Improvement of monitoring of tertiary filtration with particle counting

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Abstract Nowadays filtration processes are still monitored with conventional analyses like turbidity measurements and, in case of flocculation–filtration, with phosphorus analyses. Turbidity measurements have the disadvantage that breakthrough of small flocs cannot be displayed, because of the blindness regarding changes in the mass distributions. Additional particle volume distributions calculated from particle size distributions (PSDs) would provide a better assessment of filtration performance.

Lab-scale experiments have been executed on a flocculation–filtration column fed with effluent from WWTP Beverwijk in The Netherlands. Besides particle counting at various sampling points, the effect of sample dilution on the accuracy of PSD measurements has been reflected. It was found that the dilution has a minor effect on PSD of low turbidity samples such as process filtrate. The correlation between total particle counts, total particle volume (TPV) and total particle surface is not high but is at least better for diluted measurements of particles in the range 2–10 μm. Furthermore, possible relations between floc-bound phosphorus and TPV removal had been investigated. A good correlation coefficient is found for TPV removal versus floc-bound phosphorus removal for the experiments with polyaluminiumchloride and the experiments with single denitrifying and blank filtration.

Keywords Flocculation; fractionation; multimedia filtration; particle counting; phosphorus removal; tertiary filtration

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Introduction

Further removal of particles from wastewater will be of growing interest for reducing the environmental impact of the discharges of effluent (Graaf, 2001). Almost all wastewater treatment plants (WWTPs) in The Netherlands have been improved in order to remove phosphorus and suspended solids to a very low level. A typical quality of the effluent after conventional activated sludge treatment is given with $P_{tot} < 2$ mg/L and SS < 10 mg/L, while the (future) standards are aiming at $P_{tot} < 0.15$ mg/L. Therefore, tertiary filtration (discontinuous or continuous) chemically enhanced for phosphorus and suspended solids removal receives more attention in pilot investigations as well as practical applications.

Monitoring of the filtration process and performance generally occurs with turbidity measurements. Turbidity is a quantitative measure of remaining un-dissolved solids. It is generally used to indicate malfunctions within the treatment process. It is typically determined using 90° scattered light principle in compliance with EN ISO 7027 (ISO, 1999).

By passing optical radiation through a dispersing system, dispersed solids reduce radiation power by transforming it into another form of energy. This effect is called absorption. The ratio of penetrating to emitting radiation is measured as turbidity. The measuring principle already explains the disadvantage of this analysis: turbidity is an overall parameter of the analyzed sample; mass distributions of particles are not displayed.

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Particle analyses on the other hand can provide, as a qualitative measure, particle mass distributions. The two techniques of light extinction (light blocking) and light scattering are available for particle counting (Hargersheimer and Lewis, 1998). This article focuses only on measurements conducted with the light extinction principle. Light extinction devices measure the change of light intensity caused by a particle as it passes through a light beam. The light intensity is transformed into a voltage pulse which can be related to particle size. The result is a report of the number of particles according to size through data acquisition, as the particle size distribution (PSD) (Lawler, 1997). With the assumption of spherical particles in the sample a particle volume distribution (PVD) can be calculated based on the PSD.

Nowadays particle analyses in water and wastewater treatment are mainly used for assessment of malfunction in membrane filtration systems. In the last decade research regarding wastewater treatment had been conducted to evaluate the surplus value of particle counting for the monitoring of filtration performance and in relation to other measured parameters in which the relation to bacteria counts predominates.

Kobler and Boller (1997) compared the results of the particle analysis conducted for different tertiary wastewater filtration systems (deep bed, cloth drum, continuous up-flow and cell filters) to other quality parameters, which are related to small particles such as microorganisms and heavy metal concentrations. A relation between the removal of small size particles ($< 8\,\mu m$) and bacteria counts as well as copper was found. Chavez *et al.* (2004) studied the correlation between the number or volume of particles and the concentration of fecal coliforms, Salmonella and helminth ova in jar tests using coagulation–flocculation for raw and chemically treated wastewater.

However, the correlation with the general monitored parameter turbidity seems an important issue as an indicator of the present concentration of suspended solids.

Particle counting is influenced by different factors: faults, such as operational errors or user inaccuracy, the particle destroying effect of the analytical method itself and the concentration of particles in the sample. In the case of high concentration, particles can be overlooked behind others while passing the measuring cell. Or, small particles next to each other in a short distance can be analysed as larger compounds. Effluent from a wastewater treatment contains a large amount of particles of different origin in various sizes. Therefore dilution might be an option to gain more representative measurements.

In the evaluation of filtration performance the calculation of the total particle volume (cumulative particle volume) removal can be suitable. This calculation indicates the suspended solids removal efficiency based on particle analyses. On the other hand the removal of particle-bound phosphorus also represents a kind of filtration performance. In the sense that suspended solids concentration includes particle-bound phosphorus, a relationship between the two filtration performance indicators is expected.

In order to investigate these ideas, dual media filtration experiments at lab-scale had been executed in co-operation with the Delft University of Technology and the water board Hoogheemraadschap Hollands Noorderkwartier at the WWTP Beverwijk in The Netherlands (Miska and Broek, 2005). The effluent of this treatment plant is rather typical for a modern wastewater treatment plant in The Netherlands.

Methods and materials

A multimedia lab-scale (inner diameter 12.9 cm; lower layer: quartz sand Ø 0.8–1.25 mm, height 40 cm; upper layer: anthracite Ø 1.6–2.5 mm, height 80 cm) filtration installation was operated at filtration rates of 10 and 20 m/h, using iron(III) chloride (dosing: 3–4 mgFe/l) and polyaluminiumchloride (dosing: 2–3 mgAl/l) for flocculation. The installation had also been used for blank filtration (without dosing),

single denitrification and simultaneous denitrifying flocculation–filtration to investigate the influence of the biomass on the removal performance of phosphorus and suspended solids (Miska *et al.*, 2006). Table 1 gives a summary of the conducted experiments during the research period.

During the experiments orthophosphate analyses of grab samples from three sampling points, WWTP-effluent (EF), flocculated WWTP-effluent (AF) and process filtrate (FI), have been conducted, on two fractions: first, the raw sample; and secondly, over a 0.45 μm cellulose acetate filter pre-filtered sample, to investigate the actual concentration of floc-bound and dissolved phosphorus. In the preparation of the raw sample for the orthophosphate analyses the sample is treated with sulphuric acid. In that sense, the particle-bound phosphorus will be dissolved again and analysed as orthophosphate. Comparison between the phosphorus concentration in the raw sample and pre-filtered sample provides the concentration of particle-bound phosphorus.

Turbidity measurements of grab samples of the three sampling points took place with a HACH turbidimeter.

Furthermore, PSD measurements have been executed for the raw and diluted samples EF, AF and FI, with the dilution rate of 3 (dilution ratio: sample × demineralised water = 1 × 2), using a MetOne PCX particle counter with measuring range 2–100 μm counting particles in 0.5 μm increments. The used sensor is based on the light extinction principle with a particle concentration upper limit of 10,000 per millilitre. This concentration limit is that number of particles at which 90% will go through the sensor individually and 10% will be coincident.

With the PSD, particle volume distributions (PVDs) and cumulative particle volume (CPV) have been calculated per increment under the assumption of spherical measured

Table 1 Process operation during research period

Period	Operation	Process parameter
22/06/04 – 26/06/04	Clean bed filtration	Filtration rate 20 m/h (262 l/h); backwash with water and air
29/06/04 – 12/07/04	Clean bed filtration	Filtration rate 10 m/h (131 l/h); backwash with water and air
29/07/04 – 10/08/04	Flocculating filtration	Filtration rate 10 m/h (131 l/h); $FeCl_3$ dosing 3–4 mgFe/l; backwash with water and air
23/08/04 – 27/09/04	Flocculating filtration	Filtration rate 10 (131 l/h) or 20 m/h (262 l/h); $FeCl_3$ dosing 3–4 mgFe/l or PAX dosing 2–3 mgAl/l; backwash with water and air
28/09/04 – 12/10/04	Denitrifying filtration	Filtration rate 6.5 m/h (90 l/h); methanol dosing 240 ml/h and 180 ml/h; backwash with water and air
13/10/04 – 01/11/04	Denitrifying flocculating filtration	Filtration rate 10 m/h (131 l/h); methanol dosing 180 ml/h; $FeCl_3$ dosing 2–4 mgFe/l; backwash with water and air
02/10/04 – 09/11/04	Denitrifying flocculating filtration	Filtration rate 10 m/h (131 l/h); methanol dosing 180 ml/h; PAX dosing 2–3.5 mgAl/l; backwash with water and air
10/11/04 – 11/11/04	Denitrifying filtration	Filtration rate 10 m/h (131 l/h); methanol dosing 90 ml/h; backwash with water and air
12/11/04 – 18/11/04	Flocculating filtration	Filtration rate 10 m/h (131 l/h); $FeCl_3$ dosing 3–4 mgFe/l; backwash with water and air
22/11/04	Blank filtration	Filtration rate 10 m/h (131 l/h); backwash with water and air

particles. Furthermore, the calculated total particle counts (TPC), total particle volume (TPV) and total particle surface (TPS) are also used in this article.

The results of diluted samples had been re-calculated to undiluted measurement with extraction of the eventually occurring particles in the demineralised water and inclusion of the applied dilution rate.

Results and discussion

Dilution experiments

The influence of dilution was examined by many measurements with raw and diluted samples. Figures 1 and 2 show the PSD and PVD analysis for raw and diluted EF and FI while the particle concentrations in the raw samples are below the limit of 10,000 particles per millilitre (total particle counts TPC: $TPC_{EF,raw} = 7{,}760$ counts/ml, $TPC_{FI,raw} = 1{,}871$ counts/ml). Particle counts and volume calculations are shown only for particles between 2 and 30 μm because particles in this range are generally present in the process filtrate. From there it can be concluded that the concentration of small particles between 2–10 μm increases clearly in diluted PSD measurements of the EF. Measurements of the FI containing a lower concentration of particles show almost no difference between PSD of the raw and diluted sample.

Starting from the fact that with sample dilution the concentration of particles between 2 and 10 μm increases, the rise of total particle counts between 2 and 10 μm had been calculated for the three samples EF, AF (flocculated WWTP-effluent) and FI. In total, 41 particle analyses were conducted on the raw and their diluted samples. In nine measurements (containing samples EF and AF) the particle concentration exceeded the concentration limit in the raw sample, for 13 measurements (containing samples EF and AF) the concentration limit was exceeded only for the diluted samples re-calculated to undiluted measurements and in 19 measurements (containing mainly samples FI) the total counts in the raw samples occurred below the limit. The results for different particle concentrations in the samples, partly above and below the concentration limit of the particle counter (10,000 counts/ml), are shown in Table 2.

Even if the concentration limit in the raw sample is not reached, sample dilution can result in a rise of a maximum of 40% of particle counts between 2–10 μm. In the cases of measurements exceeding the concentration limit, the counts in that size range can rise by a maximum of 56%. This strongly indicates the necessity of dilution.

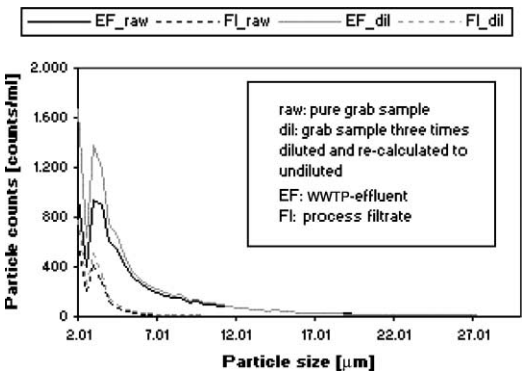

Figure 1 PSD for raw and diluted EF and FI

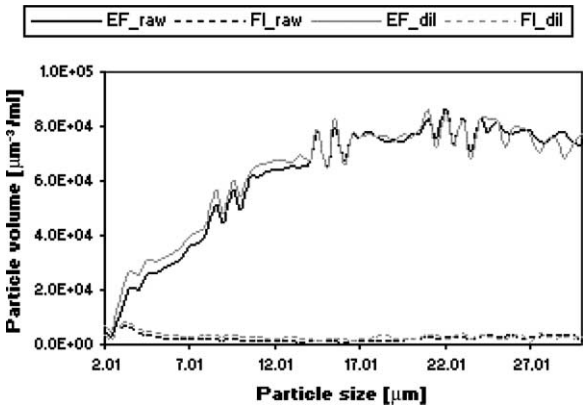

Figure 2 PVD for raw and diluted of EF and FI

Particle volumes versus numbers

TPC and TPV for the raw and diluted measurements of the 41 particle measurements were related to each other for the measuring range 2–100 μm. A linear correlation can be expected if particles are counted accurately, the idealization of spherical particles in the samples is appropriate and, most importantly, if the same particle mass distribution can be expected for each of the three samples EF, AF and FI. The correlation coefficient of TPC versus TPV is, for EF in the raw measurements, higher than in the diluted (Figure 3), contradictory to AF and FI (see in Figures 4 and 5).

The highest correlation is shown for FI, where, accordingly, coherence between TPC and TPV exists. But especially for the samples containing higher particle concentrations (EF and AF) a reasonable correlation between TPC and TPV cannot be found. Due to the differences in particle size distributions TPV may be a better parameter for further than TPC; especially in the examination of particle removal processes.

Relation with turbidity

Until now turbidity measurements have generally been related to TPC. But the light scattering not only depends on the concentration of particles but also on their total surface or volume. Therefore, the relation of TPC and TPS versus turbidity, measured in NTU, is investigated. Figures 6 and 7 show the relations of turbidity and TPC/TPS for raw and diluted measurements of particles between 2 and 100 μm over all samples EF, AF and FI, representing high turbidity samples (2.5–9.6 NTU) and low turbidity samples (0.7–3.3 NTU). The coefficient of the linear correlation for TPC versus turbidity appears to be higher than for TPS versus turbidity.

A closer look has to be given to the correlations TPC/TPS and also TPV versus turbidity between the particle size range 2–100 μm and 2–10 μm for all three samples EF, AF and FI, shown in Table 3. The correlation coefficients appear to be highest for the particle size range 2–10 μm in the diluted measurements.

Table 2 Rise of number of particles 2–10 μm in re-calculated diluted samples relative to raw samples, in [%]

Sample	>10,000 counts/ml in raw sample	<10,000 counts/ml in raw sample
EF	40.87–45.34	25.07–40.12
AF	35.83–55.76	17.37–39.62
FI		4.93–34.71

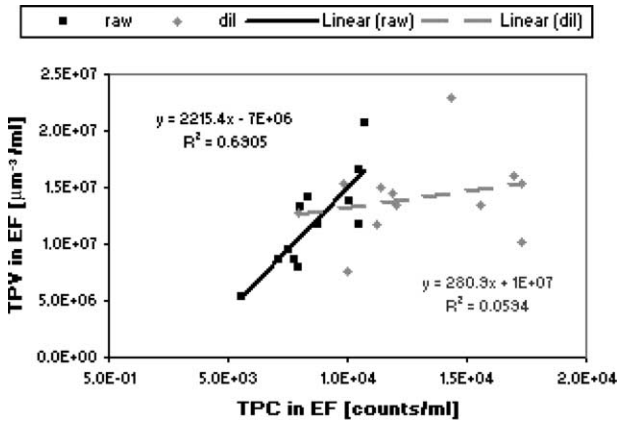

Figure 3 Correlation between TPC and TPV in EF (2–100 µm) for raw and diluted measurements

That leads us to the assumption that sample dilution is appropriate in order to obtain a more representative particle count in that lower size range. It can also be indicated that turbidity is mainly caused by particles $< 10\,\mu m$. In regard to the dependency of turbidity, TPC gives the highest correlation followed by TPS and TPV.

Nevertheless, the relation of TPC, TPS or TPV versus turbidity is valued as weak, which implies the necessity of additional particle analysis besides turbidity measurements.

Relation with phosphorus

Furthermore, the relation of floc-bound phosphorus as a part of the suspended solids and particle volume is investigated by the comparison of their removal performances. A filtration performance can be determined on the one hand from the cumulative particle volume removal and on the other hand from the floc-bound phosphorus removal, both calculated from the difference between the concentration in AF and FI. The sample AF indicates here the flocculated WWTP-effluent that is actually the process influent (especially in the case of coagulant-dosing) and therewith the reference of the ingoing floc-bound phosphorus and particle volume concentration.

The comparison of the two performances, distinguished regarding different operational conditions, provides linear correlation coefficients for TPV (2–100 µm) versus floc-bound phosphorus removal of

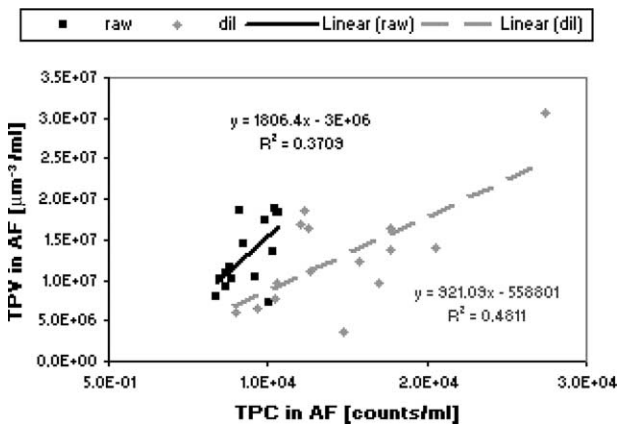

Figure 4 Correlation between TPC and TPV in AF (2–100 µm) for raw and diluted measurements

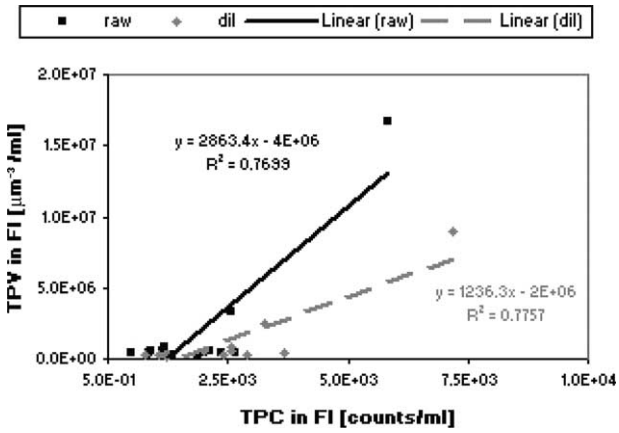

Figure 5 Correlation between TPC and TPV in FI 1

- $R^2 = 0.854$ in the experiments using PAX (including single flocculation–filtration and simultaneous denitrifying flocculation–filtration),
- $R^2 = 0.300$ in the experiments with $FeCl_3$ (including single flocculation–filtration and simultaneous denitrifying flocculation–filtration), and

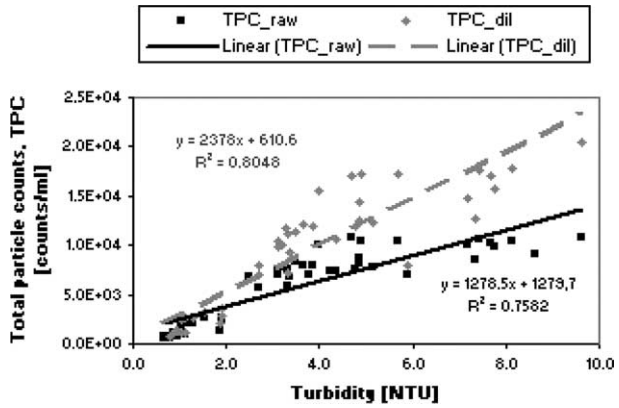

Figure 6 Relation between turbidity and TPC 2–100 µm of raw and diluted measurements

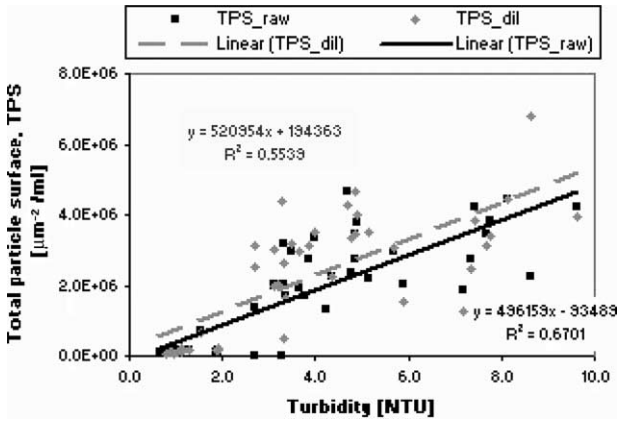

Figure 7 Relation of turbidity and TPS 2–100 µm of raw and diluted measurements

Table 3 Linear correlation coefficients for TPC/TPS/TPV for particles 2–100 μm and 2–10 μm versus turbidity [NTU]

	R^2 for TPC versus NTU		R^2 for TPS versus NTU		R^2 for TPV versus NTU	
	Raw	Diluted	Raw	Diluted	Raw	Diluted
2–100 μm	0.758	0.805	0.670	0.554	0.608	0.374
2–10 μm	0.732	0.818	0.715	0.743	0.692	0.716

- $R^2 = 0.885$ in the experiments including single denitrifying or blank (without dosing) filtration.

A possible explanation for the poor correlation between TPV and floc-bound phosphorus removal for the experiments with $FeCl_3$ might be the shrinking of flocs due to iron dosing, described in Graaf *et al.* (2001), followed by a change in floc sizes due to the filtration process.

Conclusions

Particle counting is still a questionable measurement due to the inaccuracies, which are due to the operator or the instrument itself. Even if no operational faults occur uncertainties about the analysis quality still remain. For instance particles (especially fragile flocs while coagulant dosing) can be destroyed by the turbulence in the sample distribution system of the particle counter, which negatively affects the results and complicates the conclusion to be drawn. The concentration of particles is problematic for the accurate particle counting in the sample. Also the simplification of the analysis by idealisation of spherical particles is contestable. But in the case of conducting the analysis always in the same way with the same instrument, comparing the measurements relatively and dilution of the sample for highly turbid samples, like EF, the imperfections of this analysis method can be overcome.

The measuring range 2–100 μm of the MetOne PCX particle counter seems sufficient to investigate the experiments conducted on the lab-scale flocculation–filtration column. The presented particle counts of the FI show especially the presence of particles between 2 and 30 μm. The good results for the relation of particles counted between 2–10 μm to turbidity or phosphorus measurements shows the suitability of the particle counter used and its measuring range.

The described results represent the importance of sample dilution in order to count particles between 2–10 μm more accurately even in samples with a particle concentration below the limit of 10,000 counts/ml. The correlation between TPV versus TPC between 2–100 μm increases in diluted measurements for samples AF and FI but not for EF. Interpretation of particle counting by particle size distributions is weak compared to particle volume distributions, because for highly turbid samples TPC can be related to greatly varying TPVs.

The dilution also positively affects the correlation of TPC versus turbidity, TPV versus turbidity and TPS versus turbidity, where the range 2–10 μm provides the highest correlation compared to the range 2–100 μm. That leads to the indication that turbidity is caused by particles < 10 μm.

While relating the floc-bound phosphorus removal to the TPV a good linear correlation coefficient had been calculated for the experiments with polyaluminiumchloride PAX (including single flocculation–filtration and simultaneous denitrifying flocculation–filtration) and the experiments including single denitrifying or blank (without dosing) filtration. For the experiments using $FeCl_3$ only a poor correlation had been found.

After collecting these results the surplus value of particle counting in the improvement of the monitoring of tertiary wastewater filtration is not contradicted. Based on the results presented in this article further pilot investigations have been conducted since February 2005 in co-operation with Delft University of Technology, Water Management and Sewage Service Amsterdam and Witteveen + Bos Consulting Engineers on the WWTP Horstermeer in Nederhorst den Berg, The Netherlands.

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