

## ADSORPTIVE DESULFURIZATION OF MODEL FUELS WITH LOW-COST ADSORBENT IN DYNAMIC CONDITIONS

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### ABSTRACT

*The adsorption capacity of pyrolyzed rice husks was investigated with respect to individual model solutions of benzothiophene (BT) and dibenzothiophene (DBT), and a model mixture containing benzothiophene, dibenzothiophene and 4, 6 - dimethyldibenzothiophene (4, 6 - DMDBT). The adsorption process was carried out under dynamic conditions. The influence of temperature and initial concentration of sulfur compounds in individual model fuels was investigated. The highest adsorption capacity was achieved at 60°C and the highest initial sulfur concentration in the model fuels. Carrying out the adsorption at higher temperatures leads to an increase in the degree of desulfurization. The adsorption selectivity of the pyrolyzed rice husk from mixture model fuel decreases in the order DBT > 4, 6 - DMDBT > BT.*

*Keywords: desulfurization; dynamic adsorption; pyrolyzed rice husks, model fuels.*

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### INTRODUCTION

Despite the emergence of new alternative energy sources, fossil fuels continue to be the primary energy source. It is predicted that by 2040, they will still be the dominant energy source [1]. The consumption of fossil fuels leads to serious air pollution, the formation of greenhouse gases, acid rain, etc., due to the presence of sulfur compounds [2, 3]. The total sulfur content is one of the most important indicators affecting the quality of petroleum products [4]. Therefore, strict regulations have been proposed to reduce the sulfur content to 10 and 15 ppm based on the rules of the European Union (EU) and the United States Environmental Protection Agency (USEPA) [5 - 7]. The implementation of these directives is a serious challenge and requires the improvement of the purification technologies applied for this purpose. In addition to being efficient, these methods must also ensure a balanced price for the final product.

Petroleum and its products contain a variety of sulfur compounds, classified mainly into four groups: mercaptans, sulfides, disulfides, and thiophene-based structures [2, 8]. These sulfur compounds pose

significant challenges during refining due to their tendency to deactivate catalysts and accelerate corrosion of process infrastructure such as metal surfaces, pipelines and pumps.

Their removal requires advanced treatment methods. Among them, hydrodesulfurization is the predominant technique used in refineries [9 - 12]. It involves catalytic reactions conducted at elevated temperatures and high hydrogen pressures that effectively reduce the sulfur content of fuel streams and improve product quality [13].

Several alternative and complementary methods to traditional hydrotreating have been developed to reduce sulfur compounds in petroleum fuels. These include biodesulfurization using microorganisms [14 - 16], extractive [17 - 19] and oxidative [20 - 23] desulfurization, selective adsorption [24 - 26], and others. Among these alternative methods, selective adsorption is considered the most economical approach to solving the problem. The advantages of this method include high efficiency, the possibility of conducting the process without the participation of hydrogen under relatively mild conditions, simplicity of technology and equipment, low capital and operating costs, as well as

safety in carrying out the process.

Various carbon-containing materials obtained after processing agricultural and food industry waste can be used as adsorbents [27]. In this way, two problems related to environmental protection are solved: products with practical application and added value are produced, and the costs of waste disposal and management are reduced. In the present study, rice husks, which are a significant agricultural waste, were used as the starting material for the preparation of the adsorbent. Their pyrolysis produces a composite C/Si<sub>2</sub>O material with a developed mesoporous structure and surface characteristics that make it suitable for the desulfurization of liquid fuels [28, 29].

The subject of this work is to study the influence of conducting the process under dynamic conditions, since real industrial adsorption processes are carried out precisely in a dynamic mode. The adsorption capacity of pyrolyzed rice husks was studied with respect to individual model solutions of benzothiophene and dibenzothiophene, and a model mixture containing benzothiophene, dibenzothiophene and 4, 6, -dimethyldibenzothiophene, in order to consider, the competitive influence of sulfur compounds in the fuel. The influence of temperature on the degree of desulfurization was also considered.

## EXPERIMENTAL

### Materials and methods

#### *Preparation and characterization of the adsorbent*

The starting material for the preparation of the adsorbent used in the present study was rice husks obtained from the Pazardzhik region, Bulgaria. The raw husks were pyrolyzed at 480°C, the pyrolysis conditions, as well as the structural characteristics of the obtained pyrolysis residues, have been described in previous articles [30 - 32]. Summary data are presented in Table 1.

Pyrolysis rice husks are characterized by high porosity and total pore volume. They have a diverse porous structure, which is one of the conditions for good adsorption capabilities of the material. It is known that in addition to good surface characteristics, for a good adsorbent, its surface functional groups are also important for its effectiveness. In the process of pyrolysis of rice husks, volatile compounds are released because of the destruction of the lignocellulose matrix. Some of these compounds are deposited on the surface of the

Table 1. Characteristics of pyrolyzed rice husks.

Characteristics	Value
Total pore volume, cm <sup>3</sup> g <sup>-1</sup>	1.05
Micropore volume, cm <sup>3</sup> g <sup>-1</sup>	0.005
Porosity, %	57.1
Total pore surface, m <sup>2</sup> g <sup>-1</sup>	5.9
Average pore diameter, mm	0.72
Average pore diameter, nm	4.1
Bulk density, g cm <sup>-3</sup>	0.54
B.E.T. specific surface area, m <sup>2</sup> g <sup>-1</sup>	253
Mass yield, wt. %	38
Carbonaceous material/ash ratio	0.96

solid pyrolysis residue and determine the chemistry of its surface. In previous studies, the composition of acetone-extracted fluids from pyrolysis rice husks has been quantitatively and qualitatively determined [30]. Based on GH-MS analysis, it has been proven that these fluids are mainly hydrocarbons containing carboxyl groups, included in a straight chain or in condensed nuclei, carbonyl groups and phenol type hydroxyl groups.

### Adsorptive desulfurization

#### *Preparation of model fuels*

To consider the influence of the sulfur concentration in the starting model fuels on the degree of desulfurization and the adsorption capacity of the material, individual model solutions of n-octane with a controlled content of benzothiophene and dibenzothiophene were prepared (Table 2). When preparing the solutions, it was noted that the sulfur content in benzothiophene is 23.84 % and in dibenzothiophene 17.37 %. The starting solutions were prepared by measuring the required amount of the respective sulfur-containing compound in a glass flask and adding up to 100 g with n-octane. The percentage of sulfur in the solutions was determined by Sindie OTG Sulfur Analyzer using monochromatic wavelength dispersive X-ray fluorescence (MWDXRF), XOS.

To determine the affinity of pyrolyzed rice husks for various sulfur compounds, a model mixture of benzothiophene, dibenzothiophene and 4, 6-dimethyldibenzothiophene in n-octane was prepared, with a total sulfur content of 600 ppm (Table 3). The sulfur compounds in the model mixture were analysed on an Agilent Gas Chromatograph (7890 A) equipped with a sulfur chemiluminescence detector. Initially,

Table 2. Initial sulfur content into the individual model fuels.

Compound	Amount (S ppm)		
BT	1170	2070	4550
DBT	1268	2404	3536

Table 3. Composition of the mixture model fuel.

Model fuel, 600 ppm		
S from BT	S from DBT	S from 4, 6 - DMDBT
25.1 %	41.7 %	33.2 %
150.3 ppm	250.4 ppm	199.3 ppm

the temperature in the column was maintained at 50°C for 3 min, then increased by 6°C min<sup>-1</sup> until reaching 300°C. This temperature was maintained for 5 min. The sulfur compounds in the model mixture were identified qualitatively and quantitatively.

### Dynamic adsorption

The adsorption experiments were conducted under dynamic conditions and temperature variations in the range between 20 - 60°C. To the study, 3 g of adsorbent with particle size below 0.4 mm, previously dried to a constant weight, was poured into a glass tube with a diameter of 11 mm. The model fuel passed through the adsorbent layer at a rate of 0.5 mL min<sup>-1</sup> until a volume of 20 mL was collected in the receiver. The adsorption capacity of the adsorbent was calculated by the Eq. (1).

$$a = \frac{(C_0 - C_i) \cdot V \cdot \rho \cdot 10^{-3}}{m}, \text{ mg S g}^{-1} \quad (1)$$

where:  $C_0$  - initial sulfur concentration in the fuel sample, wppmS;  $C_i$  - final sulfur concentration after treatment, wppmS;  $\rho$  - fuel density, g cm<sup>-3</sup>;  $V$  - volume of the fuel exposed to the sorbent, mL,  $m$  - mass of sorbent used, g.

To estimate the degree of desulfurization, Eq. (2) was used.

$$D = \frac{C_0 - C_i}{C_0} \cdot 100, \% \quad (2)$$

## RESULTS AND DISCUSSION

### Desulfurization of individual model solutions

Real industrial adsorption processes are conducted

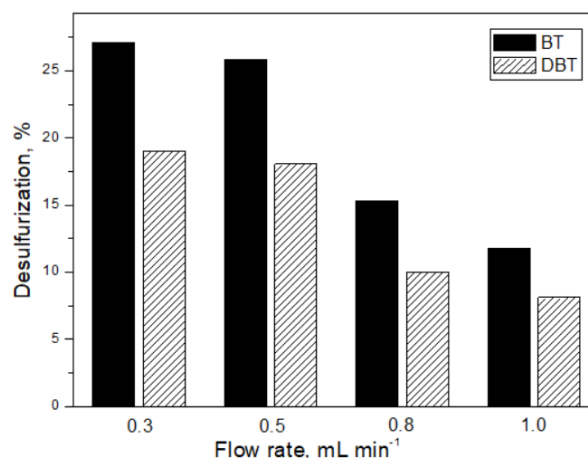


Fig. 1. Dependence of the degree of desulfurization on the flow rate.

in a dynamic mode, where the fluid comes into contact with the adsorbent for a limited period. The contact time of the adsorbate and the adsorbent is determined by the flow rate into the reactor. The degree of purification of the adsorbate is higher, the longer its contact with the adsorbent, i.e., the lower the flow rate of the purified feedstock [33]. In laboratory practice, conducting experiments in a dynamic mode aims to establish the optimal percolation rate, which enables achieving the highest degree of purification while maintaining high process productivity.

To determine the optimal flow rate for adsorption under dynamic conditions, the change in the degree of desulfurization at different initial fluid rates was investigated. The studies were conducted at 20°C with the individual model fuels with the highest BT and DBT concentrations (Fig. 1). The results obtained are expected because, at a lower flow rate, the adsorbate has a longer contact time with the adsorbent, leading to better pollutant removal. The experimentally determined optimal flow rate (0.5 mL min<sup>-1</sup>) was used for all subsequent adsorption studies in this work.

The results obtained for the adsorption capacity of pyrolyzed rice husks under dynamic conditions, with respect to benzothiophene and dibenzothiophene from individual model solutions, at different temperatures, are presented in Fig. 2.

The results show that the adsorption capacity depends on the initial sulfur concentration in the fuel and the temperature of the process. The highest adsorption capacity was achieved at 60°C and the highest initial

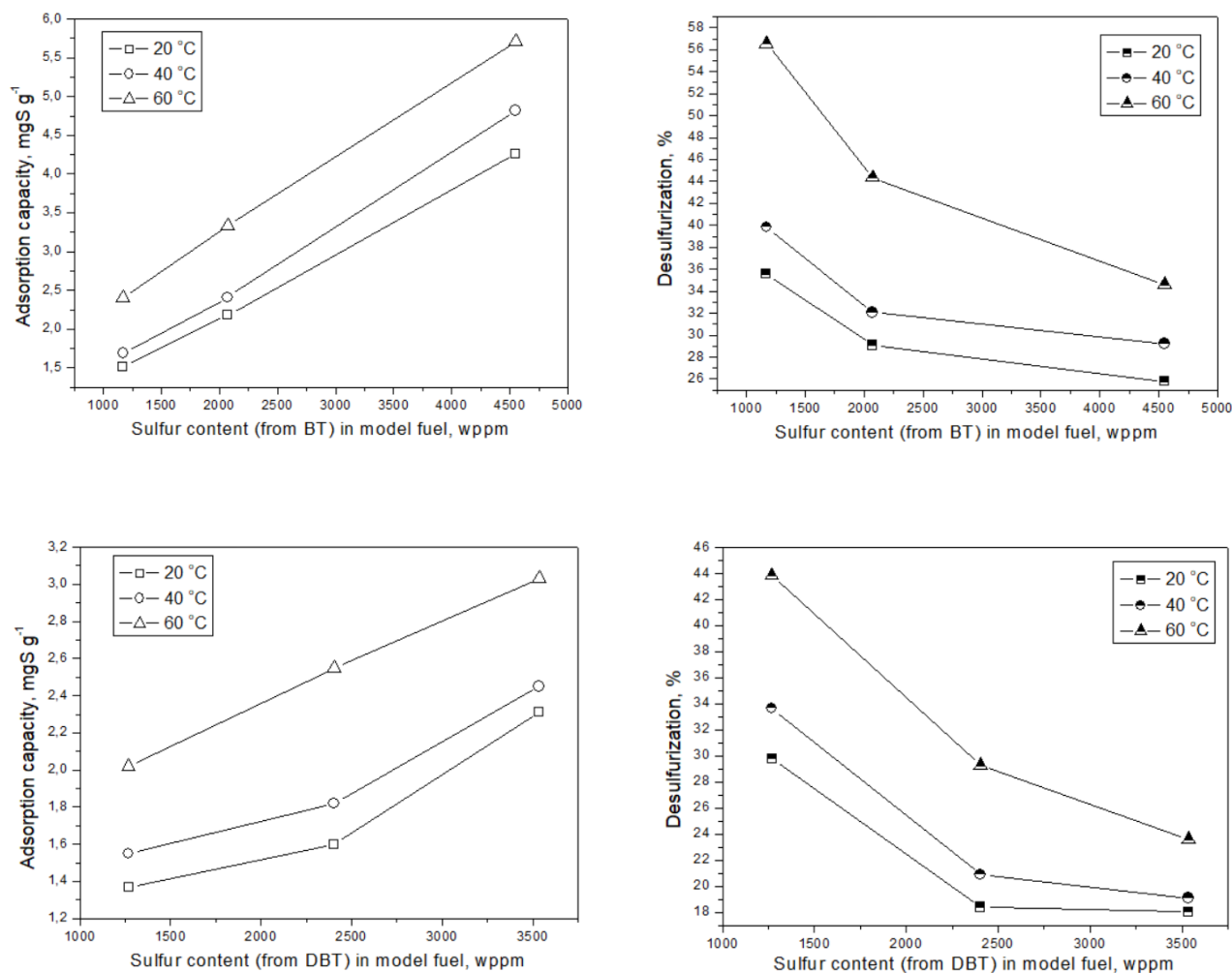


Fig. 2. Adsorption capacity of pyrolyzed rice husks concerning individual model fuels (BT and DBT) at different temperatures.

sulfur concentration in the model fuel for both sulfur compounds used. The lowest adsorption capacity of the individual model fuels was recorded at the lowest sulfur concentrations in them and at the lowest temperature of the adsorption process (20 °C).

Regardless of the adsorption temperature, the degree of desulfurization of the model fuels decreases with increasing sulfur concentration in the starting solutions. These results can be explained by the fact that high initial sulfur concentrations in the starting model fuels lead to faster saturation and blocking of the active adsorption centers of the pyrolyzed rice husks. Carrying out the adsorption at higher temperatures (40 °C and 60 °C) leads to an increase in the degree of desulfurization. In a previous study related to the adsorption of thiophene compounds under static conditions, using pyrolyzed rice

husks, it was found that the adsorption process is due to valence forces [34]. However, the results obtained in the present study prove that the adsorption process is not only physical in nature, but some chemical interactions in the liquid/solid system probably also occur.

The adsorption studies conducted with individual model fuels show that relatively high adsorption capacity of pyrolyzed rice husks is observed at low initial sulfur concentrations in the fuel. This means that the adsorbent can be successfully used for deep purification of hydrocarbon oil fractions containing weakly reactive thiophene compounds.

#### Desulfurization of model fuel containing benzo-thiophene, dibenzothiophene and 4, 6-dimethyldibenzothiophene

Due to the conclusion made above that pyrolyzed

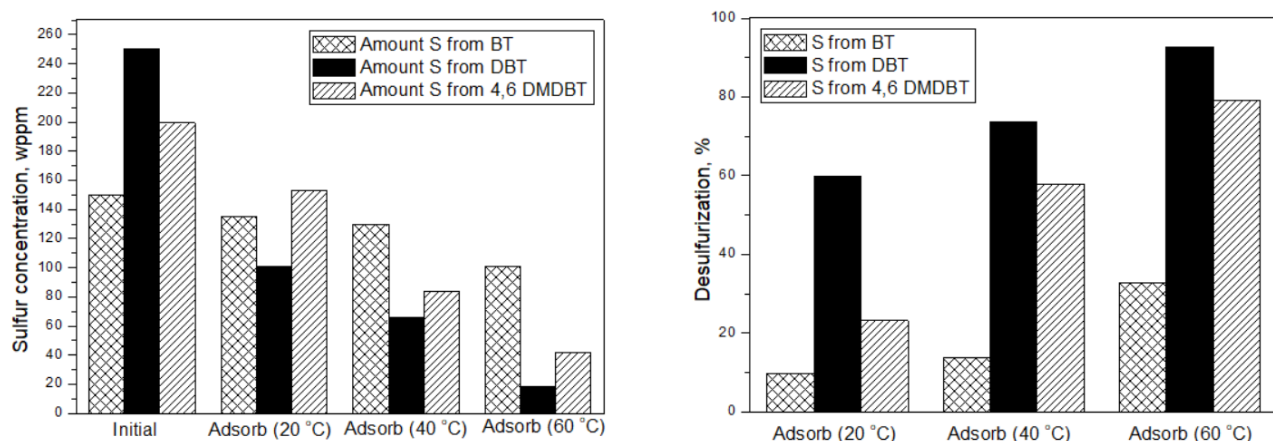


Fig. 3. Adsorption capacity of pyrolyzed rice husks concerning model mixture at different temperatures.

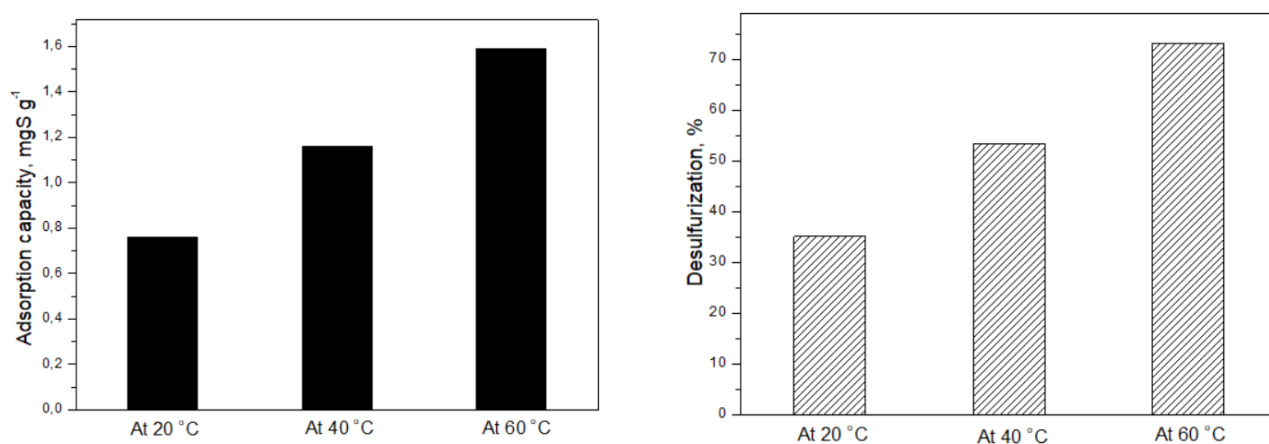


Fig. 4. Composition of the mixture model fuel determined with GC-MS analysis after adsorption at different temperatures.

rice husks can be used for the further purification of weakly reactive thiophene compounds, the model mixture prepared for analysis had a total sulfur concentration of 600 ppm. From the results obtained, after carrying out the adsorption process, the adsorption capacity of the pyrolyzed rice husks as well as the degree of desulfurization were calculated (Fig. 3).

The results presented in Fig. 3 are like those obtained when conducting dynamic adsorption with individual model fuels, namely that with increasing process temperature, both the adsorption capacity and the degree of desulfurization increase.

The composition of the mixture model fuel determined with GC-MS analysis is presented in Fig. 4. The results show that in the presence of benzothiophene, dibenzothiophene, and 4, 6-dimethyldibenzothiophene

in the model mixture, the adsorbent from pyrolyzed rice husks exhibits the best affinity for dibenzothiophene at all three temperatures of the adsorption process. For all three thiophene compounds, the degree of desulfurization increases with increasing temperature during the adsorption process. According to the obtained results, the selectivity of the used adsorbent decreases in the order dibenzothiophene > 4, 6-dimethyldibenzothiophene > benzothiophene. These results differ from those obtained in the adsorption of the individual model solutions, in which pyrolyzed rice husks have the best adsorption capacity with respect to benzothiophene. This can be explained by the fact that the molecular size of benzothiophene is smaller compared to those of dibenzothiophene and 4, 6-dimethyldibenzothiophene and this allows a larger amount to be adsorbed in the

pores and on the surface of pyrolyzed rice husks.

According to the studies of Zhou et al., the selectivity of the adsorbent depends not only on its type and surface characteristics (specific surface area and pore size distribution), but also on the surface functional groups of the adsorbent [35]. In the same study, the influence of surface oxygen-containing functional groups on the adsorption selectivity of the material was proven. According to the same author, the adsorption selectivity of the studied adsorbents increases in the order: benzothiophene < naphthalene < 2-methylnaphthalene < dibenzothiophene < 4-methyldibenzothiophene < 4, 6-dimethyldibenzothiophene. The higher degree of desulfurization of dibenzothiophene, compared to 4, 6-dimethyldibenzothiophene, by the model mixture, in our case, is probably since the size of the molecule does not allow it to enter the pores of the adsorbent and to bind to the active centers. It is also possible that the two methyl groups in the 4, 6-dimethyldibenzothiophene molecule sterically hinder the sulfur atom, making its sorption difficult.

## CONCLUSIONS

In this work, the application of pyrolyzed rice husks for adsorptive desulfurization in dynamic conditions was investigated. It has been proven that the initial concentration of sulfur compounds and temperature affect the adsorption capacity of the adsorbent. The highest adsorption capacity was achieved at 60°C and the highest initial sulfur concentration in the model fuel for both sulfur compounds was used. The positive effect of temperature suggests that, in addition to physical adsorption, chemical interactions between the adsorbent and the sulfur compounds may also contribute to the overall mechanism.

In the adsorption of individual model fuels, the adsorbent showed better capacity towards benzothiophene. However, in a competitive environment of sulfur compounds, pyrolyzed rice husks showed the best affinity towards DBT. The selectivity of the used adsorbent decreases in the order DBT > 4, 6 - DMDBT > BT.

Future research on dynamic adsorption should systematically investigate and establish critical parameters that govern process efficiency and adsorption performance. Key aspects requiring further examination include the bed height of the column, the breakthrough

point, and the exhaustion time of the adsorbent. A comprehensive analysis of these factors will enhance the fundamental understanding of adsorption dynamics and facilitate the optimization of industrial applications through process control and mass transfer efficiency.

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