

Removal of selected dyes on activated carbons under flow-through conditions

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ABSTRACT

Dyes and pigments are important organic pollutants in the aquatic environment. They are emitted into wastewater from various industries, mainly from the dye, textile, cosmetic and paper industries. Dyes can be removed from wastewater by one of the most effective methods of wastewater treatment – adsorption on activated carbons. The aim of this study was to determine the efficiency of dye removal from aqueous solutions using an adsorption process. The dynamic adsorption of two dyes were studied: the cationic Aniline blue and the anionic Acid bud. The study used two commercial activated carbons – ROW 08 Supra and WG-12 and was carried out under flow-through conditions in filter columns. The initial concentration of the dyes tested was $C_0 = 100$ mg/L. The flow direction of the solution containing the dye solution was from top to bottom with flow velocities of 3, 6 and 9 m/h. Tests were carried out until a dye concentration equal to 0.9 of the initial concentration was obtained in the column effluent. Based on the laboratory results obtained, so-called exit curves were plotted. Dynamic dye adsorption values were high, especially for ROW 08 Supra activated carbon. Longer protective times were obtained when the cationic dye Aniline blue was sorbed.

Keywords: Activated carbon; Adsorption; Dyes; Wastewater treatment

1. Introduction

One of the important aspects of the struggle for a clean environment is the care for the purity of water, which is subject to pollution as a result of human activities in technological, urban and processing processes. Modern water pollution is not only solid waste, wastewater of biological origin, but also, or rather mainly, wastewater from a variety of chemical processes, as a result of which pollutants enter into permanent reactions with water [1–3].

Particularly dangerous for the environment is the appearance in wastewater of heavy metal compounds, chemically active substances, toxic and radioactive substances, and very complex chemical compounds that are difficult or even impossible to remove using traditional treatment methods. The process of treating such contaminated wastewater requires specialized solutions that allow not only the

chemical purification of water, but also the restoration in it of the basic components necessary for the proper functioning of the hydrosphere.

Dyes are used in a very wide range of industries, which automatically makes them sites of wastewater generation from dyeing and coloring processes. An example of such sources of wastewater from dye production are plants producing a variety of paints and varnishes. This group of plants includes manufacturers of emulsion paints, oil paints, acrylic paints for the construction industry, the automotive industry or the aforementioned clothing and textile industry [4–6].

Dyes are organic compounds with complex chemical structures, characterized by a certain toxicity to aquatic organisms, and their presence in industrial wastewater is a huge problem, not least because of their low biodegradability [7–9]. More than 100,000 industrial dyes are

currently in use, and their annual production is estimated at 7×10^5 Mg [10–14].

The textile industry produces wastewater that contains a range of soluble and insoluble substances, including unreacted dyes and other substances used at various stages of the production process. The effective use of dyes in the production process in the manufacturing process is sometimes less than 60% [15,16]. The remainder, along with other substances, enters the wastewater, which increases the values of biochemical oxygen demand, chemical oxygen demand, total dissolved solids and other parameters [17–20].

The environment (lakes, rivers, ponds) is negatively affected by discharged industrial wastewater containing significant amounts of hard-to-degrade dyes, which, depending on the type, give water a more or less intense, undesirable color. In the case of coloured organic compounds, colouring of the water can usually be seen at levels below 1 mg/g [21]. The need for decolorization involves the use of many complicated processes and, consequently, an increased cost of treatment. Despite advancing purification techniques, the number and type of applied dyes are increasing at the same time, making the process largely difficult and slowing down. The efficiency of treatment application becomes higher the more clean water is recovered and the more effectively the pollutant load in the discharged wastewater is reduced [22].

Despite the use of newer and newer technologies, it is hard to protect water resources from dyes. This is a broad group of pollutants that have a negative impact on the environment. The utility of water for domestic purposes decreases with the amount of polluting dye. By the high accumulation of dye from, for example, wastewater from printing plants or fabric dyeing plants in the water, light access is reduced [23]. The result is a disturbed self-purification process and the inability of plants to take in sufficient light and aquatic organisms – fish [24–26].

Water that is contaminated with dyes can also cause skin irritation, respiratory infections, or gastrointestinal problems [27], as well as allergies [28].

Dyes can also cause allergic reactions and have mutagenic and carcinogenic effects [29,30].

The intensive development of the production of dyes, pigments and paints, has contributed to the creation of various types of wastes, which, penetrating into the environment, pose a serious threat to aquatic ecosystems [31,32]. Dyes are also used in the food industry, in the production of, among other things: beverages and confectionery [33,34].

Also, the chemical industry and plants involved in the production of cosmetics, pharmaceuticals and plastic products use a variety of dyes in the production process to color their products.

Despite the wide range of knowledge in many fields regarding the treatment of dye wastewater, there is still no concrete way to solve this problem. The main courses of action for this purpose are the following methods: chlorination, ozonation, coagulation, chemical precipitation, adsorption, electrochemical processes, membrane methods and biological treatment [34,35]. The adsorption process is often used in the purification of water from organic compounds, including dyes [36–38]. Of the various sorbents widely available on an industrial scale, activated carbon is

the most widely used. They are characterized by a well-developed specific surface area, a porous structure and the possibility of being regenerated when the sorption capacity is exhausted. Porous carbon materials can be used in granular, granular or particulate form. These materials adsorb unwanted substances by chemical or physical adsorption. Once their adsorption capacity is exhausted, they undergo regeneration processes. An additional advantage of activated carbons is that they can be modified. Adsorption is therefore a promising process for the treatment of contaminated wastewater, including those containing dyes [39–45].

Studies of adsorption under dynamic (flow) conditions start at a small scale, which are then reproduced at a large scale. Performing dynamic adsorption studies on a laboratory scale enables the correct selection of parameters such as:

- the flow rate of the dye solution through the filter column,
- concentration of the dye to be tested,
- the height of the activated carbon bed in the column,
- the mass of the selected adsorbent.

The study of the adsorption properties of carbonaceous materials in aqueous solutions is mainly based on adsorption static, while adsorption dynamics is carried out to a much lesser extent. It seems that for practical adsorbent use, knowledge of adsorption dynamics is essential.

On the basis of dynamic adsorption studies under laboratory conditions, it is possible to determine whether and to what extent a given adsorbent can be used to purify water or wastewater from organic compounds, including dyes. Therefore, the aim of the present study was to investigate the dynamic adsorption of two dyes (Aniline blue and Acid bud) from aqueous solutions on two activated carbons (ROW 08 Supra and WG-12).

The scope of work included the preparation of the activated carbons used for the study and conducting the study under dynamic conditions – in a filter column filled with activated carbon. Dynamic adsorption studies were carried out until the bed was saturated. The time to adsorption capacity breakthrough, the time to adsorption capacity depletion, the height of the mass exchange zone, the velocity of the mass exchange zone and the dynamic adsorption capacity of the activated carbons used were determined.

2. Experimental set-up

2.1. Sorbate

The anionic dye, Acid bud and the cationic dye, Aniline blue, produced by BORUTA – ZACHEM KOLOR (Poland), were selected for the study.

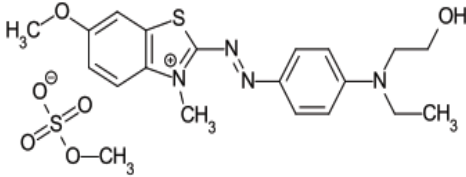
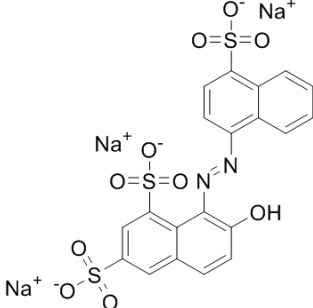
Table 1 presents the characteristics of the dyes used in the laboratory tests.

2.2. Sorbent

For the laboratory tests, two types of activated carbon were used as adsorbent material filling the filter columns:

- ROW 08 Supra – is a granular and refined carbon that can be thermally regenerated. It is produced from peat by Dutch Company NORIT,

Table 1
Characteristics of dyes

Type of dye	Name of dye	Structural formula
Cationic	Aniline blue	
Anionic	Acid bud	

- WG-12 – granulated coal, produced from hard coal by the Polish company GRYFSKAND sp. z o.o. from Hajnówka.

Activated carbons used in the study are obtained through the application of steam-gas activation. They are mainly used for drinking water treatment.

Table 2 presents the characteristics of the basic physical and chemical properties of used activated carbons, given by their manufacturers.

2.3. Experimental methodology

Adsorption dynamics studies were conducted in a 2.6 cm diameter filter column filled with a 25 cm layer of activated carbon. Before the actual tests, distilled water was passed through the carbon bed for 1 h, after which the bed was left saturated for 24 h. Only after this time, a solution of the appropriate dye was passed through the adsorbent layer. Filtration was carried out in the top-down direction at 3, 6, and 9 m/h, corresponding to volume fluxes of, respectively: 26.6, 53.1 and 79.7 mL/min. The initial concentration of the dyes tested was $C_0 = 100$ mg/L. The filtrate was sampled at 30 min intervals. The concentration of dye in the filtrate was determined by Thermo Electron Corporation HELIOS α spectrophotometer using previously prepared standard curves. The wavelengths corresponding to the adsorption maximum of a given dye were, respectively:

Aniline blue $\lambda = 609$ nm,

Acid bud $\lambda = 520$ nm.

Dynamic adsorption studies were carried out until the bed was saturated. As it is stated in literature [46,47], if dye concentration in the filter reaches 90% of the initial

Table 2
Physico-chemical properties of ROW 08 Supra and WG-12 activated carbons

Indicator	Value	
	ROW 08 Supra	WG-12
Specific surface area, m ² /g	796	1,005
External surface, m ² /m ³	3,208	2,292
Bulk density, g/L	380	420
Mechanical strength, %	98	98
Granule diameter, mm	0.8	1.2
Water adsorption, mL/g	0.97	0.82
Ash content, %	5.94	11.00
pH of aqueous extract	8.6	10.1
Methylene number, LM, mL	30	30
Iodine adsorption, LJ, mg/g	1,096	1,050

concentration, this moment is considered as bed saturation. On the other hand, bed breakthrough occurs when the dye concentration in the filter reaches a concentration equal to 10% of the initial concentration.

Based on the laboratory results obtained, the velocity of displacement of the mass exchange zone along the height of the activated carbon layer (u) was calculated using Eq. (1):

$$u = \frac{h}{t_k - t_p} \quad (1)$$

where u – velocity of the mass exchange zone along the height of the activated carbon layer, cm/h; h – height of mass exchange zone, cm; t_p – time to achieve breakthrough by the adsorbent bed, min; t_k – time until the bed exhausts its adsorption capacity, min.

Eq. (2) was used to calculate the dynamic adsorption:

$$a_d = \frac{C \cdot t_p \cdot Q}{V} \tag{2}$$

where a_d – dynamic adsorption, mg/L; C – concentration, mg/L; t_p – time to achieve breakthrough by the adsorbent bed, h; Q – solution flow rate through the carbon bed, L/h; V – volume in the activated carbon column, L.

The results shown are the average of 3 measurements.

3. Results and discussion

3.1. Adsorption of Acid bud

Figs. 1 and 2 show bed breakthrough curves for Acid bud using ROW 08 and WG-12 activated carbons from aqueous solutions at applied flow velocity of 3, 6 and 9 m/h.

The results of the study of adsorption of Acid bud from solution show that a higher flow rate of the solution through the filter column causes a faster depletion of the adsorption capacity of the tested carbon and a decrease in dynamic adsorption. Exhaustion of the adsorption capacity for ROW 08 activated carbon at the applied flow velocity of 3m/h occurred after 45.5 h, while for the flow velocity of 9 m/h it was 9.5 h. The second adsorbent used also showed faster depletion for the bed with the higher flow velocity, for which, at a flow velocity of 3 m/h, the column operating time was 31.5 h, while for a flow velocity of 9 m/h, the

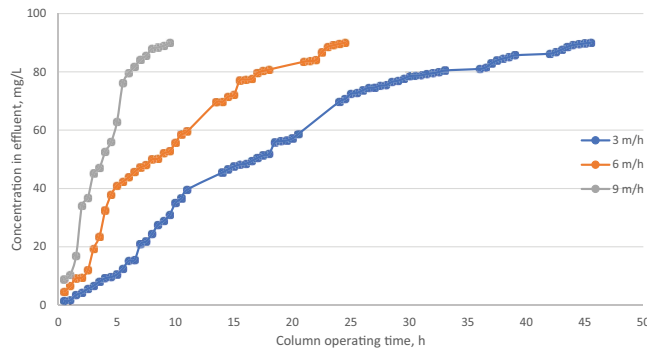


Fig. 1. Breakthrough curve of the activated carbon bed ROW 08 for the Acid bud.

time decreased to 7 h. Of the carbons used in the study, adsorption on ROW 08 activated carbon is much longer than for WG-12 activated carbon. Table 3 shows the obtained adsorption results of the dye used.

3.2. Adsorption of Aniline blue

The test results for Aniline blue are shown in Figs. 3 and 4 in the form of graphs of breakthrough curves using ROW 08 and WG-12 activated carbons from aqueous solutions at applied flow velocity of 3, 6 and 9 m/h.

The results obtained by adsorption of Aniline blue show that the higher speed of carrying out filtration increases the breakthrough time of the adsorption capacity.

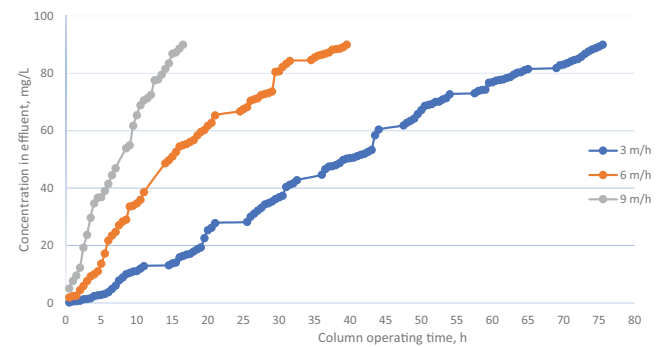


Fig. 3. Breakthrough curve of the activated carbon bed ROW 08 for the Aniline blue.

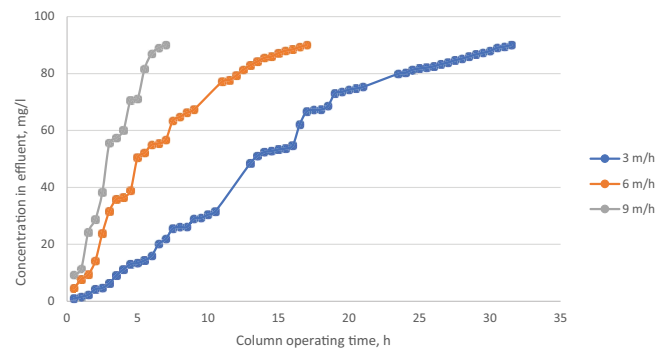


Fig. 2. Breakthrough curve of the activated carbon bed WG-12 for the Acid bud.

Table 3
Adsorption of Acid bud

Type of carbon	Flow velocity	Time to adsorption capacity breakthrough		Time to adsorption capacity depletion		Mass exchange zone sliding velocity	Dynamic adsorption		
		m/h	h	min	h		min	cm/h	g/L
ROW 08	3		5.06	303.33	45.5	2730	0.62	6.07	15.96
	6		2.72	163.33	24.5	1,470	1.15	3.27	8.60
	9		1.06	63.33	9.5	570	2.96	1.27	3.34
WG-12	3		3.50	210.00	31.5	1,890	0.89	4.20	9.99
	6		1.89	113.33	17.0	1,020	1.65	2.27	5.40
	9		0.78	46.67	7.0	420	4.02	0.93	2.21

Table 4
Adsorption of Aniline blue

Type of carbon	Flow velocity	Time to adsorption capacity breakthrough		Time to adsorption capacity depletion		Mass exchange zone sliding velocity	Dynamic adsorption	
	m/h	h	min	h	min	cm/h	g/L	mg/g
ROW 08 Supra	3	8.40	504.00	75.5	4,536	0.37	10.08	26.51
	6	4.39	263.33	39.5	2,370	0.71	5.27	13.86
	9	1.83	110.00	16.5	990	1.70	2.20	5.79
WG-12	3	5.78	346.67	52.0	3,120	0.54	6.93	16.49
	6	3.06	183.33	27.5	1,650	1.02	3.67	8.73
	9	1.22	73.33	11.0	660	2.56	1.47	3.50

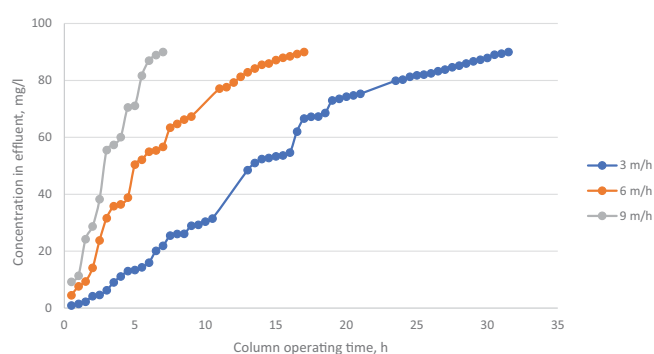


Fig. 4. Breakthrough curve of the activated carbon bed WG-12 for the Aniline blue.

In the case of activated carbon ROW 08 at a speed of 3 m/h, this time was 75.5 h, and with an increase in the flow rate to 9 m/h, this time decreased to 16.5 h. We can see the same with respect to activated carbon WG-12 where, at the smallest applied speed of solution flow through the column, the time of adsorption capacity depletion was 52 h, while for the speed of 9 m/h this time decreased to 11 h. Table 4 also shows that the sliding speed of the mass exchange zone increases with increasing speed. Compared to the second cationic dye used, the filtration of Aniline blue is shorter on both activated carbons used.

From the output curves (isoplan) the following were read working time of the deposit up to the breakthrough point t_p and up to depletion t_w and the speed of movement of the mass exchange zone mass “ u ” (Tables 3 and 4).

The activated carbons used in the study (ROW 08 and WG-12) effectively removed the selected dyes (Aniline blue and Acid bud). Adsorption proceeding under dynamic conditions strictly depends on many factors, including the rate of water flow through the filter bed, which directly affects the contact time of the flowing solution with the activated carbon. The results of dynamic adsorption studies of two different dyes on activated carbons are shown in the figures as breakthrough curves. The curves obtained show that, irrespective of the type of dye used, the order of bed breakthrough was similar for the activated carbons tested. This phenomenon is closely related to the speed of the flowing solution. Similarly, the saturation time of the tested activated carbons followed a similar order as the bed breakthrough

time. Regardless of the type of dye used, shorter saturation times were obtained for activated carbon WG-12. However, comparing the dyes, longer saturation times were obtained when Aniline blue was sorbed. The longer protection times obtained for ROW 08 coal can be explained by its larger external surface area. Total adsorption capacities of carbons of activated carbons in relation to the Acid bud determined under flow conditions ranged between 3.34 and 15.96 mg/g for ROW 08 coal, while the dynamic sorption capacities for WG-12 coal ranged from 2.21 to 9.99 mg/g. Slightly higher adsorption capacities of the carbons were obtained for Aniline blue, which ranged from 5.79 to 26.51 mg/g (ROW 08) and from 3.50 to 16.49 mg/g (WG-12).

Dynamic adsorption studies were carried out until the carbon bed was saturated. If the dye concentration in the filter reaches 90%–95% of the initial value, this moment is often considered bed saturation, while bed breakthrough occurs when the dye concentration in the filter reaches a value equal to 10% of the initial concentration [48]. In the present study, measurements were taken until the concentration in the effluent reached 90% of the initial concentration, or 90 mg/L. The authors of the paper [49], who conducted studies on the sorption of a dye called Acid Orange 7 under dynamic conditions, found that increasing the flow rate of the solution flowing through the filter column results in a decrease in the adsorption efficiency of the dye. Therefore, it is important to determine the correct flow rates through the adsorption bed to remove as much dye as possible. This was confirmed in their study by the authors of the paper [50], who conducted research on the adsorption of methylene blue on activated carbon obtained from agricultural waste. They found that the adsorption rate was affected by the water flow rate, but also by the height of the bed.

4. Conclusions

Adsorption is a very useful method for purifying water from organic compounds, including dyes, and activated carbon is one of the most important adsorbents used for this purpose on an industrial scale. Thanks to their highly developed porous structure, very large specific surface area and pore volume, activated carbons are able to adsorb large quantities of harmful dyes contained in wastewater and can be regenerated once their adsorption capacity is exhausted.

Activated carbons are very effective in removing both anionic and cationic, which were used in laboratory studies. The effectiveness of their removal depends on the type of activated carbon used.

Of the activated carbons used in the study, the most effective adsorption under dynamic conditions was on ROW 08 Supra activated carbon. The longest time to complete depletion of the sorption capacity were obtained on this activated carbon.

An increase in filtration speed results in faster depletion of adsorption capacity, which results in obtaining shorter times for the use of activated carbons.

The adsorption method is easy to use, stable in performance and low in cost and does not cause secondary pollution or change the structure of the pollutants in the dye wastewater, so it is widely used in wastewater treatment [51].

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