

FTIR AND GAS CHROMATOGRAPHIC CHARACTERIZATION OF C9 HYDROCARBONS IN PYROLYSIS DISTILLATE SEPARATED AT 159 °C USING SULZER PACKING

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ARTICLE INFO

Dates:

Received: 19.09.2026

Revised: 28.09.2026

Accepted: 30.09.2026

DOI:

10.66960/jof.3093-8899.00039

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ANNOTATSIYA

Ushbu ishda 159 °C da Sulzer strukturali nasadkali kolonka yordamida piroliz distillatidan ajratilgan uglevodorod fraksiyasini FTIR-spektroskopiya va gaz xromatografiyasi usullari bilan tahlil qilish maqsad qilingan. Ushbu tadqiqotda Sulzer strukturali nasadkasi yordamida 159 °C da ajratilgan piroliz distillatining tarkibi o'rganildi. Ajratilgan fraksiya strukturaviy xususiyatlari va uglevodorod tarkibini aniqlash maqsadida Furye-transformatsiyali infraqizil (FTIR) spektroskopiya va gaz xromatografiyasi usullari yordamida tahlil qilindi. FTIR spektrida alifatik, to'yinmagan va aromatik uglevodorod tuzilmalariga xos yutilish polosalari kuzatildi. Gaz xromatografik tahlil natijasida bir nechta C9 uglevodorodlari aniqlandi va ular orasida 1H-indene ustunlik qildi. Aniqlangan C9 uglevodorodlarining umumiy nisbiy miqdori 8,70% ni, 1H-indene ulushi esa xromatografik maydonning 8,02% ini tashkil etdi. Natijalar 159 °C da Sulzer nasadkasi orqali fraksion haydash C9 aromatik uglevodorodlarining sezilarli qismini o'z ichiga olgan fraksiyani olish imkonini berishini ko'rsatdi. Natijada, ajratilgan fraksiya keyingi tozalash va neft-kimyoyarayonlarida foydalanish uchun istiqbolli oraliq mahsulot sifatida qoralishi mumkin.

Kalit so'zlar: piroliz distillati, C9 uglevodorodlari, Sulzer nasadkasi, fraksion haydash, uglevodorod fraksiyasi, FTIR spektroskopiyasi, gaz xromatografiyasi

АННОТАЦИЯ

Цель данного исследования — проанализировать с помощью ИК-фурье-спектроскопии и газовой хроматографии углеводородную фракцию, выделенную из пиролизного дистиллята при температуре 159 °C в колонне, оснащенной регулярной насадкой Sulzer. В данной работе исследован состав пиролизного дистиллята, выделенного при 159 °C с использованием структурированной насадки Sulzer. Выделенную фракцию исследовали методами инфракрасной спектроскопии с преобразованием Фурье (FTIR) и газовой хроматографии для определения ее структурных характеристик и углеводородного состава. В FTIR-спектре наблюдались характерные полосы поглощения, соответствующие алифатическим, ненасыщенным и ароматическим углеводородным структурам. По результатам газохроматографического анализа были обнаружены несколько C9-углеводородов, среди которых преобладал 1H-инден. Суммарное относительное содержание идентифицированных C9-углеводородов составило 8,70%, при этом содержание 1H-индена достигало 8,02% от общей хроматографической площади. Полученные результаты показывают, что фракционная перегонка при 159 °C с использованием насадки Sulzer позволяет получать фракцию, содержащую заметную долю ароматических C9-углеводородов. Следовательно, выделенная фракция может рассматриваться как перспективный промежуточный продукт для дальнейшей очистки и использования в нефтехимической переработке.

Ключевые слова: пиролизный дистиллят, C9-углеводороды, насадка Sulzer, фракционная перегонка, углеводородная фракция, FTIR-спектроскопия, газовая хроматография

ABSTRACT

This study aims to analyze, using FTIR spectroscopy and gas chromatography, a hydrocarbon fraction separated from pyrolysis distillate at 159°C in a column equipped with Sulzer structured packing. This study investigated the composition of the pyrolysis distillate fraction separated at 159 °C using Sulzer structured packing. The isolated fraction was characterized by Fourier transform infrared (FTIR) spectroscopy and gas chromatography to determine its structural features and hydrocarbon composition. The FTIR spectrum showed characteristic absorption bands associated with aliphatic, unsaturated, and aromatic hydrocarbon structures. Gas chromatographic analysis identified several C9 hydrocarbons, among which 1H-indene was predominant. The combined relative content of the identified C9 hydrocarbons reached 8.70%, while 1H-indene accounted for 8.02% of the total chromatographic area. The results demonstrate that Sulzer-packed fractional distillation at 159 °C can produce a fraction containing a measurable proportion of C9 aromatic hydrocarbons. Consequently, the separated fraction may be considered a potential intermediate for further purification and petrochemical processing.

Keywords: pyrolysis distillate, C9 hydrocarbons, Sulzer packing, fractional distillation, hydrocarbon fraction, FTIR spectroscopy, gas chromatography

How to cite:

Imanova N.Sh., Bekturdiyev G.M., Kodirov O.Sh., Allambergenov A.D. (2026). FTIR AND GAS CHROMATOGRAPHIC CHARACTERIZATION OF C9 HYDROCARBONS IN PYROLYSIS DISTILLATE SEPARATED AT 159 °C USING SULZER PACKING. *Journal of Future*, 2(6), 1–5. <https://doi.org/10.66960/jof.3093-8899.00039>

Introduction

The increasing demand for hydrocarbon resources and the need for more efficient utilization of secondary hydrocarbon streams have intensified research into thermochemical conversion and subsequent fractionation technologies [1-3]. Pyrolysis provides an important route for converting complex hydrocarbon feedstocks into liquid products containing a broad range of hydrocarbons that can potentially serve as fuels, blending components, or chemical feedstocks [4, 5]. However, the direct use of pyrolysis distillates is often limited by their complex composition and relatively broad boiling range [6]. Consequently, controlled fractionation followed by detailed compositional analysis is required to identify valuable hydrocarbon fractions and determine their potential applications [7-10].

Pyrolysis-derived liquid products commonly contain paraffinic, olefinic, cycloalkane, and aromatic hydrocarbons with different carbon numbers [11]. Among these components, the C9 hydrocarbon fraction is of particular interest because it may contain valuable aromatic and unsaturated compounds that can be used as intermediates in petrochemical processes [12]. Detailed analysis of C9 fractions is therefore important for evaluating their chemical value and determining suitable routes for further processing [13, 14]. Previous chromatographic investigations have demonstrated that C9 fractions can contain numerous individ-

ual hydrocarbons, and that reliable identification requires a combination of chromatographic retention characteristics and spectrometric information [15]. For example, a detailed investigation of pyrolysis-gasoline C9 fractions identified 39 compounds with concentrations above 0.2%, while the relative standard deviation of the quantitative GC analysis was reported to be below 5% [16-18].

The chemical composition of pyrolysis distillates is strongly affected by both the origin of the feedstock and the conditions of thermal conversion [19]. Feedstock composition determines the types of primary pyrolysis products, while temperature, residence time, heating rate, and secondary reactions influence the transformation of larger hydrocarbon molecules into lighter products [20]. As a result, the composition of a target C9 fraction cannot be assumed solely from its boiling range [21]. Experimental fractionation and instrumental characterization are necessary to establish the actual distribution of hydrocarbons in each separated fraction [22]. This consideration is particularly important for complex pyrolysis distillates derived from regional hydrocarbon resources, including feedstocks originating from the Ustyurt region [23].

Fractional distillation is one of the most practical methods for separating multicomponent hydrocarbon mixtures according to volatility [24]. The separation performance of a packed column depends on vapor-liquid contacting, mass-transfer efficiency, packing geometry, pressure drop, and operating conditions. Sulzer structured packings are widely

used in separation technology because their geometry promotes effective vapor–liquid contact while maintaining comparatively low pressure drop [25]. Experimental studies comparing structured and random packings have shown that packing characteristics can significantly influence height equivalent to a theoretical plate (HETP) and overall separation efficiency [26]. Sulzer's Mellapak-type structured packings are specifically designed to provide high mass-transfer efficiency and improved column performance [27].

The selection of the distillation temperature is also critical when attempting to isolate a particular hydrocarbon fraction. A recent study of atmospheric distillation of pyrolysis distillate investigated fractions obtained at 152, 159, and 170 °C and demonstrated a clear dependence of C9 fraction yield on the selected temperature. The reported C9 fraction yields were 11.11% at 152 °C, 4.59% at 159 °C, and 4.72% at 170 °C, indicating that relatively small changes in fractionation temperature can substantially affect the amount of C9-containing material recovered. These findings demonstrate the importance of carefully selecting the separation temperature when developing a process for the recovery of a target hydrocarbon fraction [28–30].

Instrumental analysis is essential for evaluating the chemical characteristics of the separated product [31]. FTIR spectroscopy provides rapid information concerning the structural features of hydrocarbon mixtures through characteristic absorption bands associated with C–H stretching and deformation and vibrations of unsaturated and aromatic structures [32–33]. Gas chromatography, in contrast, enables separation of individual volatile components and determination of their relative abundance [34]. Studies devoted specifically to C9 fractions have successfully combined GC, GC–MS, and infrared spectroscopic information to improve compound identification and overcome difficulties associated with co-elution and similar mass spectra [35]. Thus, the combined use of FTIR and gas chromatography provides complementary information for characterizing complex pyrolysis-derived hydrocarbon fractions [36].

Despite the considerable interest in the chemical utilization of pyrolysis liquids, the characterization of a C9-enriched fraction obtained from pyrolysis distillate using Sulzer packing at 159 °C remains insufficiently documented [37]. In particular, there is a need to relate a defined fractional-distillation temperature and packed-column configuration to the structural and compositional characteristics of the resulting hydrocarbon fraction [38]. Therefore, this study investigates the fraction obtained from pyrolysis distillate at 159 °C using Sulzer packing. The separated fraction was characterized by Fourier transform infrared (FTIR) spectroscopy and gas chromatography to identify its principal structural features and determine the composition and relative contribution of C9 hydrocarbons. The study aims to assess the suitability of Sulzer-packed fractional distillation for obtaining a C9-containing hydrocarbon fraction and to provide a basis for its further petrochemical utilization [39, 40].

Research Methods

Pyrolysis Distillate

The material investigated in this study was a liquid pyrolysis distillate obtained from hydrocarbon feedstock originating from the Ustyurt region. The distillate contained a complex mixture of hydrocarbons with different molecular structures and boiling characteristics. Before fractionation, the sample was homogenized and stored in a tightly sealed glass container to minimize the loss of volatile components.

Fractional Distillation Using Sulzer Packing

Fractional distillation was performed using a laboratory-scale packed distillation unit equipped with Sulzer structured packing. A predetermined mass of pyrolysis distillate was introduced into the distillation flask, and the system was assembled with the packed fractionation column, thermometer, and condenser.

The distillation was conducted under atmospheric pressure. The pyrolysis distillate was gradually heated, and the temperature of the vapor phase was continuously monitored. The fraction distilling at approxi-

mately 159 °C was separately collected in a receiving vessel. After collection, the fraction was cooled to room temperature and transferred to a sealed container until further analysis.

The Sulzer packing facilitated repeated vapor–liquid contact within the fractionation column and enhanced the separation of components according to their volatility. The use of a structured packing was intended to improve contact between the liquid and vapor phases and thereby obtain a comparatively narrow hydrocarbon fraction around the selected distillation temperature.

Determination of Fraction Yield

The yield of the fraction collected at 159 °C was determined gravimetrically.

FTIR Spectroscopic Analysis

The separated fraction was analyzed by Fourier transform infrared (FTIR) spectroscopy to characterize its principal molecular structural features. The FTIR spectrum was recorded over the appropriate mid-infrared spectral range. The absorption bands were used to identify characteristic vibrations associated with hydrocarbon structures. The functional groups and structural characteristics of the pyrolysis distillate fraction were investigated using a PerkinElmer Spectrum Two Fourier transform infrared (FTIR) spectrometer. Model Spectrum Two (Std), Spektral diapazon 8300–350 cm^{−1}, Detektor Standart (LiTaO₃), Optik material KBr, Optik resolution 0.4 cm^{−1}, To'liqin sonini o'rnatish aniqligi ±0.5 cm^{−1}.

Particular attention was given to the spectral regions corresponding to aliphatic C–H stretching and bending vibrations, as well as vibrations associated with C=C and aromatic structures. The obtained spectrum was used as complementary information for interpretation of the chromatographic composition of the separated fraction

Gas Chromatographic Analysis

The hydrocarbon composition of the fraction obtained at 159 °C was determined by gas chromatography (GC). The sample was injected into the chromatographic system under controlled operating conditions, and the volatile components were separated according to their retention behavior. Instrument model: 7000D, Triple Quadrupole GC/MS, Ion source: Electron impact, 150–350 °C, Ionization type: Electron ionization (EI), Quadrupole, monolithic, hyperbolic, Mass range: 10–1050 am

Individual chromatographic peaks were identified by comparison of retention times and, where applicable, by comparison with reference data or a spectral/library database. The relative content of the identified components was calculated from the corresponding chromatographic peak areas.

Special attention was given to compounds containing nine carbon atoms (C9 hydrocarbons). The relative contribution of C9 hydrocarbons to the total chromatographic composition was determined by summing the peak-area percentages of the identified C9 hydrocarbon components.

Data Processing

The FTIR and GC data were evaluated together to obtain a comprehensive characterization of the fraction separated at 159 °C. FTIR spectroscopy was used to establish the principal structural characteristics of the hydrocarbon mixture, whereas gas chromatography was used to determine the individual component distribution and the relative contribution of C9 hydrocarbons.

The results obtained using Sulzer structured packing at 159 °C were subsequently used to assess the effectiveness of the selected fractionation conditions for recovering a C9-containing hydrocarbon fraction from the pyrolysis distillate.

Result and Discussion

Fractional distillation of the pyrolysis distillate using Sulzer structured packing enabled the isolation of a hydrocarbon fraction at 159 °C. The obtained fraction was subsequently subjected to instrumental characterization to determine its structural features and hydrocarbon composition. First, Fourier transform infrared (FTIR) spectroscopy was applied to characterize the functional and structural groups present in the sepa-

rated fraction. The FTIR spectrum of the fraction obtained at 159 °C is presented in Figure 1.

The FTIR spectrum of the pyrolysis distillate fraction separated at 159 °C using Sulzer packing is shown in Figure 1. The analysis was performed to identify the characteristic infrared absorption regions of the compounds present in the isolated hydrocarbon fraction. The resulting spectrum provides structural information that can subsequently be correlated with the detailed compositional data obtained by gas chromatography.

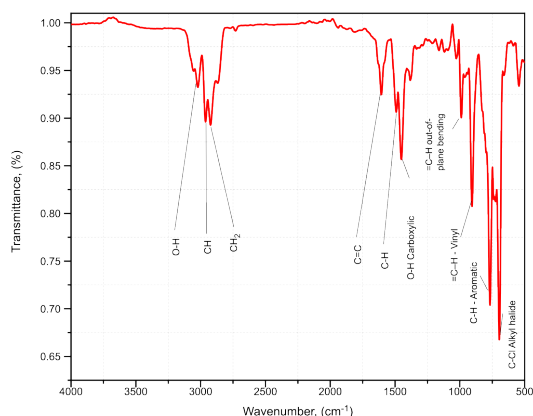


Figure 1. FTIR spectrum of the pyrolysis distillate fraction separated at 159 °C using Sulzer structured packing.

A pronounced group of absorption bands appears in the region of approximately 3000–2800 cm^{-1} , which is characteristic of C-H stretching vibrations of aliphatic CH_3 and CH_2 groups. The relatively distinct absorptions in this region indicate that saturated hydrocarbon structures constitute an important part of the separated fraction.

The spectrum also contains a band in the vicinity of 1600 cm^{-1} , which can be associated with C=C stretching vibrations of unsaturated structures and/or aromatic ring vibrations. This feature suggests the presence of unsaturated and potentially aromatic hydrocarbons in addition to saturated compounds.

Absorption bands in the approximate 1460–1370 cm^{-1} region are attributable mainly to C-H deformation vibrations of CH_2 and CH_3 groups. These bands are consistent with the presence of aliphatic hydrocarbon chains and substituted hydrocarbon structures.

The lower-wavenumber region below approximately 1300 cm^{-1} contains a series of complex and relatively intense absorption features. This fingerprint region can include C-C and C-H skeletal and deformation vibrations and is particularly useful for differentiating between structurally diverse hydrocarbon components. The complexity of this region is consistent with the multicomponent nature of the pyrolysis-derived fraction.

Overall, the FTIR spectrum indicates that the fraction separated at 159 °C contains predominantly aliphatic hydrocarbon structures together with unsaturated and aromatic components. However, FTIR spectroscopy alone does not allow reliable identification or quantitative determination of individual C9 hydrocarbons. Therefore, gas chromatographic analysis is required to establish the detailed composition and relative content of the C9 fraction.

Gas Chromatographic Analysis

Gas chromatographic analysis was performed to determine the individual hydrocarbon components present in the fraction separated at 159 °C using Sulzer structured packing. The chromatographic method provided detailed information on the retention behavior and relative abundance of the compounds present in the isolated fraction. The obtained chromatogram is presented in Figure 2, while the identified components and their corresponding relative peak areas are summarized in Table 1.

The combined chromatographic data were used to evaluate the hydrocarbon composition of the separated fraction and, in particular, to determine the contribution of **C9 hydrocarbons** to the total composition. Detailed interpretation of the chromatographic peaks and quantitative assessment of the C9 components are discussed below based on the results presented in Figure 2 and Table 1.

The gas chromatographic analysis showed that the fraction obtained at 159 °C using Sulzer structured packing contained several hydrocarbons with nine carbon atoms. The identified C9 hydrocarbons were mainly represented by 1H-indene, which appeared at three retention times with relative contents of 3.99, 2.57, and 1.46%, respectively. Their combined contribution was therefore 8.02%.

In addition, benzene, 1-propenyl-, a C9 aromatic hydrocarbon, was detected with a relative content of 0.68% at a retention time of 9.892 min.

Consequently, the total relative content of the identified C9 hydrocarbons was 8.70%. The predominant component was 1H-indene, accounting for 8.02 percentage points, or approximately 92.2% of the identified C9 hydrocarbon contribution:

Thus, the C9 hydrocarbon fraction obtained at 159 °C was characterized predominantly by indene-type aromatic hydrocarbons, while the contribution of 1-propenylbenzene was comparatively small.

The gas chromatographic results demonstrate that the fraction separated at 159 °C using Sulzer structured packing contains a complex mixture of hydrocarbon and heteroatom-containing compounds. Within this mixture, several compounds corresponding to the C9 carbon-number group were detected, with 1H-indene representing the major C9 hydrocarbon component. The repeated detection of 1H-indene at different retention times indicates that indene is an important constituent of the isolated fraction.

The total relative content of the identified C9 hydrocarbons was calculated as 8.70% from the chromatographic peak areas. The contribution of 1H-indene was 8.02%, corresponding to approximately 92.2% of the identified C9 hydrocarbon content, whereas 1-propenylbenzene accounted for the remaining 0.68%. This distribution demonstrates that the C9 hydrocarbon fraction is strongly dominated by an indene-type aromatic structure.

The relatively high proportion of indene can be associated with the thermal transformation of larger hydrocarbon structures during pyrolysis. Aromatic and condensed-ring compounds can form through secondary reactions of unsaturated pyrolysis vapors, including cyclization and dehydrogenation processes. Consequently, the presence of indene in appreciable concentration is consistent with the complex aromatic character typically encountered in pyrolysis-derived liquid products.

The use of Sulzer structured packing at 159 °C also plays an important role in the fractionation process. Efficient vapor–liquid contact within a structured packed column can improve the separation of components with relatively close boiling characteristics. Therefore, the composition observed in the collected fraction reflects not only the original composition of the pyrolysis distillate but also the selective fractionation achieved under the applied distillation conditions.

The results further demonstrate the complementary roles of FTIR spectroscopy and gas chromatography. FTIR provides information on the principal structural features of the separated fraction, whereas GC offers a more detailed view of the individual volatile components and their relative abundances. In the present study, the combination of these analytical approaches made it possible to establish both the general hydrocarbon character of the fraction and the quantitative contribution of the identified C9 hydrocarbons.

Overall, the 8.70% relative content of identified C9 hydrocarbons indicates that the fraction separated at 159 °C contains a measurable and potentially valuable proportion of C9 compounds. The predominance of indene suggests that further purification or selective fractionation could be considered when the objective is to recover aromatic C9 hydrocarbons for subsequent petrochemical applications.

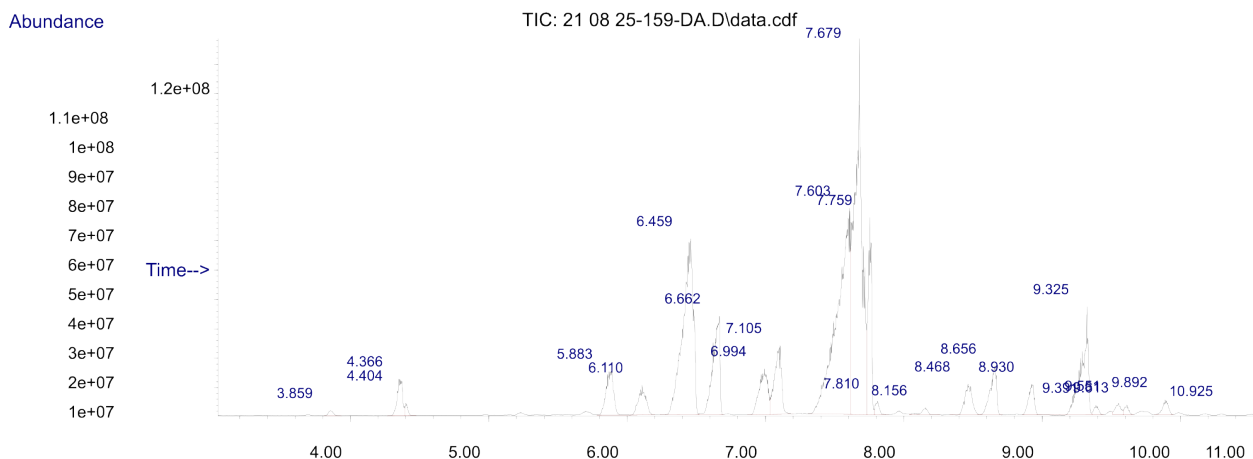


Figure 2. Gas chromatogram of the pyrolysis distillate fraction separated at 159 °C using Sulzer structured packing.

Table 1. Gas chromatographic composition of the pyrolysis distillate fraction separated at 159 °C using Sulzer structured packing.

No.	Retention time, min	Relative content, %	Tentative compound identification
1	3.859	0.21	3-Cyano-4-(2-nitrophenyl)-1,2,4-oxadiazin-5(6H)-one
2	4.366	1.63	Benzonitrile
3	4.404	0.27	Benzeneacetonitrile, α -ethyl-
4	5.883	2.75	4-Methylbenzonitrile
5	6.110	1.72	2-Nitrophenyl isocyanate
6	6.459	15.49	(5-Phenyl-tetrazol-2-yl)-acetic acid (3-nitro-benzylidene)-hydrazide
7	6.662	5.68	Styrene
8	6.994	3.20	Tetracyclo[3.3.0.0(2,4).0(3,6)]oct-7-ene-4-carboxylic acid, methyl ester
9	7.105	3.99	1H-Indene
10	7.603	21.95	1,3-Benzenediacetonitrile
11	7.679	22.81	1,2,5-Thiadiazole-3,4-diamine
12	7.759	5.71	4-(6,7-Dihydro-5H-benzo[1,2,5]oxadiazol-4-ylideneaminooxymethyl)-benzonitrile
13	7.810	0.44	1-Octanol
14	8.156	0.39	1H-Indene, 3-methyl-
15	8.468	1.85	Diisopropyl sulfide
16	8.656	2.57	1H-Indene
17	8.930	1.46	1H-Indene
18	9.325	5.65	1,8-Naphthosultone
19	9.391	0.36	1H-Indole, 1-methyl-
20	9.551	0.51	Benzonitrile, 4-methoxy-
21	9.613	0.35	2,3-Dihydro-1H-inden-1-one
22	9.892	0.68	Benzene, 1-propenyl-
23	10.925	0.33	1H-Indole

Conclusion

The pyrolysis distillate fraction separated at 159 °C using Sulzer structured packing was successfully characterized by FTIR spectroscopy and gas chromatography. The GC results showed a total relative content of 8.70% identified C9 hydrocarbons, with 1H-indene as the predominant component (8.02%). These results confirm that the selected fraction contains a significant proportion of C9 aromatic hydrocarbons and may be suitable for further petrochemical processing and purification.

Acknowledgements

The authors express their sincere gratitude to the Tashkent Institute of Chemical Technology for providing the necessary scientific and technical support, laboratory facilities, and research resources that contributed to the successful completion of this study.

Author Contributions

Nargiza Sh. Imanova contributed to the conception and design of the study, experimental work, data collection, and interpretation of the results. Gulomboym. Bekturdiyev contributed to the experimental investigations, data analysis, and interpretation of the obtained results. Both

authors contributed to the preparation and revision of the manuscript and approved the final version.

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