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## THE INFLUENCE OF THE ADSORBENTS' ELECTROKINETIC POTENTIAL ON THE ADSORPTION PROCESS OF HUMIC SUBSTANCES

The paper presents the analysis of the structure of humic substances and their behaviour in water environment. Adsorption process of humic substances on three following adsorbents was tested: natural bentonite, activated bentonite and activated carbon Norit CN 1. There was found the dependence of electrokinetic potential on adsorption ability of adsorbent particles. The presence of multivalent cations in water is an important factor influencing the course and efficiency of sorption process.

### 1. INTRODUCTION

Humic substances are macromolecules, commonly present in water environment. They are formed during humification of plant and animal materials such as lignin, proteins, pectins, polysaccharides, tannins [1]. These materials are in different states of decomposition and constitute the mixture of high-molecular polymers which exhibit the properties of organic acids, and their structure is unknown.

The solubility of humic substances in diluted acids and bases is the basis on which they can be classified as follows:

- humic acids soluble in base solution and precipitated in solution of hydrochloric acid,

- fulvic acids soluble in water, acid solutions (pH~1) and basic solutions,
- humins that are insoluble neither in acid solutions nor in basic solutions.

According to Kononowa (KOWAL, ŚWIDERSKA-BRÓŻ [2]) humic substances may be divided into:

- humic and ulmin acids that are soluble in bases,
- kreon and apokreon acids (fulvic acids) that are soluble in water,
- hymatomelanic acids that are soluble in alcohol,
- insoluble humins and ulmins.

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Humic substances are important in the process of water treatment because of their health-safety and aesthetic values as well as the problems, which they cause in particular single processes of water treatment (among other things they adsorb themselves on granular activated carbon decreasing its absorbing capacity). On account of humic substances adsorption properties, particularly adsorption of hydrophobic organic compounds such as pesticides, phthalates and polychlorinated biphenyls (PCBs), their removal from treated water is necessary. Humic substances are also considered to be responsible for forming toxic trihalomethanes (THMs) and other chloro-organic substances during water chlorination.

Humic substances show a great ability to form complexes with heavy metals. Solution including  $100 \text{ mg/dm}^3$  of fulvic acids may cumulate  $8.4 \text{ mg Fe}^{3+}$  and  $4.0 \text{ mg Al}^{3+}$  in  $1 \text{ dm}^3$  [1]. ŚWIDERSKA-BRÓŹ [3], [4], who examined the interactions of lead, copper, nickel and zinc with humic acids, found that their affinity decreased in the following descending order:  $\text{Pb}^{2+} > \text{Cu}^{2+} > \text{Zn}^{2+} > \text{Ni}^{2+}$ . At the same time she showed that the presence of humic acids improved removal of zinc and copper from the water (the complexes were formed and then removed at low pH). However, ions of zinc and nickel were mainly removed as sparingly soluble inorganic compounds.

The authors proved [5] that humic acids were easier to remove from water of increased hardness.

However, we are unable to remove efficiently humic substances from water if their structure and properties are not known as they and the character of adsorbent surface determine the effectiveness of adsorption process.

That is why the goal of the work is to analyse the structure of humic substances and their behaviour in water environment and to define correlation between process of humic adsorption and electrokinetic potential of adsorbent particles.

## 2. STRUCTURE AND ADSORPTION OF HUMIC SUBSTANCES

Many authors have investigated the configuration and chemical structure of humic substances. However, neither their configuration nor their properties have been univocally determined so far.

The following functional groups have been found in humic substances [1]: carboxylic, phenolic, alcoholic, methoxyl, carbonyl, etheric and estrone. Carboxylic and phenolic groups dominate in fulvic acids, which according to Oden (DOJLIDO [1]) make 87.2% of humic substances. Their molecular weights fluctuate – according to Orłow (DOJLIDO [1]) from 700 to 800,000, and according to Steelink (KOWAL, ŚWIDERSKA-BRÓŹ [2]) from 2,000 (for readily soluble forms) up to 50,000 (for slightly soluble forms). Other authors report the following ranges: 500–250,000 [6] and 1,000–1,000,000 [7].

The sizes of particles in humic acids according to Beufelspacher and Flaig (KOWAL, ŚWIDERSKA-BRÓŹ [2]) depend on pH of environment and most often they equal to 8–10 nm.

Most researchers express the opinion that humic substances are long-chain molecules with aliphatic and aromatic segments with different kinds of chain branching. Polar functional groups in structures form H-bonds although cross-linking is also possible. The most possible configuration of humic substance molecules, especially of humic acids, are loose coils, which in water solutions form roughly spheroidal units whose mass densities are higher in their central parts. Polar groups are oriented to the outside, and hydrophobic – to the inside of the coils. Such a structure is supported by the fact that in dry humic substances the polar groups are oriented possibly maximally to the inside and the humic substances show a non-polar character [8].

Similar opinion about configuration of humic substances may be found in the publication by SEIN et al. [9], who, on the basis of molecular structure of humic acids proposed by STEELINK and TNB (Temple-Northeastern–Birmingham) (SEIN et al. [9]), have constructed their own model with chiral centres and the lowest possible energy of geometry (figure 1).

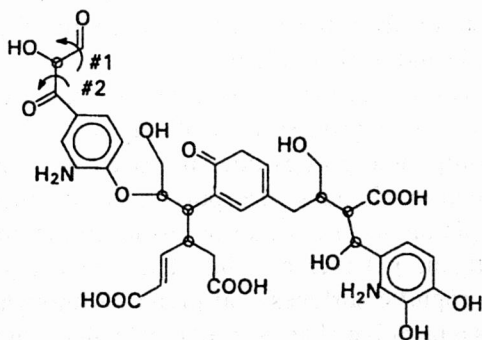


Fig. 1. Molecular structure of humic acid monomers with chiral centres [9]

Different opinion about the structure of soluble humic acids is presented by CONTE and PICCOLO [10]. They think that humic acids are rather small subunits loosely connected with one another, mainly attached to one another by weak hydrophobic forces. The sizes of particles are only apparently large, because it has been discovered that acids are reversibly disintegrated as a result of sudden change of pH of the medium from acidic to alkaline. The stability of such loosely bound structures is connected with their entropy. Removal of water particles from humic substances decreases a total particle energy. According to the authors of this paper, this hypothesis is not compatible with those results which confirm the occurrence of functional groups in humic acids. The groups, depending on pH and solution content, make acid particles electronegative or electropositive. However, adsorption due to hydrophobic forces, i.e. inter-particle forces, may occur also in the case of apolar elements. Those dependencies are particularly important for small particles about which Conte and Piccolo write.

Although adsorptive properties of humic acids are generally known, an available literature provides only sparse information about adsorption kinetics and desorption of humic acids.

PINHEIRO and others [11], who used alternating current voltammetry for investigation of humic acids' adsorption on the interface of Hg-H<sub>2</sub>O, showed that adsorption kinetics was influenced not only by transport, but also by surface reactions such as electrostatic interactions.

However, AVENA and KOOPAL [12] using refractometer to measure adsorption on the surface of ferric oxide proved that the rate of the process rapidly decreased after first 100 seconds. In the solution with salt concentration of 0.1 M, humic acids' concentration of 50 mg/dm<sup>3</sup> and at pH 3.25, about 1.5 mg/m<sup>2</sup> of substances were absorbed for the first 100 s. Afterwards, the process run much slower for many hours.

AVENA and KOOPAL [11] gave particular attention to the first, fast stage of humic acids' adsorption. They proved that in adsorption process proceeding on hydrophilic surfaces (ferric oxide and aluminium) and on hydrophobic surfaces (polystyrene and SiO<sub>2</sub> gel) such factors as pH, humic acids concentration and solution ionic strength were important. It was discovered that initially kinetics of adsorption was rather a complicated process, which depended on surface properties and solution conditions. The structure of humic acids is such that many carboxylic and phenolic groups protrude from the molecules, therefore the attachment of humic acids to aluminium oxide and ferric oxide is fast.

On a hydrophobic surface, where adsorption takes place due to van der Waals' forces, that process is relatively slow. This is caused by the fact that majority of hydrophobic groups does not occur on the edge of the particle and only after some structural changes adsorption may take place. In such a case, also a noticeable effect of pH on an initial rate of adsorption is observed. The rate of the process also drops with the pH increase, but that decrease starts at pH approaching 4 (in the case of hydrophilic surfaces – at pH>5). Although it is possible that the surface of SiO<sub>2</sub> gel has a negative charge at high pH values, the similarity in the behaviour of humic acids on both hydrophobic surfaces suggests that electrostatics is not essential in this case. This fulfills the expectations that the remaining -OH groups on the silicon dioxide surface are resistant to deprotonation [11].

The increase in pH is associated with the the increase in a negative charge of humic acids. Highly charged hydrophilic groups occurring outside of the particle shield hydrophobic groups, thus they make impossible their connecting to the hydrophobic surface. A substantial decrease of adsorption rate is observed at pH>7. At such a value of pH further increase of charge of humic acids particle does not accelerate the attachment process. At salts' concentration higher than 0.2 mole/dm<sup>3</sup> the aggregation of particles of humic acids is important and if in such a case an electrostatic repulsion between adsorbent surface and adsorbate does not occur, the adsorption process, independently of the type of the surface [11], is limited by diffusion.

Similar conclusions were drawn by MAZET and SERPAUD [13] who investigated the adsorption process of humic acids on the surface of [Al(OH)<sub>x</sub>]<sup>n</sup> flocs formed earlier in coagulation of kaolinite by aluminium sulphate. They also distinguished two adsorption stages: the first – fast, lasting up to 20 minutes, after which humic

substances of the concentrations of 15, 10 and 5 mg/dm<sup>3</sup> were removed in 48%, 52% and 80%, respectively. In this stage, the interaction between positive charge of the flocs and negative charge of polyanionic humic substances takes place. The second stage is much slower and may be referred to the exchange of OH<sup>-</sup> ions between flocs and anions' sites of humic substances.

Because of their structure, i.e. presence of various hydrophobic and hydrophilic groups, humic substances may influence other substances in water environment. In the paper [14], a quantitative model has been described. It enables anticipation of the influence of dissolved humic substances on adsorption of other organic substances, sometimes harmful to human health, e.g. pesticides, phthalates, PCBs. These compounds are adsorbed on solid phase. Adsorption of fluoranthene, an example of soluble organic substances, on clay surface in the presence or absence of humic substances was investigated. It appears that soluble humic substances release organic solvent, whose adsorption coefficient is higher than theirs, but they decrease the adsorption of solvent whose adsorption coefficient is lower than theirs.

The above investigations testify to a great interest of researchers in humic substances. If we know real structure of humic substances and their interaction with other compounds present in natural water environment and with anthropogenic contaminants introduced in larger and larger quantities to natural water bodies, we will be able to understand the mechanisms of their influence on environment, to anticipate their possible interactions, hence to increase effectiveness and economy of their removal in water treatment processes.

### 3. METHODS

Review of literature shows that humic substances may be labile in water environment, which is strictly connected with their structure and physicochemical composition of water environment. In order to define the correlation between the adsorption process of humic substances and their structure, we decided to examine their adsorption on three different adsorbents of different electrokinetic potentials. They can be itemized as follows:

1. *Natural bentonite*. Montmorillonites, silty minerals, are its basic elements. Montmorillonites are characterized by excess of negative charge, which is balanced with inter-packet cations. However, part of those cations may be found on the edges of those packets where they are connected with Si-OH groups whose protons are capable of ion exchanging. Properties of bentonites depend on inter-packet cations [15].

2. *Activated bentonite*. It is obtained in laboratory by activating natural bentonite by means of hydrochloric acid. This activation is based on the exchange of multivalent inter-packet cations for protons.

3. *Powdered hydrophilic activated carbon Norit CN 1*. It is produced by activating powdered carbon by phosphoric acid and then used for adsorption of organic compounds of high-molecular weight and large molecules of colouring compounds.

The sequence of investigation was as follows:

• *Extraction of humic acids.* Humic acids for investigation were extracted from river sediments. Detailed procedure of their extraction is presented in figure 2, which is prepared on the basis of information presented in articles [16], [17].

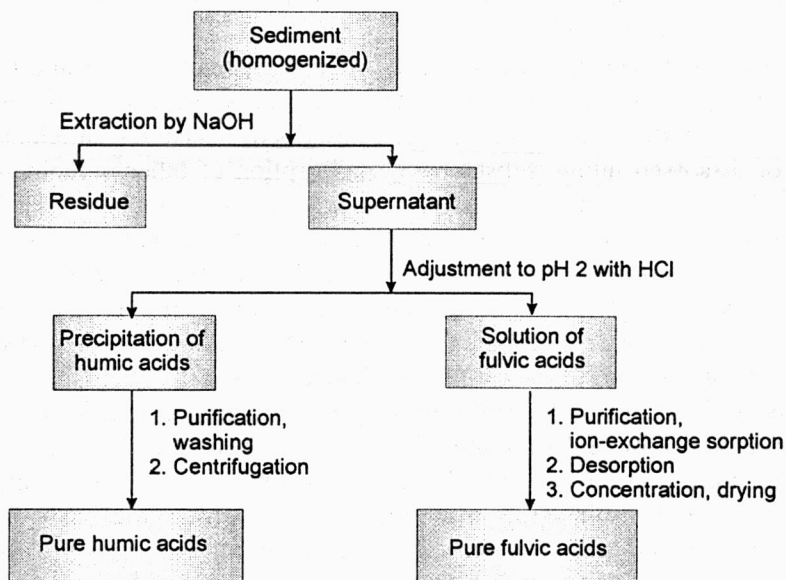


Fig. 2. Diagram of humic substances extraction from sediments

• *Activation of natural bentonite.* A portion of natural bentonite was poured twice with the solution of HCl (1+1) and heated for two hours up to 100 °C. Then bentonite was rinsed with distilled water until neutral reaction was obtained and dried at a temperature of 105 °C to a constant weight.

• *Measurement of electrokinetic potential.* Electrokinetic potential of the particles of adsorbents being tested was determined using the parabola method. Measurements were carried out for 9 depths of measurement cell, using Zetametr EKB 1.1 made by EKOBUDEX according to the method described in [18].

The sample examined, whose concentration reached 0.5 g/dm<sup>3</sup>, was placed in quartz measurement cell equipped with electrodes. After applying a voltage to the electrodes, the movement of particles was observed. The velocity of this movement was the sum of electrophoretic velocity and electroosmotic velocity of fluid. Because of variable movement directions of solvent in crosswise axis of the cell, the distribution of particles' velocity had a parabolic shape.

In order to calculate the value of electrokinetic potential, the integration method was applied. This is the most detailed method of microelectrophoretic measurements. It allows – on different successive levels – the measurement of time that is necessary for a particle to cover a definite distance. Thereafter an average time, velocity and electrophoretic mobility  $R_{ef}$  are calculated. Next, using Simpson's equation, theoretical values of electrophoretic mobility are calculated. Also the differences

between  $R_{ef}$  determined for a given level and theoretical  $R_{ef}$  are calculated and given as relative error. Average electrophoretic mobility is converted into potential  $\zeta$  according to the following dependence:

$$\zeta = 113097.34 \frac{\eta}{D} R_{ef},$$

where:  $\eta$  – fluid viscosity [P],  $D$  – dielectric constant.

• *Kinetics of adsorption process of humic acids on chosen adsorbents.* Adsorption of humic acids on natural bentonite, activated bentonite and hydrophilic activated carbon Norit CN 1 was investigated. Those adsorbents in the form of aqueous suspension were added to the sample. Humic acid solution was prepared on the basis of distilled water or tap water. Tap water on account of the content of  $\text{Ca}^{2+}$  and  $\text{CO}_3^{2-}$  ions showed buffering activity and maintained constant pH about 7.5 of solution. Tap water was characterized by total hardness of  $7.0 \text{ mval/dm}^3$ , alkalinity of  $4.0 \text{ mval/dm}^3$ , oxygen consumption of  $1.6 \text{ mg/dm}^3 \text{ O}_2$ . Contents of iron and chloride amounted to  $0.1 \text{ mg/dm}^3$  and  $35.8 \text{ mg/dm}^3$ , respectively. Electrolytic conductivity of water was  $\kappa = 692 \text{ } \mu\text{S/cm}$ .

In order to prepare a humic acid solution, fulvic and humic acids were mixed in proportions: 60% of fulvic acid and 40% of humic acids. This mixture was prepared 24 hours before investigation.

Kinetics of adsorption process was tested in conical flasks filled with humic acids' solution added in the concentration of  $40 \text{ mg/dm}^3$  and in the amount of  $200 \text{ mg/g}$  of adsorbent. Then adsorbent was added. Control sample without adsorbent was also prepared. Then, the flasks were tightly closed and samples mixed for one an hour. After some specified time a part of well mixed sample was taken, centrifuged at 3000 rpm for 15 minutes to get rid of the adsorbents, and then the colour of clear solution, which was an indirect determinant of the quantity of humic acids residing in solution, was determined. The investigation was carried out until equilibrium was achieved, i.e. for 9 days.

#### 4. DESCRIPTION AND ANALYSIS OF RESULTS

Figure 3 and table 1 present the results of research of electrokinetic potential of the adsorbents' particles tested. Analysis of the results obtained show that adsorbents under specified constant conditions (pH 7.5, adsorbent concentration of  $0.2 \text{ g/dm}^3$ ) are characterized by different negative values of electrokinetic potential:

- natural bentonite –  $\zeta = -37 \text{ mV}$ ,
- activated bentonite –  $\zeta = -56 \text{ mV}$
- activated carbon Norit CN 1 –  $\zeta = -59 \text{ mV}$ .

Activated bentonite has larger negative electrokinetic potential than natural bentonite due to ion exchange during chemical treatment of bentonite with hydrochloric acid. In this

process, multivalent cations are eluted from negative active centres and replaced by protons. As a result, a smaller compensation of negative charge of active centres occurs.

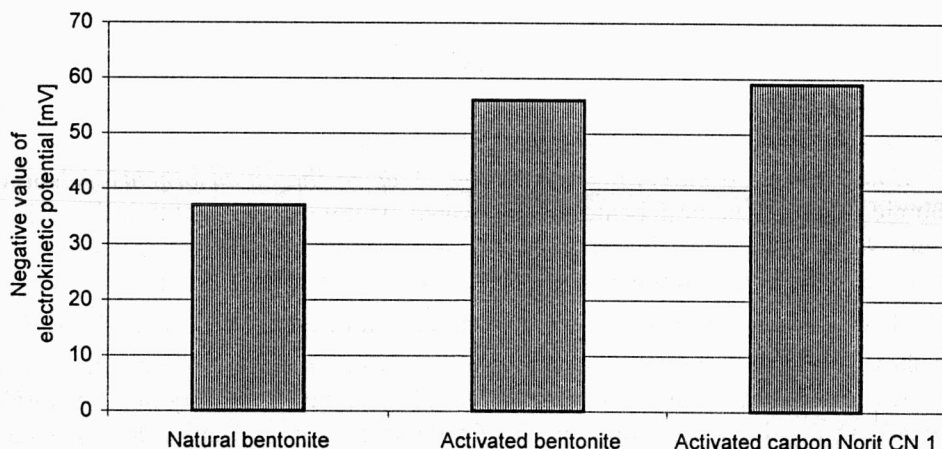


Fig. 3. Electrokinetic potential of the adsorbents tested in the solution of pH 7.5

Table 1

Electrokinetic potential of the adsorbents tested at pH 7.5

Kind of adsorbent	$\zeta$ [mV]	$R_{ef}$ [ $\mu\text{m}\cdot\text{cm}/\text{V}\cdot\text{s}$ ]	$E$ [V/cm]	$T$ [K]	Standard deviation
Natural bentonite	-38.67	-2.48	16.48	288.8	5.20
Activated bentonite	-55.47	-3.39	16.47	286.6	4.38
Activated carbon Norit CN 1	-58.98	-3.75	16.46	288.4	3.74

On account of negative electrokinetic potential all three adsorbents at pH 7.5 should show affinity to electropositive particles and ions. The structure of humic acids' monomers [9] shows that they contain mainly carboxyl and phenol groups and only a little amount of amine groups. Carboxylic groups dissociate to active carboxylic ions at pH as low as 4.6–4.9. Phenolic groups dissociate in alkaline solutions and also develop electronegative character. However, amine groups at low pH exhibit electropositive character. Due to the presence of aliphatic and aromatic fragments humic acids are of local apolar character. That is why it may be assumed that those acids are heteropolar compounds. Depending on the solution pH and anions connected to the polar groups (multi- or single-valued), humic acids may be adsorbed on electronegative, electropositive and apolar surfaces.

Such a character of humic acids enables their mutual connecting due to electrostatic forces which with the change of pH rapidly decrease or increase, hence agglomeration of dispersed particles or dispersion of aggregates formed due to



electrostatic forces may take place. Such a property of humic acids causes their big lability and increases their adsorption capacity. That is why humic acids are carriers of heavy metals, pesticides, THMs and other substances of different chemical structures.

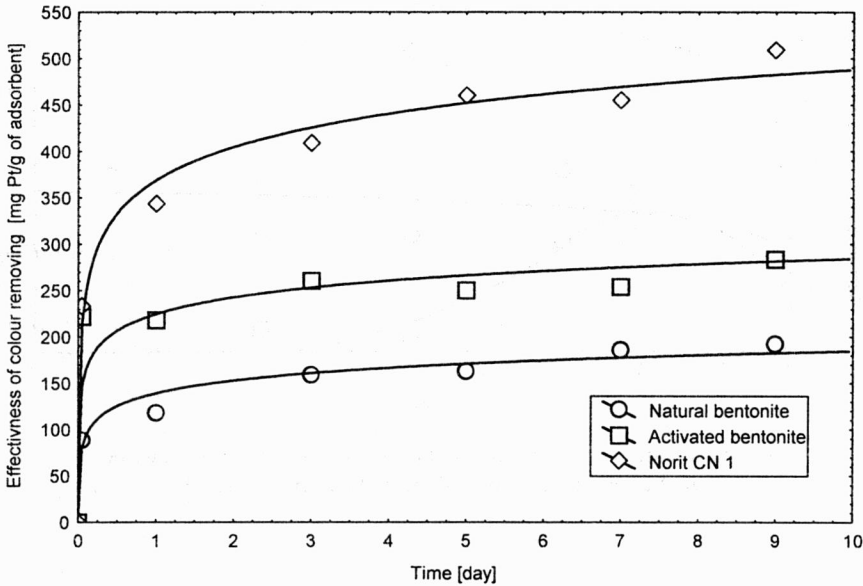


Fig. 4. Kinetics of adsorption of humic acids diluted in tap water in the amount of 200 mg/g of adsorbent, pH 7.5

Investigation of adsorption of humic substances on 3 adsorbents (figure 4) show the dependence of that process on the value of electrokinetic potential of adsorbent particles. The greater the negative value of electrokinetic potential of particles, the more humic acids are adsorbed on them. However, it is interesting that electrokinetic potential of adsorbent particles is negative. So, the effect of electrostatic repulsion by negative carboxylic ions should take place. The results obtained show that adsorption of particles of humic acids on electronegative surface of adsorbent occurs. Two reasons may be responsible for the dependence obtained. First, humic acids have positive functional groups, which are in ionised form at pH 7.5, which is not very probable because amino groups are ionised at low pH. Second, tap water contains cations, which are easily adsorbed on the interface of adsorbent-adsorbate changing electronegative character of the adsorbent surface or adsorbate into electropositive one. Anions and electronegative particles are easily adsorbed on such a surface. This dependence is confirmed by other researchers, e.g. [15], showing an increase of humic acids precipitation together with the increase of water hardness, hence the increase of concentration of cations, mainly  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ .

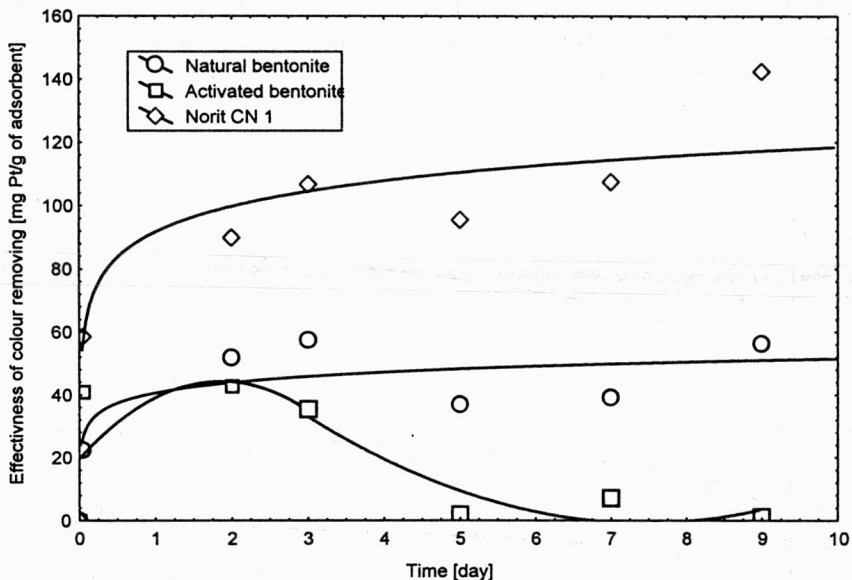


Fig. 5. Kinetics of adsorption of humic acids diluted in distilled water in the concentration of 200 mg/g of adsorbent, pH 7.3

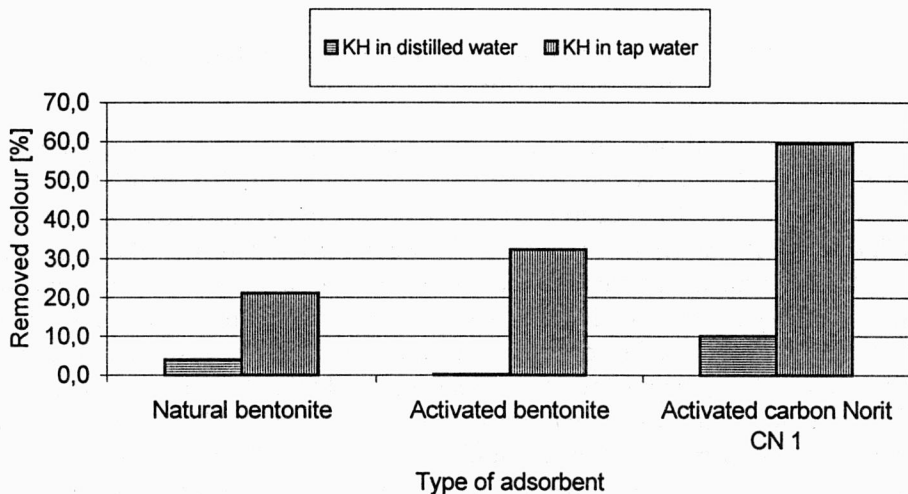


Fig. 6. Adsorption of humic acids on some selected adsorbents after five-day contact time. Concentration of humic acids diluted in distilled water and tap water was 200 mg/g of adsorbent, pH 7.3

In order to check this dependence, adsorption of humic acids dissolved in distilled water of electrolytic conductivity  $\kappa = 2.50 \mu\text{S}/\text{cm}$  was examined (figure 5). On the basis of the results obtained, the relationships presented in figure 6 were established

and compared with the results obtained for tap water (table 2). Analysis of the results confirms the hypothesis that humic acids are adsorbed on electronegative adsorbent particles, and in this way removed from water. The effectiveness of the process in the presence of small quantity of multivalent cations (distilled water) was as low as 0–10%, while in the tap water it amounted to 20–60%. This proves that multivalent cations make adsorption of humic acids on electronegative surface of adsorbent easier. So, the conclusion should be drawn that effectiveness of adsorption may be increased by applying sorbent as the load in the coagulation process with aluminium and iron salts.

Table 2

Adsorption of humic acids on chosen adsorbents after five-day contact time. Concentration of humic acids diluted in distilled water and tap water in the amount of 200 mg/g of adsorbent, pH 7.3

Adsorbent	HA diluted in distilled water			HA diluted in tap water		
	Colour [mg Pt/dm <sup>3</sup> ]	Colour "0" [mg Pt/dm <sup>3</sup> ]	Removed colour [%]	Colour [mg Pt/dm <sup>3</sup> ]	Colour "0" [mg Pt/dm <sup>3</sup> ]	Removed colour [%]
Natural bentonite	180.2	187.6	3.9	121.7	154.4	21.2
Activated bentonite	187.2	187.6	0.2	104.5	154.4	32.3
Activated carbon Norit CN 1	168.5	187.6	10.2	62.3	154.4	59.7

The results obtained confirm at the same time our opinion about the hypothesis of CONTE and PICCOLO [10] proposed to explain formation of aggregates of particles of humic acids. On the basis of up-to-date investigation the conclusion may be drawn that agglomeration of particles of humic acids takes place mainly as a result of electrostatic interactions of functional groups of different charges or as a result of adsorption of multivalent cations on negative functional groups.

## 5. CONCLUSIONS

On the basis of the investigation carried out the following conclusions may be drawn:

- In water solution of pH 7.5, the adsorbents tested have negative electrokinetic potential.
- The value of electrokinetic potential depends on adsorption ability of adsorbent particles.
- Adsorption of humic acids on electronegative surface of adsorbent particles takes place by electropositive amine groups of humic acids or as a result of adsorption of multivalent cations on the adsorbent–adsorbate interface.

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WPŁYW POTENCJAŁU ELEKTROKINETYCZNEGO ADSORBENTU  
NA PROCES ADSORPCJI KWASÓW HUMUSOWYCH

Zanalizowano strukturę substancji humusowych i ich zachowanie w środowisku wodnym. Zbadano proces adsorpcji substancji humusowych na trzech adsorbentach: bentonicie naturalnym, bentonicie uaktywnionym i węgla aktywnym Norit CN 1. Stwierdzono, że istnieje zależność między wartością potencjału elektrokinetycznego a zdolnością adsorpcyjną cząstek adsorbentu. Istotnym czynnikiem wpływającym na przebieg i skuteczność procesu sorpcji była obecność w wodzie wielowartościowych kationów.