Dóra Laky

Arsenic removal by coagulation

Theses of the PhD dissertation

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1. Introduction, aim of the research

In the second half of the 1990s the National Public Health Institute within the frame of the NEKAP program (National Environmental-Health Action Program) carried out a survey concerning the water quality of the supplied water in Hungary. According to this survey, around 1.4 million people at about 400 settlements are supplied with drinking water above the 10 µg/L standard set by the 98/83 EU Directives.

In Hungary the coagulation-flocculation method is the most often applied arsenic removal technology. At the end of the 1980s, beginning of 1990s, arsenic removal technologies were installed at those settlements, where the previous 50 µg/L standard was not fulfilled. These technologies were based on the coagulation method.

Coagulation is a widely used technique for arsenic removal. The surface charge of the colloid particles are neutralized by adding metal-salts to the water, therefore aggregation can take place. After that, the formed flocs are removed from the water by solid/liquid phase separation (sedimentation, filtration). When this method is applied, arsenate is also able to build into the flocs, thus arsenate is also removed from the water when the flocs are removed by phase separation.

Adsorption technology can be also used for arsenic removal. However, the special characteristic of the Hungarian subsurface waters – especially the relatively high orto-phosphate concentration – might hinder arsenic removal when special adsorbents for arsenic removal are applied. The exhaustion of these adsorbents often occurs much earlier than the expected time.

At many settlements in Hungary only simple iron and manganese removal takes place currently. If the raw water contains arsenic, the presence of the *in-situ* coagulant cause arsenic removal to some extent, however the 10 µg/L standard cannot be fulfilled. Upgraded, modified technology is needed at these settlements, and also at those waterworks, where the previous standard is met (50 µg/L), but current one is not fulfilled.

Many research works deal with the coagulation technology for arsenic removal, however often generalization is carried out based on the removal efficiency under certain water quality conditions. Such generalization is for example the determination of the needed metal : arsenic mass ratio to achieve the 10 µg/L standard value or stating arsenic removal efficiency for a certain type of coagulant. Such general recommendation can be extremely misleading, because the raw water characteristics highly influence the arsenic removal efficiency.

Earlier research work extensively deal with the effect of several water quality parameters on arsenic removal (e.g., Holm, 2002; Kelemen, 1991; Liu et al.,

2007; Meng et al., 2000). However, in order to explore the basic processes, the applied circumstances are often not typical for drinking water treatment (e.g., extremely high initial arsenic concentration or coagulant dose). Therefore, the aim of my research was to study the arsenic removal efficiency under the typical Hungarian water quality (pH, orto-phosphate, bicarbonate, silicate concentration, organic material content) conditions.

The removal of arsenic-containing flocs can be carried out by traditional rapid sand filtration (if it is necessary, sedimentation before filtration) or by membrane technology. In Hungary the operational costs of the membrane technologies are still relatively high compared to the traditional methods. Therefore, in the dissertation, the applicability of rapid sand filtration was investigated in order to remove the arsenic-containing flocs. The goal was to determine the particulate-arsenic removal efficiency of the simple rapid sand filtration, to study whether the 10 μg/L standard value can be achieved.

2. Methods

The experiments were carried out in two levels: in laboratory, batch-scale experiments (jar tests) and also in pilot-scale experiments. The batch-experiments were conducted with different types of synthetic water (model systems made by distilled water, by tap water of Budapest, by untreated raw water from Budapest and by raw water from Hortobágy-Szásztelek), and also by natural waters containing arsenic (raw water from Hajdúbagos, Pocsaj). The aim of the batch-experiments was to study the efficiency of different oxidants and coagulants, to explore the basic processes and also to investigate the influencing effect of several water quality parameters. The laboratory batch experiments carried out by raw water from Hajdúbagos and Pocsaj gave moreover the basis for determining the operational parameters (initial chemical dosages) of the pilot-scale experiments.

Jar-tests were performed in the following way: the water was introduced into 1 L beakers, and coagulant was added with rapid mixing (350 rpm) for 1 minute, followed by slow mixing (20 rpm) for 10 minutes and settling for 20 minutes. To evaluate the efficiency of arsenic removal, samples were taken from the clarified water, and they were filtered through membrane of 0.45 μm pore size. To investigate the effect of silicate on the floc-formation, the samples were filtered through 0.2 μm pore size membrane as well. Arsenic concentration was measured from the membrane-filtered water. The temperature was 22-27 °C during the laboratory experiments.

In order to quantify the influencing effect of the studied water quality parameters, linear regression analysis was carried out using the results from the laboratory experiments carried out by synthetic water made from distilled water.

The pilot-scale experiments were carried out in Hajdúbagos and Pocsaj. The pilot plant operated under pressure with two separate lines of $1 - 1$ m³/h maximum flow rate. Each line consisted of four fast-mixing (137 rpm) reactors for chemical dosing, one slow-mixing (14 rpm) flocculator reactor and rapid sand filter. The filtration velocity was modified by changing the actual flow rate. The evaluation of the role of mixing in the arsenic removal process was possible by installing by-pass pipes into the pilot plant.

Arsenic was measured from the sand filtered water and in most cases from the membrane filtered water as well (the sand filtered water was filtered through membrane of 0.45 µm pore size). The efficiency of rapid sand filtration was evaluated by comparing the total and dissolved arsenic concentration after rapid sand filtration.

3. Results

3.1 Water quality parameters influencing arsenic removal (results of the laboratory batch-scale experiments)

The aim of the first batch-scale experiments was to compare the efficiency of different oxidants and coagulants. Out of the three studied coagulants (ferric-chloride, aluminum-sulphate and Bopac), ferric-chloride was proved to be the most efficient in case of all studied water quality parameters. Ferric-chloride was able to remove arsenite (As(III)) as well to some extent, however it was more efficient in case of the arsenate (As(V)) form. The coagulants containing aluminum are not able to remove the As(III) form (in case of low organic content; around 1 mg/L COD_{Mn}).

Except air, all the studied oxidants (ozone, sodium-hypochlorite and potassium-permanganate) were all able to oxidize As(III) to As(V) efficiently. When sodium-hypochlorite is applied and the raw water contains ammonium as well, chlorine dose far below the breakpoint dose could efficiently oxidize As(III).

The pH of the raw water significantly effects the dissolved-solid transition of arsenic through influencing the solubility of the metal-hydroxide flocs. In case of high initial arsenic concentration (200 – 300 µg/L) and wide pH range (5.0 – 11.0), the arsenic removal efficiency varied between 17 % and 96 %. The best removal efficiency (for both ferric and aluminum coagulants) was found in the 7.0 – 7.5 pH range. When narrower, more typical pH interval was applied (7.0 – 7.8) with lower initial arsenic concentration values (70 µg/L) the effect of pH was still evident (at lower pH better removal efficiency was found), however under these circumstances the influencing effect was not that significant. The buffering capacity of the Hungarian waters are relatively high (10 meq/L alkalinity is not uncommon), therefore significant amount of acid is needed to

decrease the pH. As a consequence, modifying the raw water pH is usually not an economic solution in Hungary; instead the coagulant dose has to be increased in order to compensate the undesirable effect of high initial pH.

Phosphate is widely present in subsurface waters in Hungary. Investigations proved that the efficiency of arsenic removal is highly influenced by the presence of orto-phosphate (Holm, 2002). The reason is that the phosphate concentration is higher than the arsenate concentration with one order of magnitude; therefore they occupy most of the sorption sites on the metal-hydroxide flocs, which hinders arsenic adsorption. My laboratory experiments carried out by synthetic water proved that 0.15 mg/L PO_4-P concentration can already significantly increase the remaining arsenic concentration. The negative effect of phosphate ions on the arsenic removal efficiency is more significant at lower coagulant dosages and at higher pH values.

If the water contains orto-phosphate, it is not sufficient to simply add more ferric-coagulant according to the number of moles of orto-phosphate (assuming that phosphate is precipitated in the $FePO_4$ form). In the presence of phosphate special Fe(III)-hydroxide precipitates are formed, with which the arsenate cannot contact. The coagulant dose has to be increased in order to achieve a certain Fe(III) : PO_4-P [mg/mg] ratio, above which the arsenic removal can effectively take place. In case of model systems made by tap water or distilled water this ratio is around 10 mg Fe(III) / mg PO_4-P in the 7.5 – 8.0 pH range.

For one hand, inorganic carbon (H_2CO_3, HCO_3^- and CO_3^{2-}) content has advantageous affect, since it contributes to the buffering capacity of the water and metal-hydroxides are able to be formed. At least three times more hydrogen-carbonate ion moles are needed than the dosed moles of metal-salts. However laboratory experiments carried out at 7.0 and 8.0 initial pH (dosing 0.018 mmol/L Fe^{3+}) showed that HCO_3^- above 1 mmol/L has negative effect on arsenic removal, since the arsenic concentration in the treated water increased as the HCO_3^- concentration increased. At higher pH values (> 8.0) and in the presence of 5 – 10 mmol/L HCO_3^- the residual arsenic concentration was around 20 times higher than in case of 0.125 – 0.5 mmol/L HCO_3^-. In case of higher alkalinity the final pH values are significantly higher which results in decreased efficiency in the arsenic removal process. In this way alkalinity has indirect effect on arsenic removal by influencing the final pH value. The analysis presented in chapter 3.2 proved that this is the mechanism how inorganic carbon influences the arsenic removal processes.

According to Liu et al. (2007) the adsorption and surface complexation of silicate on ferric hydroxide result in decreased ζ potential. The decreased ζ potential of ferric hydroxide precipitates increased the repulsive forces between precipitates, which inhibited further particle agglomeration. Their results indicated that silicate increases repulsive charges and decreases surface sites

available for arsenic, rather than facilitating the formation of tiny ferric colloids with adsorbed arsenic, which are able to go through the 0.45 μm pore size membrane filters. My laboratory experiments carried out with model waters made from distilled water confirmed the results of Liu et al. (2007). The size of the formed Fe(III)-hydroxide flocs in silicate-rich systems were mostly in the 0.2 μm – 0.45 μm range, therefore 15 mg/L SiO_2 content already resulted in significant amount of arsenic in the 0.45 μm filtrate. This effect was even more significant at higher pH values. By increasing the coagulant dose, the target arsenic concentration (10 μg/L) was finally achieved, however compared to the results with silicate-free systems, around 2.5 – 3.5 times higher iron dose was needed to reach this value in case of 30 – 50 mg/L SiO_2 content.

According to the results of Kelemen (1991) organic content has significant effect on the needed coagulant dose. The organic material gets in contact with the dosed metal-salts (colloid-stabilizing and complex-forming effect), therefore the amount of coagulant – which can be used for arsenic removal – decreases. Comparing the results of the experiments carried out with high (~ 13 mg/L COD_{Mn}) and low (~ 1 mg/L COD_{Mn}) organic content, it can be stated that significantly (even with one order of magnitude) higher coagulant dosage might be needed in case of the same initial arsenic concentration but different organic content. The huge difference in the needed metal : arsenic molar ratio emphasizes that the technology planning cannot be based on the initial arsenic concentration only, since the other water quality parameters – mostly the organic content, orto-phosphate and silicate concentration – basically modify the coagulant dose needed to fulfill the arsenic standard.

3.2 Predicting the residual arsenic concentration

After evaluating the role of the water quality parameters individually (pH, phosphate, silicate concentration, alkalinity, coagulant dose) my goal was also to investigate the aggregate effect of them and to determine the most important influencing factors. The aim was moreover to build up a robust model, which could predict the needed ferric coagulant dose in order to achieve the 10 μg/L standard value for a given raw water quality.

For the analysis, results from experiments with distilled water were used, where the initial arsenate concentration was 50 – 60 μg/L, ferric-chloride coagulant and *in-situ* floc formation was applied. Earlier results showed that due to the presence of natural organic matter, the needed coagulant dose could be higher with even one order of magnitude. However, due to the different characteristics of organic materials, their effect on arsenic removal could be very different; therefore organic material content was excluded from the analysis. The following water quality parameters were considered in the analysis: final pH value (pH after coagulation), silicate, phosphate, inorganic carbon concentration and ferric coagulant dose. As the first step the aim was to set up an equation,

which considers the aggregate effect of these parameters and the residual arsenic concentration (in case of 50 – 60 μg/L initial arsenate concentration) can be roughly estimated. After the first analysis, it turned out that the hydrogen-carbonate concentration had no influence on the residual arsenic concentration. The resulted equation is the following:

Residual (dissolved) As [μg/L] = $\qquad\qquad\qquad\qquad\qquad\qquad$ (1)

– 94.44 + 31.14 · PO₄-P [mg/L] + 14.71 · pH + 0.55 · SiO₂ [mg/L] – 5.80 · Fe [mg/L]

In the equation the pH after coagulation is a variable. However this value depends on the initial pH, alkalinity and also the coagulant dose. I worked out the methodology how to determine the final pH after coagulation as a function of initial pH, alkalinity and ferric-coagulant dose.

The partial correlation values were calculated in order to see the significance of each variable. The most important influencing factor proved to be the silicate concentration and the next one was to ferric-chloride dose. The multiple correlation value was 0.86 ($R^2 = 0.74$), which means strong correlation between the independent variables (pH, PO_4-P, SiO_2, Fe) and the dependent variable (residual dissolved arsenic concentration).

The investigated formula was verified by 24 independent additional experiments with different types of initial water quality parameters. The calculated and measured residual arsenic concentration values were in very good agreement ($R^2=0.88$). This means that the investigated formula is capable of estimating the residual arsenic concentration in the studied concentration range.

The needed coagulant dose to achieve the 10 μg/L standard value can be estimated by rearranging equation (1):

Fe [mg/L] ≥ -18.01 + 5.37 · PO₄-P [mg/L] + 2.54 · pH +0.09 · SiO₂ [mg/L] $\qquad$ (2)

When equation (2) is applied, iteration is needed to determine the needed coagulant dose. As a first step a final pH value has to be set (which is lower than the initial pH), and than by using equation (2), the needed ferric coagulant dose can be calculated. For this coagulant dose, final pH can be determined by using the equations, graphs presented in the dissertation. Than this new final pH value is put into equation (2) and a new coagulant dose is determined. The procedure has to be continued until the difference between the final pH values is negligible.

Since the concentration of natural organic matter is not included in the presented model, only the minimally required coagulant dose can be estimated. If the organic content is significant ($COD_{Mn} > 2$ mg/L) higher coagulant dosages are needed than the estimated value.

3.3 The results of the pilot experiments

One aim of the pilot experiments was to examine whether the arsenic-containing flocs can be efficiently removed by traditional rapid sand filtration. As a first step of these experiments, the optimal type and dosage of chemicals (oxidants and coagulants) were determined in order to securely achieve the 10 µg/L standard value. The pilot experiments were carried out at two different sites with two distinct raw water qualities. The optimal type and dosage of chemicals were different at the two sites. The first experiments were carried out at Hajdúbagos, where potassium-permanganate and ferric-chloride proved to be the most efficient oxidant and coagulant. At Pocsaj, with double concentration of COD_{Mn} and higher pH, the sodium-hypochlorite + ferric-chloride combination was found to be the most efficient and around double coagulant dose was needed to reach the standard arsenic value. These results call the attention that for each raw water quality, individual preliminary experiments are needed to determine the required chemical type and dose.

At Pocsaj, the rapid sand filter containing 1-2 mm particle size sand proved to be applicable to remove the formed flocs. By considering 600 g/m² filter loading, the calculated and observed filtration cycles were in good agreement: when 5.2 m/h filtration velocity was applied, the length of the filtration cycles was around 14 hours. After 14 hours, the iron concentration in the rapid sand filtrate started to increase.

The modification of the mixing program (applying no rapid mixing or no flocculator tank) did not change the residual arsenic concentration in the filtrate in short term. However, when only the flocculator tank was excluded, the important role of slow mixing was already observed (by regular sampling between two filter-backwashes) in long term. By excluding the flocculator tank, the operational time between two filter-backwashes decreased to the half.

By summarizing the experiments related to rapid sand filtration, it can be stated that by applying proper particle-size (1-2 mm), filtration velocity (5.2 m/h) and mixing program, the traditional rapid sand filtration technology is able to remove the arsenic containing flocs efficiently.

Parallel to the pilot experiments, laboratory batch experiments were also carried out. The results of the batch and pilot experiments were in good agreement, which means that the preliminary batch experiments give good basis for the pilot and full-scale design process.

4. Theses

The two main groups of arsenic removal technologies are the adsorption and coagulation methods. Due to the special characteristics of the Hungarian subsurface waters (mostly the high phosphate concentration) many adsorbents, which were specially developed for arsenic removal and efficiently work abroad, do not work well in Hungary. Therefore in my research I focused on the applicability of the coagulation technology, whether this technique can sufficiently lower the arsenic concentration to the 10 μg/L standard value.

Figure 1 demonstrates the research areas and the applied methods of each thesis.

1st thesis: From the results of laboratory batch-scale and also pilot experiments I have concluded that during the design of an arsenic removal technology based on coagulation, it is not sufficient to consider the initial arsenic concentration only. Since the other pollutants (besides arsenic; mostly organic material, orto-phosphate and silicate, which also contact the coagulant) are present in one or even more orders of magnitude higher concentration than arsenic, the needed coagulant dose is dependent on the concentration of these compounds and not the arsenic concentration. When the technology is designed and the needed coagulant dose is determined, there are three levels in the planning process, which has to be carried out in the following order:

- **Modeling: the optimal coagulant dose is determined based on the raw water quality data**
- **Laboratory batch-scale experiments: in order to refine the optimal dose of chemicals (by using the results of the modeling step)**
- **Pilot-scale experiments: in order to refine the optimal dose of chemicals (by using the results of the laboratory batch-scale experiments), and to determine operational parameters.** [10, 13, 20]

The dissertation details these three technology-planning steps: the 5th thesis presents the results related to modeling. The relation between the laboratory-batch and pilot experiments is detailed in the 6th thesis. The 2nd, 3rd and 4th thesis detail the effect of alkalinity, orto-phosphate and silicate concentration on the arsenic removal efficiency based on laboratory batch-scale experiments
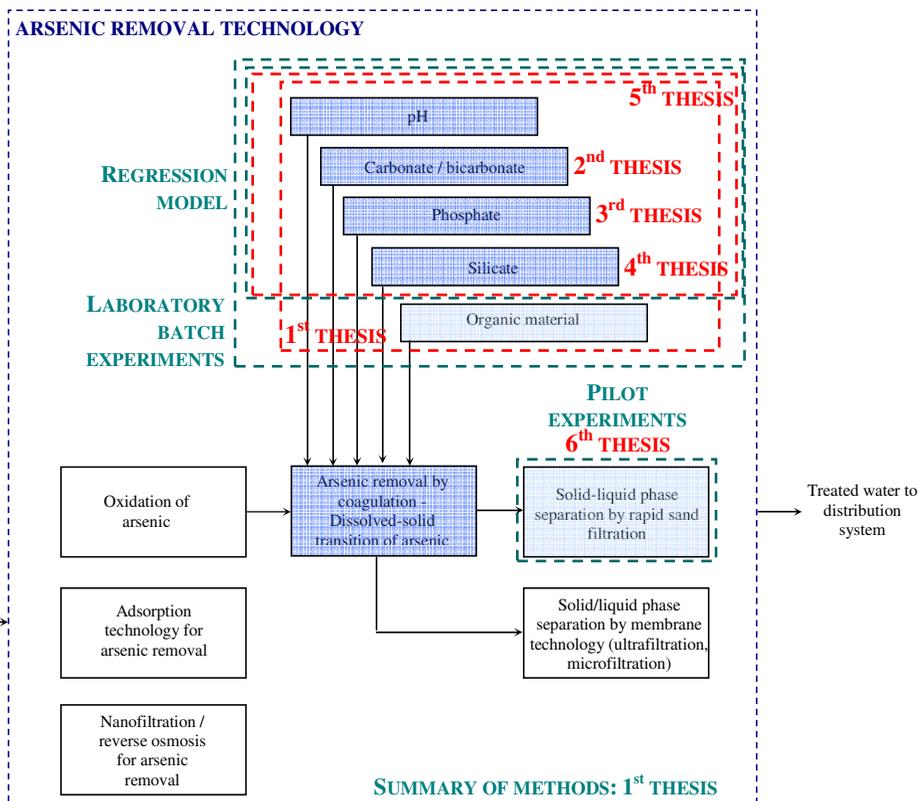
Figure 1 – The research areas and applied methods related to each thesis (blue colour marks those areas, with which the dissertation deals in detail, light blue marks those areas, which are also concerned in the dissertation, but not in detailed way)

In Hungary, due to the geological conditions, the buffering capacity of the waters is relatively high; it often reaches 10 meq/L. Therefore I have studied the effect of alkalinity on arsenic removal.

2nd thesis: Based on the results of laboratory batch-scale experiments I have concluded that with fixed coagulant dose and increasing alkalinity, the residual arsenic concentration increases. When 0.018 mmol/L (1 mg/L) Fe(III) coagulant dose in synthetic water made of distilled water was applied, 0.125 mmol/L HCO_3^- concentration was needed in order to achieve sufficient ferric-hydroxide floc formation, however above 1 mmol/L HCO_3^- concentration, the presence of hydrogen-carbonate

already increased the residual arsenic concentration. Based on my results, alkalinity has indirect effect on arsenic removal by influencing the final pH after coagulation. In case of higher alkalinity the final pH is higher, which results in decreased arsenic removal efficiency. In case of high initial pH values (> 8.0), and sufficient alkalinity (HCO_3^- = 5 – 10 mmol/L) the residual arsenic concentration can be 20 times higher compared to the residual arsenic concentration at low alkalinity (0.125 – 0.5 mmol/L HCO_3^-). [18, 19, 23]

The relatively high orto-phosphate concentration (0.8 mg PO_4 /L concentration is not uncommon) is a special characteristic of the Hungarian raw waters originating from the deep-confined aquifer. When adsorption technology is applied for arsenic removal, there is a competition between phosphate and arsenate for the adsorption sites; therefore often the adsorption technology is not able to remove arsenic efficiently. I carried out laboratory experiments in order to see to what extent orto-phosphate concentration can influence the other arsenic-removal technology, the coagulation.

3rd thesis: Based on the results of laboratory batch-scale experiments I have concluded that if coagulation technology is applied for arsenic removal, the presence of 0.15 mg/L PO_4-P already has significant effect on arsenic removal. If the water contains orto-phosphate, it is not sufficient to simply add more ferric-coagulant according to the number of moles of orto-phosphate (assuming that phosphate is precipitated in the $FePO_4$ form). In the presence of phosphate special Fe(III)-hydroxide precipitates are formed, with which arsenate cannot contact. The coagulant dose has to be increased in order to achieve a certain Fe(III) : PO_4-P [mg/mg] ratio, above which the arsenic removal can effectively take place. In case of model systems made by tap water or distilled water this ratio is around 10 mg Fe(III) / mg PO_4-P in the 7.5 – 8.0 pH range. [18, 19, 23]

Earlier researchers have concluded that silicate hinders the formation of ferric-hydroxide flocs with sufficient size, and moreover arsenic cannot contact with these flocs. In laboratory batch experiments I studied whether the negative effect of silicate can be compensated by increasing the coagulant dose.

4th thesis: When coagulation experiments are carried out with silicate and arsenate containing synthetic water, the size of the formed ferric-hydroxide flocs is mostly in the 0.2 μm – 0.45 μm range. By increasing the coagulant (ferric-chloride) dose in model systems made by distilled water, the 10 μg/L standard value for arsenic can be reached, however in the presence of 30 – 50 mg/L SiO_2 around 2.5 – 3.5 times higher coagulant dosages are needed compared to silicate-free systems. [19, 23]

5th thesis: After studying the effect of raw water quality parameters (pH, alkalinity, orto-phosphate and silicate concentration, coagulant dose) individually, linear regression analysis was carried out in order to see which are the most important influencing factors. The silicate concentration of the raw water and the ferric-chloride coagulant dose proved to be the most important factors. Than an equation was set up and verified, which is able to predict the residual arsenic concentration as a function of the orto-phosphate and silicate concentration of the raw water, the final pH and ferric-chloride coagulant dose in case of 50 – 60 μg/L initial arsenate concentration. In the verification step the modeled and measured arsenic concentration values were in good agreement, which means that the formula is able to predict the efficiency of arsenic removal by coagulation for a given raw water quality. [19, 20, 23]

Besides the investigation of the role of different water quality parameters in laboratory, pilot-scale experiments were carried out with natural, arsenic containing water, originating from deep confined aquifer. The role of the pilot experiments was to investigate important questions related to operation (filtration velocity, mixing), and moreover to see whether the results of the laboratory batch experiments are comparable to the results of the pilot experiments.

6th thesis: Based on the results of the pilot-scale experiments I have concluded that the traditional rapid sand filtration is able to remove the arsenic containing flocs efficiently (by applying 1-2 mm sand particles and 5.2 m/h filtration velocity) if proper mixing (fast mixing followed by slow mixing) is also applied. By excluding the flocculator tank, the operational time between two filter-backwashes decreased to the half. By carrying out simultaneous pilot and batch experiments with the same raw water I have concluded that the results of the batch and pilot experiments were in good agreement, which means that the preliminary batch experiments give good basis for the pilot and full-scale design process. [16]

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