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Procedia Engineering 117 (2015) 264 – 272

**Procedia  
Engineering**[www.elsevier.com/locate/procedia](http://www.elsevier.com/locate/procedia)

International Scientific Conference Urban Civil Engineering and Municipal Facilities,  
SPbUCEMF-2015

## Electrokinetic and Electroconductivity Properties of Filtering Material Aqualat

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### Abstract

For Aqualat filtering material, which is supposed to be used for electroadsorption post-treatment of waters, three methods of zeta-potential determining were applied: streaming potential method, microelectrophoresis and method implemented by Zetatrac analyzer. Besides, the conductive properties of the material were determined in a wide range of electrolyte concentrations.

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Peer-review under responsibility of the organizing committee of SPbUCEMF-2015

*Keywords:* Anthracite; elektroadsorption; wastewater; electrokinetic potential; streaming potential; electroconductivity; microelectrophoresis; Zetatrac

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### 1. Introduction

Multifactor anthropogenic load on the environment in Russia is still rather high. The rapid and large-scale water, air and soil pollution continues. In spite of the wide range of methods for water and air purification, new methods are being developed and new materials and designs are used.

Filtration and dynamic adsorption methods on one hand are classical methods, but on the other hand, they could be updated by using new natural and synthetic carriers and sorbents and by their use in a new capacity (e.g., bases for catalysts) and / or in combination with other impact techniques, such an electric or magnetic fields [1–5]. So in

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recent years electroadsorption method [6-23] or capacitive deionization (CDI) have become increasingly used (CDI is an electrosorption process that removes inorganic ions by charge separation and acts as a "flow through" capacitor) [24-32].

The electrosorption method is based on studies of A.N. Frumkin et al. carried out in the middle of XX century [33].

Most papers related to the electrosorption method or CDI dedicated to the removal of ions. However carbon aerogel (CA) electrodes were used in CDI not only for metal ions, but for bacteria, and some organic pollutants removal [22].

Typical requirements for electrodes in CDI-processes are:

- large surface area
- low electrical resistance
- good polarizability

Such requirements are satisfied, for example, by carbon aerogel [22,34], which has a high porosity, large surface area (400–1000 m<sup>2</sup>/g); controlled pore size (less than 2 nm to remove inorganic cations such as Na<sup>+</sup>, K<sup>+</sup>, and 2–50 nm for the removal of larger inorganic and organic ions) and low resistance.

In Russia, activated carbons are mainly used for electroadsorption removal of ionic impurities from natural and waste waters. For example, activated carbon BAU exposed to an electric field was applied for desalination [6]. Current leads were from platinum metal (stainless steel, titanium). Backfill of the adsorbent was located in the interelectrode space separated by a dielectric membrane. Purified water was fed into the interelectrode space. Under the influence of an electric field cations were attracted to the surface of one volume electrode and anions – to the surface of another. In addition to inorganic ions removal there also took place water purification from organic impurities, collapsing in an electric field. Regeneration of the coal backfill was performed by switching the polarity of the electrodes.

Agaveva et al. [12, 17] used sulfonated coal, coals KM and OKM-2 to extract lithium ion from the thermal waters of Dagestan. The influence of current density, cathodic polarization and lithium cations concentration in solution on the amount of adsorption on sulfonated coal and coal OKM-2 were investigated. It was shown that at anodic polarization of the coals lithium ions were completely desorbed.

Tsvetnov et al. [9, 18, 19] carried out a comparative study of charging of electrical double layer (EDL) of activated carbon fibers Aktilen-A and B-Aktilen, granular carbon "Sibunit" P-709 and C-1 coal. The electrochemical properties of carbon materials were studied by potentiodynamic pulses method at small potential deviations from the stationary value ( $\pm 50$  mV), when it occurs mainly the charging of the electric double layer (EDL) as well as in considerable overvoltage accompanied by Faraday process of water molecules discharge (polarization curves).

Usually the electroadsorption study begins from EDL state investigation, definitions of electrokinetic potential (Zeta-potential) of electrode-solution interface in assay solutions [21].

Despite the fact that the activated carbons generally comply with the requirements for electroadsorption materials (large surface area, low electrical resistance, good polarizability) their mechanical strength is lower than that of the feedstock, which has been activated.

It is known that the mechanical strength of coal depends on the chemical composition and moisture content and increases from brown coals to hard coal and anthracites [35] accompanied by changing the degree of coalification (metamorphism). The presence of surface functional groups determines not only the hardness of coal, but also other physicochemical properties of coal, such as electrical conductivity, strength of supramolecular structure packaging [36].

Anthracite is characterized by high degree of metamorphism and low content of mineral component. It is characteristically for anthracite strong predominance of structures which consist of carbon (high condensable aromatic structures, graphite-net) [37]. The proportion of aromatic carbon, a component of coal increases from 0.75 to 0.99, respectively, for brown coal and anthracite [38]. The surface of coal with a high degree of metamorphism is mostly hydrophobic, and the structure of EDL of its surface is fundamentally different from EDL of coal with low and medium degree of metamorphism [36]. Information about the electro-surface properties of unmodified anthracite is extremely small.

Unique low-ash anthracites became the feedstock for production of filter material (AQUALAT) with improved physicomachanical properties as compared to conventional materials [39].

A distinctive feature of the Aqualat filter material is low ash content (5%) and low sulfur content (1%). An important indicator of the suitability of anthracite as filtering materials is their ability to crack at crushing to the grain of isometric shape (spherical or cubic). Grains close in form to the cube or spheres, parallelepipeds and various types of pyramids, prisms with technological form coefficients of 1.8 and heterogeneity coefficients of 1.47 are predominant in Aqualat filter loading. The homogeneity of the grain size and their shape provides uniform and a large amount of intergranular space. Practically there are no grains with flapjack, needle and scale form, that excludes "windage" of grains and their removal during regeneration of load. Due to high mechanical strength (grindability  $\leq 1.6\%$ , abrasability  $\leq 0.14\%$ ), less intensity and duration of washing, there is no destruction of filter material in the process of rebuilding its properties [39,40].

As noted above, electrosurface and electroconductive properties of the filtering material or adsorbent are determinative for the possibility of its use in elektroadsorption. Therefore, the aim of work was the study of electroconductive properties of carbon filtering material Aqualat, as well as the determination of zeta potential in conditions of pH and concentrations values of indifferent electrolytes close to that of natural and wastewaters in the last stages of post-treatment [41-43].

## 2. Results and discussion

We used Aqualat filtering material with fractions 0.6-1.2 mm, 1.0-1.8 mm and powdered to micron size. The filtering material was washed before operation and boiled in distilled water.

Working solutions were prepared in distilled water with a conductivity not higher than  $3 \cdot 10^{-4} \text{ Ohm}^{-1} \cdot \text{m}^{-1}$ . As indifferent electrolyte we used NaCl with chemically pure qualification. 0.1M NaOH solution was used to prepare the solutions with pH in neutral and weakly alkaline region.

Electroconductivity of filtering material was defined as inverse value of resistivity of the sample equilibrated by appropriate electrolyte solution. Silicone tube with an inner diameter of 80 mm was placed vertically, the bottom hole was temporarily blocked by the adapter sleeve with a membrane and the away hose with clamp, tightly filled with filtering material. Then the resulting "flexible column" was placed horizontally by means of two tripods with a small whipping in order to prevent electrolyte solution flow out of the tube. Fitting was removed, metal plates connected to the test leads of analog digital multimeter APPA 101 (APPA Technology Corporation, Taiwan) were introduced into the wet granular loading into both open ends of the tube to a depth of 2-3 mm. Resistance was measured multiple times. In parallel the electrical conductivity of used electrolytes was measured by a Portable Multi-Range Conductivity / TDS Meter HI 8733N (HANNA Instruments, Germany).

Three methods were tried out to measure the electrokinetic (zeta) potential ( $\zeta$ -potential).

### 2.1. The method of the streaming potential [44–46],

Which is considered as one of the most reliable methods for determining of zeta potential [45]. A simplified scheme of the apparatus is shown in Fig. 1. As recommended by Grigorov O.N. et al. [45] silver chloride electrodes were used to measure of streaming potential as well as reversible copper electrodes [46]. The electrodes were connected to the terminals of the potentiometer (used Multimeter APPA 101).

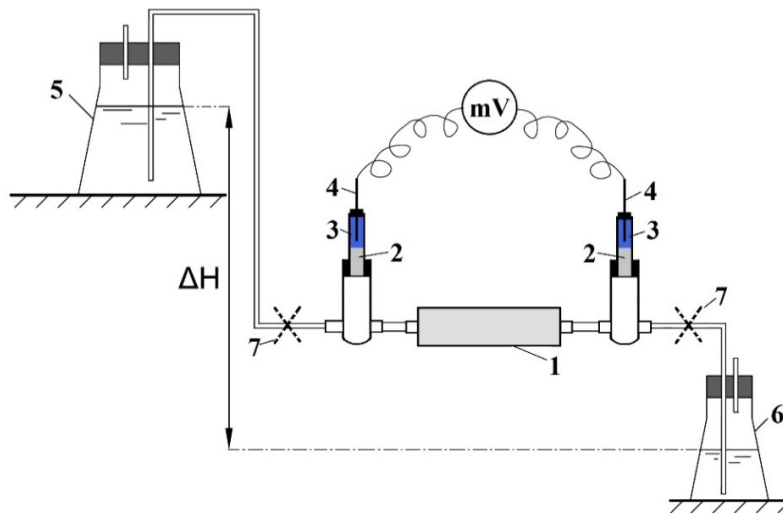


Fig. 1. Simple drawing of streaming potential apparatus: 1 – tube with Aqualat; 2 – agar-agar; 3 – copper sulfate solution; 4 – copper electrode; 5 – flask with feed electrolyte solution, 6 – flask for receiving of elapsed solution, 7 – clamps.

Measurements were performed during the flow of the electrolyte solution through the tube filled with filter material Aqualat sequentially in two directions. The pressure was created with water column. For each pressure (each value of solution heights difference in the source and the receiving tank) we firstly measured the potential difference between the electrodes without pressure  $\pi_0$  (at least 5 times), i.e. asymmetry potential (using clamps 7), testing its stability over time. Then we removed the clamps 7, organizing the flow of the solution, and after the establishment of the equilibrium measured the potential  $\pi_1$ . Then we again interrupted the flow with clamps 7 and after the establishment of equilibrium measured the final value of potential asymmetry  $\pi_{0fin}$ . The measurements were carried out at 3–4 values of height ( $\Delta H$ ). Then we changed the direction of the solution streaming and after flowing of sufficient electrolyte repeated the measurements.

When measurements of potentials were completed, we measured electroconductivity and pH of the solutions, then calculated the average of measured values at each point, and then found the values of the difference  $E = (\pi_1 - \pi_{0fin})$ , divided to the corresponding values of  $\Delta H$ . Then we calculated the average values for each direction and the average between them.

The zeta potential was calculated (without correction for surface conductivity) according to (Eq. 1):

$$\zeta = \eta * \varkappa * E / (\varepsilon * \varepsilon_0 * \Delta H * k), \quad (1)$$

where  $\eta$  – the dynamic viscosity, ( $\eta = 1.0050 \text{ mPa}\cdot\text{s}$ ),  $\varkappa$  – specific electrical conductivity, ( $\text{Ohm}^{-1}\cdot\text{m}^{-1}$ ),  $\varepsilon$  – dielectric constant of water (80.4 at 20 degrees on Celsius),  $\varepsilon_0$  – the permittivity of free space (a vacuum) ( $8.85 * 10^{12} \text{ F/m}$ ),  $k$  – conversion factor (1 cm of water column = 98.1 Pa).

## 2.2. Measurement of zeta potential with Zetatrac analyzer (Microtrac Inc., USA).

Zetatrac determines Zeta Potential by measuring the response of charged particles to an electric field. In a constant electric field particles drift at a constant velocity. Through the velocity, the charge and Zeta Potential can be determined. Zetatrac utilizes a high frequency AC electric field to oscillate the charged particles. The Brownian motion power spectrum is analyzed with the Nanotracer controlled reference technique of particle sizing to determine the Modulated Power Spectrum. This is a component of the power spectrum resulting from the oscillating particles. Zeta Potential is calculated from the MPS signal. The particle mobility (velocity per electric field), charge and particle size were also determined [47].

The sample for zeta potential measurement were prepared as follows: triturated Aqualat filter material was placed into the electrolyte solution, vortexed, allowed to stand for 10 minutes, the supernatant was decanted and it was filtered through filter paper. The filtrate was passed through a track membrane (pore diameter 0.2 microns) using a nozzle for syringe or the filter cells (200 ml) [48,49]. Microparticles deposited on the membrane were washed with a small volume of the filtrate to the concentrated (or by reverse current). The samples obtained were stirred well and placed into the analyzer cell.

Instrument readings were checked by the standard model of colloidal alumina.

### 2.3. Microelectrophoresis.

Microelectrophoresis method was applied to determine the electrophoretic mobility of the particles in the Aqualat samples prepared according to the procedure described above. Setting with laser light is described in [50,51]. The method uses light scattering by colloidal and suspended particles [52]. We measured the mobility of particles of the filtrate after track membranes, and particles of the concentrate added to filtrate in a concentration of 0.05-0.2 percent by volume. In our work we used a modified Abramson cell. Electrophoretic velocity of the particles was determined by the method of stationary levels (at a depth equal to the 0.21 of height of flat capillary cell).

The electrophoretic mobility ( $U_{e.ph.}$ ,  $m^2 \cdot V^{-1} \cdot s^{-1}$ ) was determined by the Eq. 2,  $\zeta$ -potential ( $\zeta$ , V) – by Eq. 3.

$$U_{e.ph.} = \frac{\Delta X \cdot \varepsilon \cdot S}{t \cdot I}, \quad (2)$$

where  $\Delta X$ , – the distance at which the particle moves in an electric field, (m), during the time  $t$ , (s),  $\varepsilon$  – the specific electrical conductivity of liquid, ( $\text{Ohm}^{-1} \cdot \text{m}^{-1}$ ),  $S$  – cross sectional area of chamber, ( $\text{m}^2$ ),  $I$  – current strength, (A).

$$\zeta = \varepsilon \cdot U_{e.ph.} / \varepsilon \varepsilon_0 \cong 14.17 \cdot 10^5 \cdot U_{e.ph.} \quad (3)$$

The designations in Eq. 3 are given above, the numerical coefficient is calculated for standard conditions.

Table 1 shows the results of resistance measurement and conversion calculation to the electrical conductivity of Aqualat filtering material placed into the silicone tube in diameter of 8 mm. The length of the filled part of the tube is equal to 387 mm. Aqualat has previously been balanced by electrolyte solutions of different concentrations. Resistance values were averaged over six measurements. When calculating the absolute error was taken into account instrumental error (APPA 101), equal to resistance measurement  $\pm (0.7\% + 2 \text{ counts})$ .

Table 1. Results of measurement of Aqualat resistance and conversion calculation to the electrical conductivity

Parameters	NaCl concentration [M]		
	$10^{-3}$	$10^{-2}$	$10^{-1}$
R [kOhm]	300±10	178.6±1.9	88.8±2.6
$\varepsilon$ [ $\text{Ohm}^{-1} \cdot \text{m}^{-1}$ ]	0.0258±0.0009	0.0431±0.0009	0.0868±0.0025

The obtained values are close to the limit values of conductivity ( $0.03 \text{ Ohm}^{-1} \cdot \text{m}^{-1}$ ) of anthracite dispersions in their critical content in the alkaline earth metal carbonates, providing continuous formation of chain streamlines [53]. With increasing of electrolyte concentration the conductivity of Aqualat filtering material packed in this electrolyte increases.

The values of zeta potential obtained by streaming potential method are presented in Tab. 2.

Table 2. Results of measurement of Aqualat zeta potential, (mV), by streaming potential method

pH at the input	pH at the output	NaCl concentration [M]	
		$10^{-3}$	$10^{-2}$
5.9	6.05-6.2	-58±6	

7.1-7.3	7.05-7.1	-64±6	
5.6	5.8-6.1		-8.9±0.9
7.25	7.05-7.15		-9.3±0.9
8.2-7.9	7.6-7.7		-10.6±1.1

From the data shown in Tab. 2 we can see that in the neutral pH range at concentrations of NaCl, equal  $10^{-3}$ – $10^{-2}$  M, zeta potential values are negative, which is in good agreement with the data presented by Lopanov A.N. [36]. Falling of zeta potential module with an increase in the concentration of electrolyte is also consistent with his data [36], but the difference between the values obtained for Aqualat is significantly higher than for anthracite in the [36] (-46 and -40 mV for KCl concentrations of  $10^{-3}$  and  $10^{-2}$  M respectively). This difference will be also significant when taking into account the surface conductivity with allowance surface conductivity. From the pH values given in Tab. 2 we can also see that Aqualat shows buffer properties as described in reference [36].

Significantly lower values of  $\zeta$ -potential in  $10^{-3}$  M NaCl solution at pH 7.2 were obtained with Zetatrack analyzer (Fig. 2). However, it should be noted that these data were episodic and could not be repeated, because concentration index required for the method when loading the sample, reached the desired level for the obtained samples of Aqualat colloidal concentrate (particle size about 1 micron – see Fig. 2) only once.

By microelectrophoresis method we also determined  $\zeta$ -potential for particles of filtrate (their size was 120 nm according to Zetatrack) and for particles of concentrate added into another portion of filtrate at a volume ratio of 1 to 600 and 1 to 3000. The obtained values were:  $-(15 \pm 3)$  mV for filtrate particles;  $-(24 \pm 5)$  mV and  $-(22 \pm 4)$  mV for concentrate. Validity of data was confirmed by standard alumina dispersion, by which a check is made for Zetatrack. According to the certificate data for standard 0.2% dispersion of alumina and electric conductivity equal to  $90 \cdot 10^{-4} \text{ Ohm}^{-1} \cdot \text{m}^{-1}$  the value of  $\zeta$ -potential is equal to  $+(50 \pm 5)$  mV. Upon dilution of the alumina sample in 1000 times with an aqueous solution of NaCl with electrical conductivity equal  $92 \cdot 10^{-4} \text{ Ohm}^{-1} \cdot \text{m}^{-1}$ , pH 5.8, microelectrophoresis method gave a value  $+(40 \pm 8)$  mV.

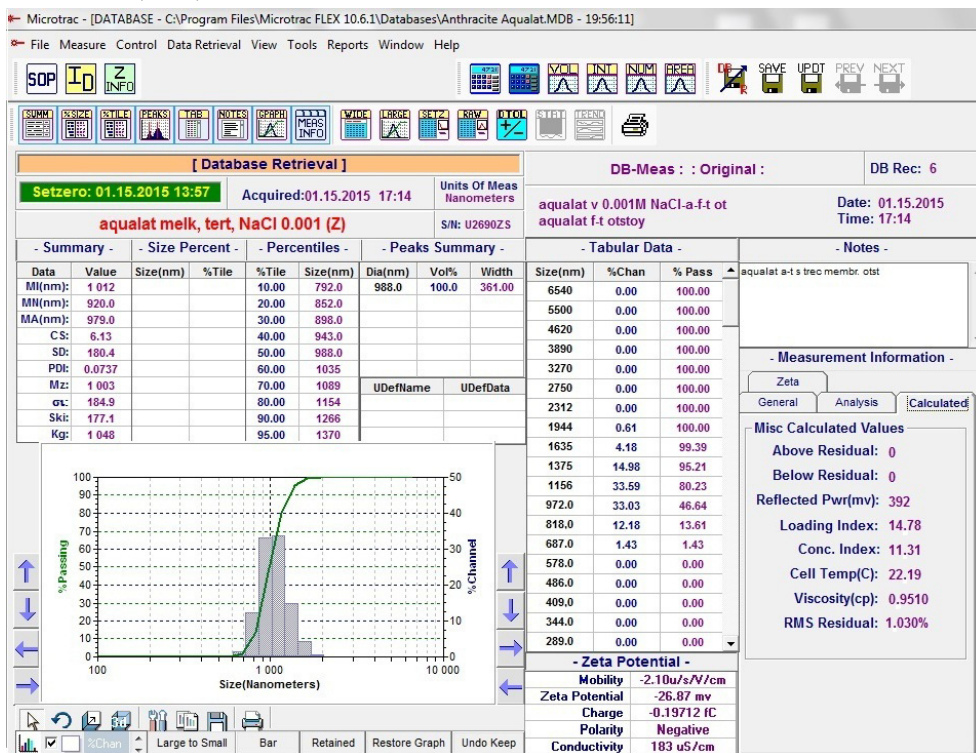


Fig. 2. Report of zeta potential measurement with simultaneous determination of particle size distribution on the device Zetatrack for particle dispersion of Aqualat material

### 3. Conclusion

Thus, three methods were tested for determining the zeta potential of Aqualat filtering material. For particles packed into the dense layer streaming potential method showed the results comparable with the values known from the scientific literature for anthracite (from which Aqualat was made). For dilute dispersions of grated Aqualat we can recommend microelectrophoresis method, but it requires a certain sample preparation.

Also the values of electrical conductivity of Aqualat filter material disposed in a dense layer in NaCl solutions were also determined over a wide range of its concentrations. The data obtained supposed to be used for organization the electrosorption post-treatment of waste (natural) waters with Aqualat filtering material.

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