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# Impact of MBR flux enhancer on floc size distribution, dewaterability and shear stability

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ABSTRACT

This study aims at a better understanding of the effects of flocculants and adsorbents on membrane bioreactor (MBR) mixed liquor. A total of 12 different additives (metal salts, chitosans, starches, synthetic polymers and PAC) were tested with regards to their impact on particle size distribution in activated sludge. Of further interest was the shear stability and dewaterability of sheared sludge. This was tested in a range of shear rates dominating in MBR. For most additives, a significant effect on the capillary suction time (CST) was observed. Most additives formed aggregates that were stable in the tested shear range (0–4000 $s^{-1}$). Nevertheless, only the tested chitosans and polymers were able to significantly increase the volume based particle size (up to 127%). In order to examine the long term effect of shearing on particle size three of the tested additives were surveyed in pilot plant experiments. Here the increase in particle size was only 17–18% for the tested polymers. In lab scale tests these polymers had caused an increase of approx. 50%.

***Keywords:*** Adsorption; Flocculation; Flux enhancer; MBR; PSD

## 1. Introduction

The number of membrane bioreactor (MBR) installations for municipal and industrial application has increased dramatically over the last decade [1]. Nevertheless, membrane fouling still restricts the wider application and better acceptance of MBR technology. Besides traditional strategies for fouling reduction like the optimization of hydrodynamic parameters in terms of an higher air scour, pulse pause operation, backflush or other, the addition of flocculants or coagulants to the activated sludge can enhance the filterability as shown in various publications [2–4].

According to theoretical models, the permeation flux in microfiltration is based on particle deposition and the back-transport from the membrane into the bulk [5]. From the Carman-Kozeny equation it can be seen that the particle size has a strong impact on the hydraulic diameter of the formed channels and thus on the specific resistance of the cake layer.

Kwon et al. evaluated the effect of different particle sizes on the cross-flow microfiltration of monodisperse dispersions of spherical polystyrene latex particles and found the critical flux decreased with decreasing particle size [6]. Similar results were also found in real MBR sludge where the filtration resistance increased with decreasing particle size [7]. By the addition of flocculant or adsorbent to MBR sludge it is thought to be possible to reduce fouling by forming larger particles or reducing the amount of the very small, colloidal matter.

In the recent past, several additives have been evaluated for their impact on activated sludge in MBR.

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For example the addition of activated carbon does not only result in lower membrane fouling, but may also positively affect the removal efficiency [3,8,9]. Satyawali and Balakrishnan [10] observed a less dense cake layer and a more uniform gel layer in a PAC added mesh equipped bioreactor. From literature studies they concluded that especially the ratio between PAC and sludge floc size has a strong impact on the efficiency of the PAC dosing.

Song et al. compared the impact of ferrum and alum dosing on the MBR performance [11]. While both additives improved the phosphorous removal and the filtration resistance, ferrum was not further tested as it strongly decreased the pH. Furthermore they observed a shift in the particle size distribution (PSD): the mean volume based particle diameter shifted from 45 μm for the untreated sludge to 57 μm if 30 mg/L alum were added to the sludge.

Wu and Huang [12] evaluated the effect of polymeric ferric sulphate (PFS) on the filtration performance in MBR. They attributed the enhanced filtration on the removal of supernatant organics and the enlargement of flocs (approx. 62 μm and 76 μm in the reference and the PFS added MBR, respectively).

Wu et al. investigated the effect of different monomeric and polymeric coagulants [13]. As polymeric coagulants offer more positive charges they found a higher removal of supernatant organic matter and a better enlargement of sludge floc size. This also resulted in a lower membrane fouling.

Another comparative study was conducted by Ji et al. [14]. Therefore the performance of aluminium sulphate, PFS and chitosan addition were evaluated in parallel MBRs. They found that the filtration enhancement was influenced by of the soluble and colloidal foulant removal and the flocculation efficiency. The specific filtration resistance $\alpha$ decreased with increasing particle size $d$ and decreasing fractal dimension $d_f$ (characterising the compactness of the floc).

Especially cationic polymers are very effective in flux enhancing by increasing the floc size and the porosity of the cake layer on the membrane as shown by Hwang et al. [15]. They studied the architecture of the filtration cake layer formed with and without the addition of a cationic polymer to the MBR. Even though the cake of the cationic polymer added reactor was thicker than in the reference it was more even and porous. The extracellular polymeric substances (EPS) concentration was significantly lower in the supernatant but higher in the filter cake when the polymer was added. Also the mean particle size was significantly higher: 179 μm in comparison to 10 μm in the control. By adding the cationic polymer the fouling was effectively decelerated.

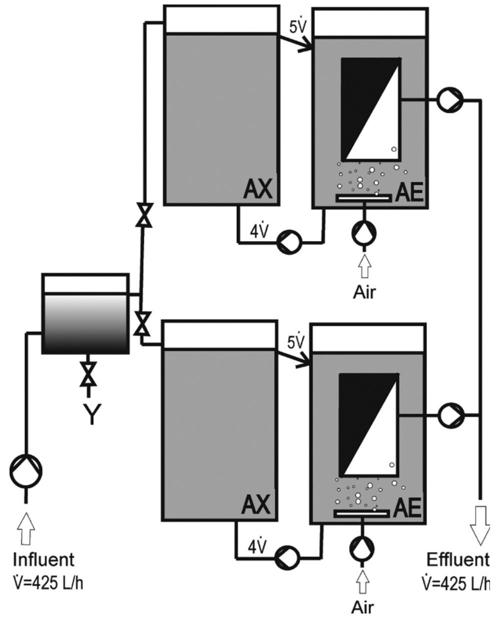


Fig. 1. Flow sheet of the parallel pilot plants.

As can be seen from the literature, the stability of the formed flocs strongly depends on pH, concentration of the flocculant, molecular weight, charge density and several other system parameters [16]. Especially hydrodynamic forces like shear forces play an important role in the floc stability [17]. For the long term effectiveness of a flocculant it is therefore important to know the shear stability of the formed aggregates.

In the presented study, 12 different additives (natural and synthetic polymers, activated carbons and metal salts) were tested for their impact on MBR activated sludge particle size. The PSD was determined for the reference sludge and the treated sludge (prepared in accordance with [2]) for different shearing rates. In order to evaluate the dewaterability, the capillary suction time (CST) was determined for the treated and untreated sludge exposed to different shearing rates. The results of these lab scale tests were then compared to findings in a pilot scale system.

## 2. Material and methods

### 2.1. Pilot plants

In order to investigate the effect of flux enhancing chemicals in MBRs, two identical pilot plants have been set up (Fig. 1). Each plant consists of two 1 $m^3$ tanks with a working volume of approx. 0.8 $m^3$. The pilot units are located in a 20′ sea container on the grounds of a pumping station of the Berliner Wasserbetriebe thus drawing combined municipal wastewater from Berlin city centre as influent. After the settler used

Table 1
Tested additives and their optimal dosing concentration (according to previous works [2,18]).

| Substance class | Producer | Abbreviation | Product | $c_{opt}$ ($mgL^{-1}_{MLSS}$) |
|---|---|---|---|---|
| Metallsalt | Ciba | PACl | Magnasol 5108 | 100 |
| | Merck | $FeCl_3$ | – | 85 |
| Chitosan | France Chitine | C-1 | Chitosan 221 | 200 |
| | France Chitine | C-2 | Chitosan 652 | 250 |
| Activated carbon | Norit | PAC-1 | SA Super | 450 |
| | PICA | PAC-2 | Picahydro LP 27 | 5000 |
| Polymer | Nalco | Poly-1 | MPE-50 | 500 |
| | Kurita | Poly-2 | MP L 30 | 500 |
| | Adipap | Poly-3 | Adifloc KD 451 | 70 |
| | Adipap | Poly-4 | Adifloc KD 452 | 70 |
| Starch | Rhodia | Sta-1 | Jaguar C162 | 300 |
| | Tate&Lyle | Sta-2 | Mylbond 168 | 1500 |

as a sand trap for the removal of larger particles, the wastewater flows into a stirred anoxic chamber. The following tank is aerated and equipped with a 22 $m^2$ membrane module (A3 Water Solutions, Germany, PVDF, nominal pore size 0.2 μm). TMP, flux, DO, T and pH in the membrane chamber are registered on-line. The systems have been in operation since October 2006. While different flocculants were dosed in one system, the other served as a reference.

The chemical oxygen demand (COD) and total nitrogen (TN) concentrations of feedwater were around 780 and 95 mg/L, respectively.

The hydraulic retention time (HRT), sludge retention time (SRT) and aeration rate were 7-8 hours, 13 days, and 17 $m^3/h$, respectively.

### 2.2. Tested additives

Table 1 summarizes information about the additives used. The optimal dosage was determined on the basis of SMP removal and filterability in previous tests [2,18]. Out of 30 initially tested additives, 12 additives were chosen for this study. The selection was done according to SMP removal efficiency. In addition, at least one chemical from each category (metal salt, chitosan, activated carbon, synthetic polymer and starch) was chosen in order to evaluate different physiochemical effects. For the starches and the chitosans a special preparation was needed. Starch was dissolved in boiling water and cooled down before use. The chitosan flocs or powder were wetted with distilled water and then dissolved by adding lactic acid. This solution was prepared 2 or 3 days before the experiments took place in order to have a highly activated chitosan solution. All other chemicals were dissolved/wetted in 50–60 mL distilled water in the respective weights and added to 5 L sludge. After strong initial mixing the solution was given at least 1 h time for reacting and/or the formation of larger flocs. During this time the solution was only mixed moderately from time to time.

### 2.3. Shearing of the sludge

Activated sludge was freshly sampled daily from the reference pilot (without additive dosing) described above. Each day the untreated reference mixed liquor was measured in comparison to mixed liquor spiked with one of the additives shown in Table 1. The samples were then sheared at defined shear rates between 0 and 4000 $s^{-1}$ for 5–60 min in a rotational viscometer (Type Viscotester VT 550, Haake GmbH, Karlsruhe, Germany) with a gap width of 0.35 mm. Approx. 10 mL of sludge were sheared in the viscometer. From own measurements and approximations a representative shear rate in a plate and frame module can be expected to be in the range of 2000 $s^{-1}$. The temperature of the mixed liquor was between 24°C and 29°C with a temperature of 26°C for most samples. The values presented here are not temperature corrected.

### 2.4. Measurement of PSD

For each shear rate the PSD was measured in a Mastersizer S (Malvern Instruments GmbH, Herrenberg, Germany) with sample presentation unit, pump, stirrer and UV for a more homogenous mixture of the sample. A 300RF lense was used, allowing the measurement of particle sizes ranging from 0.05 to 900 μm. The PSDs were evaluated based on volume and number of particles.

*2.5. Measurement of CST*

The dewaterability of the different mixed liquor samples was evaluated measuring the CST. A Triton Model 200 was used (Allied Colloids GmbH, Hamburg, Germany) with CST papers and cartridge for good dewaterable sludge. The CST was measured for the unsheared sample and mixed liquor sheared at 4000 $s^{-1}$ for 5 min.

## 3. Results

*3.1. Shearing tests at lab scale*

Table 2 shows the CST, viscosity and medium particle size for different shearing rates. The increases for the most significant parameters were calculated for the treated mixed liquors in comparison to the reference.

*3.2. Impact on CST*

From Table 2 it can be seen that most additives have a positive effect on the dewaterability of the mixed liquor. This improvement did not always correlate with an increase in particle size. This might be due to effects on other sludge characteristics as, e.g. the fractal dimension $d_f$. This parameter is used to describe the morphology of the sludge floc. The higher the fractal dimension the more spherical and compact are the sludge flocs, while a low value refers to a more loose and linear structure. The fractal dimension was not evaluated in this study.

It should be noted that the manufacturer of the CST apparatus generally states that the accuracy of this measurement is not very high and only a deviation of more than 20% is significant. Nevertheless, the agreement for the duplicate samples was generally very good (medium standard deviation of 5%). Shearing at 4000 $s^{-1}$ for 5 min decreased the CST in most cases slightly, for PAC-2 and Poly-4 the deviation was higher with about 33% and 26%, respectively.

*3.3. Impact on particle size*

The PSD and the medium particle size (volume based distribution) were generally not or only slightly changed if shear stress was applied. Except for Poly-4 and Sta-1 were a 17% and 13% reduction in the mean volume particle size was observed. It should be noted that the sheared sludge volume was quite small (10 mL). Replicate measurements have therefore been conducted, which did not show any significant differences.

While $FeCl_3$ and alum were not effective for the formation of larger flocs at the applied concentration, especially the tested polymers and chitosan strongly increased the volume based particle size. Chitosan C-1 had the strongest effect with a median volume based particle size of 143 μm. It was observed that the chitosan flocculated sludge tended to form bacillary flocs that were oriented in the rotating direction of the viscometer at lower shear rates. These may form a quite dense layer on the membrane. At higher shear rates this phenomenon was not so distinctive probably due to a brake up of the rod-shaped flocs. These results are also in accordance with literature, where a strong increase in particle size was observed for chitosan added MBRs [14,20]. It was also found that the fractal dimension was strongly decreased if chitosan was added indicating that the flocculated sludge has a more loose and linear structure [14]. This also agrees with the bacillary flocs observed in this work and the strong decrease in CST.

The tested activated carbons did not change the volume based PSD. These findings are in accordance with Satyawali and Balakrishnan [10], who stated that the enlargement of flocs by PAC addition is generally given, if the PAC offer a support for biofilm growth (size of PAC particles is in the range of the sludge or larger). The PAC used in this study is in the range of 8–35 μm and thus much smaller than the sludge flocs.

Oppositional to the volume based results are the data for the number based distributions. Except the activated carbons and the starches the additives did not increase or even decreased the mean number based particle size. This is also shown in Fig. 2b and d. As literature generally refers to an increase of the volume based particle size if a flocculant is added [11,15,20] it should be discussed critically which distribution is of more significance for the fouling in MBR.

*3.4. Impact on viscosity*

The dynamic viscosity of all samples decreased with increasing shear rate as expected, due to the pseudoplastic rheology of activated sludge. The viscosity measured at 718 $s^{-1}$ was either decreased (up to 46%) or increased (up to 13%) if the sludge was spiked with an additive. These differences were diminished at the higher shearing rate of 4000 $s^{-1}$, where the deviation was only up to $\pm 8\%$.

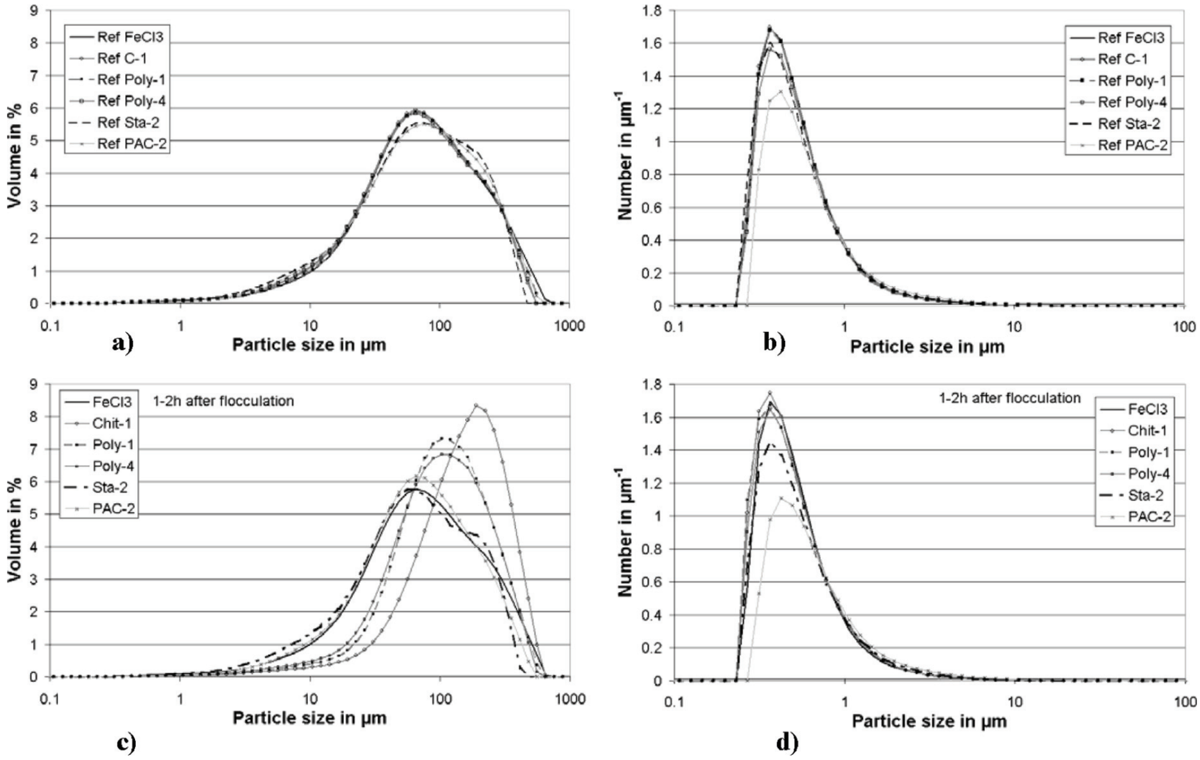


Fig. 2. Particle size distribution for selected additives and respective reference sludges without shearing: (a) and (c) volume based, (b) and (d) number based.

### 3.5. Volume versus number based PSD

The volume and number based PSDs for unsheared activated sludge samples with and without the addition of additive is shown in more detail in Fig. 2a–d. Although these measurements were conducted within 10 days, the PSDs for the reference activated sludge changed slightly from day to day. Especially for the last two measurements (Sta-2 and PAC-2) the reference sludge showed a different PSD due to problems in plant operation.

As was already indicated by the mean particle sizes in Table 2 the volume based PSD did not shift to larger particles if a metal salt, PAC or a starch was added to the mixed liquor. Naturally the total volume of particles increased if PAC was added. The addition of a cationic polymer or the chitosan shifted the volume based PSD to larger particle sizes. If the results are evaluated according to the number based PSD the results are different. As small particles in the range of 0.1–10 µm can be found in a high number in activated sludge but make up only a very small amount of the total volume, this region is better resolved in the number based PSD. Also this fraction is thought to be mainly responsible for fouling as it is in a similar range than the membrane pores in microfiltration. Fig. 2d clearly shows that the tested polymers and the $FeCl_3$ do not have an influence in this region. Naturally the number of small particles is reduced if an activated carbon is added, as can be seen in case of PAC-2. Also Sta-2 causes a decrease of the number of small particles.

### 3.6. Impact of shear rate on PSD

In Fig. 3 the impact of different shearing rates and times is shown for activated sludge flocculated with

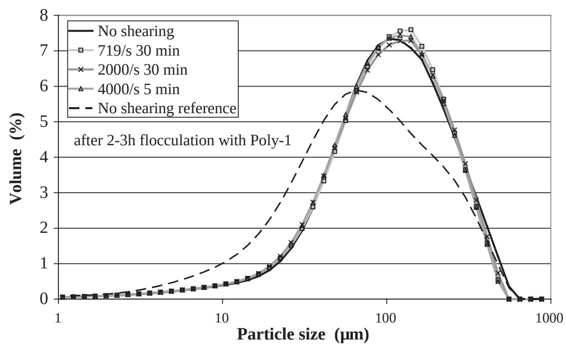


Fig. 3. Impact of shearing on PSD for Poly-1 flocculated mixed liquor.

Table 2
Effect of additive on CST, dynamic viscosity and medium particle size for 5 min shearing at rates of 0, 718 and 4000 $s^{-1}$.

| | CST0 (s) | Increase (%) | CST4000 (s) | μ718 (mPa s) | μ4000 (mPa s) | d(v 0.5) 0 (μm) | Increase (%) | d(v 0.5) 4000 (μm) | d(n 0.5) 0 (μm) | Increase (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| Ref | 50 | | 51.5 | 6.02 | 3.18 | 66.8 | | 66.2 | 0.628 | |
| FeCl3 | 43.8 | 12 | 50.5 | 5.07 | 3.28 | 66.9 | 0 | 67 | 0.629 | 0.2 |
| Ref | 40.2 | | 44 | 3.88 | 2.87 | 65.5 | | 64.2 | 0.629 | |
| PACl | 40.7 | −1 | 39.6 | 4.14 | 2.85 | 65.1 | −1 | 65.2 | 0.625 | −0.7 |
| Poly-1 | 10.7 | 73 | 12.5 | 4.41 | 2.9 | 100.6 | 54 | 96.22 | 0.616 | −2.2 |
| Ref | 39.9 | | 40.7 | 3.84 | 2.99 | 63.3 | | 61.5 | 0.627 | |
| C-1 | 10.6 | 73 | 10.6 | 2.07 | 3.24 | 143.6 | 127 | 135.8 | 0.601 | −4.4 |
| C-2 | 13.7 | 66 | 15.4 | 4.18 | 2.93 | 78.6 | 24 | 80.2 | 0.594 | −5.5 |
| Ref | 43.2 | | 47.1 | 4.32 | 3.1 | 63.1 | | 62.4 | 0.661 | |
| Poly-2 | 24.6 | 43 | 24.5 | 3.87 | 2.93 | 65.4 | 4 | 64.5 | 0.649 | −1.7 |
| Poly-3 | 11.4 | 74 | 12.05 | 3.06 | 2.86 | 88.5 | 40 | 84.2 | 0.627 | −5.4 |
| Poly-4 | 12.9 | 70 | 16.3 | 3.49 | 3.01 | 93.9 | 49 | 77.5 | 0.628 | −5.3 |
| Ref | 33.3 | | 33.3 | 2.97 | 2.69 | 64.9 | | 62.5 | 0.656 | |
| Sta-1 | 24.1 | 28 | 22.6 | 3.17 | 2.75 | 70.1 | 8 | 61 | 0.688 | 4.7 |
| Sta-2 | 26.1 | 22 | 23.9 | 3.36 | 2.74 | 58.5 | −10 | 57.8 | 0.712 | 7.9 |
| Ref | 33.1 | | 31.4 | 3.06 | 2.76 | 67.7 | | 67 | 0.821 | |
| PAC-1 | 31.8 | 4 | 36.1 | 2.77 | 2.54 | 68.5 | 1 | 68.6 | 0.839 | 2.2 |
| PAC-2 | 24.3 | 27 | 32.3 | 3.22 | 2.73 | 63.2 | −7 | 62.4 | 0.918 | 10.6 |

Poly-1. The impact of shearing time on PSD of the reference sludge was negligible for shearing rates of 719 $s^{-1}$ and 2000 $s^{-1}$ and times between 1 and 20 min (data not shown). Here no significant change in PSD was found for all tested shearing rates.

In Fig. 3 it is obviously shown that even for shearing times of 30 min and rates of 2000 $s^{-1}$ the distribution is not changed significantly. The formed aggregates seem to be quite stable, at least in the shearing range that can be expected in plate and frame modules.

### *3.7. PSD vs. filtration enhancement*

It should be noted that previous studies have shown results for the filterability that do not correlate directly with the PSD data presented in this work. Koseoglu et al. [2] evaluated the filterability and the critical flux for activated sludge that was spiked with additives that were also used in this work. They found a good filterability and a strong increase in the critical flux value if a cationic polymer was added to the sludge. These results are also confirmed by the strong increase in mean particle size observed in these experiments. When alum or ferric chloride was added to the mixed liquor the filterability was not improved considerably, which also correlates with the fact that $FeCl_3$ and alum did not change the PSD significantly. It is quite surprising that the addition of Chit-1, that resulted in very large flocs in this work, did not change the critical flux in the work presented by Koseoglu et al. As reported above, chitosan formed bacillary flocs in the shearing direction and these may cause problems on the membrane. Generally it is assumed that larger flocs form a more porous filter cake on the membrane. The contrary might be the case for the rod-shaped flocs formed under shear if chitosan was added.

### *3.8. Long term tests in pilot plant*

In the pilot plant shown in Fig. 1 the additives Poly-1, Poly-4 and Sta-2 were tested over a period of several weeks in order to understand their effects on the sludge, elimination characteristics and membrane fouling. These additives were selected due to positive effects on filterability and mixed liquor supernatant composition found in lab scale batch trials. Furthermore, they had no toxic effects on the biomass as reported in previous studies [2,19,21]. Further information concerning this pilot plant study can be found in [22].

Table 3 shows the mean volume based particle size during the three measurement campaigns with different additives. While Poly-1 and Poly-4 showed a 50% increase in mean particle size during the short term tests (Table 2) the increase in mean particle size was

Table 3
Increase of particle size in pilot plant.

| | d(v 0.5) | Increase (%) |
|---|---|---|
| Reference | 52.5 | |
| Poly-1 | 61.5 | 17.1 |
| Reference | 67.7 | |
| Sta-2 | 72.1 | 6.5 |
| Reference | 89.4 | |

only 17.1% and 18.6% in the pilot plant for Poly-1 and Poly-4, respectively. A different trend was observed for the tested starch. While it reduced the particle size about 10% in lab tests it caused a 6.5% increase in particle size in the pilot plant.

These results make clear that a transfer of results obtained from small scale, short term tests to a pilot system is not possible. This was also found in previous study with regards to permeability and cleaning effects [23]. Although shearing showed no strong impact on the particle size in lab scale tests for Poly-1 (Fig. 3) it might be one of the causes for the lower increase in particle size with additive in the pilot plant. Poly-4 treated sludge on the other hand already showed some dependency on the shear rate in lab tests. The shear stress for these trials was approximated for the shearing that might be expected in a plate and frame module. Nevertheless further shearing occurs in a full MBR system due to pumps, stirrers, tubing, etc. Furthermore the dosing location, plant geometry and influent composition have a large impact on the particle size. These factors were not accounted for in the lab tests.

## 4. Conclusions

A total of 12 different additives (metal salts, chitosans, starches, synthetic polymers and PAC) were tested with respect to their impact on PSD in activated sludge. Several of these additives effectively increased the particle size, especially the tested chitosans and polymers, which increased the volume based particle size up to 127%. Ferric chloride, the tested alum and the activated carbons did not change the particle size significantly. Nevertheless, except the tested alum and one of the tested PACs all additives increase the dewaterability of the sludge. It can therefore be concluded that other factors besides the particle size play an important role for the dewaterability.

Shearing at 4000 $s^{-1}$ for 5 min decreased the CST in most cases slightly, for PAC-2 and Poly-4 the deviation was higher with about 33% and 26% respectively.

Also the PSD and the medium particle size (volume based distribution) were generally not or only slightly changed if shear stress was applied, except for Poly-4 and Sta-1 where a 17% and 13% reduction in the mean particle size was observed.

The long term tests in the pilot plant did not show a comparable increase in volume based particle size if a chemical was added there. While both tested polymers showed an increase in particle size of around 50% in lab tests, the increase was only 17–18% in the pilot plant. These results make clear that a transfer of small scale, short term tests to a pilot system is not possible. The wastewater influent composition, plant geometry, dosing strategy and additional shear stress due to pumps and mixers might be an explanation for this.

As the number and volume based PSD give strongly different results concerning the effectiveness of an additive, both should be evaluated in order to have more complete information.

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