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Adsorption Of Acid Dyes On Active Carbon

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1. Introduction

Mechanical, physico-chemical, chemical and biological methods are used for treatment of post-dyeing wastewaters [1,2,3,4,5]. Each of the above methods allows for reduction of substances' concentration contained in the wastewaters [19÷25]. Many years of practical experience in municipal wastewater treatment plants indicates that the post-dyeing wastewaters do not change their colour, or that the change of colour is negligent after biological treatment. However, some research work has indicated [6,8] that there are admissible concentrations of dyes at which biological treatment of wastewaters can be successfully applied. Therefore, it is necessary to partly or fully remove dyes from wastewaters before they can be delivered to the biological treatment plant. This can be achieved through application of simple physico-chemical methods.

Effectiveness of physico-chemical methods varies and depends on the type of dyes being eliminated [9,10,11,12,13]. Each dye is, in most cases, a complex organic compound. It has chinoid and functional groups which give it either electropositive or electronegative nature depending on pH of the solution. Total elimination of dyes from their wastewaters can be achieved by application of coagulation together with sorption [14,15]. The coagulation should be performed through addition of two reagents in a specifically determined proportioning sequence. The dyes from the solution are precipitated together with a sediment which is a product of a reaction between the reagents (coagulants) supplied to the solution, therefore, this process is more a co-precipitation than coagulation.

The author [7,14,15], having done extensive research work, noted that there is a correlation between the dyes co-precipitation and adsorption process and their chemical structure and adsorbent surface potential. It was shown that dyes are precipitated with coagulants due to formation of dye hydroxo-complexes which can form a real solution or precipitate in the form of easily sedimentating deposit. Addition of calcium hydroxide, at the second stage, to the solution increases pH therefore, increases precipitability of the sediment (e.g. ferric hydroxide) on which adsorption or co-precipitation of dyes (or their complexes) can proceed. The author has tested a considerable group of dyes selected in random [17,14,15]. However, huge amount of dyes being manufactured worldwide makes further research work necessary. In particular, it seems advisable to check the assumption that acid dyes can easily be sorbed on electronegative surface of active carbon due to their chemical composition and tendency to colour fibres in acid reaction.

2. Experimental

The process of sorption was statistically tested assuming presupposed dye concentration, pH and varying time of contact between the sorbate and adsorbent (0÷300 seconds). The concentration of dyes was being changed from 250 to 1.25 mg/dm^3. The dye containing solution was mechanically agitated and the impeller velocity was approx. 60 r.p.m. After given time of adsorption the sample was being filtered through a medium hardness filter and the filtrate thus obtained was being analysed.

The resultant factors were as follows: transmittance (percent value of light transmission) at wavelength λ=410, 530 and 610nm and concentration of the dye remaining in the solution, mg/dm^3.

The amount of the dye remaining after sorption was being determined for respective dyes from reference curves indicating variability of transmittance as a function of dye concentration.

The electrokinetic potential is a factor determining indirectly the adsorbent surface potential. Hence its value depending on pH, dye type and concentration was being measured. These tests were performed on a zetametre with application of the parabola method which is based on measurement of electrophoretic mobility at nine depth levels of the testing cell. At least fifteen measurements for moving active carbon particles were performed at each level. The total amount of measurements making one result of the electrokinetic potential was around 135. The block diagram of the zetametre used for testing ζ potential has been shown in Figure 1.

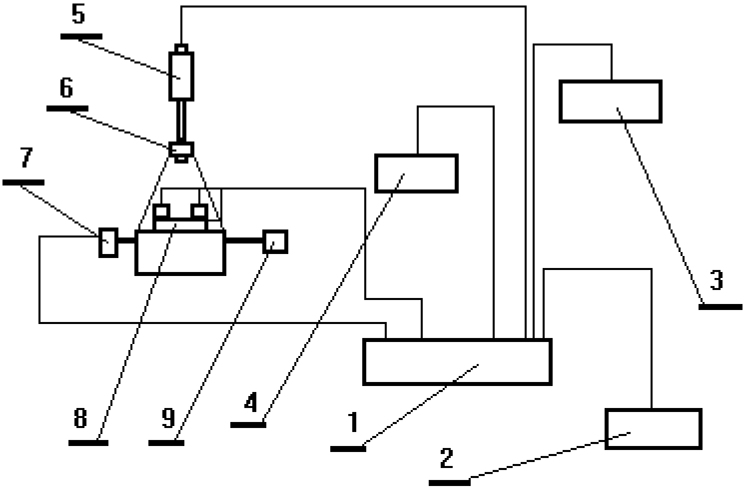


Fig. 1. Zetameter block diagram: 1 - microcomputer, 2 - keyboard, 3 - monitor, 4 - printer, 5 - camera, 6 - microscope, 7 - stepper motor, 8 - casing for the cell containing electrodes and temperature sensor, 9 - microscope power supply

Rys. 1. Schemat blokowy zetametru: 1 - mikrokomputer, 2 - klawiatura, 3 - monitor, 4 - drukarka, 5 - kamera, 6 - mikroskop, 7 – silnik krokowy, 8 – obudowa celki z elektrodami i czujnikiem temperatury, 9 – zasilacz mikroskopu

3. The characteristic of the sorbent and dyes used for testing

The sorbent used for testing was active carbon O-3. This product is made of high quality, selected, vegetable material.

The sorbent had the following properties [16]:

- discolouration force MB (milligramme number) 270-370 mg,
- degree of adsorption LM (methylene number) at least 14 ml,
- maximum moisture content 8%,
- pH=7,
- dust particle size,
- filtration ability – very good.

Tests indicated that active carbon 0-3 had a negative electrokinetic potential within pH scope 1.2÷13.1; see Figure 2, Table 1. It can be concluded therefrom that sorbent surface has an excess of negative charge and shows affinity to a positive charge. The graphic relationship between ζ potential and pH shown in Figure 1 can be described with a straight line equation:

$$\zeta = -0.83\text{pH} - 11.68 \tag{1}$$

Table 1. Testing effect of pH on active carbon ζ potential at concentration c=0.5 mg/dm^3
Tabela 1. Badania wpływu pH na potencjał ζ węgla aktywnego przy stężeniu c=0,5mg/dm^3

pH	Temperature	Current intensity E	Electrophoretic mobility R_{ef}	ζ	$ζ_{av}$
	K	V/cm	$\frac{\mu m \times cm}{V \times s}$	mV	mV
1.20	303.95 303.10 303.10	7.80 7.80 7.80	-10.52 -10.40 -10.65	-112.24 -112.19 -112.36	-112.26
2.5	308.80 300.60 299.90	13.86 13.86 13.86	-1.50 -1.47 -1.50	-16.11 -18.15 -18.60	-17.62
3.10	298.20 297.30 296.10	13.87 13.87 13.87	-0.87 -0.87 -0.84	-11.57 -11.41 -11.33	-11.44
4.40	294.10 295.10 294.60	13.87 13.87 13.87	-0.97 -0.89 -0.94	-13.53 -12.22 -12.99	-11.91
5.40	297.10 296.40 294.50	13.87 13.87 13.87	-1.19 -1.15 -1.11	-15.72 -15.62 -15.43	-15.59
6.30	292.70 293.40 293.60	13.87 13.87 13.87	-1.07 -1.10 -1.11	-15.38 -15.59 -15.72	-15.56
7.05	293.10 293.40 292.70	13.87 13.87 13.87	-1.24 -1.21 -1.16	-17.13 -17.98 -17.66	-17.39
8.40	292.70 292.90 293.20 293.10	13.87 13.87 13.87 13.87	-1.25 -1.31 -1.38 -1.32	-18.00 -18.75 -19.60 -18.87	-18.54
9.20	292.90 293.20 292.90	13.87 13.87 13.87	-1.39 -1.56 -1.47	-19.95 -19.98 -19.76	-19.90
10.30	293.90 293.50 293.20	13.87 13.87 13.87	-1.32 -1.47 -1.31	-18.54 -20.78 -18.61	-19.31

11.20	296.70	13.87	-1.22	-17.27	-17.66
	296.40	13.87	-1.28	-17.75	
	294.30	13.87	-1.24	-17.96	
12.10	295.10	13.86	-1.48	-20.25	-22.01
	295.40	13.86	-1.46	-20.12	
	295.00	13.86	-1.70	-23.39	
	294.30	13.86	-1.74	-24.29	

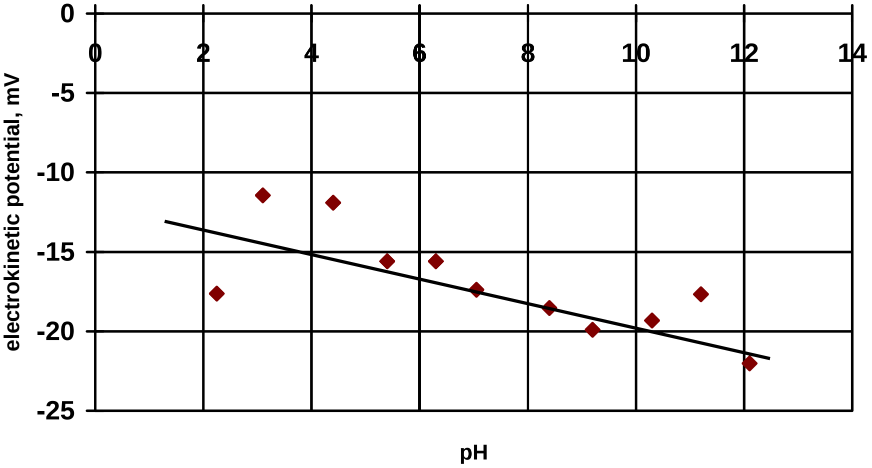
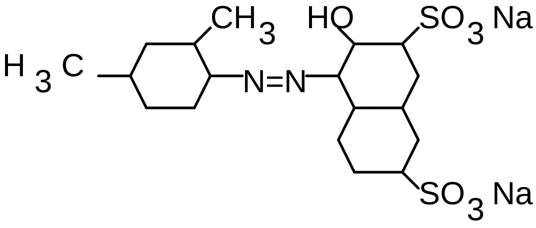


Fig. 2. The influence of the solution reaction on the O-3 activated carbon electrokinetic potential value

Rys. 2. Wpływ odczynu roztworu na wartość potencjału elektrokinetycznego węgla aktywnego O-3

The following dyes were tested: Acid Red FBL, Acid Scarlet D, Acid Marine Blue N, Acid Alizaryne Green G, Acid Ponceau 2RL, Acid Blue R. The mentioned dyes are used for colouring wool and they feature average light fastness and show the highest affinity to protein fibres in acid solutions [17,18].

Acid dyes have chromophore and auxochrome groups which feature either electronegative or electropositive character depending on pH of the solution. Having analysed chemical composition of the exemplary Acid Ponceau 2RL (1) one can see that the dye has two negative sulfo groups which dissociate in a wide pH range (both acid and alkaline reaction) and one hydroxyl group dissociating in alkaline reaction. The remaining groups ionise in acid medium (with excess of protons), showing electronegative character. Location of the negative groups on one side of the molecule and the positive ones on the other side, characterises that molecule as a dipole.



(1)

Acid Ponceau 2RL

3. Results and discussion

The tests have been carried out in two stages. Firstly, influence of selected factors on the process of adsorption has been tested and this was followed by testing effect of solution concentration and pH on electrophoretic mobility and ϕ potential of the sorbent. Reference curves indicating variations in transmittance depending on dye concentration in the solution have been prepared before commencement of the tests. The curves were necessary for determination of amount of dye remaining in the solution after a given process. It has been assumed that in the adsorption process no substances migrate from the adsorbent to the solution and the value of transmittance depends on the quantity of dye in the solution; this value varies due to dye adsorption on adsorbent surface.

The analysis of the results of sorption process of Acid Red FBL on active carbon O-3, as showed in Figures 3 and 4, indicates that in the first 50 seconds active carbon adsorbs a considerable amount of the dye and concentration thereof in the solution is close to the adsorption equilibrium condition. Such relationship has been obtained for all the tested initial concentrations of the dye, i.e. c=125; 25; 12.5; 2.5; 1.25 mg/dm^3. The pH of the solution has some significance here; the lower the pH, the less amount of the dye gets adsorbed. For example, if the initial concentration of the dye was c=25 mg/dm^3, the transmittance for wavelength κ=550 nm after 60 seconds of sorption time increased at pH=5.1 up to 84.8% and at pH=7.8 up to 23.7% (i.e. 61.1% less). Such significant difference in transmittance cannot arise from pH effect on colour of Acid Red FBL. The difference in transmittance observed is, therefore, the result of change of affinity of the dye to adsorbent surface.

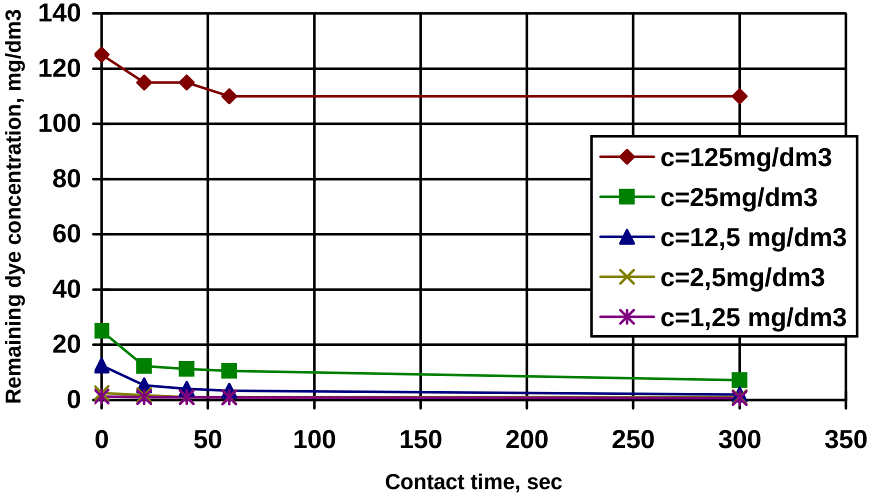


Fig. 3. Effect of adsorption time and initial concentration *c* of Acid Red FBL on amount of the dye remaining in the solution pH=6.5

Rys. 3. Wpływ czasu adsorpcji i początkowego stężenia Czerwieni Kwasowej FBL *c* na ilość pozostałego barwnika w roztworze pH=6,5

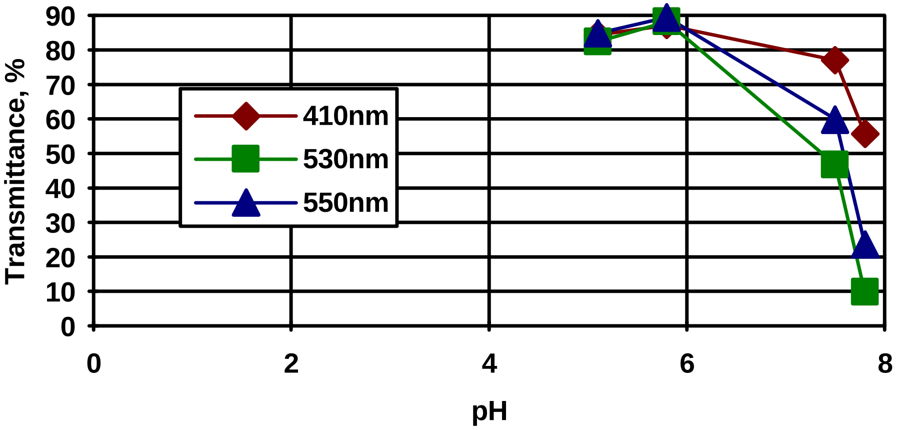


Fig. 4. Effect of Acid Red FBL pH, concentration 25 mg/dm^3 on dye sorption on active carbon O-3

Rys. 4. Wpływ pH Czerwieni Kwasowej FBL, stężenie 25 mg/dm^3 na sorpcję barwnika na węglu aktywnym O-3

Similar relationships have been obtained for other tested dyes. Adsorption of Acid Marine Blue GGR (see Figures 5 and 6) also proceeded quickly and after 50 seconds a condition close to the adsorption equilibrium was reached. The best adsorption results, for all wavelengths under studies (410, 530, 550, 610nm), were obtained for pH<7.1.

Adsorption of Acid Scarlet (see Figure 7 and 8) on active carbon was the most effective during approx. 50 seconds. The effect of pH on sorption is quite significant; the difference in transmittance for λ=550 nm with pH changing from 4.2 to 7.1 was almost 60%.

Comparable relationships were obtained for Acid Ponceau 2RL (see Figures 9 and 10). For all the tested initial concentrations of the dye (125; 25; 12.5; 2.5; 1.25 mg/dm^3) a condition close to absorption equilibrium has been reached just after 50 seconds. Once the adsorption process was being carried out in acid reaction (pH=4.2), transmittance for all the tested wavelength reaching approx. 90% was observed. As soon as pH of the solution was elevated up to 7.5, adsorbtion of the dye on active carbon clearly has decreased and for wavelength equal to 550 nm it was merely 46.6%. In the case of Acid Blue the effect of pH on transmittance was considerably lower (see Figure 12). Having changed pH from 4.1 up to 7 (λ=550 nm) transmittance has changed by 15%. The optimum period for sorption of Acid Blue was 50 seconds (see Figure 11). Similar results of adsorption for Acid Alizarin Green R on active carbon have been observed (see Figures 13 and 14).

Summing up, the tests performed showed that factors like contact time and initial concentration of the dye influence the process of acid dyes sorption on active carbon O-3. As contact time increases, so does the amount of dye on sorbent. Considerable amount of the dye adsorbs just after 50 seconds and the equilibrium amount of given dye is closely dependent on the initial concentration. The higher the initial concentration, the more of the dye gets adsorbed.

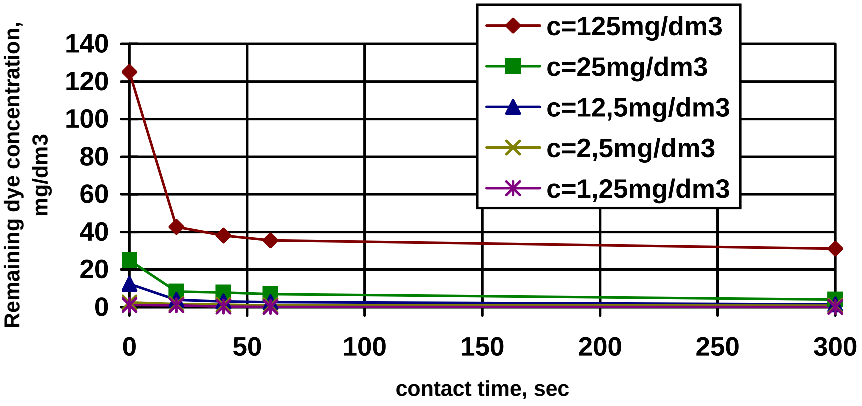


Fig. 5. Effect of adsorption time and initial concentration *c* of Acid Marine Blue GGR on amount of the dye remaining in the solution pH=6.5

Rys. 5. Wpływ czasu adsorpcji i początkowego stężenia Kwasowego Błękitu Morskiego GGR *c* na ilość pozostałego barwnika w roztworze pH=6,5

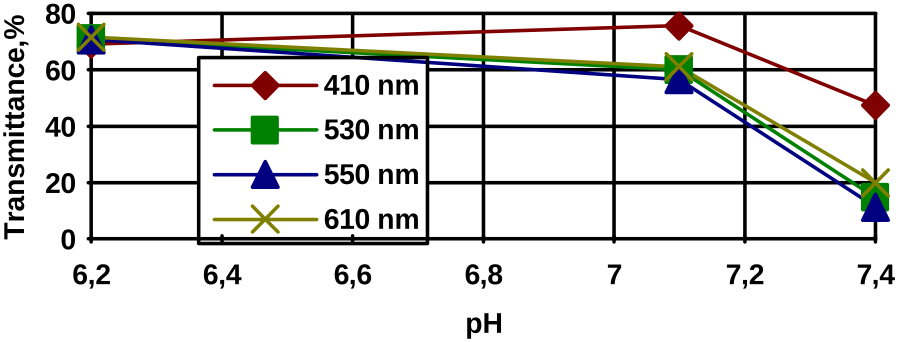


Fig. 6. Effect of Acid Marine Blue GGR pH of concentration 25 mg/dm^3 on dye sorption on active carbon O-3

Rys. 6. Wpływ pH Kwasowego Błękitu Morskiego GGR, stężenie 25 mg/dm^3 na sorpcję barwnika na węglu aktywnym O-3

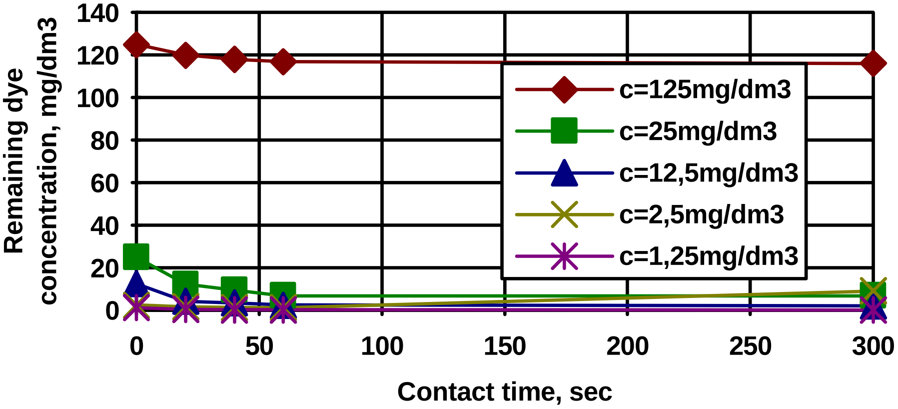


Fig. 7. Effect of adsorption time and initial concentration *c* of Acid Scarlet on amount of the dye remaining in the solution pH=6.5

Rys. 7. Wpływ czasu adsorpcji i początkowego stężenia Szkarłatu Kwasowego *c* na ilość pozostałego barwnika w roztworze pH=6,5

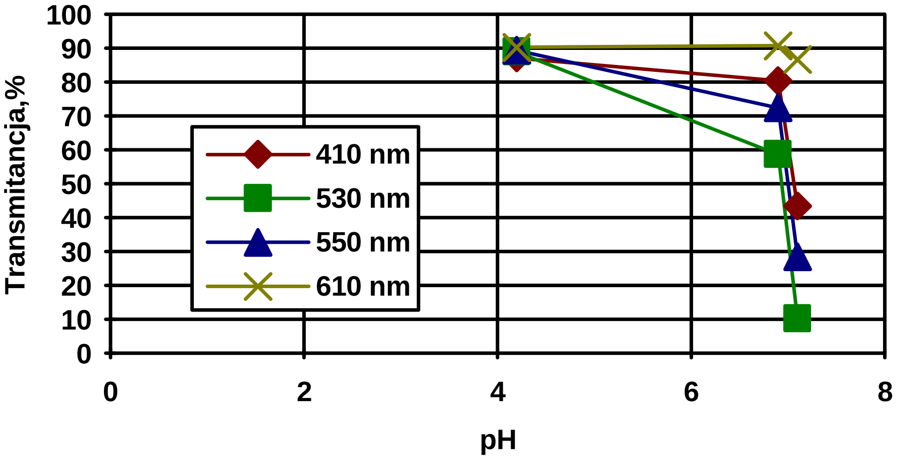


Fig. 8. Effect of Acid Scarlet pH of concentration 25 mg/dm^3 on dye sorption on active carbon O-3

Rys. 8. Wpływ pH Szkarłatu Kwasowego, stężenie 25 mg/dm^3 na sorpcję barwnika na węglu aktywnym O-3

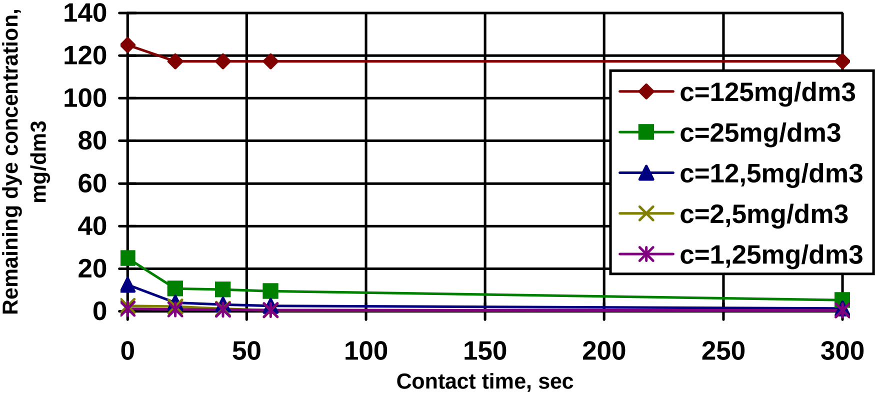


Fig. 9. Effect of adsorption time and initial concentration *c* of Acid Ponceau 2RL on amount of the dye remaining in the solution pH=6.5

Rys. 9. Wpływ czasu adsorpcji i początkowego stężenia Pąsu Kwasowego 2RL *c* na ilość pozostałego barwnika w roztworze pH=6,5

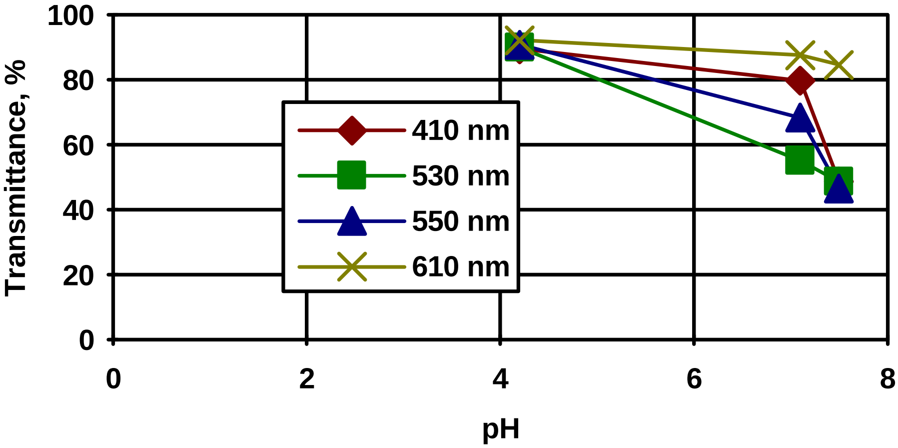


Fig. 10. Effect of Acid Ponceau 2RL pH of concentration 25 mg/dm^3 on dye sorption on active carbon O-3

Rys. 10. Wpływ pH Pąsu Kwasowego 2RL, stężenie 25 mg/dm^3 na sorpcję barwnika na węglu aktywnym O-3

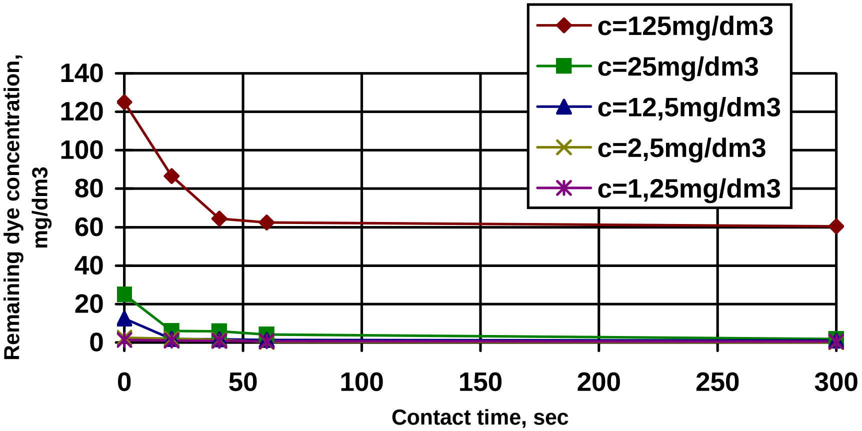


Fig. 11. Effect of adsorption time and initial concentration *c* of Acid Blue on amount of the dye remaining in the solution pH=6.5

Rys. 11. Wpływ czasu adsorpcji i początkowego stężenia Błękitu Kwasowego *c* na ilość pozostałego barwnika w roztworze pH=6,5

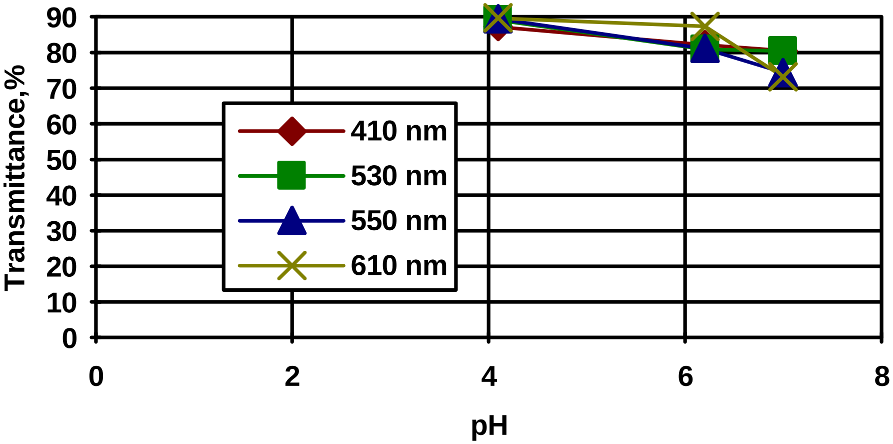


Fig. 12. Effect of Acid Blue pH of concentration 25 mg/dm^3 on dye sorption on active carbon O-3

Rys. 12. Wpływ pH Błękitu Kwasowego, stężenie 25 mg/dm^3 na sorpcję barwnika na węglu aktywnym O-3

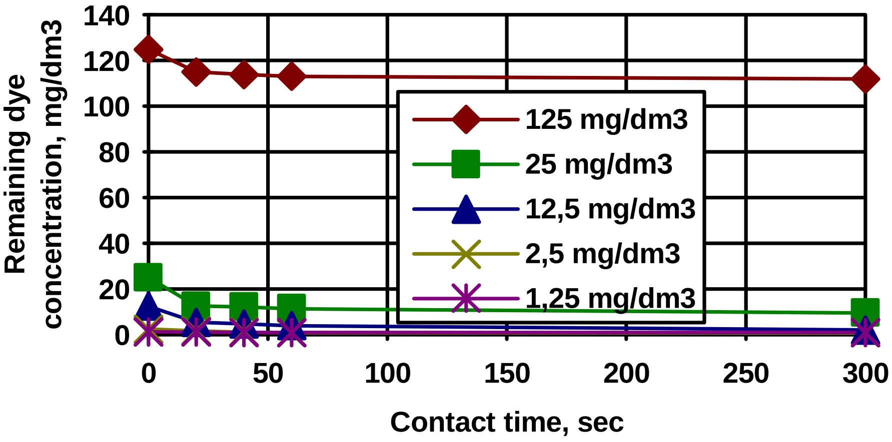


Fig. 13. Effect of adsorption time and initial concentration *c* of Acid Alizarin Green R on amount of the dye remaining in the solution pH=6.5

Rys. 13. Wpływ czasu adsorpcji i początkowego stężenia Kwasowej Zielni Alizarynowej *c* na ilość pozostałego barwnika w roztworze pH=6,5

The tests showed that acid dyes get sorbed best at pH<7. Acid Red FBL was least sorbed at pH=7, Acid Marine Blue at pH 7-8, Alizarin Green at pH=7.5 and Acid Ponceau 2RL at pH=7.45. Similar relationships have been obtained for Acid Blue R. This phenomenon is closely connected with the stereo-structure of acid dyes which are applied for fibre colouring in acid dying solution. The results of our tests confirm relationship between the dye structure and sorbent surface potential. Adsorption of dyes on active carbon can only be effected through positive functional groups which get activated in environment with excess of protons. For example, in the case of Acid Ponceau, which has a dipole type structure, adsorption occurs due to attachment of the positive functional groups to the negative-charged sorbent surface. On the other hand, the negative sulfo functional groups will be directed outside the sorbent-dye complex surface giving it an electronegative character. This was confirmed by tests of active carbon electokinetic potential following sorption of Acid Ponceau 2RL (see Table 2). The electrokinetic potential was negative and its absolute value was higher than that for the active carbon electrokinetic potential in distilled water, i.e. before dye sorption.

Table 2. Active carbon electrokinetic potential in presence of Acid Ponceau 2RL
Tabela 2. Potencjał elektrokinetyczny węgla aktywnego w obecności Pąsu Kwasowego 2RL

Concentration	pH	Temperature K	Current intensity	Electrophoretic mobility	ζ	ζ_{av}	ζ from equation (1)
125	4.2	290.7 291.7 291.9	13.86 13.86 13.86	-0.95 -0.93 -0.90	-14.31 -13.64 -13.09	-13.08	-15.17
125	7.05	293.2 292.6	13.87 13.86	-1.27 -1.12	-17.11 -17.24	-17.18	-17.53
25	7.45	292.2 292.6 292.3	13.96 13.87 13.87	-1.33 -1.28 -1.29	-19.34 -18.50 -18.65	-18.83	-17.86
25	7.50	292.1 292.6 292.6	13.86 13.86 13.87	-1.33 -1.28 -1.29	-19.41 -18.49 -18.52	-18.81	
125	7.20	293.0 293.1 293.0	13.87 13.86 13.87	-1.33 -1.30 -1.28	-19.01 -18.30 -18.52	-18.55	-17.66

4. Conclusions

The following conclusions can be drawn from tests of acid dyes sorption on active carbon O-3 and from the analysis of the results obtained:

- Active carbon electrokinetic potential is negative in wide scope of pH variations thus indicating that the surface potential of active carbon is also negative.
- The relationship between active carbon O-3 electrokinetic potential and pH is linear and can be presented in the form of an equation as follows: $\zeta = -0.83\ \mathrm{pH} - 11.68$ [mV].
- Acid dyes get adsorbed on the negative surface of active carbon.
- Acid dyes are best adsorbed on active carbon O-3 in acid reaction, i.e. in presence of excess of protons.
- Adsorption of acid dyes is strictly dependent on dyes chemical structures composition and adsorbent surface potential.

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Adsorpcja barwników kwasowych na węglu aktywnym

Streszczenie

Zbadano sorpcję na węglu aktywnym wybranej grupy barwników kwasowych. Mechanizm adsorpcji barwników na powierzchni węgla aktywnego wyjaśniono na podstawie składu chemicznego poszczególnych barwników oraz powierzchniowym potencjale węgla aktywnego.

Autor [7,14,15], po wykonaniu rozległych badań, zauważył, że istniej korelacja pomiędzy koprecypitacją barwników i procesem adsorpcji a ich strukturą chemiczną i potancjałem powierzchniowym adsorbentu. Pokazano, że barwniki są strącane przez koagulanty poprzez tworzenie hydroxo-kompleksów barwników, które mogą tworzyć prawdziwy roztwór lub sedymentować

w postaci osadu łatwo opadającego. Dodatek wodorotlenku wapnia, w drugim etapie, do roztworu zwiększa pH a co za tym idzie zwiększa strącalność osadu (np. wodorotlenku żelaza) na którym może zachodzić adsorpcja lub koprecypitacja barwników (lub ich kompleksów). Autor przebadał znaczną grupę losowo wybranych barwników [17,14,15]. Jednakże, ogromna ilość barwników produkowanych na całym świecie powoduje konieczność prowadzenia dalszych badań. Szczególnie wydaje się wskazanym sprawdzenie założenia, że barwniki kwasowe mogą być łatwo sorbowane na elektroujemnej powierzchni węgla aktywnego z powodu ich składu chemicznego i tendencji do barwienia włókien w odczynie kwasowym.

Proces przebadano statystycznie przyjmując założone stężenia barwników, pH i zmienny czas kontaktu sorbatu z adsorbentem (0÷300 sekund). Stężenie barwników zmieniano od 250 do 1,25 mg/dm^3. Roztwór zawierający barwnik wstrząsano mechanicznie przy prędkości około 60 1/min. Po określonym czasie adsorpcji próbkę filtrowano przez filtr o średniej twardości i w ten sposób otrzymany filtrat został poddany analizie.

Czynniki wynikowe były następujące: transmitancja (procentowa wartość transmisji światłą) przy długości fali λ=410, 530 i 610nm oraz stężenie barwnika pozostałego w roztworze, mg/dm^3.

Ilość barwnika pozostałego po procesie sorpcji oznaczano dla poszczególnych barwników z krzywych wzorcowych pokazujących zmienność transmitancji w funkcji stężenia barwnika.

Potencjał elektrokinetyczny jest wskaźnikiem określającym pośrednio potencjał powierzchniowy adsorbentu. w związku z tym zmierzono jego wartość w zależności od pH, typu barwnika i stężenia. Badania te wykonano na zetametrze.

Z badań nad sorpcją barwników kwasowych na węglu aktywnym O-3 oraz analizy otrzymanych wyników można wyciągnąć następujące wnioski:

- Potencjał elektrokinetyczny węgla aktywnego jest ujemny w szerokim zakresie zmian pH co wskazuje, że potencjał powierzchniowy węgla aktywnego jest również ujemny.
- Zależność pomiędzy potencjałem elektrokinetycznym węgla aktywnego O-3 i pH jest linowa i może być przedstawione w formie następującego równania: ζ= -0,83 pH - 11,68 [mV].
- Barwniki kwasowe adsorbują się na ujemnej powierzchni węgla aktywnego.
- Barwniki kwasowe są najlepiej adsorbowane na węglu aktywnym O-3 w odczynie kwaśnym, tj. w obecności nadmiaru protonów.
- Adsorpcja barwników kwasowych ściśle zależy od składu chemicznej struktury barwników i potencjału powierzchniowego adsorbentu.