Investigation of MIEX[®] resin sedimentation in the aspect of particle sizes remaining in the supernatant

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ABSTRACT

The aim of the study was to evaluate the effectiveness of the MIEX® resin sedimentation process, both new and repeatedly used in hybrid water treatment MIEX®DOC + microfiltration (MF) or ultrafiltration (UF) processes (repeatedly regenerated). The tests consisted of performing 1 h sedimentation of the suspension resin in nonflow conditions and analysis of supernatant water at different process durations. The suspension weight analysis was performed on filters (of porosity 0.45 μ m) and granulometric analysis (using a laser granulometer) was accomplished as well. Particles size measurements showed that the average particle size (d50) of the new MIEX® Resin was approximately 120 μ m. The repeated use of resin in the water treatment caused significant reduction in suspension grain size (d50 = 31 μ m), which could be the result of their abrasion or crumbling during exploitation. The sedimentation, the process efficiency was 99%. For the repeatedly used, regenerated resin the process went forward at much slower rate and 99% effectiveness was obtained scarcely after about 11–12 min of sedimentation. It was established that particles of the new resin, larger than 50 μ m, settled much faster than the corresponding particles of the regenerated resin. The smallest particles (<50 μ m) settled faster and more effectively in the case of regenerated resin. The soltaned test results allowed to determine the optimal sedimentation process parameters as well as the selection of particles with specific, the most favorable sizes (e.g., the rejection of the smallest particles to limit phenomena of results and water purification system MIEX®DOC + MF/UF).

Keywords: MIEX[®] resin; Sedimentation; Size of particles; Granulometric analysis

1. Introduction

One of the basic processes used in the classical water treatment is sedimentation. It serves to remove the falling suspension particles with a density greater than the density of water from the water. In water treatment systems, sedimentation is primarily used to remove the natural suspension present in captured water (pretreatment) and to remove the suspension produced during coagulation of colloidal impurities. The effectiveness of the sedimentation process depends on many factors, among which the most important are the sedimentation properties of the suspension particles (particle concentration, their shape, size, and density) and technological parameters of the process (velocity and direction of water flow, sedimentation time, and temperature) [1,2].

The sedimentation process, that is, the falling of particles, can also be used for restoration of ion exchange resin dosed to the water, which works in the form of a suspension in water, not in the form of an ion exchange bed. Such resins include the commercial MIEX[®] resin, which is used to remove (in the ion exchange process) dissolved natural organic substances (precursors by-product disinfection) and

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inorganic substances from water [3]. In addition to the ion exchange properties, the resin additionally has a built-in magnetic factor in the grain structure that allows for its fast agglomeration and easier sedimentation. In a classic water purification system, ion exchange using MIEX® resin is carried out on a resin suspended in water and takes place in a fully stirred tank/reactor, supplied with raw water. Next, the water with the resin flows to the settling tank, where it is separated in the sedimentation process. The settled (compacted) resin is partially recycled to the reaction chamber with the addition of a fresh and regenerated resin, and the remaining part is continuously directed to regeneration process. In a conventional technological system, the supernatant liquid after sedimentation of the resin flows to the sand filters, where it is polished [4,5]. It has to be kept in mind, that the efficiency limit of sand filters in retention of fine particles is $2-10 \ \mu m$ [1], so they may be inadequate if smaller particles are present in the supernatant. Therefore, the effectiveness of the advanced system can be increased, using the microfiltration (MF), ultrafiltration (UF), or nanofiltration processes [6,7].

In the case of UF and MF, the dominant mechanism of separation of particles present in water is the sieve mechanism. In this case, the separation of substances involves physical sieving, and the efficiency of the process depends on the porosity of the membrane and the size of the particles being removed. To achieve a satisfactory separation process, the size of particles removed from the water should be larger than the pore size of the membrane. Moreover, the presence of particles with dimensions comparable with those of membrane pores in water can lead to irreversible blocking of membranes [8,9]. This process is one of the main factors limiting the use of membrane techniques in practice. In this case, the knowledge of the particle size found in the liquid fed to the membrane system (in particular the finest particles) should be the basic criterion for the selection of the separation technique (e.g., type and pore size of the membrane).

The aim of this study was to evaluate the effectiveness and course of the process of sedimentation of MIEX[®] resin in the aspect of resin particle sizes remaining in the supernatant. The research concerned a new and repeatedly used resin in water purification (repeatedly regenerated).

2. Materials and methods

The subject of the research was Magnetic Ion Exchange Resin MIEX® that is produced by Orica Watercare Inc. (Australia). Polyacrylic resin grains of an average size of 150–180 µm (according to the producer specification) characterize with a water content of about 64% and specific crosslinking skeleton, in which micromagnets are built in. Small sizes of resin grains, approximately five times smaller in comparison with classic resin, results in a very high surface area and the ion exchange capacity are 0.40 val/L. The relatively high water content in resin grains makes the penetration of macromolecules and ion exchange easier, while the magnetic component of the grains enables fast agglomeration and sedimentation of resin grains [6,10].

Resin samples were prepared along with the volumetric method of the settled procedure by their gravity densification to a constant volume [11]. Five new resin portions and five portions of regenerated resin (the resin 10 times regenerated with 10% NaCl solution) with the same volume of 10 mL each were prepared for the tests. The determined volumes of resin were measured with laboratory cylinders of the same capacity. The method of measuring the resin consisted of filling a cylinder with graduated resin and leaving it to settle and compaction. The suspension in the cylinder was topped up until the level of resin thickened in the cylinder reached a volume of 10 mL.

The sedimentation process was performed in laboratory, nonflow conditions. The granulometric measurements and the quantitative measurements of the particle content/ concentration in the overlying water were made for the following sedimentation times: 2.0; 3.0; 5.0; 10.0; 15.0; 20.0; 30.0; 45.0; and 60 min. The process was carried out at the temperature around 20°C.

The granulometric tests consisted of the measurement of the particle size of the resin remaining in the supernatant after a given sedimentation time. In these tests the sedimentation process was carried out in glass vessels (beakers), with a capacity of 1.0 L each, separately for each sedimentation time. The total liquid depth in the vessel was approximately 16.0 cm. 10.0 mL of concentrated resin suspension was introduced into each beaker and refilled with demineralized water to a volume of 1.0 L. Then, for about a minute, the solution was vigorously stirred (80-90 rpm) using a paddle mixer to distribute the slurry evenly throughout the entire volume of water. After stopping the agitator, the sedimentation process began. After the specified sedimentation time, all excess water was withdrawn (about 0.75 L, about 3.0 cm above the sediment layer, and about 12.0 cm below the liquid surface). The supernatant water was collected using a vacuum pump. For the granulometric measurements, the entire volume of supernatant water was used. After granulometric measurements, the supernatant water was again combined with the separated precipitate and the solution was reused in subsequent tests (for other sedimentation times).

Quantitative studies of the sedimentation process consisted of determining the content of the suspension in the supernatant liquid. These tests were performed separately after granulometric studies. The entire resin suspension (separately new and regenerated) used in the granulometric studies (a total of 5.0 L, with a 50 mL content of thickened resin) was poured into a glass vessel with a capacity of 5.0 L, in which the sedimentation process was carried out. Similarly to the granulometric studies, the suspension was vigorously stirred using a paddle stirrer (for about 1 min) to distribute evenly the suspension throughout the entire volume of water. During mixing, a sample of approximately 70 mL of the solution was taken to determine the initial concentration of the suspension. After stopping the agitator, the sedimentation process began. After the specified sedimentation time, about 100-250 mL of supernatant water was taken (using a vacuum pump) from a depth of about 15 to 20 cm below the water surface. For the longest sedimentation times, larger amounts of supernatant water were collected (for 30 and 45 min, approximately 500 mL, and for 60 min approximately 900 mL). Samples of the overcharged water collected, and the solution taken with the resin during mixing (after measuring the volume of the sample taken), were filtered through cellulose filters with a porosity of 0.45 µm.

To measure sizes of particles, the laser diffraction particle size analyzer Mastersizer Hydro 2000MU (Malvern Instruments Ltd., UK) was used. The measuring range of the analyzer is between 0.02 and 2,000 µm. The instrument uses two laser lights: red and blue ones. The measuring technique is based on the laser diffraction phenomenon (LALLS - Low Angle Laser Light Scattering [12]). The laser beam, which passes through a sample is scattered by particles dispersed in solution. Determination of particles sizes is an indirect result of the calculation of their volume. The instrument provides results as particle size distribution, which illustrates percentage share of particles with equivalent diameter in relation to the total volume of all particles in the sample (by volume) [12-14]. The equivalent diameter is defined as the diameter of the sphere with the same volume as the measured particle. According to Bushell et al. [14], this method is appropriate for particles characterized by low values of refractive index and for particles with loose structure. This method proves itself as well in particles size measurement applied in surveys over purification of water and wastewater [15,16]. The measurements were taken with following settings: particle = default, refractive index = 1.520, absorption index = 0.1; dispersant = water, refractive index = 1.330; analysis model = general purpose; and sensitivity = normal. Measurement time for each laser light (red and blue) was equal to 5 s, whereas single measurement took in total approximately 16 s. Five measurements were taken for each sample.

In addition to the particle size distribution, characteristic values such as d10, d50, and d90 were calculated, as well as the largest and smallest particles observed during the measurement. The characteristic values (d10, d50, and d90) are defined as: d10 – particles of this and smaller diameters constituting 10% of the total volume (mass) of all particles in the sample, d50 (average particle size) – particles of this and smaller diameters constituting 50% of the total volume

(mass) of all particles in the sample), and d90 – particles of this and smaller diameters constituting 90% of the total volume (mass) of all particles in the sample.

The assumption was made that the density of the resin was constant, regardless of the size of the particles, and thus the distribution of the particles of the suspension in volume (relative to the volume of the suspension particles), obtained during granulometric measurements, was also a weight distribution (relative to the mass of the suspension).

Each time the term dimension or size of particles is mentioned in this study, it means the equivalent diameter of the particles, which value is the average of the measurement interval with the range of approximately $\pm 7\%$ of given value.

3. Results and discussion

Measurement of particle dimensions using a laser granulometer showed that the particle size for a new MIEX® resin ranged from about 0.3 µm to about 390 µm (Fig. 1, Table 1) (the smallest particles below 10 µm were estimated indirectly after several minutes of sedimentation). The average particle size (d50) of the new resin amounted to around 117 μ m, so it was a bit smaller than the 180 µm size reported by other researchers [17,18]. The other characteristic dimensions were gauged respectively as: d10 approximately 48.6 µm and d90 approximately 214 µm. Almost 90% of all suspension particles (by volume and by weight) were larger than 50 µm, while 60% of particles were larger than 100 µm. Particles smaller than 10 µm were estimated for less than 1.0% of all particles in the suspension, while the smallest particles, smaller than 1 µm, accounted most probably for less than 0.1% of all suspension particles.

Repeated use of the resin resulted in significant changes in the particle size of the suspension, wherein a reduction of the particle size was observed in general (Fig. 1). The particle size ranged from about 0.5 μ m to over 500 μ m. The grain



Fig. 1. Size of MIEX® resin particles, new and repeatedly used (regenerated).

Time	MIEX® resin – new (µm)					MIEX [®] resin – regenerated (µm)				
Min	Min.	Max.	d10	d90	d50	Min.	Max.	d10	d90	d50
0	0.30	390.0	48.6	213.6	117.4	0.51	513.1	12.5	73.1	31.0
2	0.45	170.2	9.3	80.0	33.9	-	-	-	-	_
3	0.45	148.3	6.5	64.6	27.5	0.45	148.3	11.2	60.6	27.0
5	0.45	148.3	4.7	75.1	24.3	0.51	98.0	10.5	47.1	22.4
10	0.45	112.5	4.3	55.6	20.8	0.45	98.0	10.7	49.6	24.1
15	0.45	112.5	3.7	46.8	17.7	0.89	56.4	10.2	36.7	21.1
20	0.39	98.0	3.5	37.4	16.6	1.03	56.4	10.4	35.8	20.7
30	0.45	42.8	2.8	28.3	13.1	0.45	74.3	9.5	42.5	21.3
45	0.34	37.2	2.2	24.4	11.1	0.45	74.3	9.1	40.1	20.2
60	0.34	56.4	2.1	28.5	11.3	0.45	74.3	9.5	42.0	21.3

Table 1 Granular composition of MIEX[®] resin new and regenerated during sedimentation

fragmentation was indicated by characteristic sizes, which were significantly reduced in relation to new grains of resin. And so, the size d10 was about 12.5 μ m, average diameter d50 about 31 μ m and d90 about 73 μ m. Thus, the size of the regenerated resin particles decreased almost four times in comparison with the new resin. This fragmentation, however, did not apply to the smallest particles, because in the case of a regenerated resin, the smallest particles were about 0.5 μ m in size, while particles of new resin were almost half the size of the regenerated resin. Particles with dimensions larger than 50 μ m constituted only to about 21% in the case of regenerated resin, and larger than 100 μ m – less than 4% (by weight) of all particles in the suspension. Particles smaller than 10 μ m were about 5.0% of all particles in the suspension.

The reduction in size of repeatedly recycled resin grains may be the result of their abrasion or crumbling during use. The lack of the smallest particles in the resin suspension $(0.3-0.5 \ \mu m)$ was probably associated with the method of the used suspension separation from the solution, which took place in the sedimentation process (the smallest particles settled at the slowest rate and remained in the supernatant fluid). It was difficult, however, to explain the presence of the largest particles in the suspension of regenerated resin, larger than the largest particles in the new resin, which was in contradiction to the general trend, indicating the fragmentation of the suspension as a result of use. It possibly was due to the conglomeration of some grains and formation of larger, relatively permanent agglomerates.

The indications showed that despite the preparation of the same volume of new and regenerated resin, the mass of prepared resin samples was different. In the case of the new resin, the total dry matter of the prepared resin was 11.0g/50 mL (220g dry matter/L), and of the regenerated resin 6.08g/50 mL (122g dry matter/L). Thus, the bulk density of the new resin was almost twice as high as the repeatedly used resin. It means that the actual dose of the repeatedly regenerated resin, measured by the volumetric method, in conversion to the resin dry weight could be as much as twice smaller (by 45%) than for the new resin. Most probably, the higher bulk density of the new resin was the result of the larger (stronger) magnetic properties of the new resin in comparison with the regenerated resin. It can be assumed that, due to the stronger magnetic attraction, the new resin seeds were packed more densely than the seeds of the repeatedly regenerated resin. Molczan [11] showed that the swelling of saturated resin was greater than that of fresh resin, which could partly explain the smaller mass of resin repeatedly used, however, in the case of the presented tests, the resin repeatedly used after the last use was regenerated with NaCl solution, so it was not saturated.

The sedimentation process of new and repeatedly regenerated MIEX® resin is shown in Fig. 2. Sedimentation of both, new and repeatedly regenerated resin was a very effective process. The new resin settled very quickly. Already after the first 2 min of sedimentation, the suspension content in the overlying water (by weight) accounted for only about 2% of the initial content (98% efficiency), and after 5 min sedimentation the process efficiency reached almost 99% and slightly above 1% of the initial suspension (Fig. 2) remained in the supernatant. The prolongation of sedimentation resulted in a further, though much slower, decrease in the content of the suspension, the content of which in the supernatant liquid after 60 min of sedimentation was less than 0.2% of the initial suspension. MIEX® repeatedly regenerated resin settled at a much slower pace than the new resin. After 2 min of sedimentation, the process efficiency was only about 60%, and after 5 min - 85%. 99% efficiency was obtained only after about 11-12 min of sedimentation. However, further sedimentation proceeded much faster than for the new resin (in the same time interval), so after 60 min, the efficiency of sedimentation of the regenerated resin equaled with the efficiency of sedimentation of the new resin and about 0.2% of the initial suspension remained in the supernatant fluid. Those results are consistent with Molczan [11] observations, which proved that the sedimentation period for the fresh MIEX® resin was relatively short. Relative stabilization of the volume of the resin sludge layer occurred after about 5 min of sedimentation, and after 20 min no further changes in sediment volume were observed.

The results of measurements of the sizes of particles remaining in the supernatant, using a laser granulometer, are shown in Fig. 3 and Table 1. During sedimentation of both new and regenerated resin, in every subsequent measurement carried out in the overlying water even smaller



Fig. 2. The course of sedimentation (by weight) of new and regenerated MIEX[®] resin (Mo – dry weight of resin before sedimentation; M – dry weight of resin after chosen sedimentation period).

particles remained, wherein this process was much faster in the case of the new resin. So in the case of the new resin, for the first 5 min of sedimentation, the average particle size (d50) remaining in the supernatant water decreased almost five times (to about 24 μ m) (Fig. 3, Table 1). At that time, particles larger than 100 μ m, total mass of which in the solution before sedimentation accounted for 60% of the total mass of all suspension particles, were almost completely removed and constituted only about 3% of weight of all particles remaining in the supernatant. In the longer sedimentation, the average particle size remaining after sedimentation systematically decreased and eventually stabilized after about 45 min at about 11 μ m.

During the sedimentation in the supernatant water, the percentage of the smallest particles in the total mass of all particles systematically increased. Particles, smaller than 10 μ m in size, which accounted for less than 1% of the total mass of all particles in the suspension prior to sedimentation, already after 5 min of sedimentation accounted for 20% of the mass of all suspended particles, and at the end of sedimentation (after 45–60 min) was almost 50% of weight of all particles remaining in the overlying water. This indicated on the quick sedimentation of the largest particles and the smaller, slowly settling particles remained in the supernatant water.

In the case of regenerated resin, the systematic reduction of the particle size of solids remained in the overlying water was only observed for the first 15–20 min of the process. The average particle size (d50) decreased by only about one-third from 31 μ m in the solution before sedimentation to about 21 μ m (Fig. 3, Table 1). In longer sedimentation (20–60 min), the average particle size observed in the overlying water did not change significantly. During the sedimentation of the regenerated resin, in the overlying water, similarly to the new resin, a systematic mixing of the largest particles and an increase in the percentage share of the smallest particles was observed. However, these changes did not take place as quickly as in the case of the new resin. The smallest particles, smaller than 10 μ m in size, which in the suspension before sedimentation constituted to about 5% of the total mass of all particles, only at the end of sedimentation (after 45–60 min) doubled their share, to approximately 10%.

It was also characteristic that the average particle size (d50) of solids, which remained in the overlying water during the sedimentation of regenerated resin, never reached such small dimensions as in the case of the new resin, although in the suspension before sedimentation this dimension was much smaller than in case of the new resin. For the new resin, the average particle size during sedimentation decreased to approximately 11 μ m, while d50 for the regenerated resin stabilized at 20–21 μ m, so it was nearly twice as large. This result confirmed the observations obtained during the preparation of resin samples – the same volume of regenerated resin. It is possible that the lower density of the repeatedly used resin was due to the reduced magnetic properties of the particles, so that they were less bounded together.

Using the results of both, weight and granulometric measurements, it was possible to calculate the approximate absolute mass of particles of specific sizes remaining in the overlying water during sedimentation. These calculations allowed to follow the course of sedimentation for various fractions of new and regenerated resin particles. The absolute mass of particles of specified size is calculated as: product, percentage of particles of a specified size (from granulometric measurements), and mass of resin remaining in the overlying water after a given sedimentation time. The sedimentation results of particular fractions of the new and the regenerated resin obtained on the basis of these calculations are shown in Fig. 4.

The comparison of the courses of sedimentation of various fractions of new and regenerated resin particles was quite surprising. It turned out that the smallest particles of



Fig. 3. Size distribution of new and regenerated MIEX® resin after different sedimentation times.



Fig. 4. The sedimentation curves of selected fractions of new and regenerated MIEX[®] resin (Mo – dry weight of a selected resin fraction before sedimentation; M – dry weight of a selected resin fraction after selected sedimentation period).

regenerated resin (<10 and 10-50 µm) settled faster and more efficiently than the corresponding new resin particles (Fig. 4). During the first 15-20 min of sedimentation, particles of regenerated resin with dimensions smaller than 50 µm were removed in almost 99%, and after 60 min of sedimentation less than 0.3% of these particles remained in the supernatant. In the case of a new resin, after 15 min of sedimentation in the overlying water there remained almost 20% of particles with dimensions less than 10 µm and over 4% of particles with dimensions of 10-50 µm, and after 60 min of sedimentation in the overlying water there still remained about 10% of particles smaller than <10 µm and about 1.5% of particles smaller than 50 µm. These results seem to contradict the results of weight measurements, indicating on a much faster sedimentation of the new resin (Fig. 2). This can be explained by the method of recovering the spent resin after the purification process, which took place in the sedimentation process (the most slowly settling particles remained in the overlying water and were not recycled for reuse). Hence, those (among the smallest) particles, that settled faster, most likely of higher density (e.g., with lower porosity), were able to be reuse. In addition, small particles of repeatedly used resin could have more regular shapes, which also could promote faster sedimentation. Another factor causing a faster sedimentation of the smallest particles of the regenerated resin could be the larger dimensions of these particles in relation to the new resin. The granulometric analysis showed that the smallest particles of regenerated resin (<10 μ m) were dominated by particles only slightly smaller than 10 μ m (more than half of them had sizes greater than 5.0 μ m, and the smallest particles were larger than 0.5 μ m). However, in the case of a new resin, about two-third of the particles smaller than 10 μ m were smaller than 5.0 μ m, and the smallest particles were smaller than 5.0 μ m.

On the other hand, new resin particles of size between 50 and 100 μ m, as well as those largest, with size over 100 μ m, settled much faster than particles of the corresponding sizes, but originated from the regenerated resin. This high velocity of sedimentation of the largest particles, in combination with the high content of these particles in the total mass of the new resin, determined the faster sedimentation of the new resin, especially in the first minutes of the process (particles larger than 50 μ m constituted more than 90% of the total mass of all particles), while for regenerated resin particles above 50 μ m gave only about 25% of the total mass of all particles.

The obtained test results indicate that sedimentation can have a positive effect on the membrane filtration process. Although direct sedimentation does not remove the smallest and most unfavorable for the membrane processes particles from the water, but even a short, several-minute long sedimentation can reduce the amount of suspension fed to the membranes, thus limiting their blocking. In addition, indirect sedimentation can be used for modification of the particle sizes of the resin. Selecting the time of sedimentation of the resin (in particular, proper shortening of the process) enables the rejection of the smallest, most disadvantageous from the membrane filtration point of view particles. Such modification of the resin grain size by sedimentation can be performed by preparing a new resin (before the first use), as well as by preparing the resin after every cleaning and regeneration process. Thanks to such actions, the particle size of the resin can be selected, so that membranes with a higher porosity (hydraulically more advantageous) can be used, or the phenomenon of irreversible blocking of membranes by the smallest particles can be reduced.

4. Conclusions

- The laser granulometer, used for measuring particle sizes can be a useful tool for sedimentation characterization. Using the results of granulometric and quantitative measurements, it was possible to qualitatively and quantitatively follow sedimentation of specific fractions of suspension particles.
- Measurements of particle size showed that repeated use of MIEX[®] resin resulted in a significant reduction of particles of the bigger size, which could be the result of abrasion or crushing of grains during reuse.
- The actual dose of the repeatedly regenerated resin, measured by the volumetric method, in conversion to the resin dry weight, can be as much as almost twice smaller than for the new resin.
- Sedimentation of both, new and regenerated resin, was a very effective process. For the new resin, after the first 5 min of sedimentation, the efficiency of the process was 99%. Resin repeatedly regenerated settled much slower, and 99% efficiency was obtained after about 11-12 min of sedimentation. During the sedimentation of both, new and regenerated resin, in each subsequent measurement, smaller and smaller particles remained in the supernatant water, and the process proceeded much faster in the case of the new resin. It was found that particles of new resin with larger sizes (above 50 µm) settled much faster than the corresponding particles of regenerated resin. However, the smallest particles (<50 µm) settled faster and more effectively in the case of regenerated resin. This may mean that the smallest particles of the regenerated resin had a more compact structure and a larger specific mass than the corresponding, smallest particles of the new resin.
- The obtained test results allow to determine the optimal parameters of the sedimentation process, as well as to use this process to select the particles with specific, most advantageous sizes (e.g., rejection of the smallest particles that could be a potential threat due to blocking of membranes used to separate the resin from clean water).

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