Removal of Acid Textile Dye from the Aqueous Solution by a New Adsorbent Obtained from Waste Cotton from the Garment Plant

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Summary: The paper investigates the possibility of removing acid dye from the aqueous solution by an adsorption process on new adsorbent prepared from waste cotton textile from the ready-made garment industry. It is a waste generated during the cutting of the layers of cotton knitwear, which is practically the product from the textile cutting process. The obtained adsorbent is a bulk material with heterogeneous porous particles, of ragged shapes. In particle interiors, there are pronounced cracks, cavities and channels that form the basis of microporous material. The qualitative and quantitative characterization of the obtained adsorbent shows that this is a relatively porous material where the carbon is dominant in the chemical composition. The results of textural properties of new adsorbent from cotton knitwear waste show different parameters which with their numerous values characterize the specific surface area, pore volume or pore diameter. It can be said that the obtained new adsorbent has micropores and small mesopores, which produce a high specific surface area. During adsorption, the longer contact time causes a greater amount of dye on the adsorbent, i.e. with the duration of the adsorption process the dye concentration in the solution decreases. A number of isotherms of two- (*Langmuir*, *Freundlich* and *Jovanovic)*, three- (*Toth*, *Sips* and *Radke-Prausnitz)*, and four-parameter models (*Fritz-Schlunder* and *Marczewski-Jaroniec)* were used to describe the adsorption process. The four-parameter isotherms are best covered by experimental points and most accurately describe the events of adsorption of acid dye on the surface and in the interior of the new adsorbent particles obtained from ready–made garment cotton waste. The results of this research suggest the possibility of practical application in the decolorization of the colored waste waters of the textile industry providing a contribution to protecting the environment from both an economic and a practical point of view.

Keywords: adsorbent; acid dye; adsorption; modelling; ready-made garment.

Introduction

Some industries, such as textile, leather, paper or food industries, are extensive dye consumers. The dyes can cause environmental hazards due to the presence of a large number of contaminating substances, such as toxic organic residues, acids, bases or inorganic substances. Some of the dyes are carcinogens and mutagens because they have previously been generated from hazardous chemicals such as benzidine, metals, etc. The discharge of colored liquid waste in the receiving waters not only affects their aesthetic nature, but also impedes the transmission of sunlight and thus reduces photosynthetic activity, which disturbs the natural balance affecting the aquatic world and the food chain [1,2].

One of the main methods for removing pollutants from waste waters is the use of porous solid absorbents. The properties of porous materials, which make them useful for water treatment, are high porosity and surface, as well as the physical and chemical nature of internal absorptive surfaces. Adsorbents, such as activated carbons are the most commonly used in separation and purification processes. Lately, researchers have been intensifying their efforts to find alternative adsorbents that would replace expensive activated carbons. Industrial waste materials are one of the potentially cheap adsorbents for the removal of metals and organic matter [3-5]. In the adsorption fields, several hybrid materials have been used to remove dyes. The new technology was used to develop chitosan/polyamide nanofibers, with potential to remove dyes from aqueous media. The nanofibers have a semicrystalline structure, with several functional groups and diameter from 100 to 500 nm [6]. In another case, natural clay was grafted with 3-aminopropyl-trietoxisilane for obtaining an organic-inorganic hybrid material. This material was successfully used as adsorbent for removal of acid anionic dyes from aqueous solutions. Also, natural pozzolan, an amorphous silicate-based material of volcanic origin was modified by using 3-aminopropyl-triethoxysilane as a grafting agent. The functionalized materials were used successfully for the dye removal from aqueous solutions using batch-contact adsorption [7].

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This paper deals with the modelling of adsorption of textile acid dye from aqueous solution to new adsorbent that is made from waste cotton textile from the ready-made clothing factory. Textile waste is a by-product formed during the cutting process in fabricating cotton garments. The goal is to utilize this waste textile material, transform it into potential adsorbent and use it to remove textile acid dye, for example, after textile dyeing.

Experimental

New adsorbent obtained by chemical and physical modification of cotton waste from the garment factory was used. After collection, the waste is cut into as small pieces as possible, and washed with detergent (Felosan NKB) in hot distilled water (60°C). After drying at 100°C, the chopped waste was treated with $CaCl_2$ solution (bath ratio 1: 100, 30 g·dm^{-3} $CaCl_2$) for 48 h at room temperature, after squeezing, the samples were dried (150°C) to dry. So dried samples are packed in aluminium foil and are used for annealing at 450°C, 1.5 h. It is followed by cooling and partial shredding (mortar and pestles), rinsing with distilled water and neutralizing with sodium bicarbonate. After drying (100°C) and grinding the sample is ready for experiment of removing the acid dye from the solution.

The adsorption test was performed in glass reaction vessels in which the new adsorbent was suspended in the solution of acid dye (adsorbate). The reaction vessels are placed on a shaker with a circular motion (120 rpm) at a temperature of 20°C and held for specific time. The amount of adsorbent was constant, 1 g, while the solution in a constant volume of 0.1 dm^3 contained acid dye of the suggested initial concentrations, 50, 100, 150, 200, 250, 300, 350 and 400 mg·dm^{-3}.

Considering that there are eight different initial dye concentrations, as well as wanting to save time and materials, an attempt has been made to remove the dye concentrations of 150, 250 and 350 mg·dm^{-3} and only work with the remaining initial dye concentrations. This was decided on the basis of the diagram in Fig. 1, where the graph with all concentrations is dominated, and in the right-hand corner there is a graph with a reduced number of initial dye concentrations. It is noticed that there are no major differences in the shape of a diagram and a curve that links all the experimental points, which can be checked mathematically on the basis of the middle squares of deviations after fitting.

So, it was decided to work with the next initial dye concentrations: 50, 100, 200, 300 and 400 mg·dm^{-3}.

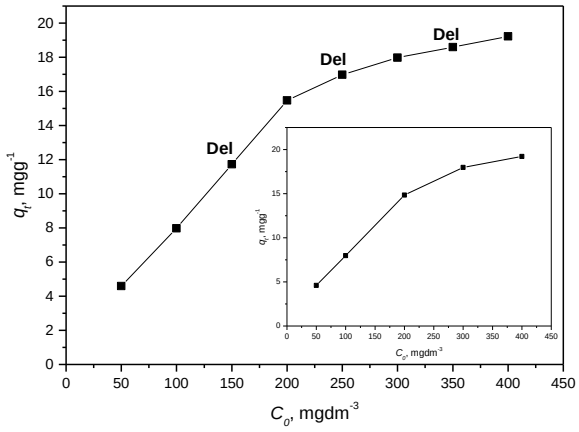

Fig 1: Graph in graphics for the choice of work initial dye concentrations.

The treatment time, with continuous shaking, was 3, 6, 10, 20 and 30 min. The pH of the aqueous solution of the dye was 2-3. The equilibrium time of acid dye adsorption on new adsorbent was achieved after 30 min, and with the prolongation of the treatment time, the amount of adsorbed dye was not significantly changed (Fig. 2).

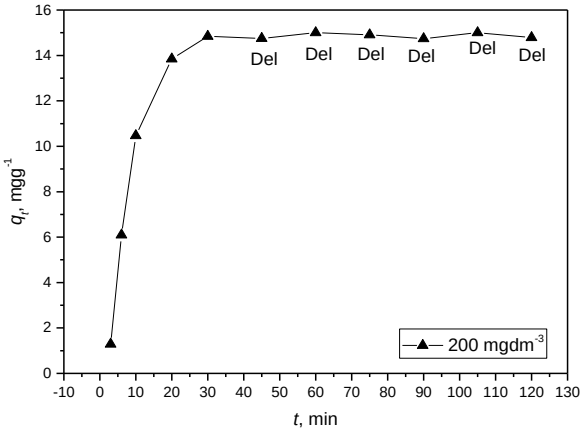

Fig. 2: Graph for the choice of equilibrium time of the adsorption.

The used acid dye belongs to the group of dyes with one sulpho group and one phenylamine (aniline) functional group, Fig. 3. The color index is C.I. Acid Blue 113, and it is completely soluble in water and ethanol, but poorly soluble in acetone and insoluble in other solvents. It is used for dyeing wool, polyamide and silk fibers, paper and leather [8].

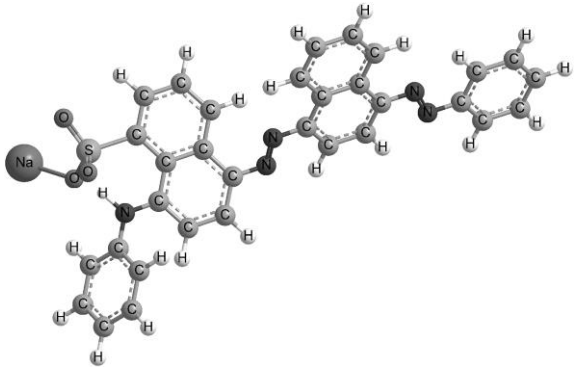

Fig. 3: The structure of the used acid dye C.I. Acid Blue 113 [5].

Measurement of the absorption of the solution was done on the spectrophotometer UV-VIS spectrophotometer (Cary 100 Conc UV-VIS, Varian) at 590 nm (maximum wavelength of the spectrum of the used dye solution).

The degree of dye removal was calculated from dye concentration before and after the treatment [9]:

$$Dye\ removal = \frac{C_0 - C_t}{C_0} \times 100 \qquad (1)$$

where: C_0 and C_t were initial dye concentration and dye concentration in time t (mg·dm^{-3}), respectively. The amount of adsorbed dye (adsorbate) per unit mass of the adsorbent at time *t* or at equilibrium, $q_{t,e}$ (mg·g^{-1}) was determined by the expression [9]:

$$q_t = \frac{(C_0 - C_t) \times V}{w};\ q_e = \frac{(C_0 - C_e) \times V}{w};\ (2)$$

where: *w*, mass of the adsorbent (g); *V*, solution volume (dm^3); C_e, dye concentration at equilibrium (mg·dm^{-3}).

SEM measurements were performed on the Tescan Mira3 microscope (Czech Republic) using the secondary electron detector and the EDS detector was used to measure characteristic X-rays of the main elements. The EDS system provided a quick estimation of the elemental composition of the sample-adsorbent.

Nitrogen adsorption-desorption isotherms were determined using the Micromeritics ASAP 2020 instrument. The samples were degassed at 150°C for 10 h under reduced pressure. The specific surface area of the samples was calculated according to Brunauer, Emmett, Teller (BET) method from the linear part of the adsorption isotherms of nitrogen. The total pore volume (V_{tot}) was given at *p/p0*=0.998. The volume of the mesopores was calculated according to the Barrett, Joyner and Halenda method from the desorption branch of isotherm. The volume of micropores was calculated from alpha-S plot.

Results and Discussion

The obtained adsorbent is a bulk material with heterogeneous porous particles, of ragged shapes. In particle interiors, there are pronounced cracks, cavities and channels that form the basis of microporous material. Otherwise, porosity is characteristic of the adsorbent, which is made possible by the presence of particles of very irregular shapes, high degree of amorphization and a large number of voids in the structure. The micrograph in Fig. 4 gives an appearance with magnification of ×200. In addition to the difference in shape, the pores vary also in their availability for the adsorbate molecules that can be associated with the fact that they can be closed, open only at one end or at both ends, and can be isolated or interconnected [10].

In a similar paper, which deals with adsorption of acid dye on adsorbent obtained from plant residues and biological waste, it has been established that the SEM images revealed the formation of irregularly shaped and sized cavities in non-uniform particles of the adsorbent [11]. In other case, the adsorbent that was prepared by chemical activation of the sludge collected from tannery wastewater treatment plant, had a similar appearance to the surface of its particle based on micrographs [12].

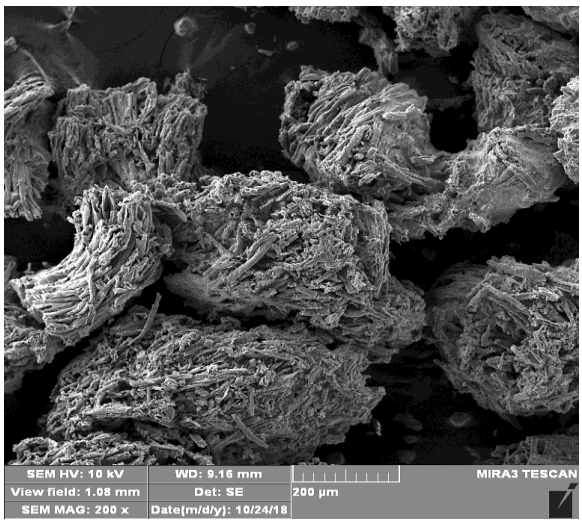

Fig. 4: SEM micrograph of the used adsorbent.

Table-1: Textural characteristics of new adsorbent obtained by N_2 adsorption and desorption analysis.

Specific surface area S_p, $m^2 \cdot g^{-1}$	Total pore volume V_{total}, $cm^3 \cdot g^{-1}$	Volume of mesopores V_{meso}, $cm^3 \cdot g^{-1}$	Volume of micropres V_{micro}, $cm^3 \cdot g^{-1}$	Average pore diameter D_{sr}, nm	Pore diameter occupying the largest volume D_{max}, nm
48.66	0.0999	0.0866	0.0189	10.0	7.9

The EDS system allows a quick estimation of the elemental composition of the sample. The results show the quantitative composition of new adsorbent, i.e. percentage of one element in relation to the sum of all detected elements. C (61.64 %), O (32.42 %), Na (1.18 %), Cl (2.25 %), Ca (2.51 %) were detected, and the individual percentages of each element meant that in 100 g of all detected elements there are exactly as many grams of each element as shown.

Based on data from the EDS analysis, as it is expected, carbon dominates, while the greater presence of oxygen is related to metal oxides (Na), and there is also a possibility of reaction of carbon with oxygen from the air.

Similar results were obtained by authors who prepared adsorbent (activated carbons) from different types of cellulosic waste materials. Thus, in adsorbent from plant waste the following chemical composition was found: C (53.13 %), O (46.36 %), Al (0.2 %), Ca (0.12 %), Si (0,12 %) and K (0.07 %) [13] or in adsorbent (activated carbons) from rice straw: C (64.80 %), O (17.00 %), S (0.11 %), N (2.75 %), H (2.75 %) and ash (13.60 %) [14].

The textural properties of new adsorbent from cotton ready-made waste are given in Table 1. The results show different parameters which with their numerous values characterize the specific surface area, pore volume and pore diameter.

It can be said that the obtained new adsorbent has micropores and small mesopores, which produce a high specific surface area. The largest part of the volume occupies pores with diameter of 7.9 nm, one part of the volume occupy also pores with diameter of 4.05 nm as well as micropores.

Similar texture properties have also adsorbent from different research. Thus, when analyzing adsorbent from textile waste-sludge, specially made for removing textile dyes, it was found that, for example, the values of the specific surface area range [15] from 90 to 221 $m^2 \cdot g^{-1}$ or with adsorbent made from oak leaves, pine needles, walnut shell, sunflower stalk or shells of hazel, the specific surface area is 15.5, 34, 21, 31, 27, 60 $m^2 \cdot g^{-1}$, respectively [16].

The selected dye C.I. Acid Blue 113 has good solubility due to the presence of one sulpho group, and shows a good tendency towards the adsorbent with which it is in contact, since it has a weak tendency toward the aqueous phase. Starting from the fact that the applied dye molecule is linear, and also contains a system of conjugated double bonds, it can be easier and more likely to be bound to the adsorbent relative to a dye molecule that is not linear and has no conjugated system of bonds. Further, the dye molecule of C.I. Acid Blue 113 can have a higher substantivity for the adsorbent, since it has 2 benzene rings in its structure lying in one plane (planar structure). The presence of sulpho group gives a negative charge to this dye, the aromatic ring (in aniline) decreases the basicity of the amine depending on the substituents, and the presence of the amino group increases the reactivity of the aromatic ring due to an electron donor [8].

The influence of the initial dye concentration on its dye removal or amount of removed dye was tested at a concentration range of 50–400 $mg \cdot dm^{-3}$ (Fig. 5). By increasing the initial dye concentration, dye removal is reduced in all cases. The initial concentration of dye in the solution gives an important driving force to overcome the mass transfer resistance between the aqueous and solid phase.

According to the curves in the diagram of Fig. 5, the dye removal is expected to be greatest at lower initial concentrations, representing an apparent observation, since in absolute amount of the dye removed is greatest at the highest initial concentrations, which is clearly seen from the diagram in Fig. 6.

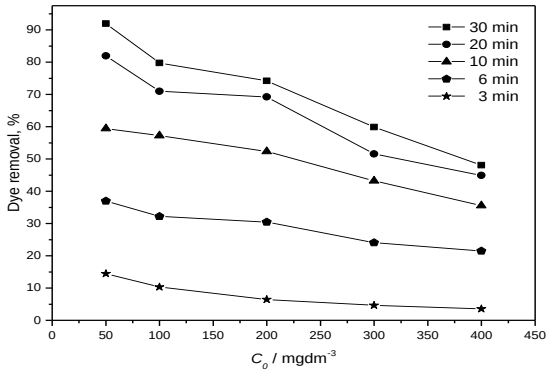

Fig. 5: Removed acid dye depending on the initial concentration during adsorption.

Fig. 6 shows a change in the adsorbed amount of dye per unit of the adsorbent mass during adsorption for different initial dye concentrations. The diagram from this picture confirms that the amount of adsorbed dye increases with the duration of adsorption, and that the highest adsorption is observed at the highest initial concentrations.

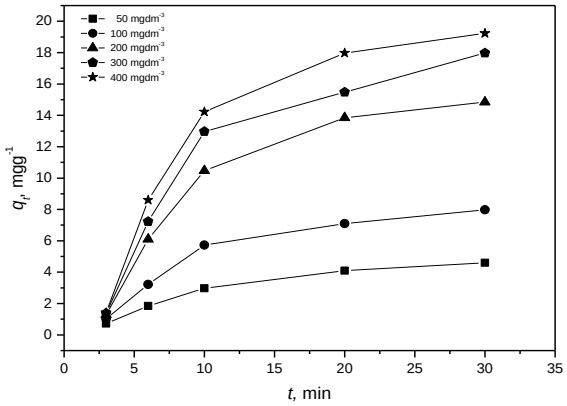

Fig. 6: The change of the adsorbed amount of acid dye to new adsorbent during adsorption.

Similar behavior in terms of the degree of dye removal of azo dye on adsorbent obtained from biological waste is presented in other studies. For example, it was found that during adsorption, a decrease in the pH of the solution from 10 to 2 causes an increase in the percentage of dye removal from 39.11 to 49.73. Also, the adsorption occurred fast within the first 30 min and the rate gradually decreased until equilibrium, as the adsorption capacity of the dye progressively increases with the C_e increase [17].

Adsorption isotherm is essential for investigating the adsorption process. An analysis of isothermal data by their fitting, over different isothermal equations, is an important step towards finding a suitable model that can be used to control and monitor the adsorption process. In this study, two-, three- and four-parameter isothermal models were used to describe the adsorption procedure by nonlinear fitting to experimental points using the OriginPro software.

To evaluate the success of the fitting, a coefficient of determination is used, which represents a relative measure of representativity or measure of the usefulness of the model.

The diagram of Fig. 7 represents a non-linear interpretation of the two-parametric isothermal models of *Langmuir*, *Freundlich* and *Jovanovic*, showing the dependence of the q_e parameter in relation to the equilibrium dye concentration of C_e.

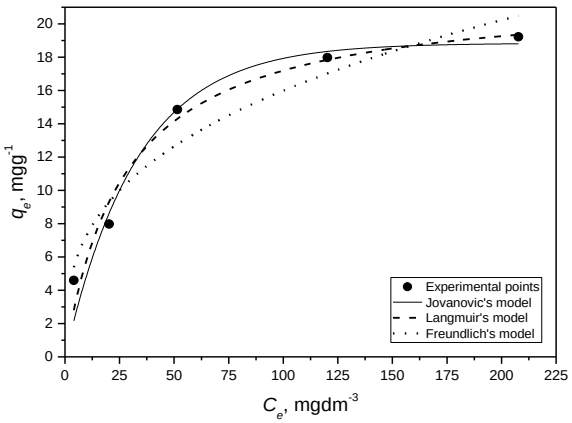

Fig. 7: Nonlinear forms of two-parametric adsorption isotherms for the system acid dye–new adsorbent.

According to Table 2, the coefficient of determination (R^2=0.967) of *Langmuir's* isotherm has the highest value compared to the coefficient of determination of other isotherms, which is favorable because it is the most effective in nonlinear simulation of isothermal adsorption of acid dye on new adsorbent. This claim is also confirmed by the visual inspection of curves from the diagram in Fig. 7. Thus, according to these results, the uniform adsorption energy on the surface dominates without the trans-migration of adsorbates in the surface of the adsorbent. The adsorption system is more homogeneous than it is heterogeneous and there is no direct relationship between the concentration of the adsorbate in the solution and the concentration of the adsorbate on the surface of the adsorbent.

Based on the results, *Jovanovic's* adsorption model follows immediately behind *Langmuir's* isotherm, which is expected, given that this model is very similar to *Langmuir's*. It is a monolayer adsorption, which also allows some mechanical contacts between adsorbed and desorbed dye molecules.

The weakest result shows *Freundlich's* model, which also has a high R^2 (0.943), but is certainly weaker compared to other two-parameter models.

Table-2: Analytical expressions of two-parameter nonlinear models for adsorption of acid dye on new adsorbent

Mathematic expression of the model	Model parameters		R^2
Langmuir: $q_e = \frac{0.79 \times C_e}{1 + 0.036 \times C_e}$	Q_0, mg·g^{-1}	b, dm^3·mg^{-1}	0.967
	21.90	0.036	
Freundlich: $q_e = 3.36 \times C_e^{0.34}$	K_F, mg·g^{-1} (mg·L^{-1})$^{1/n}$	n	0.943
	3.36	2.95	
Jovanovic: $q_e = 18.84 \times \left(1 - e^{-0.03 \times C_e}\right)$	q_m, g·mg^{-1}	K_J, dm^3·mg^{-1}	0.959
	18.84	0.030	

Table-3: Analytical expressions of three-parameter non-linear acid dye adsorption models on new adsorbent.

Mathematic expression of the model	Model parameters			R^2
Toth: $q_e \frac{14.61 \times C_e}{\left(12.87 + C_e^{0.91}\right)^{1.1}}$	q_{m_T}, mg·g^{-1}	K_T	t_T	0.968
	14.61	12.87	0.91	
Sips: $q_e = \frac{1.52 \times C_e^{0.76}}{1 + 0.06 \times C_e^{0.76}}$	q_{m_S}, mg·g^{-1}	K_S	m_S	0.974
	25.32	0.06	0.76	
Radke-Prausnitz: $q_e = \frac{2.81 \times 10^{46} \times C_e^{0.34}}{1 + 3.37 \times C_e^{-0.66}}$	$q_{m_{RP}}$, mg·g^{-1}	K_{RP}	m_{RP}	0.943
	8.35·10^{45}	3.37	0.34	

The same isotherms of a two-parameter character were used in a similar study, where it was confirmed that *Langmuir's* isotherms are dominant in the adsorption of acid dyes on an adsorbent made from pinewoods compared to *Freundlich* or *Jovanovic's* model [18].

Fig. 8 shows a diagram of three-parameter nonlinear isothermal models of *Toth*, *Sips* and *Radke-Prausnitz*, showing the dependence of the q_e parameter in relation to the equilibrium dye concentration of C_e. Table 3 presents the quantitative data of these models with mathematical expressions and model parameter values.

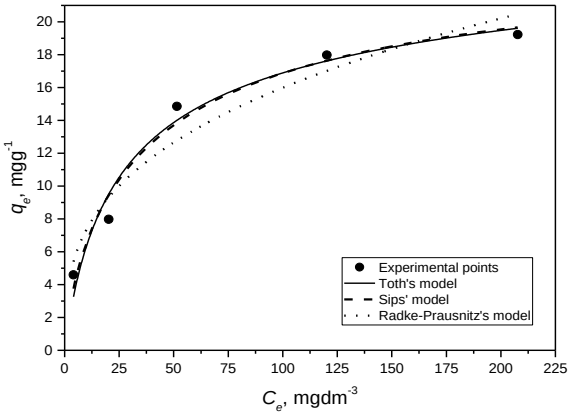

Fig. 8: Nonlinear forms of three-parameter adsorption isotherms for the system acid dye–new adsorbent.

Based on Fig. 8 and the results in Table 3, it is noticeable that the *Sips* nonlinear isothermal adsorption model best covers experimental points, which is confirmed by the numerical value of the coefficient of determination (R^2=0.974). Next to it are *Toth's* and *Radke-Prausnitz's* isotherms, according to the values of the same coefficient that determine the usefulness of the model.

To the same conclusion came the researchers who compared the same three-parameter isotherms, the *Sips* model went to victory, i.e. proved to be the most effective in adsorption of acid dyes on new adsorbent obtained from bean pods from the bean genus, as precursors [19].

Sips isotherm is a combination of *Langmuir* and *Freundlich's* equation for the prediction of heterogeneous adsorption systems. At low adsorbent concentrations, the *Sips* model is reduced to *Freundlich*, while at high concentrations (the case in this study) it predicts the monolayer adsorption capacity characteristic of *Langmuir's* isotherm [19].

The *Toth* model of isotherm, which follows in importance, has been developed to improve the *Langmuir* equation and is useful in the description of heterogeneous adsorption systems that satisfy the low and high concentration values [19].

The *Radke-Prausnitz* isotherm has several important properties, which makes it preferable in most adsorption systems at low concentrations of adsorbates. As this was done here with a higher concentration of adsorbates, this model yields the weakest result. Also, *Radke-Prausnitz's* model can be transformed into another model under certain conditions. At a low concentration, this model is reduced to the *Langmuir* model, while at higher concentrations the *Radke-Prausnitz* model is translated into *Freundlich's* [19].

In general, the three-parameter nonlinear isothermal models have better statistical indicators (R^2) of the success of covering experimental points when compared to two-parameter models.

The nonlinear forms of the four-parameter adsorption isotherms of *Fritz-Schlunder* and *Marczewski-Jaroniec* are presented in the diagram in Fig. 9. According to the curves of this diagram as well as on the results from Table 4, it is noticed that both models showed excellent results, especially if one looks at the values of the coefficients of determination that for these two models differ only in the third decimals (0.982 and 0.987).

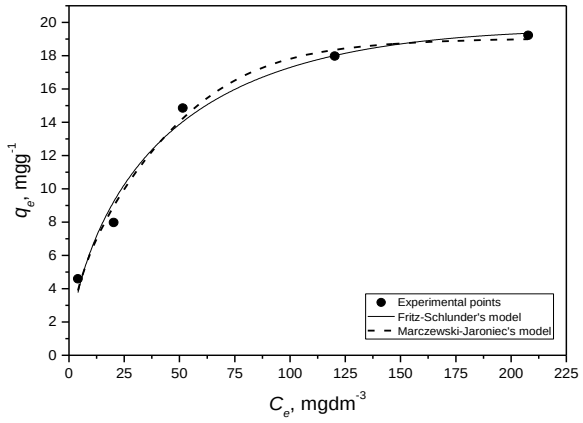

Fig. 9: Nonlinear forms of four-parameter adsorption isotherms for the system acid dye–new adsorbent.

Table 4: Analytical expressions of four-parameter nonlinear models for acid dye adsorption on new adsorbent.

Mathematic expression of the model	Model parameters				R^2
Fritz-Schlunder:	A_{FS}	B_{FS}	a_{FS}	b_{FS}	
$q_e = \frac{1.65 \times C_e^{0.61}}{1 + 0.0058 \times C_e^{1}}$	**1.65**	**0.0058**	**0.61**	**1**	**0.982**
Marczewski-Jaroniec:	$q_{m_{MJ}}$, **mg·g^{-1}**	K_{MJ}	n_{MJ}	m_{MJ}	
$q_e = 67.97 \times \left(\frac{(0.011 \times C_e)^{3.55}}{1 + (0.011 \times C_e)^{3.55}} \right)^{0.04}$	**67.97**	**0.011**	**3.55**	**0.14**	**0.987**

In the study of other researchers using the same four-parameter models, excellent results were obtained in the adsorption of base dye on new adsorbent made from cotton seeds, the coefficients of determination were R^2=1, for the *Fritz-Schlunder* model and R^2=0.993 for the model *Marczewski-Jaroniec* [20].

Compared to the two- and three-parameter models, the four-parameter isotherms of *Fritz-Schlunder* and *Marczewski-Jaroniec* best cover experimental points and most accurately describe the events of adsorption of acid dye on the surface and interior of the porous particles of new adsorbent obtained from the ready-made cotton waste.

This is expected if one takes into account that both four-parameter models have empirical equations that can respond to a wide range of experimental results due to the large number of coefficients (parameters) in isotherms.

Fritz-Schlunder's four-parameter empirical model includes the characteristics of *Langmuir* and *Freundlich's* isotherm [20].

Marczewski-Jaroniec's isotherm has a similarity with *Langmuir's* isotherm and is recommended based on the assumption of local *Langmuir* isotherm and the distribution of adsorption energy at active sites on the adsorbent [20].

Conclusion

Using calcium chloride, as an activating agent, a thermochemical conversion of waste cotton cuttings from the garment plant into powdered new adsorbent was carried out. The elemental composition of the produced adsorbent is dominated by carbon, which is in line with the theoretical data for active carbon, indicating that waste textiles can reasonably be considered a suitable coal precursor for conversion to activated carbon.

Based on the obtained results, it should be pointed out that new adsorbent obtained from waste ready-made cotton textile can be an effective adsorbent for removal of acid dyes from aqueous solution with a promising tendency of application also under industrial conditions.

Longer contact time means a higher amount of dye on new adsorbent, i.e. with the duration of the adsorption process the dye concentration in the solution decreases. The amount of adsorbed dye increases with the duration of adsorption, the highest adsorption is observed at the highest initial concentrations.

The *Langmuir* model is the most efficient of two-parameter isotherms in non-linear simulation of adsorption of acid dye on new adsorbent. The *Sips* model is the most efficient of the three-parameter isotherms in non-linear simulation of adsorption of acid dye to new adsorbent. The *Marczewski-Jaroniec* model is the most efficient of the four-parameter isotherms and is the most effective of all isotherms

compared in this study of non-linear simulation of adsorption of acid dye to new adsorbent.

According to the achieved effects, the results of this research suggest the possibility of practical application in the decolorization of the colored waste waters of the textile industry, providing a contribution to protecting the environment from both an economic and a practical point of view.

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