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## Optimising the adsorption characteristics of spent coffee grounds by thermal and chemical activation

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**Abstract.** The aim of this study was to optimise the adsorption parameters of methylene blue on spent coffee grounds by combining thermal exposure and orthophosphoric acid treatment. The study was carried out using a spectrophotometric method, infrared spectroscopy, and a method for determining the specific surface area by water vapour adsorption. A possible mechanism for the adsorption of the cationic dye methylene blue on coffee grounds has been proposed. It was found that an increase in the processing temperature wasn't led to a linear increase in the specific surface area and adsorption characteristics of spent coffee grounds. It was discovered that coffee grounds' adsorption properties were inferior to those of untreated samples at temperatures of 400°C and 800°C. However, thermal activation of spent coffee grounds at 600°C led to a 72% increase in the specific surface area (from 561 to 958 m<sup>2</sup>/g). Treatment of the waste with 60% orthophosphoric acid solution increased the specific surface area by 23% (up to 690 m<sup>2</sup>/g) compared to untreated coffee grounds. The most effective in terms of adsorption characteristics of the adsorbent was the combined treatment of coffee grounds with orthophosphoric acid followed by thermal activation at 200°C, which provided a maximum specific surface area of 1078 m<sup>2</sup>/g and water vapour adsorption of 0.543 g of water per 1 g of sample, exceeding the characteristics of some commercial activated carbon samples. Under these conditions, the highest removal efficiency of methylene blue from model solutions was achieved – 57% in 30 min and almost 90% of the dye in 180 minutes of contact. The proposed conditions for the modification of spent coffee grounds make it possible to obtain an effective biosorbent for wastewater treatment from organic dyes, which is of practical importance for solving environmental problems in the textile and printing industries and the rational use of coffee production waste

**Keywords:** biosorbent; coffee waste; thermal modification; phosphate activation; methylene blue

### Introduction

Despite controversial evidence on the health benefits of coffee drinks, N. Zhao *et al.* (2024) indicated that their popularity was growing globally and, in some countries, where coffee had become part of the cultural tradition. G.D. Gebreeyessus (2022) stated that coffee was currently the most popular beverage after water and was the best-selling commodity after oil and oil products. According to the Coffee Market Report (2024), global coffee consumption in 2024 was reported at 177.0 million 60 kg bags, up 2.2% from the 2023. The per capita

consumption of coffee per year in North America was about 5.0 kg, in Europe – 4.2 kg, in Asia and Oceania – 0.58 kg. According to K. Johnson *et al.* (2022), only about 30% of the weight of coffee beans can be extracted into a coffee drink, and 70% ended up as spent coffee grounds, which were mostly disposed of as waste. H. Ahmed *et al.* (2024) noted that spent coffee grounds (SCGs) were solid waste generated after coffee was brewed using various methods. About 6 · 10<sup>6</sup> tonnes of SCGs were produced annually worldwide,

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half of this amount was from the production of instant coffee. Small coffee shops, restaurants, and individuals generated 50% of the SCGs produced globally.

Spent coffee grounds were mostly not recycled, but disposed of in solid waste landfills. Greenhouse gasses including carbon dioxide and methane are released, when the organic components of SCGs break down anaerobically. As noted by G. La Scalia *et al.* (2021), greenhouse gas emissions from coffee waste decomposition had a significant impact on climate change and atmospheric degradation. However, the implementation of innovative processing technologies allowed to transform the environmental challenges associated with the disposal of SCGs into a number of promising opportunities for resource-efficient use of this raw material. In this regard, the high content of lignin, cellulose, hemicellulose, proteins, lipids and other bioactive substances in spent coffee grounds made it a valuable and cheap raw material for further processing. P.J. Ong *et al.* (2023) proposed the possibility of using spent coffee grounds to create structural, energy-saving, and packaging materials. S. Hechmi *et al.* (2023) studied the possibility of using SCGs for composting and soil structuring.

The use of coffee waste as a biosorbent had gained worldwide attention due to a number of physical and chemical properties, including the unique porous structure of spent coffee grounds. Because heavy metals and dyes are easily dispersed, toxic, can build up in the human body, and cannot biodegrade, treating water of these contaminants has proven to be a very difficult problem. Coffee waste had a large specific surface area and can be used as an alternative to traditional adsorbents such as activated carbon, silica gel and zeolites. A significant advantage of adsorbents

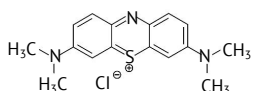
based on coffee waste was their low cost, availability and natural origin. The study by V. thi Quyen *et al.* (2021) examined the use of coffee grounds as an adsorbent for water treatment. In the work of A. Skorupa *et al.* (2023), it was proposed to use raw coffee grounds for the adsorption of dyes, heavy metals, microplastics and other pollutants. Many scientific works focused on the systematic study of the influence of key parameters of the adsorption process, such as adsorbent dosage, solution pH, contact time, temperature, on the efficiency of dye removal. Dye adsorption parameter optimisation on wasted coffee grounds was a challenging problem that required more work.

The aim of this study was to optimise the adsorption parameters of methylene blue, a hazardous dye commonly used in the textile and printing industry, onto SCGs by combining thermal exposure and orthophosphoric acid treatment.

## Materials and Methods

The coffee grounds used for the study were those that had been brewed in a coffee machine in the canteen of Lutsk National Technical University during 2024. The coffee grounds were washed with hot water until the washing liquid became colourless. Distilled water was used to rinse the garbage one last time. After being completely cleaned, the coffee waste was dried for 24 hours at 100°C to 110°C in an oven. For chemical activation of the spent coffee grounds, H<sub>3</sub>PO<sub>4</sub> (60%) was used. To study the adsorption characteristics, a methylene blue solution with a concentration of 20 mg/l of the initial solution was used, which was prepared before each adsorption test. The main characteristics of methylene blue were given in Table 1.

**Table 1.** Main characteristics of methylene blue

Chemical structure	Molecular formula	Chemical name	Molecular weight (g/mol)	$\lambda$ , nm
	C <sub>16</sub> H <sub>18</sub> ClN <sub>3</sub> S · 3H <sub>2</sub> O	3,7-bis (Dimethylamino)-phenothiazin-5-ium chloride	319.85	665

**Source:** developed by the author based on M. Clark (2016)

The absorption of water vapour was used to assess the adsorbent's specific surface area. Using an analytical balance, the coffee waste sample was weighed in a porcelain crucible. After weighing, the sample was placed in a desiccator, the lower chamber of which was filled with water, for 48 hours. After that, it was weighed again and the mass of adsorbed water vapour was determined. The water vapour absorption experiments were carried out in triplicate, and the average value was presented in the article.

Specific surface area, which was defined as the ratio of the interfacial surface of a substance to its mass:

$$S_A = S/m, \quad (1)$$

where  $S_A$  – specific surface area;  $S$  – interfacial surface of a substance;  $m$  – mass.

The area of the interfacial surface can be determined by the formula:

$$S = n \cdot N_A \cdot A(H_2O), \quad (2)$$

where  $n$  – amount of water substance (mol);  $N_A$  – Avogadro's constant;  $A(H_2O)$  – the area occupied by 1 water molecule ( $5.94 \cdot 10^{-20} \text{m}^2$ , based on the assumption that a water molecule was spherical and had a diameter of  $2.75 \cdot 10^{-10} \text{m}$ ).

For the physical activation, the coffee grounds were dried to a constant weight at a temperature of 100°C-110°C, after being washed with hot water. For

one hour, the coffee grind samples were maintained at 400°C, 600°C, and 800°C in a muffle furnace. A desiccator was used to bring the samples down to room temperature. A 60% orthophosphoric acid solution, made from a 90% solution, was utilised for chemical activation. After being cleaned and dried, the coffee grounds were combined with the orthophosphoric acid solution in a 1:1 ratio and allowed to activate for 48 hours in a closed flask. After activation, the treated coffee waste was rinsed with plenty of water until the rinsing water became discoloured and dried in an oven at 110°C.

The adsorption efficiency was determined on an aqueous solution of methylene blue containing 20 mg/l of the dye. The test was carried out at room temperature. Accordingly, the prepared coffee waste weighing 5 g was dispersed in 200 ml of the solution. The pH of the solutions was maintained at 7 by adding 0.1 M HCl or NaOH solutions. The suspension was continuously stirred for 10 min to ensure better contact between the pollutant in solution and the active centres of the adsorbent. After the determined contact time of the dye solution and coffee waste, the suspension was filtered and the optical density of the filtrate was determined using a photoelectrocolourimeter AP-120 APEL (Japan) at 670 nm. The amount of dye removed was determined by equation:

$$Q = \frac{C_0 - C_e}{C_0} \cdot 100\% \quad (3)$$

where  $Q$  – amount of dye removed;  $C_0$  and  $C_e$  – initial and final concentrations of methylene blue in solution (mg/l).

Adsorption tests were repeated 3 times, and the averaged adsorption value was used in the work.

## Results and Discussion

A basic pre-treatment, which included hot water rinsing and drying, was performed following the collection of coffee trash in the university canteen. W.E. Oliveira *et al.* (2008) stated that the purpose of thoroughly washing used coffee grounds was to remove impurities to avoid their influence on the adsorption process. The composition of coffee waste was important in the context of its use as a sorbent, since the properties of coffee grounds and their suitability for surface modification depend on the specific functional groups on their surface, as well as on the chemical bonds that the coffee grounds will form during adsorption with the adsorbate.

The FTIR spectrum of the spent coffee grounds was shown in Figure 1. The analysis of the coffee grounds spectrum showed the presence of intense bands of  $\nu$  CH alkyl groups (3000-2800  $\text{cm}^{-1}$ ) and a band in the region of 3650-3500  $\text{cm}^{-1}$  ( $\nu$  OH). This showed that the sample included a lot of hydroxyl OH functional groups and hydrocarbon chains. There were a lot of triple carbon-carbon  $\text{C} \equiv \text{C}$  bonds in the sample in organic chains, as evidenced by the strong absorption band in the triple bonds region (2300-2360  $\text{cm}^{-1}$ ). Ketones and esters were detected in the sample at a frequency of 1750-1735  $\text{cm}^{-1}$ , which corresponded to the absorption band of the carbonyl group  $\nu$  C=O; the absorption band of aromatic aldehydes was detected at a frequency of 1710-1685  $\text{cm}^{-1}$ . The presence of absorption bands in the region of 1605-1555  $\text{cm}^{-1}$  indicated the presence of solid amino acids.

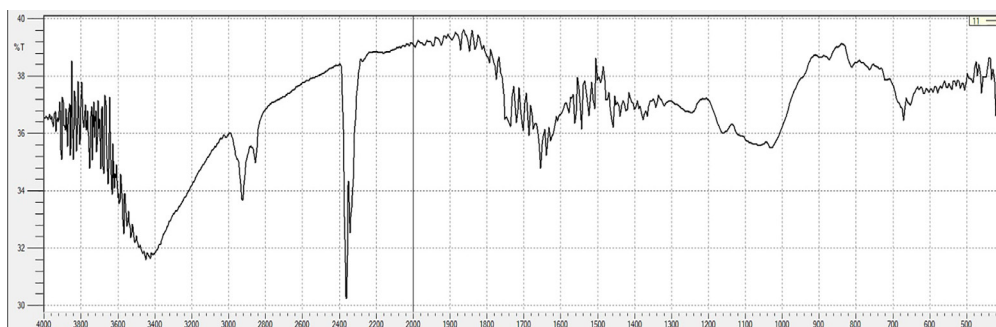
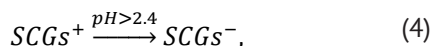


Figure 1. FTIR spectrum of spent coffee grounds

Source: developed by the author

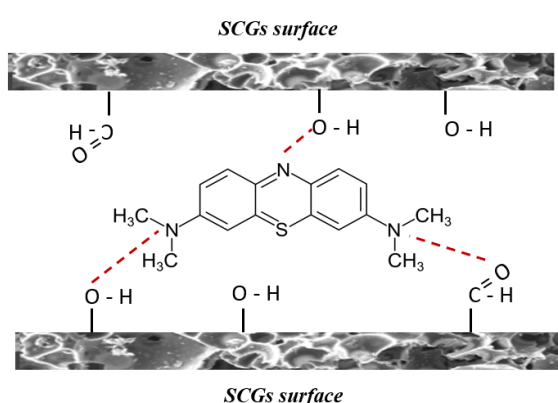
The diverse cavity and porous structures of hemicellulose, cellulose and lignin with a large number of different functional groups indicated a high adsorption capacity of spent coffee grounds for pollutants, even after simple pre-treatment. The study by Q.-A. Trieu *et al.* (2022) reported the pH of the medium as a crucial element in the methylene blue adsorption process on discarded coffee grounds. The surface charge of spent coffee waste has been determined by many scientists. In particular, R. Lafi *et al.* (2014)

indicated that for untreated coffee waste, the surface charge was negative above pH 5.5, and F. Aouay *et al.* (2024) found that, when coffee waste was treated with orthophosphoric acid, the surface charge of coffee waste became negative already at pH 2.4. The authors M. Samilyk *et al.* (2024) also used a methylene blue solution, when they studying the microbiological indicators of kefir drinks. Thus, an increase in the pH of the medium led to a change in the surface charge from positive to negative:



where *SCGs* – spent coffee grounds; *pH* – hydrogen index.

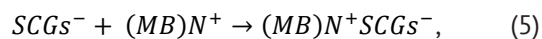
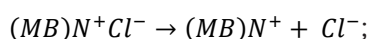
S. Bouzikri *et al.* (2022) described many factors that contribute to the adsorption of dyes on coffee waste: hydrogen bonds, electrostatic attraction, Van der Waals forces,  $\pi$ - $\pi$  bonds. The synergistic process that was the adsorption mechanism of dyes included both the physical adsorption of dye molecules onto the coffee waste substrate and the formation of chemical interactions through surface contacts. The interaction between these physicochemical processes was demonstrated by Figure 2, which depicted the suggested mechanistic pathway for methylene blue adsorption onto wasted coffee grounds.



**Figure 2.** The mechanism of methylene blue adsorption on the surface of spent coffee grounds

Source: developed by the author based on S. Bouzikri *et al.* (2022)

The electrostatic interaction of the cationic dye with the negatively charged coffee waste surface and the bonding of the dye's nitrogen of two amino groups with the oxygen of hydroxyl or carbonyl groups were the main reasons, why the dye and SCGs interact:



where *MB* – methylene blue; *SCGs* – spent coffee grounds.

Under alkaline conditions, the interaction between methylene blue and coffee grounds was optimised through robust electrostatic forces between the positive charge of the cationic dye ((*MB*)*N*<sup>+</sup>) and the

negative charge of the coffee grounds surface (*SCGs*<sup>-</sup>) (Trieu *et al.*, 2022). Physical and chemical modification methods were used to increase the adsorption activity of untreated coffee waste and optimised its properties (Kang *et al.*, 2022). A. Młynarczykowska & M. Orlof-Naturalna (2024) shown that physical techniques do not show a significant boost in the effectiveness of removing dyes and heavy metals from aqueous solutions, despite their technological simplicity. Research focused on chemical modification of spent coffee grounds surface with acids and alkalis to incorporate functional groups that enhance its ability to absorb heavy metal ions and dyes (Taleb *et al.*, 2020).

Heat treatment increased the specific surface area and encourages the conversion of cellulose, hemicellulose, and lignin into a porous structure (Chen *et al.*, 2021). O. Senneca *et al.* (2020) found that the first step in the destruction of polysaccharides, which were the basis of coffee grounds, was depolymerisation into oligosaccharides. Moreover, hemicellulose was the most labile component, decomposing at 200°C–260°C. Cellulose was relatively more stable, decomposing in the temperature range of 240°C–350°C, lignin was the most thermally stable of all, had the widest decomposition interval, and began to decompose between 280°C and 500°C. At significantly higher temperatures, the hemicellulose, cellulose, and lignin polymer chains oxidised, forming carbon monoxide, carbon dioxide, and methane. The decomposition, evaporation and depolymerisation of hemicellulose, cellulose and lignin without heat treatment and activator occur at different rates. Thus, according to H.M. Boundzanga *et al.* (2022), the mass loss of hemicellulose, cellulose and lignin at a temperature of 800°C was 76.5%, 80.9% and 55.1%, respectively.

Activated carbon was formed only as a result of heat treatment of lignin in the temperature range of 1200°C–1800°C in an environment of nitrogen and carbon dioxide. For the purpose of physical activation, the pre-cleaned coffee grounds were subjected to heat treatment in the temperature range of 400°C–800°C, and the waste was chemically activated with orthophosphoric acid without and with temperature exposure. Water vapour adsorption and specific surface area were determined for each sample. The effects of different treatment types on the specific surface area and adsorption capacity of the coffee grounds were shown in Table 2.

**Table 2.** Adsorption capacity and specific surface area of coffee grounds

Sample	Mass of adsorbed water vapour, g per 1 g of sample	Specific area <i>S<sub>A</sub></i> , m <sup>2</sup> /g
Activated carbon (Red star)	0.355 ± 0.02	705 ± 0.08
Dry raw coffee grounds	0.283 ± 0.01	561 ± 0.07
Coffee grounds after temperature treatment at 400°C	0.262 ± 0.01	515 ± 0.08
Coffee grounds after temperature treatment at 600°C	0.482 ± 0.03	958 ± 0.08
Coffee grounds after temperature treatment at 800°C	0.212 ± 0.01	422 ± 0.08

Table 2. Continued

Sample	Mass of adsorbed water vapour, g per 1 g of sample	Specific area $S_A$ , m <sup>2</sup> /g
Coffee grounds after H <sub>3</sub> PO <sub>4</sub> treatment	0.347 ± 0.03	690 ± 0.08
Coffee grounds after H <sub>3</sub> PO <sub>4</sub> treatment and temperature treatment at 200°C	0.543 ± 0.04	1078 ± 0.1
Coffee grounds after H <sub>3</sub> PO <sub>4</sub> treatment and temperature treatment at 400°C	0.402 ± 0.03	798 ± 0.08
Coffee grounds after H <sub>3</sub> PO <sub>4</sub> treatment and temperature treatment at 600°C	0.152 ± 0.01	300 ± 0.05

Source: developed by the author

Significant variations in specific surface area and adsorption capacity were observed, when the coffee grounds samples under investigation were compared. The base raw coffee grounds actually had the lowest values: the surface area was 561 m<sup>2</sup>/g and the water vapour adsorption was 0.283 g H<sub>2</sub>O/g. Exposure of the coffee grounds to 400°C did not change the porosity, which was in good agreement with the literature. At this temperature, only cellulase and hemicellulose began to decompose, while lignin decomposition had not yet begun. Increasing the processing temperature to 800°C led to a degradation of properties: the surface area decreased sharply to 422 m<sup>2</sup>/g, and the adsorption decreased to 0.212 g of H<sub>2</sub>O per 1 g of sample. The coffee grounds' structure was destroyed and their porosity decreased as a result of the high temperature. It was found that the greatest impact on the porosity of coffee grounds was caused by their treatment at a temperature of 600°C – the specific surface area increased from 561 to 958 m<sup>2</sup>/g, i.e. by 72%.

Orthophosphoric acid (H<sub>3</sub>PO<sub>4</sub>) was an effective activating agent that catalysed the dehydration of the primary material. Consequently, the lignocellulosic complex's heat degradation threshold was lowered (Ivanichok *et al.*, 2023). Activation with orthophosphoric acid enhanced the yield of the carbonised product and developed the porous structure of the material. The emission of volatile chemicals during thermochemical processes led to the creation of pores. M. Myglovets *et al.* (2014) pointed to the destruction of polymeric lignin chains and carbon oxidation. Water vapour, carbon monoxides (both carbon monoxide and carbon dioxide), and volatile phosphorus compounds were the products of these processes. The work of H.M. Boundzanga *et al.* (2022) found that during activation with orthophosphoric acid, lignin forms micropores in the adsorbent, unlike hemicelluloses and cellulose, which were responsible for the formation of mesopores in it. Therefore, treating coffee waste with an orthophosphoric acid solution will help to destroy the lignin-cellulose complex and form additional pores of different sizes. By creating phosphorus-containing surface groups, orthophosphoric acid also contributed to the stabilisation of the carbon backbone.

After treating the coffee grounds samples with orthophosphoric acid without the influence of temperature, the specific surface area of the adsorbent was

690 m<sup>2</sup>/g, an increase in surface area of 23% compared to untreated coffee grounds. The most effective was the chemical activation with orthophosphoric acid followed by a temperature treatment at 200°C. Under these conditions, the maximum performance was achieved: specific surface area of 1078 m<sup>2</sup>/g and water vapour adsorption of 0.543 g H<sub>2</sub>O per 1 g of sample, which exceeded the characteristics of some commercial activated carbon samples (705 m<sup>2</sup>/g). Studies had shown that combined chemical and heat treatment does not always provide a linear improvement in properties. For example, activation at 400°C and 600°C after chemical treatment showed a gradual decrease in adsorption capacity. The results of the correlation between the adsorption time and the amount of methylene blue elimination were displayed in Figure 3.

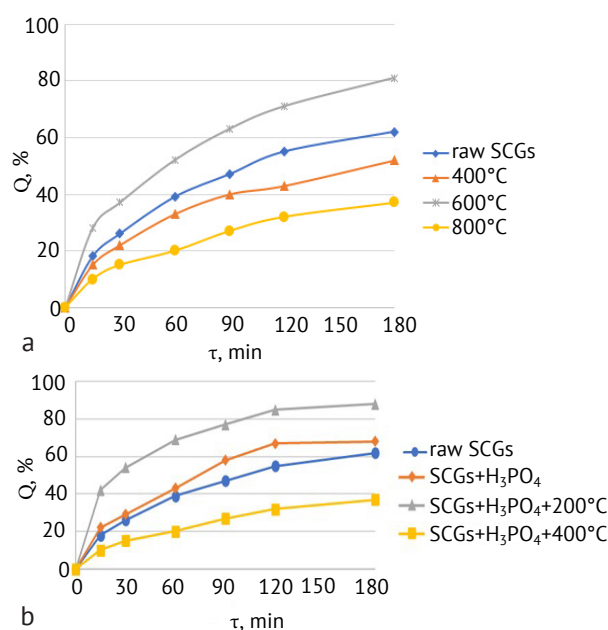


Figure 3. Dependence of methylene blue adsorption ( $Q$ , %) on time

Note: a – for thermal treated coffee grounds samples; b – for coffee grounds samples after treatment with orthophosphoric acid and combined treatment (thermal + H<sub>3</sub>PO<sub>4</sub>)

Source: developed by the author

When the adsorption characteristics of coffee grounds were examined at several temperature ranges,

the findings revealed patterns that were mostly consistent with their specific surface area value. The coffee grounds' adsorption capabilities were merely average without heat treatment, reaching only roughly 60% of the maximal value. At 400°C, there was no significant improvement in the ability to adsorb substances. The most optimal heat treatment temperature for achieving maximum adsorption performance was 600°C. Figure 3, a showed that the methylene removal reached about 80-85% of the highest possible value, which was the best result among all studied regimes. Increasing the temperature to 800°C significantly reduced the adsorption capacity of the spent coffee grounds and led to a decrease in the number of pores in the adsorbent and a decrease in the diversity and number of surface functional groups.

When compared to the unaltered samples, the adsorption capacity of coffee grounds was marginally enhanced by the chemical treatment with orthophosphoric acid. The best adsorption properties were shown by the samples after combined treatment with orthophosphoric acid and a temperature of 200°C. For these samples, a 57% adsorption rate was achieved at an adsorption time of 30 min, and almost 90% of methylene blue was adsorbed in 180 min. The optimal conditions for modifying used coffee grounds were low-temperature combined treatment of spent coffee grounds with orthophosphoric acid.

## Conclusions

The study of the adsorption characteristics of spent coffee grounds showed that modified SCGs were a promising biosorbent for wastewater treatment from organic dyes. The optimisation of adsorption properties was achieved by combined thermal and chemical activation.

The study's findings demonstrated that raising the heat treatment temperature does not necessarily result in a linear rise in the specific area and enhancement of the adsorbent's adsorption properties. It was found that

the treatment of coffee grounds at 400°C and 800°C does not improve the adsorption efficiency of methylene blue, but rather worsens it compared to untreated samples. The most effective thermal activation temperature was 600°C, which increased the specific surface area to 958 m<sup>2</sup>/g.

Treatment with a 60% solution of orthophosphoric acid improved the adsorption characteristics, in particular, increased the specific surface area by 23% (up to 690 m<sup>2</sup>/g). However, a combination of chemical and heat activation produced the best outcomes. The treatment of coffee grounds with orthophosphoric acid followed by thermal activation at 200°C resulted in a specific surface area of 1078 m<sup>2</sup>/g and water vapour adsorption of 0.543 H<sub>2</sub>Og per 1 g of sample, which exceeded the characteristics of some commercial activated carbon samples. The most successful method for eliminating methylene blue from model solutions was combined treatment, which combined thermal and chemical effects. It enabled the adsorption properties to be improved and the activation temperature to be drastically lowered. The sorbent prepared under these conditions was capable of removing 57% of methylene blue after 30 min of contact with the model solution, and 90% of the dye was removed after 180 min of adsorption.

Further research will be aimed at expanding the range of pollutants that can be removed using modified coffee grounds and investigating the possibility of multiple uses in adsorption and regeneration processes.

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## Conflict of Interest

None.

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## Оптимізація адсорбційних характеристик відпрацьованої кавової гущі шляхом термічної та хімічної активації

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**Анотація.** Метою цього дослідження була оптимізація параметрів адсорбції метиленового синього на відпрацьованій кавовій гущі шляхом поєднання термічної обробки та обробки ортофосфорною кислотою. Дослідження проводилося за допомогою спектрофотометричного методу, інфрачервоної спектроскопії та методу визначення питомої поверхні шляхом адсорбції водяної пари. Запропоновано можливий механізм адсорбції катіонного барвника метиленового синього на кавовій гущі. Було встановлено, що підвищення температури обробки не призводило до лінійного збільшення питомої поверхні та адсорбційних характеристик відпрацьованої кавової гущі. Виявлено, що адсорбційні властивості кавової гущі при температурах 400°C і 800°C були гіршими порівняно з необробленими зразками. Однак термічна активація відпрацьованої кавової гущі при 600°C забезпечила збільшення питомої поверхні на 72 % (з 561 до 958 м<sup>2</sup>/г). Обробка відходів 60 % розчином ортофосфорної кислоти підвищила питому поверхню на 23 % (до 690 м<sup>2</sup>/г) у порівнянні з необробленою кавовою гущею. Найефективнішою за адсорбційними характеристиками виявилася комбінована обробка кавової гущі ортофосфорною кислотою з подальшою термічною активацією при 200°C, що забезпечило максимальну питому поверхню 1078 м<sup>2</sup>/г та адсорбцію водяної пари 0,543 г води на 1 г зразка, що перевищує характеристики деяких зразків комерційного активованого вугілля. За таких умов досягнуто найвищу ефективність видалення метиленового синього з модельних розчинів – 57 % за 30 хвилин і майже 90 % барвника за 180 хвилин контакту. Запропоновані умови модифікації відпрацьованої кавової гущі дозволили отримати ефективний біосорбент для очищення стічних вод від органічних барвників, що має практичне значення для вирішення екологічних проблем у текстильній та поліграфічній промисловості, а також сприяє раціональному використанню відходів кавового виробництва

**Ключові слова:** біосорбент; кавові відходи; термічна модифікація; фосфатна активація; метиленовий синій