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#### RESEARCH ARTICLE

## Purification of benzene fractions and benzene from unsaturated and sulfur compounds using a novel modified natural clay-based adsorbent

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### Abstract

**Objectives.** This study aimed to create a method for purifying benzene fractions using a modified natural clay-based adsorbent, which removes both unsaturated and sulfur compounds from benzene or benzene fractions obtained by pyrolysis of petrochemical raw materials.

**Methods.** Chemical and chromatographic methods were used to conduct flow analyses before and after purification. Testing was conducted under conditions that were similar to industrial conditions. The functional characteristics of the adsorbents (moisture resistance and bulk density) were evaluated by strength tests after wetting with water and by weighing batches of the adsorbents fixed in volume.

**Results.** Three clay-based adsorbent samples were developed, and tested under laboratory conditions. Thereafter, the CS-Sorb-6890 (M) sample—an Ni-modified adsorbent—was tested under experimental industrial conditions. A positive effect was obtained, and a method for purifying benzene fractions (benzene) from sulfur compounds and olefins is proposed.

**Conclusions.** The obtained results revealed that the modified active clay-based adsorbent successfully removed sulfur and unsaturated compounds from the benzene fraction simultaneously. The total sulfur concentration was reduced to 0.00004 wt % after 90% of it was removed. The conversion of the unsaturated compounds reached 100%.

**Keywords:** benzene, benzene fraction, adsorption, purification from impurities, modified clay-based adsorbent

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## НАУЧНАЯ СТАТЬЯ

# Очистка бензольной фракции и бензола от непредельных и сернистых соединений новым модифицированным адсорбентом на основе природной глины

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## Аннотация

**Цели.** Создание способа очистки бензольной фракции (бензола) с помощью модифицированного адсорбента на основе природной глины, который позволит одновременно удалять непредельные и сернистые соединения из бензола или бензольной фракции, полученных пиролизом нефтехимического сырья.

**Методы.** Анализы потоков до и после очистки выполняли химическими и хроматографическими методами. Тестирование проводили в условиях, приближенных к промышленным. Функциональные характеристики адсорбентов (влагостойкость, насыпная плотность) оценивали с помощью испытаний на прочность после смачивания водой и методом взвешивания фиксированных по объему партий адсорбентов.

**Результаты.** Разработаны три образца адсорбента на основе глины, проведено их тестирование в лабораторных условиях, после чего образец CS-Sorb-6890 (М) – адсорбент с нанесенным Ni – прошел испытания в опытно-промышленных условиях. Получен положительный эффект, предложен способ очистки бензольной фракции (бензола) от сернистых соединений и олефинов.

**Выводы.** Достигнутые результаты свидетельствуют о том, что созданный модифицированный адсорбент на основе активной глины успешно удаляет одновременно сернистые и непредельные соединения из бензольной фракции. Удаление общей серы максимально составило 90%, и ее концентрация минимизирована до 0.00004 мас. %. Конверсия непредельных соединений достигла 100%.

**Ключевые слова:** бензол, бензольная фракция, адсорбция, очистка от примесей, модифицированный адсорбент на основе глины

**Для цитирования:** Малышкин Б.Ю., Семёнов И.П., Сазонов Д.С., Путенихин И.О., Семенихина Е.Ю. Очистка бензольной фракции и бензола от непредельных и сернистых соединений новым модифицированным адсорбентом на основе природной глины. *Тонкие химические технологии.* 2022;17(1):18–29. <https://doi.org/10.32362/2410-6593-2022-17-1-18-29>

## INTRODUCTION

Benzene is one of the most sought-after substances in the chemical industry, and it is widely used in organic synthesis. Benzene can be obtained via several methods, including pyrolysis of gasoline fractions and separation of pyrocondensate, processing of catalytic reforming products, and coking of coal.

Benzene is mainly produced from petrochemical raw materials, and the volume produced is about 1.1–1.2 mln t/year. *ExxonMobil Chemical* (USA), *Dow Chemical* (USA), and *Shell Chemicals* (USA) are the leading chemical companies in the world. *Angarsk Polymer Plant*, *Gazprom Neftekhim Salavat*, and *Nizhnekamskneftekhim* are the three largest benzene producers in Russia [1].

The benzene–toluene–xylene (BTX) fractions of liquid pyrolysis products are mainly hydrogenated to obtain benzene as the main commercial product [2]. The scheme for petrochemical benzene production (Fig. 1) includes BTX fraction separation, sequential two-stage hydrotreatment for the removal of unsaturated and sulfur compounds, catalytic or thermal hydrodealkylation, product separation in a system of distillation columns, and contact purification of the benzene fraction from unsaturated compounds and resins [3].

The adsorber is the most interesting in terms of the research task. Clay is loaded into this apparatus to separate the benzene fraction from the residual unsaturated and sulfur compounds.

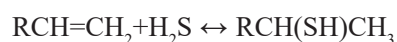
According to GOST 58415-2019, the total sulfur concentration in benzene should not exceed 0.00005 wt %. Simultaneously, the concentration of unsaturated compounds in petrochemical benzene is not directly regulated; only the “sulfuric acid color” indicator and, upon consumer request, the “bromine number” indicator are normalized, both of which indicate the unsaturated compound concentrations.<sup>1</sup>

Petrochemical raw materials contain mercaptans, sulfides, thiophenes, and other sulfur-containing compounds. In most cases, up to 10% of the total amount of initial sulfur remains in the benzene fraction. The most difficult substances to remove are thiophene derivatives. They are concentrated in the benzene fraction because their boiling point coincides with that of gasoline.<sup>2</sup>

<sup>1</sup> GOST 58415-2019. National Standard of the Russian Federation. Petrochemical Benzene. Specifications. Moscow: Standartinform; 2019.

<sup>2</sup> Glotov A.P. *Obesserivayushchie dobavki k katalizatoram krekinga nefryanogo syr'ya (Desulfurizing additives to catalysts for the cracking of petroleum feedstock)*: Cand. Sci. Thesis. Moscow: M.V. Lomonosov Moscow State University; 2016. 23 p. (in Russ).

Although the benzene production scheme includes a hydrotreating stage for sulfur removal, sulfur compounds can still be found in benzene and benzene fractions, which compromises the commercial product quality. This is due to the fact that hydrogen sulfide present in the benzene fraction after hydrotreatment interacts with olefins and paraffins to form mercaptans under hydrodealkylation reactor conditions:

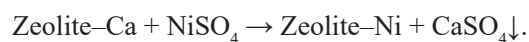


This nucleophilic addition reaction is reversible, but the equilibrium is predominantly shifted to the right because hydrogen sulfide has a high reactivity [4]. A fine posttreatment of benzene is required because of the newly formed mercaptans, which cause deviations in the total sulfur content, jeopardizing the stable sale of marketable products by the enterprise.

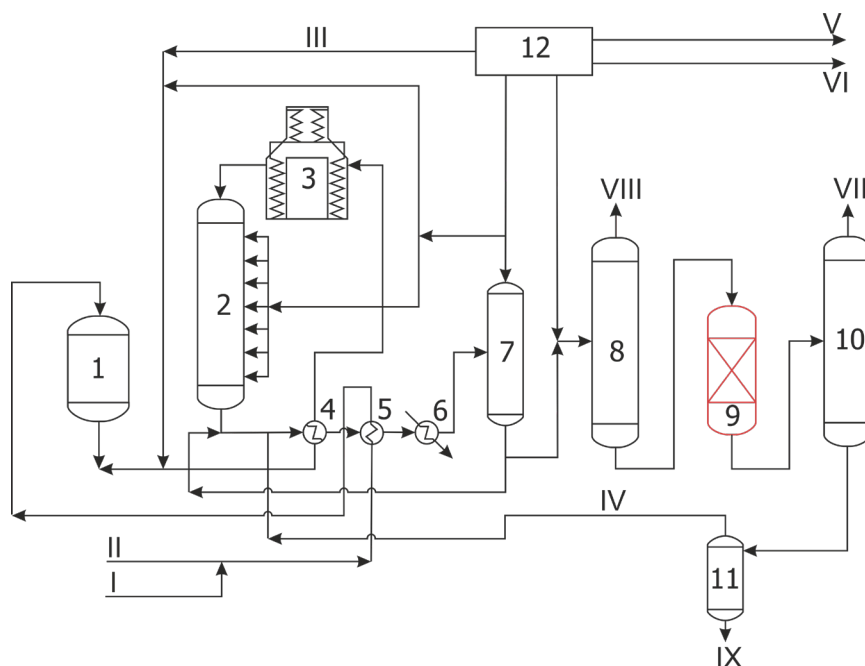
Absorption (extraction) and adsorption methods are used to capture poisons and impurities. The absorption or extraction method is used when the concentration of the absorbed substance in the initial mixture is significant, such as in the case of purifying benzene from thiophene [5]. Since there were minor unsaturated and sulfur compound impurities in the benzene fraction in our study, adsorption—the process of absorbing a substance from a gas or liquid using a solid adsorbent surface layer—was the most appropriate method.

The standard adsorbent for purifying the benzene fraction from unsaturated compounds is active clay, which undergoes alkylation and oligomerization reactions, resulting in unsaturated compounds being converted to alkyl-substituted aromatic compounds or heavy resinous compounds, which are then adsorbed by clay or separated in a benzene separation column.

Attempts have been made to modify clay-based adsorbents in order to purify the benzene fraction. For example, carbon disulfide can be removed from CaA zeolites, while thiophene can be removed from Ni- and Cu-substituted CaX and CaA zeolites according to the following reaction:



When the adsorbent is treated with nickel salt, the effectiveness of thiophene purification increases from 4% to 21.3%, while sulfuric acid modification does not have a positive effect [6]. However, there is no evidence that these adsorbents extract both unsaturated compounds and sulfur compounds simultaneously.



**Fig. 1.** Schematic of the petrochemical benzene production plant.

(1) Hydrogenation reactor, (2) hydrodealkylation reactor, (3) tubular furnace, (4 and 5) heat exchangers, (6) refrigerator, (7) gas separator, (8) stabilizer, (9) adsorber, (10) benzene recovery column, (11) toluene and biphenyl recovery column, and (12) hydrogen concentration unit.

(I) Raw materials, (II) fresh hydrogen, (III) purified hydrogen, (IV) toluene and diphenyl, (V) methane-enriched gas, (VI)  $C_{2+}$  hydrocarbons, (VII) fuel gas, (VIII) benzene, and (IX) resins and heavy hydrocarbons.

There are several reported clay-based adsorbents [7–9], which were modified in an aluminum sulfate solution. Although the adsorbent samples had a high adsorption capacity for unsaturated compounds, they did not remove sulfur compounds from the purified flow of benzene and benzene fractions.

This study aimed to develop a clay-based adsorbent and an application method that will allow it to remove both unsaturated and sulfur compounds from the benzene fraction using one apparatus.

## EXPERIMENTAL

### Adsorbent preparation

The standard adsorbent for the post-purification of benzene and the benzene fraction was made by drying natural clay, calcining it, isolating the target fraction, and activating it with acid solutions.

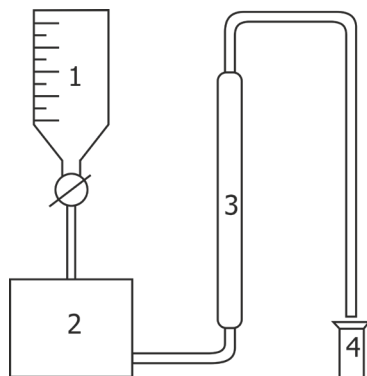
Active natural clay was impregnated to prepare the modified adsorbent for sulfur compound removal. Active clays are clays that have been subjected to the complete production cycle, which includes drying, granulation, activation, and calcination. A batch of active natural clay-based adsorbents with a grain size of 0.2–2.5 mm was loaded into an impregnating container. A solution was prepared using soluble inorganic (e.g., nitrates) or organic (e.g., oxalates or

lactates) salts of copper, zinc, and nickel to impregnate the adsorbent. The solutions were prepared using distilled water. The concentration of the solutions was selected based on the desired active metal content in the developed adsorbent. The concentrations of the impregnating aqueous solutions of organic and/or inorganic salts of copper, zinc, and nickel ranged from 5 to 400 g per 100 mL, and the ratio of the impregnating solution to the clay mass ranged from 1:2 to 1:10. The salts were selected such that when heated, the acid residue decomposes as the metal or metal oxide is deposited on the adsorbent surface.

The adsorbent batch was impregnated in an impregnating container for 1–2 h at temperatures of 20–80°C. Afterward, the impregnating solution was drained, and the adsorbent was then discharged from the drum. Subsequently, the adsorbent was dried and calcined in an oven at temperatures ranging from 250 to 550°C for 0.5–4.0 h. During calcination at this temperature, the excess of the impregnating solution was removed, while the active metal or its oxide remained on the adsorbent surface. The target adsorbent fraction with a grain size of 0.2–2.5 mm was released after sieving the calcined adsorbent through sieves. This granule size ensured that the adsorbent had an optimal specific surface area and a low pressure drop, increasing the effectiveness of using the adsorbent when it was loaded into the adsorber.

### Prepared adsorbent sample test

Laboratory tests of the active clay samples were conducted in a flow unit (Fig. 2).

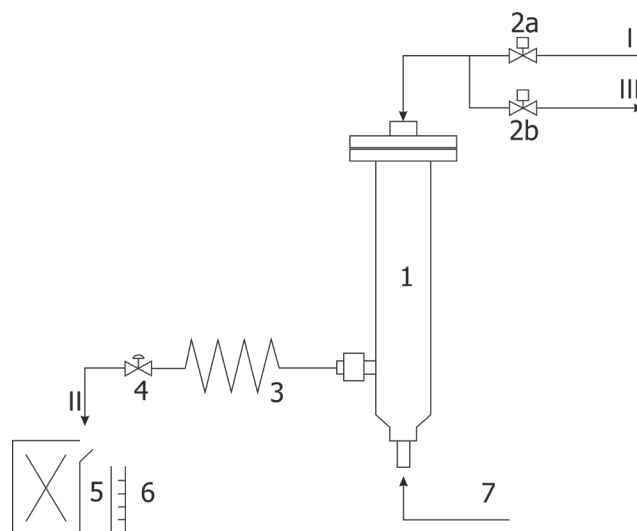


**Fig. 2.** Diagram of the laboratory setup for testing the adsorbent samples: (1) The model solution container, (2) a pump, (3) an adsorption column, and (4) a receiver.

The setup consists of a receptacle under the model solution (1), a pump (2), a glass adsorption column (3) with an inner diameter of 14 mm and a SCHOTT filter at the bottom of the inert material to fill the column in the form of a glass nozzle with a fraction size of 2–3 mm, and bottles receivers (4) with a sampling volume of 10 mL. The tests were conducted at room temperature ( $23 \pm 2^\circ\text{C}$ ). The loaded sample had a volume of 5 mL and a particle size of 0.5–1.0 mm. The sample was precalcined at a temperature of  $195^\circ\text{C}$  for 6 h and heated for 30 min to eliminate moisture. The flow rate at the outlet was  $0.55 \pm 0.05$  mL/min. Sampling was carried out for every 8 mL of the solution that passed through the adsorbent. The analyses were performed using titrimetric and gas chromatographic methods. The initial mixture was a mixture of benzene with hexene and propanethiol. Hexene and propanethiol were chosen as representative components for unsaturated and sulfur compound impurities, respectively.

Figure 3 shows the scheme of the installation that was used to test the adsorbent under experimental industrial conditions.

The prepared clay-based adsorbent (550 mL) was loaded into a direct-flow adsorber (1), which was then fed with a hot flow of benzene fraction at a temperature of  $170\text{--}230^\circ\text{C}$  and pressure of  $7.5\text{--}28.0$  kgf/cm<sup>2</sup>. A shut-off valve (2a) was installed on the flow supply line to the adsorber, allowing the supply of the benzene fraction to the reactor to be shut off. The shut-off valve (2b) was used for sampling the initial benzene fraction. Residual resins, unsaturated compounds, and sulfur compounds were removed from the flow of the benzene fraction as it passed through the clay layer. The purified flow from the adsorber (1) was cooled in an air heat exchanger (3). The flow rate was regulated by a control valve (4),



**Fig. 3.** Pilot installation of adsorbent testing. (1) Adsorber, (2a) shut-off valve in adsorber flow line, (2b) shut-off valve in sampling line, (3) air cooler, (4) valve, (5) drain tank, (6) measuring tank, and (7) inner pocket for thermocouple. (I) The flow from the BTX fraction extraction unit, (II) the purified flow of the benzene fraction, and (III) the flow for sampling the initial BTX fraction.

which was installed on the outlet line after the heat exchanger. The purified benzene fraction was drained into a canister tank (5). The drain tank (5) was replaced with a measuring tank (6) during sampling or flow measurement.

## RESULTS AND DISCUSSION

### Laboratory test results

Three samples were created for laboratory studies:

1. CS-Sorb-6891 (S), which is an adsorbent based on active clay with deposited Cu and Zn metals;
2. CS-CL200X, which is an adsorbent based on active clay without metals;
3. CS-Sorb-6890 (M), which is an adsorbent based on active clay with deposited Ni metal.

Table 1 shows the characteristics of the tested adsorbent samples.

Table 2 presents the initial composition of the flow.

Table 3 shows the results obtained under laboratory conditions when the benzene flow was purified with the adsorbents.

As shown in Table 3, the samples demonstrated different abilities in removing sulfur and unsaturated compounds from the model mixture. The CS-Sorb-6891 (S) sample exhibited the highest activity in removing sulfur compounds but did not

**Table 1.** Characteristics of the adsorbent samples

Indicators	CS-Sorb-6891 (S)	CS-CL200X	CS-Sorb-6890 (M)
Base metals	Cu, Zn	–	Ni
Metal content, %	10	–	10
Free moisture content (calcination at 105°C), wt %	1.0	2.5	4.9
Mass fraction of water (calcination at 195°C), wt %	6.4	5.4	5.4
Bulk density, g/cm <sup>3</sup>	0.76	0.81	0.81
<b>Fractional composition, %</b>			
>2.0 mm	8.5	18.4	1.9
2.0–0.25 mm	90.6	81.0	90.2
<0.25 mm	0.9	0.6	7.9

**Table 2.** Composition of the initial flow of benzene (benzene fraction)

Component	Concentration
Benzene	98.99 wt %
Hexene	1 wt %
2-Propanethiol	21–28 ppm

**Table 3.** Performance indicators of the adsorbent samples

Sample	Mass of passed raw materials, g	Concentration of sulfur compounds, ppm	Concentration of unsaturated compounds, wt %	Number of removed sulfur compounds, ppm	Number of removed unsaturated connections, wt %
CS-Sorb-6891 (S)	0.00	27.30	0.23	–	–
	98.5	0.65	0.22	26.65	0.02
	150.8	0.53	0.22	26.77	0.02
	216.3	0.57	0.23	26.73	0.00
CS-CL200X	0.00	22.99	0.20	–	–
	85.0	10.95	0.08	12.04	0.12
	98.0	12.26	0.10	10.73	0.10
	131.4	16.72	0.14	6.27	0.06
CS-Sorb-6890 (M)	0.00	28.04	0.34	–	–
	98.4	1.24	0.22	26.80	0.12
	150.1	2.56	0.24	25.49	0.10
	196.4	3.93	0.21	24.11	0.10



affect the concentration of unsaturated compounds. As expected, the CS-CL200X sample (clay without metals) was the least effective at removing sulfur compounds. The CS-Sorb-6890 (M) sample exhibited the best results; it effectively removed both the unsaturated and sulfur compounds. However, it had a slightly lower unsaturated compound removal activity than the CS-CL200X sample and a slightly lower degree of purification from sulfur compounds than the CS-Sorb-6891(S) sample at temperature 170–230°C.

### Pilot test results

Since the CS-Sorb-6890 (M) adsorbent showed good cleaning results during the laboratory studies, the decision was made to use it for pilot tests at one of the industrial enterprises. The sulfur compound concentration in the incoming benzene fraction ranged from 0.1 to 3.2 ppm. The adsorbent sample was compared with industrial R-01 and R-02 adsorbents under less favorable conditions to confirm its effectiveness. Table 4 shows the operation parameters

of the pilot plant and industrial adsorbents.

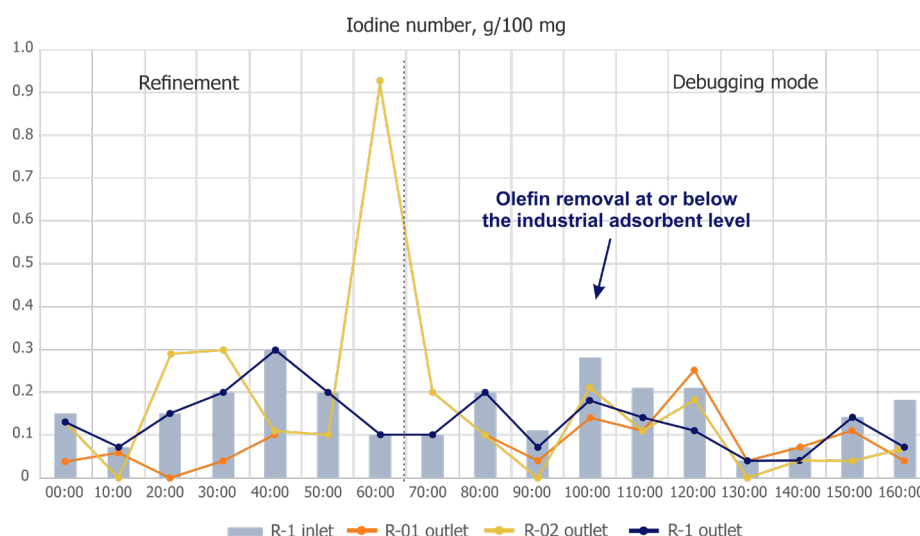
Pilot tests were conducted for 7 days (3 days for the debugging mode and 4 days for the stabilized mileage and averaged data marked in gray) are presented in Table 5.

Figures 4–6 compare the activities of the pilot and industrial adsorbents toward the removal of unsaturated and sulfur compounds.

The test results showed that the CS-Sorb-6890 (M) adsorbent removed the sulfur compounds and unsaturated hydrocarbons stably throughout the entire test period. The total sulfur concentration was reduced to an average concentration of 0.41 ppm. The industrial R-01 and R-02 adsorbents had an average sulfur compound concentration of 1.86 and 0.61 ppm at the output, respectively. The unsaturated compound concentration at the outlet of the adsorbent was determined by the iodine number indicator. The iodine number indicator ranged from 40 to 180 mg/100 g (with an average value of 100 mg/100 g) at the output. When compared to the industrial R-01 and

**Table 4.** Parameters during active clay testing

No.	Parameter	In the industrial adsorbent R-01/R-02	In the pilot adsorbent R-1
1	Consumption of benzene fraction per adsorbent	7500 kg/h	1200–1500 mL/h (20–25 mL/min)
2	Adsorbent volume in one adsorbent	22.7 m <sup>3</sup>	550 cm <sup>3</sup>
3	Volumetric flow rate	0.4 h <sup>-1</sup>	2.2–2.7 h <sup>-1</sup>
4	Temperature in the adsorbent	≥170°C	35–45°C
5	Pressure in the adsorbent	7.5–28 kgf/cm <sup>2</sup>	7.5–8.5 kgf/cm <sup>2</sup>



**Fig. 4.** Iodine number in the flow at the inlet and outlet of the adsorbents.

Table 5. Pilot test results of the adsorbers

Selection point	Initial benzene fraction (entrance to the adsorber R-1)		Output of the pilot adsorber R-1		Output of industrial adsorber R-01		Output of industrial adsorber R-02	
	Iodine number, g/100 g	Sulfur, ppmw	Iodine number, g/100 g	Sulfur, ppmw	Iodine number, g/100 g	Sulfur, ppmw	Iodine number, g/100 g	Sulfur, ppmw
00:00	0.15	2.60	0.13	1.70	0.04	0.50	0.13	0.20
10:00	0.07	3.50	0.07	2.20	0.06	6.30	0.00	0.40
20:00	0.15	2.60	0.15	0.90	0.00	1.20	0.29	0.30
30:00	0.20	2.00	0.20	1.00	0.04	0.60	0.30	2.70
40:00	0.30	1.60	0.30	4.80	0.10	5.00	0.11	1.50
50:00	0.20	3.40	0.20	2.70	0.10	8.10	0.10	0.90
60:00	0.10	3.30	0.10	2.00	0.92	1.30	0.92	0.40
70:00	0.10	2.90	0.10	0.30	0.20	0.80	0.20	0.60
80:00	0.20	2.70	0.20	1.70	0.10	1.90	0.10	0.70
90:00	0.11	3.20	0.07	1.00	0.04	2.10	0.00	1.00
100:00	0.28	0.10	0.18	0.30	0.14	1.00	0.21	1.00
110:00	0.21	0.30	0.14	0.20	0.11	1.00	0.11	0.20
120:00	0.21	2.00	0.11	0.40	0.25	0.20	0.18	0.50
130:00	0.04	2.00	0.04	0.30	0.04	0.30	0.00	0.80
140:00	0.07	2.00	0.04	0.50	0.07	3.00	0.04	0.10
150:00	0.14	1.00	0.14	0.30	0.11	3.00	0.04	0.30
160:00	0.18	2.60	0.07	0.30	0.04	4.30	0.07	1.00
Minimum	0.04	0.10	0.04	0.20	0.04	0.20	0.00	0.10
Maximum	0.28	3.20	0.18	1.00	0.25	4.30	0.21	1.00
Average	0.16	1.65	0.10	0.41	0.10	1.86	0.08	0.61



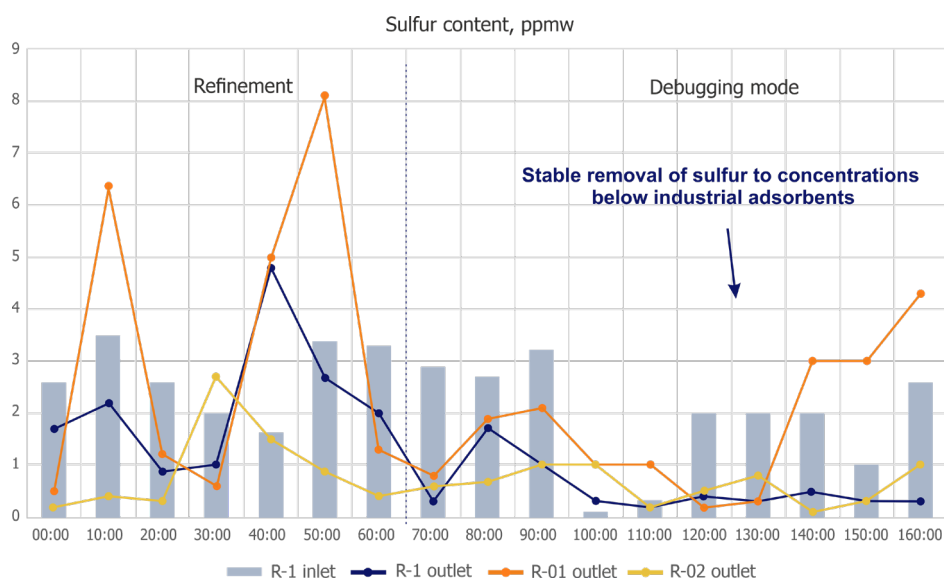


Fig. 5. Sulfur content in the flow at the inlet and outlet of the adsorbers.

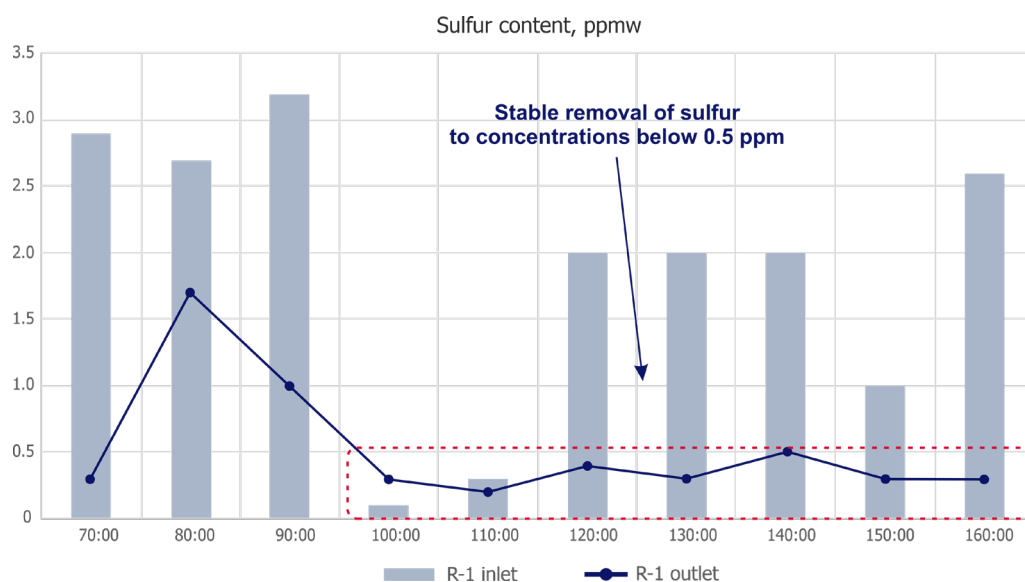


Fig. 6. Removal of sulfur by the CS-Sorb-6890 (M) sample after regime stabilization.

Table 6. Achieved adsorbent indicators during pilot tests

Indicator	Results for the CS-Sorb-6890 (M) sample in the R-1 pilot adsorber		Results for a standard adsorbent in an industrial adsorber			
			R-01		R-02	
	Average	Range	Average	Range	Average	Range
Content of unsaturated compounds (iodine number), mg/100 mL	100	40–180	100	40–250	80	40–210
Total sulfur content, ppmw	0.41	0.2–1.0	1.86	0.2–4.3	0.61	0.1–1.0

R-02 adsorbents (with a volume velocity that is 5.5–6 times higher and a temperature of ~130°C below than those of the CS-Sorb-6890 (M) adsorbent), the CS-Sorb-6890 (M) adsorbent showed similar efficiency in the removal of unsaturated compounds and higher efficiency in the removal of sulfur compounds under less favorable conditions.

## CONCLUSIONS

A modified active clay-based adsorbent, CS-Sorb-6890 (M), was created and tested under laboratory and pilot-industrial conditions. According to the laboratory test results, the studied samples had different sorption capacities for sulfur and unsaturated compounds. The CS-Sorb-6890 (M) adsorbent successfully removed olefins and sulfur compounds from the benzene fraction simultaneously under pilot testing conditions. The benzene flow had a total sulfur content of 0.41 ppm after purification, and the iodine number had a minimum value of 40 mg/100 g.

The results indicate that it is possible to purify the benzene fraction from sulfur and unsaturated compounds in only one apparatus, allowing commercial benzene of specific quality to be obtained without changing the technological production scheme and with only existing equipment.

## Authors' contribution

**B.Yu. Malyshkin** – development of the concept of scientific work, the chemical composition of the adsorbent, advice on methodology and research;

**I.P. Semenov** – development of the concept of scientific work, consultation on methodology and research, editing the text of the article;

**D.S. Sazonov** – advice on methodology and research;

**I.O. Putenikhin** – development of the concept of scientific work, consultation on methodology and research;

**E.Yu. Semenikhina** – collection and processing of material, writing the text of the article.

*The authors declare no conflicts of interest.*

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