Particle related fractionation and characterisation of municipal wastewater


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Abstract Several studies show that a more detailed characterisation of the particulate matter in municipal wastewater gives a better understanding and prediction of removal efficiencies of physical-chemical treatment techniques and the application of optimal chemical dosages. Such a characterisation should include the distribution of contaminants over various particle sizes. This article describes a method and results of experimental and full-scale investigations, conducted to determine how contaminants in wastewater are distributed over different particle sizes. For this purpose, particle size fractionations of wastewater influents originating from more than thirteen WWTP were carried out. One of these fractionations (WWTP Venray) is shown and interpreted in this article. First, the wastewaters were fractionated into 5 to 6 particle fractions (45, 5.0, 1.0/1.2, 0.45 and 0.1 µm) after which the fractions were analyzed for various water quality parameters like organic components, nutrients, salts, solids and turbidity. Based on the results the effects of removal of the different size fractions on design of the biological treatment and energy balance of a wastewater treatment plant can be assessed. The method also indicates whether a certain wastewater is efficiently treatable with physical-chemical pre-treatment methods. It is concluded wastewater fractionation on particle size is very useful, but that wastewater characteristics and particle size distributions should not be generalised, but have to be interpreted as indications for a certain average wastewater composition. To give more insight into the distribution of contaminants over particle size and the particle removal potential, a specific wastewater fractionation has to be carried out per WWTP.

Keywords Fractionation; micro particles; particle removal; particle size distribution; wastewater treatment

Introduction

Since a major part of the organic material and the nutrients in municipal wastewater is related to particulate material, advanced particle removal by physical-chemical pre-treatment, results in a lower pollutants load to subsequent treatment steps. In this way the total treatment system can be designed smaller and operated in a more energy efficient way (Ødegaard, 1998; Nieuwenhuijzen, van et al., 1998; Mels et al., 1999). Traditionally, the choice of treatment methods is mainly based on the effluent standards as well as on practical experience with the various techniques by engineering companies and waterboards. Especially the choice of adding pre-treatment steps, mainly primary sedimentation tank, preceding activated sludge systems was not, in general, based on technical knowledge and wastewater characteristics, but was made according to traditional practice and on plant size.

The applicability and efficiency of specific particle removal techniques is closely related to the particle size distribution in the water medium. Especially the distribution of contaminants over particle size is of interest. These data are wastewater specific and can differ per country or even per region due to differences in climatic circumstances, landscape and consuming habits. For example, it is stated that contaminants in wastewaters in the
Netherlands are in a larger percentage in the dissolved form compared to wastewaters from colder and mountainous regions like for example Norway or Alpine countries. The differences are supposed to be caused by the difference in slopes of the sewer systems, resulting in longer sewer retention times (Ødegaard, 1998) in flat landscapes with low slopes. In oxygen enriched wastewater in steep-slope sewers oxidation of organic matter may occur; whereas (anaerobic) pressure driven low slope sewer pipes (as are frequently used in flat countries) can contribute to a higher soluble fraction in wastewater since hydrolysis of components may occur. The supposed differences in wastewater composition caused by climatic and local conditions are verified by comparing wastewater characteristics (Nieuwenhuijzen, van, 2002).

Earlier studies (e.g. Levine *et al.*, 1991; Ødegaard, 1998) showed that a more detailed characterisation of the particulate matter in wastewater is necessary for a better understanding and prediction of removal efficiencies of physical-chemical treatment techniques and the application of optimal chemical dosages. Such a characterisation should include the distribution of contaminants over various particle sizes. Intensive wastewater characterisations have been conducted world-wide to determine biological fractions for activated sludge models, but few data are available on the size distribution of wastewater pollutants.

This article describes a methodology and results of experimental research, funded by the Dutch research organisation into water management – STOWA (STOWA, 2002), with the aim to determine how contaminants in wastewater are distributed over different particle sizes. From the obtained data, the effect of various particle removal techniques on the wastewater composition can be predicted and the size and energy requirement of the necessary post-treatment steps can be determined. The method also indicates whether a certain wastewater is efficiently treatable with physical-chemical pre-treatment methods and to what extent.

**Methods and experimental set-up**

For determination of the distribution of contaminants over particle size in municipal wastewater, a detailed size fractionation of the influent streams of thirteen wastewater treatment plants (WWTP) was carried out. The created wastewater fractions were analysed for organics, nutrients, solids, turbidity, salts and conductivity.

The wastewater inflow of the WWTPs Alphen-Noord, Amstelveen, Apeldoorn, Arnhem, Assen, Bennekom, Berkel, Boxtel, Capelle-Rotterdam, Haarlem, Hoek van Holland, Venray and Vlaardingen was sampled, fractionated and analysed. The sampling point was always selected identically for every WWTP: after grid removal and screens and directly in front of the first treatment facility (generally a sedimentation tank), since this was the desired wastewater flow to be analysed on particle related contaminants to identify the pre-treatment potential. Duplicate samples were taken on different measuring days at the same time (approximately 9.00 h and 11.30 h) and preferably during the same (dry) weather conditions.

In the first instance, grab sampling was preferred over flow proportional long term (24 h) sampling since processes like adsorption, degradation or destruction could cause changes in the wastewater composition regarding to particle size distributions. On the other hand, it was recognised beforehand that grab samples would only give an instantaneous picture of the total wastewater composition over the day. So, in a second research period also flow proportional samples were taken.

The samples were analysed immediately for temperature and pH and stored in polyethylene containers. The samples were fractionated directly on site, and the fractionated samples were sent to certified laboratories for further analysis. In case of delay during transport and after reception at the laboratory, the samples were kept under temperature control (at 4°C) to prevent changes in the wastewater composition.
The samples were fractionated over clean, pre-flushed sieves (stainless steel) and membrane filters with five different pore sizes (45, 5.0, 1.0/1.2, 0.45 and 0.1 µm) as shown in Figure 1. The filtrates of these filtration steps were analysed for COD, BOD\textsubscript{5}, nitrogen (N\textsubscript{kjeldahl} and ammonia), phosphorous (P\textsubscript{total} and P\textsubscript{ortho}), suspended solids, turbidity, and chloride. The wastewater fractions were determined by calculating the concentrations of the filtrates over a specific particle size range as summarised in Table 1. The effluents of the fractionation tests were also analysed by respirometric experiments to access the biodegradable COD for determination of the applicability of biological post-treatment (Mels et al., 2002).

In order to assess the potential size decrease of an activated sludge system when various size fractions are removed, calculations were made with the HSA methodology (ATV-A131, 1991; STOWA, 1995) using the average wastewater characteristics of Table 1. The HSA methodology is a widely used tool for designing activated sludge systems. The calculations were made for a wastewater treatment plant of 100,000-population-equivalents (p.e.) capacity with biological BOD removal and nitrification. The reactor had a minimum

**Figure 1** Schematic presentation of wastewater fractionation procedure

**Table 1** Fractions, particle sizes and analysed parameters

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Particle size range</th>
<th>Analysed parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw wastewater</td>
<td>Whole range</td>
<td>COD, BOD\textsubscript{5}, N\textsubscript{kj}, NH\textsubscript{4}, Ptot, PO\textsubscript{4}, TSS, turbidity, T, pH</td>
</tr>
<tr>
<td>Settleable-suspended</td>
<td>&gt; 45 µm</td>
<td>COD, BOD\textsubscript{5}, N\textsubscript{kj}, NH\textsubscript{4}, Ptot, PO\textsubscript{4}, TSS, turbidity, T, pH</td>
</tr>
<tr>
<td>Suspended</td>
<td>5-45 µm</td>
<td>COD, BOD\textsubscript{5}, N\textsubscript{kj}, NH\textsubscript{4}, Ptot, PO\textsubscript{4}, TSS, turbidity, T, pH</td>
</tr>
<tr>
<td>Supra colloidal</td>
<td>1.0-5 µm</td>
<td>COD, BOD\textsubscript{5}, N\textsubscript{kj}, NH\textsubscript{4}, Ptot, PO\textsubscript{4}, TSS, turbidity, T, pH</td>
</tr>
<tr>
<td>Colloidal</td>
<td>0.45-1.0 µm</td>
<td>COD, BOD\textsubscript{5}, N\textsubscript{kj}, NH\textsubscript{4}, Ptot, PO\textsubscript{4}, TSS, turbidity, T, pH</td>
</tr>
<tr>
<td>Dissolved-0.45</td>
<td>&lt; 0.45 µm</td>
<td>COD, BOD\textsubscript{5}, N\textsubscript{kj}, NH\textsubscript{4}, Ptot, PO\textsubscript{4}, TSS, turbidity, T, pH</td>
</tr>
<tr>
<td>Dissolved-0.1*</td>
<td>&lt; 0.1 µm</td>
<td>COD, BOD\textsubscript{5}, N\textsubscript{kj}, turbidity, T, pH</td>
</tr>
</tbody>
</table>

* results not shown in this paper, for more information see (Nieuwenhuijen, van and Mels, 2002)
process temperature of 10°C and a sludge concentration of 4 g/l. For all kinetic and stoichiometric parameters the default values of the HSA methodology were used.

Results and discussion

Fractionation

Since the concentration (ratios) and distribution of contaminants over particles are a wastewater specific characteristic caused by local conditions no general data are shown. In order to determine the actual particle distribution for a specific wastewater, the influent should be investigated in detail on site. More general fractionation data are presented by Van Nieuwenhuijzen and Mels (2002) and by STOWA (2002). Characteristics and differences per wastewater are discussed further on in this paper. As an (internationally) representative example, the results of the fractionation of the wastewater influent of the WWTP of Venray are presented in Table 2 and Figure 2.

The percentage of oxygen consuming components that is related to the settleable particle fraction (>45 µm) is relatively low, with a maximum of 27% and 26% for COD and BOD. Phosphorous is bound to settleable particles for only 11%. A significant part of the total COD and BOD is present in suspended and supra-colloidal particle fractions with particle diameters between 1.0 and 45 µm, namely 34% and 31%, respectively. 11% of total phosphorous is related to settleable particles, 25% to colloidal and suspended particles and 64% is soluble. For nitrogen (N\textsubscript{kjeldahl}), only 7% can be related to settleable particles and 15% to colloidal and suspended fractions; so 78% of the nitrogen is present in the soluble form (mainly ammonia).

BOD/N-ratio

When particle removal is increased within the pre-treatment, the BOD/N ratio is declining since more BOD is related to particles compared to nitrogen (see graphs on the right in Figure 2). As presented in Table 2 and the bottom right graph in Figure 3, the average BOD/N ratio of the Venray wastewater decreases from 3.7 in the raw wastewater to 2.9 due to the removal of settleable particles down to 45 µm. By total particle removal, the BOD/N ratio decreases to below 2. In diluted wastewater (during rain events) the BOD/N-ratio is already low (2.4) in the inflow and decreases even below 1.5 after complete removal of

<table>
<thead>
<tr>
<th>Fraction parameter</th>
<th>Dissolved (&lt;0.45µm)</th>
<th>Colloidal (0.45–1.0 µm)</th>
<th>Supra colloidal (1.0–5.0 µm)</th>
<th>Suspended (5.0–45 µm)</th>
<th>Settleable (&gt;45 µm)</th>
<th>Raw inflow</th>
</tr>
</thead>
<tbody>
<tr>
<td>Turbidity (NTU)</td>
<td>1 (±0.5)</td>
<td>11 (±9)</td>
<td>3 (±1)</td>
<td>113 (±20)</td>
<td>50 (±23.4)</td>
<td>179</td>
</tr>
<tr>
<td>COD (mg O\textsubscript{2}/l)</td>
<td>162 (±45)</td>
<td>30 (±12)</td>
<td>6 (±3)</td>
<td>165 (±32)</td>
<td>139 (±77)</td>
<td>501</td>
</tr>
<tr>
<td>BOD\textsubscript{5} (mg O\textsubscript{2}/l)</td>
<td>81 (±28)</td>
<td>10 (±2)</td>
<td>3 (±2)</td>
<td>62 (±23)</td>
<td>58 (±27)</td>
<td>213</td>
</tr>
<tr>
<td>N\textsubscript{ kjeldahl} (mg N/l)</td>
<td>44.5 (±4.8)</td>
<td>2.0 (±1.6)</td>
<td>0.5 (±0.5)</td>
<td>6.3 (±5.2)</td>
<td>4.5 (±4.2)</td>
<td>57.8</td>
</tr>
<tr>
<td>NH\textsubscript{4}-N (mg N/l)</td>
<td>37.8 (±2.1)</td>
<td>0.1 (±0.1)</td>
<td>0.4 (±0.4)</td>
<td>0.3 (±0.2)</td>
<td>0.8 (±0.5)</td>
<td>39.3</td>
</tr>
<tr>
<td>P\textsubscript{total} (mg P/l)</td>
<td>5.0 (±0.6)</td>
<td>0.1 (±0.1)</td>
<td>0.1 (±0.1)</td>
<td>0.3 (±0.2)</td>
<td>0.2 (±0.1)</td>
<td>5.7</td>
</tr>
<tr>
<td>Conductivity (mS/cm)</td>
<td>1.1 (±0)</td>
<td>0.0 (±0)</td>
<td>0.0 (±0)</td>
<td>0.0 (±0)</td>
<td>0.0 (±0)</td>
<td>1.1</td>
</tr>
<tr>
<td>BOD/N ratio (–)</td>
<td>2.0</td>
<td>2.1</td>
<td>2.2</td>
<td>2.9</td>
<td>3.7</td>
<td>3.7</td>
</tr>
<tr>
<td>Turbidity (NTU)</td>
<td>1% (±0)</td>
<td>7% (±6%)</td>
<td>2% (±1%)</td>
<td>62% (±11%)</td>
<td>28% (±11%)</td>
<td>–</td>
</tr>
<tr>
<td>COD (mg O\textsubscript{2}/l)</td>
<td>33% (±12%)</td>
<td>6% (±3%)</td>
<td>1% (±1%)</td>
<td>33% (±4%)</td>
<td>27% (±12%)</td>
<td>–</td>
</tr>
<tr>
<td>BOD\textsubscript{5} (mg O\textsubscript{2}/l)</td>
<td>39% (±14%)</td>
<td>4% (±1%)</td>
<td>2% (±2%)</td>
<td>29% (±9%)</td>
<td>26% (±9%)</td>
<td>–</td>
</tr>
<tr>
<td>N\textsubscript{ kjeldahl} (mg N/l)</td>
<td>78% (±11%)</td>
<td>4% (±3%)</td>
<td>1% (±1%)</td>
<td>10% (±2%)</td>
<td>7% (±9%)</td>
<td>–</td>
</tr>
<tr>
<td>NH\textsubscript{4}-N (mg N/l)</td>
<td>96% (±1%)</td>
<td>0.5% (±1%)</td>
<td>1% (±1%)</td>
<td>1% (±1%)</td>
<td>2% (±1%)</td>
<td>–</td>
</tr>
<tr>
<td>P\textsubscript{total} (mg P/l)</td>
<td>64% (±14%)</td>
<td>3% (±1%)</td>
<td>3% (±2%)</td>
<td>19% (±2%)</td>
<td>11% (±12%)</td>
<td>–</td>
</tr>
<tr>
<td>Conductivity (mS/cm)</td>
<td>97% (±1%)</td>
<td>1% (±1%)</td>
<td>1% (±0%)</td>
<td>1% (±1%)</td>
<td>0% (±1%)</td>
<td>–</td>
</tr>
</tbody>
</table>
particles. This will negatively influence the denitrification potential of the remaining wastewater. However, experimental investigations as well as model calculations (see below) indicate that the effluent composition of an activated sludge plant can comply with the effluent standard (N_{total} < 10 mg N/l) even when a very intensive pre-treatment is applied (Mels, 2001; Mels et al., 2002).

**Size decrease of activated sludge systems due to removal of different particle fractions**

An important advantage of particle removal in the pre-treatment is the possibility to decrease the BOD and nitrogen load of subsequent activated sludge (AS) systems. Moreover, particle removal in the pre-treatment will also decrease the load of inert organic matter into the activated sludge system and will thus increase the nitrifying activity per kg of sludge. In this way particle separation offers a possibility to reduce the required activated sludge volume.

Figure 3 shows the potential of volume reduction of a nitrifying AS system after removal of the various size fractions of Table 1 and compared to a system without particle removal calculated by HSA-modelling. The HSA-calculations show that due to the removal of particles in pre-treatment the volume of the AS system can largely be reduced by approx. 35% due to removal of settleable particles, and again an additional 40% due to removal of suspended and colloidal particles down to 5 µm, resulting in a total required AS volume of only 25% of the original size of the AS without particle removal.
For AS systems including denitrification, particle removal will often result in a declining BOD/N ratio since more BOD is related to particles compared to nitrogen (see graphs on the right in Figure 2). This will negatively influence the denitrification potential of the remaining wastewater.

It is a general rule of thumb that the minimum BOD/N ratio to achieve sufficient denitrification to comply with the EU effluent standards for sensitive areas (effluent N total < 10 mg/l) is approximately 2.5. The right hand graph in Figure 2 shows that this ratio is reached within the range of 5–45 µm and suggests that this ratio is reached when all particles bigger than approximately 25 µm are removed. More detailed characterisation including filters 20 or 25 µm could be included to evaluate the critical particle size in the case of WWTP Venray.

Recalculation into energy balance, space and costs
Calculating the potential energy consumption of the remaining wastewater after removal of certain particle fractions can show the effect of advanced particle removal on the energy efficiencies of a WWTP. The energy consumption of each particle removal technique has to be compared with the overall energy savings due to its removal of specific particle sizes. So the application of a certain pre-treatment technique can be counterbalanced to the resulting overall energy savings in the WWTP. In addition to energy calculations, also space requirements, chemical use, final sludge treatment, and even effluent quality can be calculated based on wastewater characterisation on particle size.

As an example, the effect of increasing particle removal in the pre-treatment on the post-treatment of the WWTP Venray is shown in Table 3. The required biological capacity of the biological activated sludge system decreases from approximately 70,300 p.e. (136 g TOD/d) without particle removal to 55,700 p.e. due to particle removal down to 45 µm (by settling). The remaining energy consumption of the post-treatment and sludge handling (calculated with DEMAS (Nieuwenhuijzen, 2002)) decreases from ca. 2,000 MWh/year to 1,700 MWh/year (saving of 330 MWh/y). Further particle removal down to 5 µm lowers the necessary biological capacity down to 37,900 p.e. (creation of extra space for 32,000 p.e. in the AS compared to treatment without particle removal) and the energy consumption decreases to 1,300 MWh/year. Further increase of particle removal down to 1 and 0.45 µm, only results in minor savings in biological capacity and energy.

Figure 3 Required volume (in percentages) of a nitrifying AS system after removal of various size fractions compared to a AS system without particle removal.

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Distribution of contaminants over particle size – specific per wastewater

Concentrations, ratios between contaminants and particle distribution in wastewater are wastewater specific and may differ per day or even per hour. Local situations (sewer type, landscape, habits, e.d.) and daily conditions (weather, climate, weekday, day hour) will influence the wastewater composition. These influences have also been observed in the analysis of the conducted wastewater fractionations per WWTP and between different WWTP. However, the shape of the distribution line of contaminants over particle size (Figure 2) is generally valid. The steep decrease over the settleable fraction (750–45 µm) and the suspended fraction (45–5.0 µm) and the flat slope of the line over the colloidal fractions (5–0.45 µm) were found in all conducted wastewater fractionations. The actual slope of the distribution line and the level of concentrations in each fraction, however, are wastewater specific.

Dissolved or not dissolved?

Since wastewaters are generally characterised in detail as input for the ASM there are disputes about the proper membrane pore size to fractionate the “dissolved” wastewater parameter (Roeleveld and van Loosdrecht, 2002). In the fractionation and particle characterisation experiments three different “dissolved” fractions were created to compare the fractions and fractionation method. Next to the 0.45 µm membrane filtration, 0.1 µm membrane filtration and precipitation with iron sulphate was used to produce the “dissolved” wastewater fraction.

From the analysis of 204 data on COD in the different “dissolved” fractions, it can be concluded that in average, the 0.1 µm membrane filtration produces a similar “dissolved” wastewater as the precipitation does. The 0.45 µm membrane filtration produces a “dissolved” wastewater that overestimates the “real dissolved” COD-fraction of the 0.1 µm membrane and the precipitation by 10%, since small colloidal particles are passing the 0.45 µm membranes. Especially for ASM-modelling, where the dissolved parameters have great impacts on the calculations, a proper definition of the dissolved fraction should be made. The experiments lead to the conclusion that the 0.45 µm separation technique should be changed to the more correct fractionation technique of 0.1 µm membrane filtration. Despite the more time consuming analyses, it is advised to apply the 0.1 µm membrane to determine the soluble part in wastewater characterisation and fractionation studies.

Conclusions

The proposed particle size fractionation and characterisation gives detailed insight into the specific distribution of contaminants over particles per wastewater and can be an important tool to assess the pre-treatment potential of a certain type of wastewater. The applied testing procedure was successfully developed and implemented on more than thirteen WWTP’s. By means of particle size fractionation the effect and applicability of a certain
(physical-chemical) particle removal technique can be derived for a specific wastewater. Moreover, the results of the test can be used to evaluate the consequences of pre-treatment for subsequent biological treatment steps and to estimate the resulting energy balance of the total treatment plant. Shape and form of the distribution line of contaminants over particle size is generally quite similar for every wastewater. However, fractionation and characterisation of wastewater show that particle size distributions should not be generalised, but should be interpreted as indications for a certain average wastewater composition of an individual wastewater source.

Based on the wastewater fractionation on particle size it is concluded that for the wastewater of the WWTP Venray particle removal down to 5 µm is efficient regarding energy consumption, space requirement and sufficient post-treatment. In this case, further particle removal in the pre-treatment seems not useful.

For optimisation of the fractionation and characterisation tests, more advanced (online) sampling, separation and analysing techniques could be implemented. For evaluation of the critical particle size regarding the remaining BOD/N ratio and potential post-treatment, a filter with a pore size of 20 µm should be added to create additional particle fractions (45–20 µm and 20–5.0 µm).

References