В пиролизном дистилляте идентифицировано 97,17 % компонентов и подтверждено преобладание аренов, доля которых составила 67,18 %. В результате анализа последовательно отобранных проб отмечены снижение доли лёгких ароматических углеводородов и увеличение доли C_9-алкилбензолов и высококипящих ароматических соединений. Наибольшая относительная доля м-этилтолуола составила 17,91 %, во фракции выше 200 °C доля 1-метилиндена составила 8,96 %, а доля азулена в отдельных пробах достигала 33,75 %. Пиролизное масло подвергали ректификации при температуре в кубе 220 °C. В ходе процесса на каждую часть отбираемого дистиллята четыре части жидкости возвращали в колонну. В результате фракция в интервале 160-180 °C была получена с выходом 25-28 %, а фракция в интервале 180-200 °C - с выходом 24-26 %. Выделенные фракции проанализированы методом ГХ-МС, определены их химический состав и распределение ароматических углеводородов в зависимости от температуры кипения. Изучено, с какими соединениями м-этилтолуол, 1-метилинден и азулен присутствуют в продуктах пиролиза. На основании полученных результатов эти три соединения были выбраны для дальнейших работ по химическому синтезу. Определено, какие фракции могут быть использованы для их раздельного выделения и очистки, а также какие примеси необходимо учитывать. Процентные значения, приведённые по результатам анализа ГХ-МС, отражают относительные доли площадей пиков на хроматограмме, а не массовое содержание веществ.*

Ключевые слова: *пиролизный дистиллят, пиролизное масло, фракционирование, ГХ-МС, 1-метилинден, азулен, м-этилтолуол, ароматические углеводороды.*

Introduction. In the petrochemical industry, during the pyrolysis of hydrocarbons, liquid secondary products are formed alongside the primary gaseous products. They contain benzene, toluene, xylenes, alkylbenzene, indene derivatives, and polycyclic aromatic compounds. The complexity of the composition of these mixtures complicates their direct processing. Therefore, it is important to first separate the liquid pyrolysis products into fractions and then determine the chemical composition of each fraction. The possibilities of assessing the hydrocarbon composition of pyrolysis gasoline using gas chromatography and predicting the distillation curve from the obtained data have been previously studied [2]. This approach demonstrates the importance of chromatographic analysis in assessing the relationship between the composition and boiling properties of hydrocarbons [3]. The gas chromatography-mass spectrometry (GC-MS) method is widely used in the study of complex hydrocarbon mixtures. This method allows for the chromatographic separation of components and the comparison of their mass spectra with library data. In particular, the NIST mass spectrum libraries are an important source of information for identifying organic compounds [4]. At the same time, the fact that the relative number of chromatographic peak surfaces does not equal the actual mass of the substances must be taken into account when evaluating quantitative results. Among the aromatic compounds in pyrolysis products, azulen and its derivatives are distinguished by their chemical structure [5]. Elwahy et al. have summarized research on palladium-catalyzed cross-linking reactions of azulen [6]. Subsequent studies have investigated the expansion of the azulen ring and the functionalization of C-H bonds [7], as well as the synthesis of alkenylazulenolactones through the oxidative cyclization of azulen-2-carboxylic acids with 1,3-dienes [8]. These studies show that the azulen skeleton can be modified with various functional groups, but the direct vinylation of azulen isolated from the pyrolysis product cannot be considered confirmed in these works [9, 10]. The technological features and catalytic processes of this direction are also covered in other studies [11].

In existing studies, the composition of pyrolysis fluids, the isolation of certain aromatic components, and their chemical transformations have been studied in various directions. At the same time, it is of practical importance to jointly evaluate the fractional distribution of m-ethyltoluene, 1-methylinden, and azulen in a specific industrial pyrolysis product. The aim of this study is to determine the hydrocarbon composition of the pyrolysis distillate and oil produced at the «Uz-Kor Gas Chemical» complex using the GC-MS method, evaluate the fractional distribution of aromatic compounds, and justify the selection of 1-methylinden, azulen, and m-ethyltoluene for further chemical research.

Objects and methods of research. The object of the study was the distillate and pyrolysis oil formed during the pyrolysis of ethane, a propane-butane mixture, and gas condensate at the «Uz-Kor Gas Chemical» complex. According to technological data, the amount of pyrocondensate accounts for 2-25 % of the total pyrolysis products. The pyrolysis oil was fractionated at atmospheric pressure in a laboratory rectification unit with a stainless steel column 1200 mm high and 15 mm in diameter. At a cubic temperature of 220 °C and a phlegm number of 4, fractions of 160-180 °C and 180-200 °C were isolated. Their yield was 25-28 % and 24-26 %, respectively. The chemical composition of the samples was determined using a GC-MS system equipped with an Agilent 5977A mass-selective detector. The compounds were identified by comparing their chromatographic retention time and mass spectra with library data. The content of components was estimated based

on the relative content of chromatographic peak areas (%). The analysis focused on the fractional distribution of 1-methylinden, azulen, and m-ethyltoluene.

Results and discussion. As a result of separating pyrolysis products into fractions and studying their composition using the GC-MS method, it was established that hydrocarbons are distributed differently depending on their chemical structure and boiling point. Initially, the total composition of the pyrolysis distillate was assessed, and at the next stage, the accumulation of aromatic compounds in individual fractions was analyzed. Particular attention was paid to the content of 1-methylindene, azulen, and m-ethyltoluene, as well as the possibility of their isolation. To determine the chemical composition of the pyrolysis distillate, the obtained sample was analyzed using the GC-MS method. As a result, components corresponding to 97.17 % of the total surface area of the chromatographic peaks were identified. Of these, 67.18 % are arenes, 23.70 % are olefins, 2.57 % are dienes, 2.08 % are cycloalkanes, and 1.64 % are alkanes. The distribution of hydrocarbons by class and number of carbon atoms is shown in Table 1.

Table 1.

Composition of pyrolysis distillate by hydrocarbon class and carbon number

Carbon number	Alkanes, %	Dienes, %	Olefins, %	Cycloalkanes, %	Arenes, %
C_5	0,80	0,89	4,91	0,19	0,00
C_6	0,22	0,41	3,87	0,41	32,94
C_7	0,25	0,14	0,84	0,45	11,23
C_8	0,12	0,08	0,18	0,48	9,75
C_9	0,04	0,10	0,04	0,15	7,56
C_{10}	0,03	0,11	9,07	0,40	5,23
C_{11}	0,18	0,69	2,95	0,00	0,47
C_{12}	0,00	0,15	1,84	0,00	0,00
Total	1,64	2,57	23,70	2,08	67,18

According to the data obtained, a high content of aromatic hydrocarbons was found in the pyrolysis distillate. In particular, the content of arenes in the C_6 group is 32.94 %, which is the highest indicator among aromatic compounds. As the number of carbon atoms increases, the relative content of arenes decreases, amounting to 11.23 % in the C_7 group, 9.75 % in C_8, 7.56 % in C_9, and 5.23 % in C_{10}. This, in turn, indicates a high content of light aromatic hydrocarbons in the distillate, as well as the presence of aromatic components boiling at high temperatures.

Also, according to the results obtained, the amount of olefins differed from arenes. Their highest content was 9.07 % in group C_{10}, 4.91 % in group C_5, and 3.87 % in group C_6. Consequently, in addition to aromatic hydrocarbons, the distillate contains unsaturated compounds of various molecular masses. Such a composition is important in the fractionation process, as it indicates that certain unsaturated components can undergo secondary reactions under heating conditions. We can see that the content of alkanes, cycloalkanes, and dienes is much lower than that of aromatic hydrocarbons. Nevertheless, their presence confirms that the pyrolysis distillate is a complex multi-component mixture. The high content of arenes allows for the study of the distillate and its fractions as a source of aromatic compounds. Therefore, in subsequent analyses, the fractional distribution of aromatic components from the total hydrocarbon composition was studied. In this process, m-ethyltoluene was first isolated from C_9 alkylbenzols, followed by 1-methylinden and azulenes found in heavier fractions, and their amounts were compared. Although a high proportion of aromatic hydrocarbons was found in the pyrolysis distillate, their composition was not uniform across all samples. In particular, the content of isomers of toluene, ethylbenzene, xylene, and m-ethyltoluene differs significantly (Fig. 1).

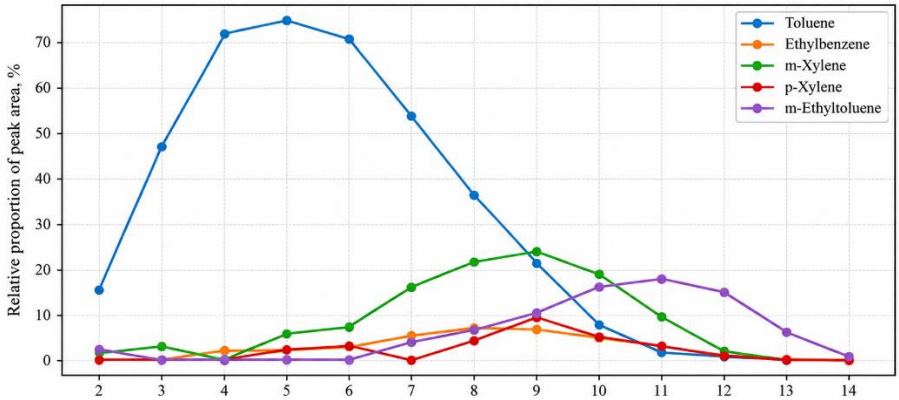

Figure 1. Changes in the relative fractions of toluene, ethylbenzene, m-xylol, p-xylol, and m-ethyltoluene across GC-MS samples.

Based on the graphical analysis, the toluene content increased in samples 2-6 and reached approximately 75 % in sample 6. In subsequent samples, its content decreased, reaching a very low level starting from sample 11. The m-Xylol content increased to sample 9 and amounted to approximately 24 %, after which it decreased. The content of ethylbenzene and p-xylol was relatively low and also varied across samples. The distribution of m-ethyltoluene deserves special attention. Its content was low in the initial samples, increasing starting from sample 8 and reaching 17.91 % in sample 11. In subsequent samples, it decreased. Consequently, samples with a predominance of toluene differ from those with a predominance of m-ethyltoluene. This circumstance indicates that the composition of aromatic compounds in the pyrolysis products is not uniform, and it is necessary to take into account the structural differences between the samples when studying C_9 alkylbenzones.

Along with light aromatic hydrocarbons, compounds with a complex structure are also found in the pyrolysis products. Therefore, in the subsequent analysis, the content of 1-methylinden and azulen in the heavier fractions was also studied. Based on this, for a detailed study of the content of aromatic hydrocarbons in various samples, the results of the GC-MS analysis of isomers of toluene, ethylbenzene, xylene, m-ethyltoluene, indene group compounds, and naphthalene were compared (Table 2).

Table 2.

Relative content of aromatic hydrocarbons in pyrolysis product samples, %

Example	Toluene	Ethylbenzene	m-Xylol	p-Xylol	m-Ethyltoluene	Indene group	Naphthalene
2	15,42	-	1,45	-	2,32	0,76	-
3	47,11	-	2,90	-	-	-	-
4	72,18	2,07	-	-	-	-	-
5	74,67	2,15	5,72	2,24	-	-	-
6	70,54	2,74	7,24	3,11	-	-	-
7	53,68	5,34	16,14	-	3,89	-	-
8	36,33	7,05	21,56	4,28	6,65	-	-
9	21,43	6,71	23,90	9,42	10,35	-	-
10	7,79	4,95	19,04	5,12	16,21	-	-
11	1,69	3,07	9,44	3,13	17,91	7,00	-
12	0,81	1,00	2,00	1,02	15,05	11,63	-
13	0,17	0,29	-	-	6,16	21,72	-
14	-	-	-	-	0,90	15,44	32,66

According to the table results, the highest toluene content was 74.67 % in sample 5. In subsequent samples, its content decreased to 0.17 % in sample 13. At the same time, the content of m-xylol in sample 9 reached 23.9 %, and m-ethyltoluene in sample 11 reached 17.91 %. These differences indicate that aromatic hydrocarbons are not found in equal quantities in all samples. The content of Indene group compounds was 7 % in sample 11, 11.63 % in sample 12, and 21.72 % in sample 13. Naphthalene was recorded in the table only in sample 14, and its relative content reached 32.66 %. Thus, in the samples with subsequent numbers, the content of toluene and xylene decreased, while the indene group compounds and naphthalene accounted for a significant share. The results obtained show that m-ethyltoluene occurs in some samples with a high relative proportion, while indene group compounds occur in other samples.

After determining the changes in aromatic hydrocarbons depending on the samples in GC-MS analyses, their boiling points and quantitative compositional proportions were compared. These data make it possible to estimate which compounds can be separated in a temperature range close to each other during the fractionation process (Table 3).

Table 3.

Relative content and boiling points of aromatic compounds in a pyrolysis product sample

Component	Relative content, %	Boiling point, °C
o-Xylol	2,05	144,4
1-Methyl-3-ethylbenzene	10,51	161,3
1,2,4-trimethylbenzene	4,82	168,0
1-Methyl-4-ethylbenzene	3,23	162,0
1,3,5-Trimethylbenzene	22,39	164,7
1,2,3-trimethylbenzene	3,86	176,0
1H-Indene	11,49	182,8
1,2-Dymethyl-4-ethylbenzene	2,01	189,75

According to the results obtained, the relative content of 1,3,5-trimethylbenzene is 22.39 %, which is the highest indicator among the presented compounds. Its boiling point is 164.7 °C, which is close to the boiling points of m-ethyltoluene (161.3 °C) and p-ethyltoluene (162.0 °C). These compounds can be difficult to completely separate from each

other by simple fractionation. The content of 1n-inden is 11.49 %, and the boiling point is 182.8 °C. Its boiling at a higher temperature helps explain the occurrence of indene group compounds in heavier fractions. However, since 1h-inden and 1-methyinden are different compounds, this result cannot be interpreted as the amount of 1-methyinden. Thus, the proximity of the boiling points of aromatic compounds to each other can lead to the combination of several components within the fractions. Therefore, in the subsequent analysis, the results of pyrolysis oil rectification and the distribution of heavier aromatic compounds were considered. In the light aromatic fractions of pyrolysis products, alkylbenzols and indene were primarily identified, while naphthalene and its alkyl derivatives accounted for a high proportion in the fraction separated at temperatures above 200 °C. The presence of 1-methylinden in this fraction was also determined by GC-MS analysis. The relative proportions of the main components are presented in Table 4.

Table 4.

The main aromatic compounds in the pyrolysis oil fraction above 200 °C

Component	**Relative content, %**
Inden	9,33
1-Methylinden	8,96
Naphthalene	41,51
1-Methylnaphthalene	8,61
2-Methylnaphthalene	16,25
1-ethylnaphthalene	1,77
1,6-dimethylnaphthalene	1,71

According to the table data, naphthalene is the main component in this fraction, accounting for 41.51 %. Among its methyl derivatives, 2-methylnaphthalene accounted for 16.25 %, and 1-methylnaphthalene for 8.61 %. The relative content of indene was 9.33%, and the 1-methylenedi selected for the study was 8.96 %. These results indicate that 1-methylinden is found together with naphthalene and its derivatives.

The detection of 1-methylinden in this fraction allows for its consideration as a promising component for further isolation and purification studies. Despite the high content of naphthalene and its alkyl derivatives in the fraction, 1-methylenediene was also detected in an amount of 8.96%. To obtain it in its pure form, additional purification of the fraction is required. To separate the aromatic hydrocarbons contained in the pyrolysis oil by boiling point, the rectification process was carried out at a cubic temperature of 220 °C and a phlegm number of 4. Fractions ranging from 160-180 °C to 180-200 °C were obtained. Their yield was 25-28 % and 24-26 %, respectively. Rectification conditions and fraction yields were determined (Table 5).

Table 5.

Rectification conditions for pyrolysis oil and fraction yield

Indicator	160-180°C fraction	180-200°C fraction
Cube temperature, °C	220	220
Product separation interval, °C	160-180	180-200
Column top temperature, °C	≤160	≤160
Output, %	25-28	24-26
Phlegm count	4	4

According to the table results, the yields of the isolated fractions under identical rectification conditions were close to each other. The yield of fractions in the range of 160-180 °C is relatively high, indicating the presence of significant amounts of boiling components in the pyrolysis oil within this temperature range. The identification of isomers of m-ethyltoluene and trimethylbenzene in previous analyses indicates the significance of this fraction for studying the composition of aromatic compounds. In the 160-180 °C fraction, C9 alkylbenzones are found, while in the 180-200 °C fraction, indeen and other higher-boiling aromatic compounds are found. ° Thus, the pyrolysis oil was separated depending on the boiling point, and the chemical composition of the fractions was studied separately.

Based on the results of fractionation of pyrolysis products and GC-MS analysis, the presence of aromatic compounds in different samples was revealed. m-ethyltoluene, 1-methylindene, and azulen were found in different amounts in the pyrolysis products. The highest relative proportion of m-ethyltoluene was 17.91 %, in the fraction above 200 °C, the proportion of 1-methylindene was 8.96 %, and the proportion of azulen in some samples was 33.75 %. Their presence in samples of different compositions indicates that the distribution of aromatic compounds during the fractionation process is not uniform. Based on the results obtained, m-ethyltoluene, 1-methylinden, and azulen were selected for further research.

Conclusion

The pyrolysis distillate and oil of the «Uz-Kor Gas Chemical» complex were investigated using the GC-MS method, the aromatic hydrocarbons in their composition were identified, and their distribution by fractions was determined. The total relative share of identified components in the pyrolysis distillate reached 97.17 %. It was established that arenes predominate with a share of 67.18 %, and the rich composition of aromatic hydrocarbons in this secondary product was confirmed by experimental results.

The presence of m-ethyltoluene,1-methylinden, and azulen in the pyrolysis products was determined by GC-MS analysis. The highest relative proportion of m-ethyltoluene was 17.91 %, in the fraction above 200 °C, it was 8.96 % for 1-methylinden, and the proportion of azulen in certain samples was 33.75 %. Thus, accurate chromatographic data on the quantitative distribution of all three compounds in the pyrolysis products were obtained. By rectifying pyrolysis oil at a cubic temperature of 220°C and a phlegm number of 4, the fraction in the 160-180°C range was isolated with a yield of 25-28%, while the fraction in the 180-200°C range was isolated with a yield of 24-26 %. The chemical composition of the isolated fractions was studied, and the distribution characteristics of aromatic components depending on the boiling point were determined. As a result of the study, the presence of 1-methylinden, azulen, and m-ethyltoluene in the pyrolysis products was confirmed by GC-MS analysis. The relative proportions of these compounds in various fractions and the aromatic hydrocarbons accompanying them were determined. Based on the results obtained, all three compounds were selected as target raw material components for further chemical synthesis research.

Foydalanilgan adabiyotlar

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