PARTICLE SIZE ANALYSIS

Rianne Okamoto

Sustainable IRES Cohort 2016

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California State Polytechnic University, Pomona

Particle Size Analysis of Decentralized Wastewater Treatment System

Rianne Okamoto

Abstract

Particle size distributions vary and proper identification of fraction characteristics can lead to a better understanding of the transformations that happen within a wastewater system. The focus of this study is a decentralized wastewater treatment plant in South Africa. Identifying chemical and physical characteristics of the various particle fractions can lead to better management and understanding of the treatment facility. The process of analyzing these particles to determine fraction sizes and characteristics includes various steps including filtration and microscopy, as well as the use of various machines like a scanning electron microscope. There is an obvious change in particle sizes throughout the system. Tracking the changes and identifying characteristics of the particle fractions and how those vary from chamber to chamber can lead to a better wastewater treatment. The total suspended solids do not decrease throughout the system but they do breakdown into different particle fractions. Microscopy images show that there may be layering on a base particle, a possible conglomeration may be carbohydrates.

1. Introduction

Wastewater is generally characterized using lumped parameters such chemical oxygen demand (COD) from particulate and dissolve matter. These parameters have worked well in categorizing and analyzing wastewater but particle size and chemical composition have become important to evaluate for a clearer idea of how specific wastewater treatment plants are operating. There have been various studies that analyze the particle size and chemical composition independently but few that have evaluated the relationship between the two. Heukelekian and Balmat (1959) conducted studies on the size and composition of particles in wastewater but since then there hasn't been much to further their work. "If different chemical compounds are predominantly associated with specific size fractions, then targeted solid–liquid separation could be used to purposefully modify the chemical composition of the organic matter in the wastewater to be treated," (Sophonsiri et al. 2004).

Particle sizes have a wide range and the only way to effectively analyze the distribution is through a combination of methods. The size of the particles in the wastewater range from settleable (e.g., >100 µm), supra colloidal (e.g., 1–100 µm), colloidal (e.g., 0.08–1 µm), and soluble (e.g., <0.08 µm) (Balmat, 1957). The figure of particle size (Levine et al., year; figure 1) (Figure 1) contains some typical constituents found in wastewater and gives their typical size fractions.

The study investigated a decentralized wastewater treatment system (DEWATS) in Newlands East just north of Durban, South Africa. The facility was set up in 2010 as a research site by Bremen Overseas Research & Development Association (BORDA), the Pollution Research Group

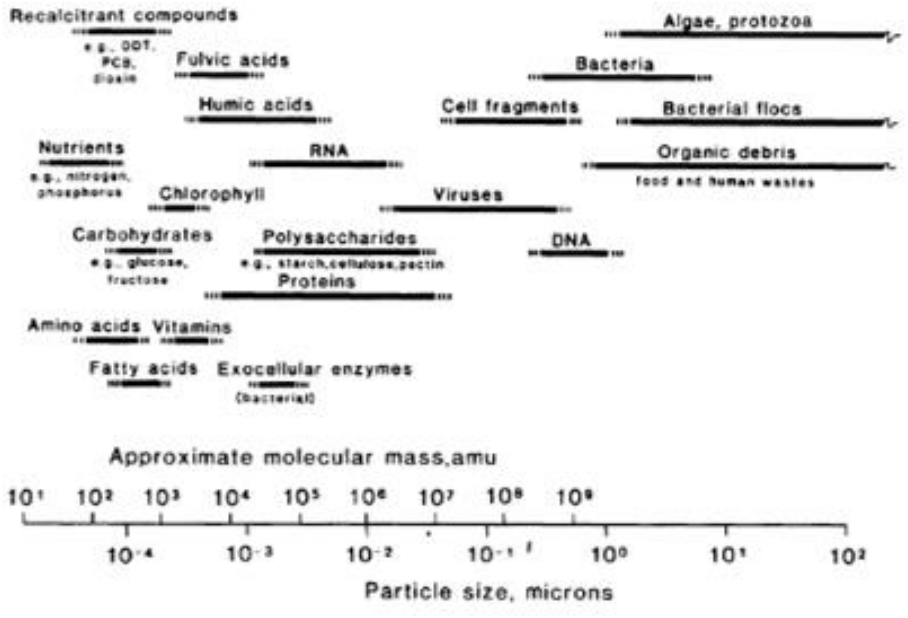

Figure 1: Figure taken from Levine et al. (1985)– Typical organic constituents in settled municipal water.

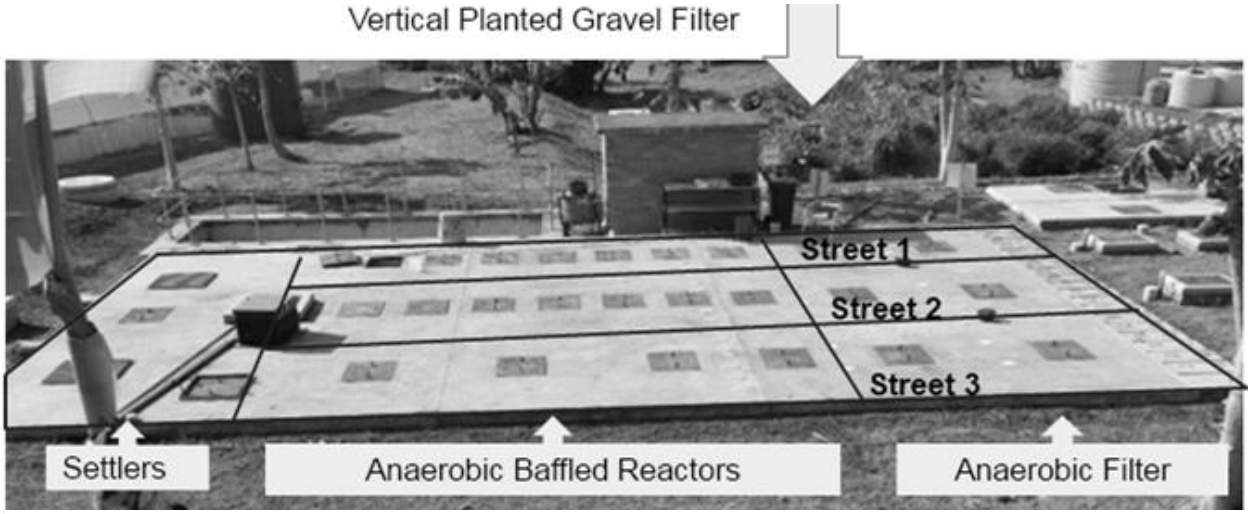

Figure 2: Layout of DEWATS System at Newlands-Mashu Research Site

at the University of KwaZulu-Natal, and the eThekwini Municipality. The domestic wastewater from 80 households is diverted from the main line to the site.

The DEWATS system is composed of 4 components the Settler, Anaerobic Baffle Reactor (ABR), Anaerobic Filter (AF), and Vertical Planted Gravel Filter (VPGF) shown in Figure 2. The system has three streets, two of which have 7 chambers in the ABR and one with 4 chambers. The baffles force the wastewater to flow up and down through the chambers. The flow of water increases the contact with the anaerobic sludge which digests parts of the organic matter in the

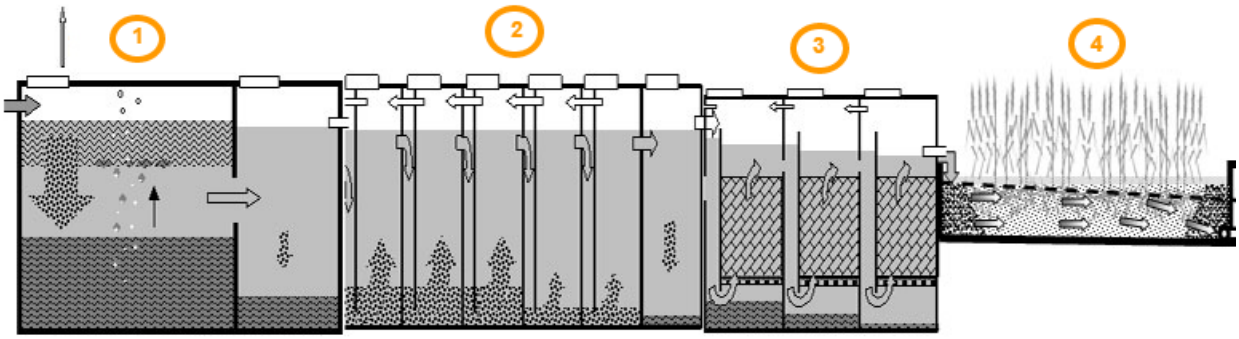

Figure 3: Layout of DEWATS System: (1) Settling Chamber, (2) Anaerobic Baffle Reactor, (3) Anaerobic Filters, (4) Vertical Planted Gravel Filter. Image borrowed from envibe website

wastewater. The AF contain coarse stones that remove the remaining particles before they move onto the VPGF. The last stage of the system is the VPGF, it provides yet another wastewater treatment using the wetlands as a removal process.

The particle sizes and percentage at which distribution change throughout the system. This study analyses the different particle fractions in three chambers, ABR 1, 7, and AF 2, in the DEWATS system and characterizes them using chemical analysis and microscopy. Proper identification and management of the particles throughout the system can lead to use of methods that will make the system more effective. Each treatment plant is going to work in a slightly different manner due to variations in diet and products use in different regions. The purpose of this research is to identify characteristics specific to this site and help create a method of analyzing the particle distributions in other sites.

2. Materials and Methods

2.1 Sampling

Samples from street 1 were taken in the mornings. Wastewater was taken from ABR 1, ABR 7 and AF, at the locations labeled as A, B and C. (Figure 4)

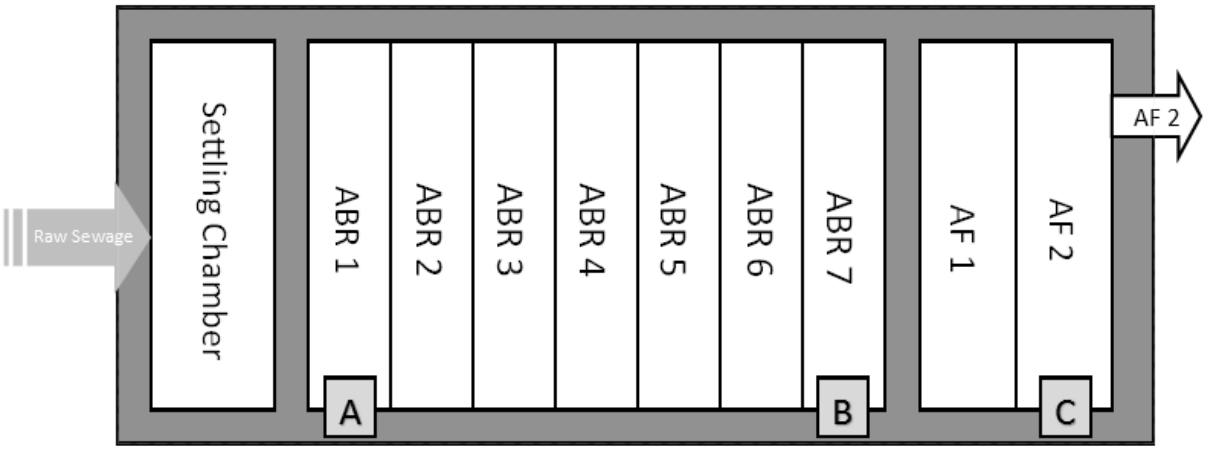

Figure 4: Schematic of Sampling Locations in Street 1

2.2 Analyses

There have been previous studies that analyzed the particle size distribution in wastewater, most notably by Sophonsiri et al. (2004) and Levine et al. (1985). Most methods used in this research were adapted from the authors listed above.

2.2.1 Initial Analysis of Particle Size

Samples collected from each chamber were analyzed using a particle size analyzer (Malvern Mastersizer, model) to graph the volume percentage of the particle classes. The particle analysis was used to determine the particle size classification expected in each chamber and the particles volume present in the sample.

2.2.2 Total Suspended and Volatile Suspended Solids

Samples were tested for total suspended and volatile suspended solids. Total solids (TSS and VSS). Solids are the suspended and dissolved matter in water. Total solids is the term used for the material left in the crucible after evaporation from the sample that is dried in an oven. Volatile Solids (organic solids) is the weight loss after ignition of the total solids.

TSS-Crucibles were labeled and then cleaned by placing in the oven at 105°C for one hour. Crucibles were allowed to cool to room temperature and weighed. Solids mass was determined by gravimetric method. Mass of empty crucibles was recorded. (W_1 = weight of crucible) Sample is mixed so the particles are suspended evenly throughout sample. Before particles settle 30 mL of the sample is measured out and added to the crucible. Mass of the crucible + sample was recorded. The crucibles with the sample are placed in the oven at 105°C for 24 hours. Crucibles and contents were cooled at room temperature and then weighed. Results are recorded. (W_2 = weight of crucible and sample after heating)

$$Total\ Suspended\ Solids = \frac{W_2 - W_1}{Volume\ of\ Sample}$$

VS- Crucibles were then placed in the furnace at 550°C for 2 hours. After cooling the crucibles were weighed again. (W_3 = weight of crucible and sample after ignition)

$$Volatile\ Suspended\ Solids = \frac{W_3 - W_2}{Volume\ of\ Sample}$$

2.2.3 Fractions of Total Suspended Solids

Samples were filtered sequentially through filter paper, diameter 47 mm, with pore sizes of 20 µm (Whatman™ 41 Ashless), 11 µm (Whatman™ 1, 70 mm Ø cut to size), 1.5 µm (Whatman™ 934-AH™, glass microfiber), 0.7 µm (Fisherbrand, TCLP), and 0,45 µm (Millipore EZ-Pak™ Membrane Filter). The 0.45 µm filter provided the soluble solution. Using a vacuum pump (Vacuubrand ME 2 NT) sample was filtered through. After each successive filtration, 10 mL of sample was set aside for further testing. Figure 5 shows filtration process. Filters were labeled and placed onto aluminum tins. Filters and tins were heated in the oven for 30 minutes at 105°C. Filters were cooled for 20 minutes before recording their mass. (W_1 = mass of back filter only) Filters were placed on filtering apparatus with a vacuum pump is attached to side arm. Sample was mixed so the particles are suspended evenly throughout sample, 60mL of sample was poured onto filter

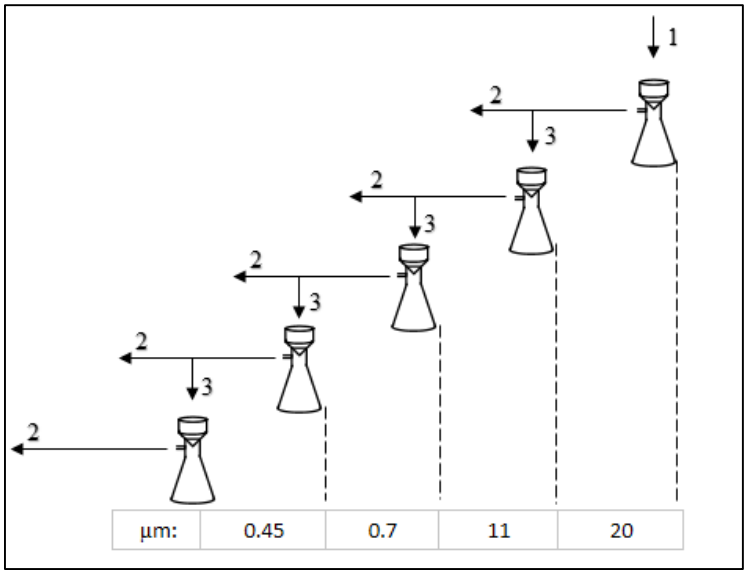

Figure 5: Schematic presentation of the sequential filtration procedure:
1—Non-settled, non-filtered, but mixed original sample, 2—sample of filtrate from the previous step, subjected to COD measurements, 3—sample of filtrate from the previous step, subjected to subsequent filtration procedure.

paper and pump was turned on. When filtration was complete paper was removed with tweezers, taking care to not tear the paper. The filter was folded into a triangular shape to seal the filter paper.

Starting with 20 µm then 11µm, 1.5µm, 0.7µm and finally 0.45µm the particle sizes fractions are removed. Ten mL samples of the filtered sample are taken and set aside for COD analysis. Filtered sample is reused, being careful to record the volume of sample used on each filter size.

Triangles were placed back on aluminum tins and placed in the oven at 105°C for 2 hours. Filters were then cooled to room temperature and weighed. (W_2 = mass of baked filters with residue)

$$Fraction\ of\ Suspended\ Solids = \frac{W_2 - W_1}{Volume\ of\ Sample}$$

2.2.5 Chemical Oxygen Demand

The Chemical Oxygen Demand (COD) expresses the amount of oxygen originating from potassium dichromate that reacts with the oxidizable substances contained in 1 l of water under the working conditions of the specified procedure. Organic and inorganic compounds oxidizable by dichromate were measured.

The COD measurements were carried out on raw samples (not-settled, not filtered, but mixed), as well as the filtered samples that were collected after each filtration steps mentioned earlier (Dulekgurgen et al., 2005).

Digester was heated to 150°C before preparing samples. Measurements were obtained by referencing the standard operating procedures for a measuring range of 100 - 1500 mg/I COD.

Empty cells were labeled according to sample, 0.3 mL of solution A, 2.3 mL of solution B, and 3 mL of the sample are added to the cells. A blank is prepared by adding 0.3 mL of solution A, 2.3 mL of solution B, and 3 mL of distilled water into an empty cell. Standardized KHP solution is used to make the standard curve, the cells are prepared using 0.3 mL of solution A, 2.3 mL of solution B, and 3 mL of the KHP. Lids are securely fastened to the cells and the contents of the cell were mixed vigorously and placed into the digester for 2 hours. After digestion was complete, the samples were cooled for 10 minutes and then swirled. Samples were cooled for another 20 minutes. See attachment A for more details of solutions used.

The spectrophotometer was turned on and set to mode 51 and the wavelength set to 605 µm for a measuring range of 100-1500 mg/l COD. The blank was inserted and the machine is zeroed. Once the blank has been tested the remaining samples can be tested using the spectrophotometer. After the last sample was measured the blank was reinserted and tested again.

2.2.6 Carbohydrate, and Protein Identification

Carbohydrates were measured using the Anthrone - Sulfuric Acid Method. Protein levels were measured using two methods: Biuret and Bradfort. The Biuret method operates under the principle that a substance with two or more peptide bonds will turn a copper (II) ion violet when present in an alkaline solution (Gornall 1946). The Bradford method is based on a shift in the absorbance of Coomassie Brilliant Blue G-250 (Bradford 1976).

Details on the methods used in identifying concentrations of carbs and proteins can be found in Bheki Mthembu's report. He conducted the analysis of the proteins and carbohydrates in the raw and filtered samples.

2.2.7 Microscopy

Analysis of the particle sizes using microscopy and observing the samples under different wavelengths of light was used to assist in identifying the composition of the particles. Different wavelengths of light and stains were used to highlight specific characteristics of the particles. Once the parameters were identified (wavelength and fluorescence ranges for specific characteristics) the raw samples and filtered samples were observed under a microscope to identify the composition of particle fractions.

The samples were observed using a Nikon Eclipse 80i under bright field, dark field, fluorescence: ultraviolet light as well as several other excitation wavelengths, and using an oil emersion method. Particles were photographed under the above listed lights, the various lights exposed different aspect of the particle that were not visible without the microscopy.

2.2.8 Elemental Composition Analysis

The Zeiss Ultra Plus Scanning Electron Microscope (SEM) was used to identify the elemental composition of the different particle fractions. The samples were scanned with a focused electron beam, with the use of the SEM images containing the topography, elemental composition of various particle sizes were obtained.

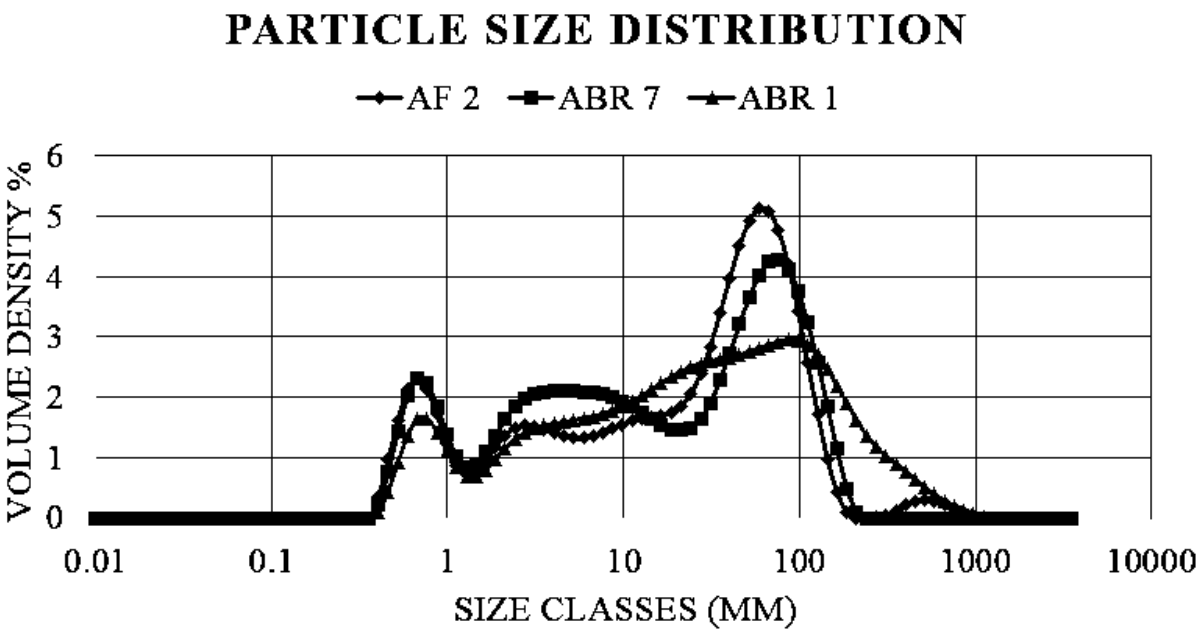

Figure 6: Particle Distribution Graph of ABR 1, ABR 7, AF 2.

3. Results

Particle sizes and the volumes present of the three sampled chambers is shown in the particle size distribution graph (Figure 6). The area under the curves represents the total solids which is fairly consistent throughout all three chambers. The peaks in the curves are representative of the size fractions and their distribution in the sample. The graph provides a qualitative view of the particle size fractions. There is a noticeable decrease in the 20 µm size class from ABR 1 to AF 2. There are also fluctuations in the other size classes throughout the system. The size distribution graph identifies the size fractions of interest for this system.

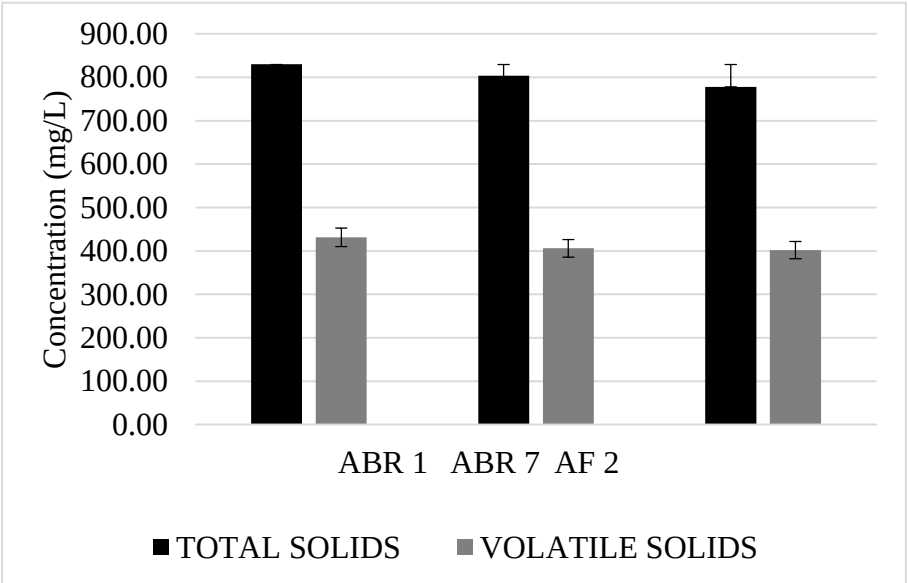

Figure 7: TSS and VSS in ABR 1, ABR 7, AF 2.

The total suspended solids and total volatile suspended solids were taken from the raw samples. The total volume of solids in the system does not change dramatically throughout the system (Figure 7). Total suspended solids were more abundant in n ABR 1, the total solids only decreases slightly in ABR 7 and AF 2. This is consistent with the size distribution graph. Volatile suspended solids has the same trend as the TSS. The goal of wastewater treatment is to remove or reduce the solids. There is not a large decrease in the overall suspended solids but from Figure 6 we can see the particle size distribution changes throughout the system.

The fractions of the suspended solids were found using the 5 filters listed in the methods. The portion of large particles (20 µm fraction) decreases from ABR 1 to AF, 2 which would suggest that the system is breaking down the particles as it is intended to do (Figure 8). The higher concentration of smaller particles in AF 2 indicates the large particles are indeed breaking down. The spike of 50-100 µm particles in AF 2, shown in Figure 4, is not represented in the fractioned concentrations. This could indicate that the large particles are loosely held together and when forced through a filter with a vacuum they are separated. The chemical oxygen demand was determined for the raw and filtered samples. As expected the COD concentrations decreased as the particle fractions, >20µm, 20µm-11µm, 11µm-1.5µm, 1.5µm-0.7µm, and 0.7µm-0.45µm, were successively removed (Figure 9). The trend of all three chamber lines verify that as particles are removed the COD concrentrations decrease.

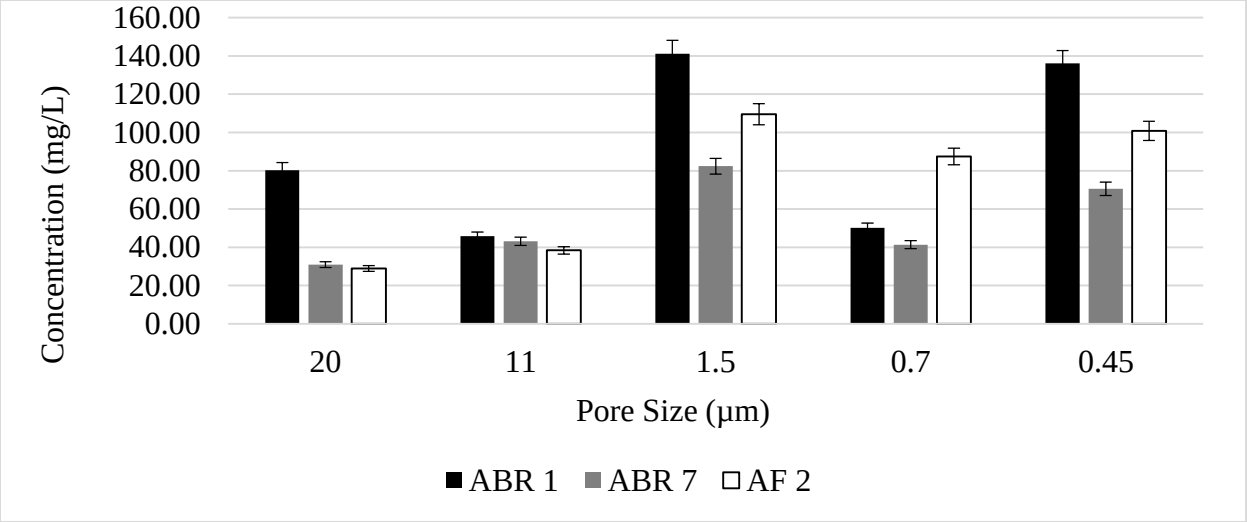

Figure 8: Concentrations of suspended solids capture on various particle fractions in ABR 1, ABR 7, AF 2.

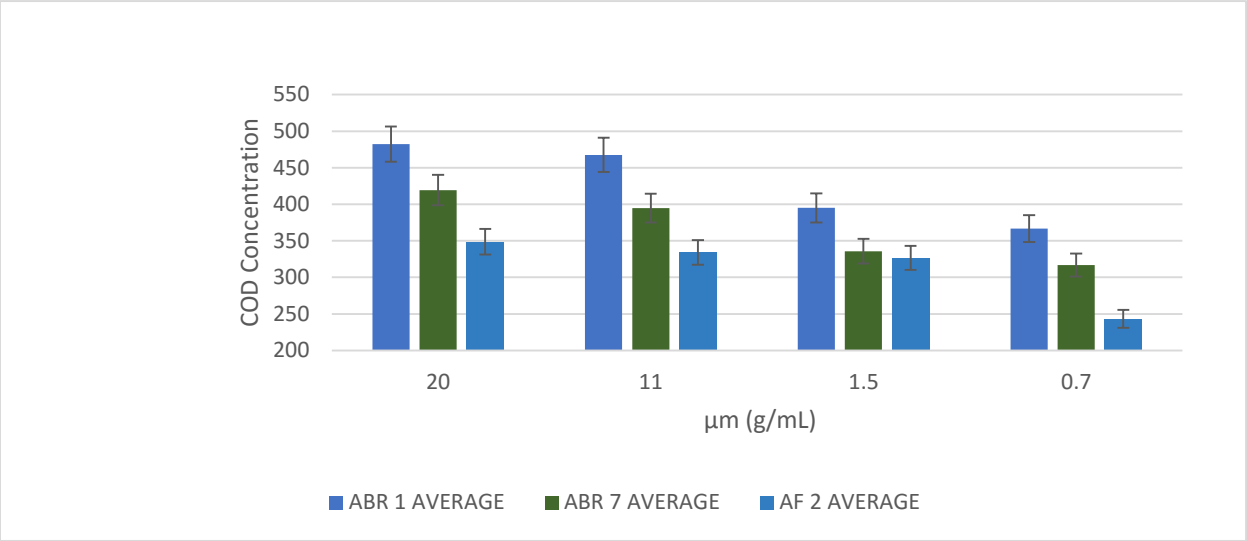

Figure 9: Chemical Oxygen Demand Concentration in each fraction

The results of the carbohydrate and protein analysis showed that both were predominant in the samples (Figure 10). Even after fractionation the carbohydrate concentration remained significant (add the concentration here). The proteins have much smaller concentrations throughout (add concentration here) the system and the filtration step removed the protein almost be completely. The 0.45 µm filter provided the soluble solution. Carbohydrates are of a smaller particle size, 10^{-4} µm to 10^{-3} µm as found in Levine et al. (1985), it is likely ultrafiltration would be required to filter out the remaining concentrations of it (Figure 1). The proteins could potentially be fully removed because their typical particle size is in our range.

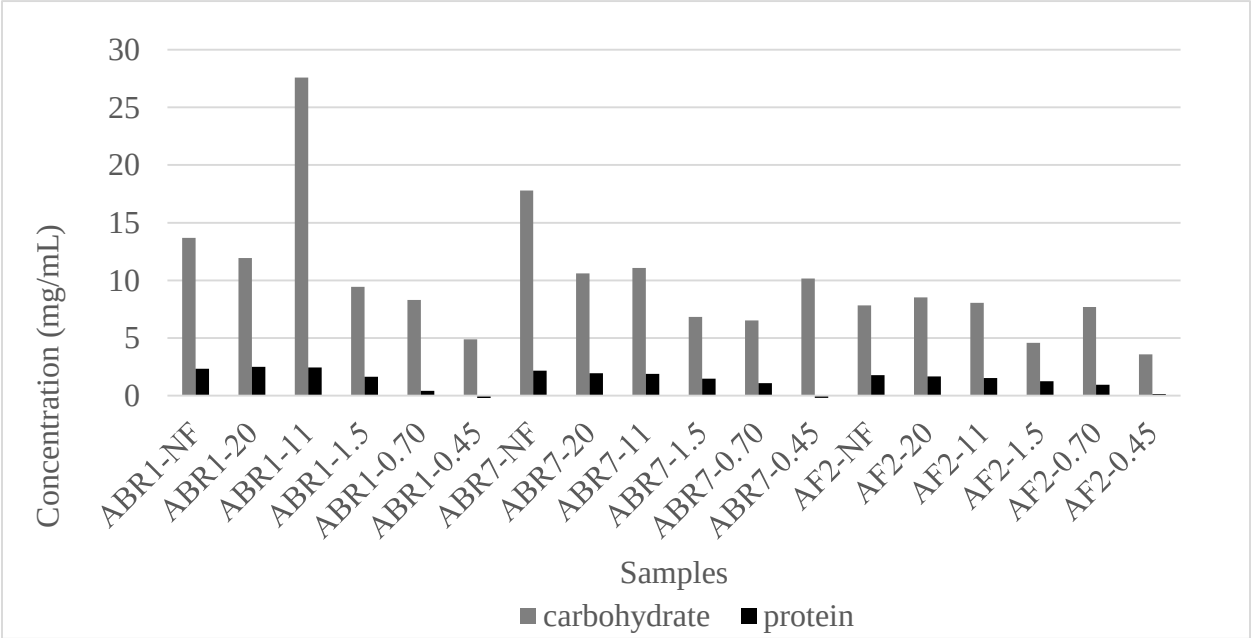

Figure 10: Carbohydrate and Protein Concentrations in Fractioned Samples.

The scanning election microscope provided the elemental composition of the surface of the sample that was analyzed. Using the SEM four different size fractions were identified from the raw sample. Figure 11 shows the composition of a portion of the particle. The percentages provided show that elemental surface composition percentages present only in the section selected. Carbon and oxygen are the most present in every image taken using the SEM. These elements are to be expected the interesting ones were the smaller percentages of aluminum and silicon present. Identifying elements present in the particles can help narrow down the tests that should be performed to get a better idea of the composition of the particle fractions. More experiments should be run to identify the source of the silicon present.

The results of observing the particles under a microscope are shown in Figure 12. Using 40x magnification it can be seen that there is a layering effect occurring on the particles. The arrows in Figure 12 point of the layering effect seen under a microscope. The different wavelengths help show the layering in multiple areas of the particle.

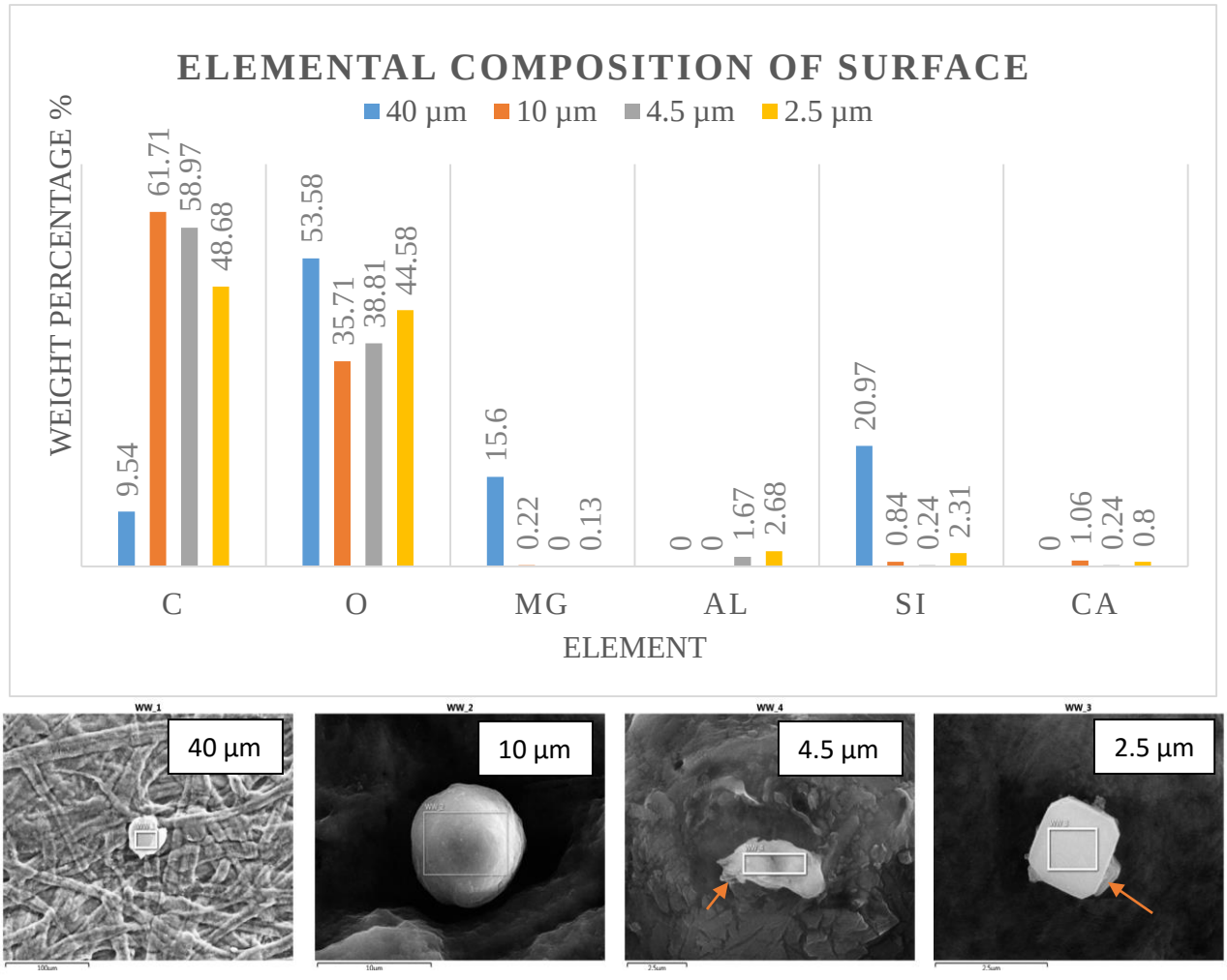

Figure 11: Results from Scanning Electron Microscope. Images in the bottom show the different shapes of the particle sizes and the top graph gives the percentages of elements present.

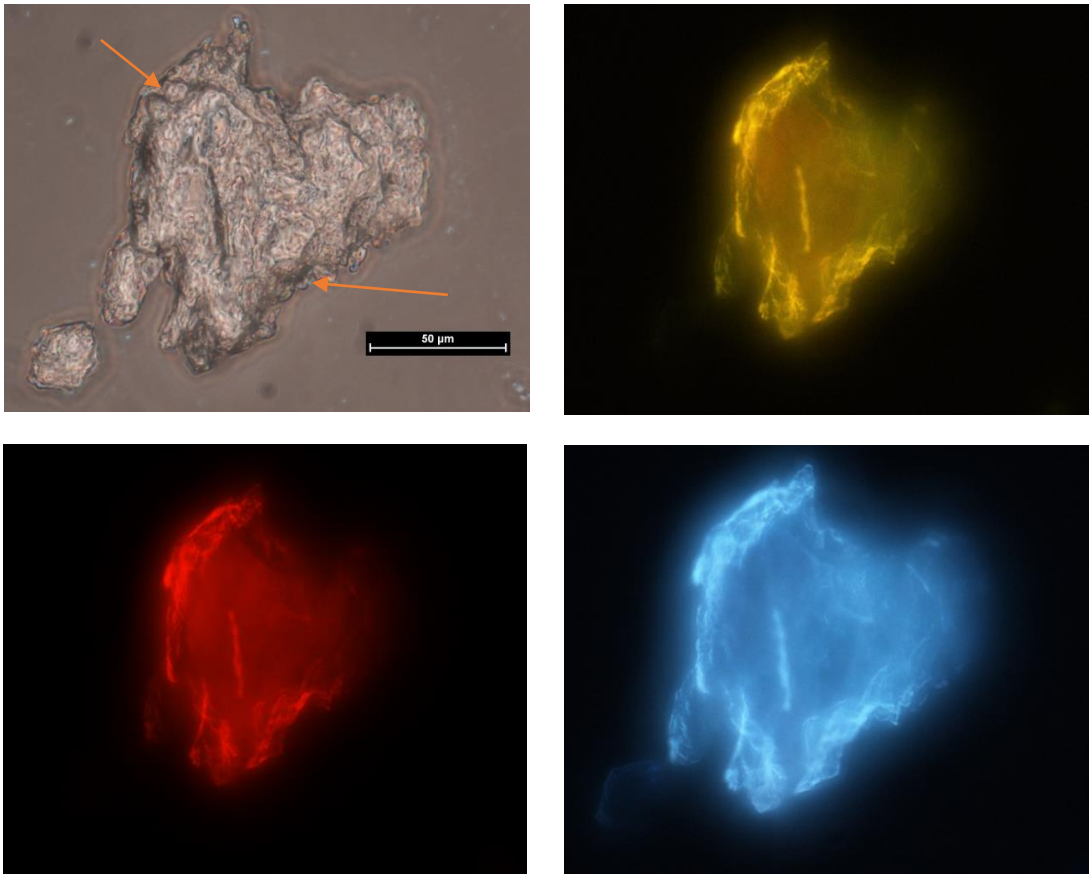

Figure 12: Microscopy ABR 1 – 40x

Discussion

The method for fractioning came from previous studies that identified the most effective way of separating the particle fractions. There have only been a few studies that have analyzed the particle fractions and characteristics in depth.

In this study chemical and physical characteristics were analyzed. COD levels and particle fractioning in the different chambers followed the expected trend. The microscopy and SEM have provided good insight into the physical characteristics of the particle fractions. Further research should be conducted to look the silicon presence. As well as verify the layering effect and reasons for it. Layering could be seen on the different particle sizes in Figure 11 and Figure 12. This might suggest that material is conglomerating onto a base particle. Further analysis of those particles would be necessary to confirm this.

The silicon may be a result of sand or it could be silica leaching from the system. The system is made of concrete and a component of concrete is silica fume. This could explain the presence of silicon found in the SEM data. But further analysis would need to be conducted to make sure it isn't common sand found on soiled clothes.

Conclusion

Wastewater samples from a decentralized treatment plant were characterized based on particle size distributions and chemical composition. The wastewater will vary with time but the method of characterizing the water should stay consistent. The results from this study have created the following conclusions:

- Confirmation that particle size changed along treatment train.
- SEM provided evidence that layering of different materials may be occurring on a base particle.
- Proteins and carbohydrates have been identified as possible conglomerates on the particles.
- Overall the total suspended solids do not decrease throughout the system but the particle sizes do breakdown.

Further analysis of the chemical characteristics of the particles can lead to the identification of the composition of the particles.

Proper identification and further characterization of the particle fractions can lead to greater understanding of the transformations that occur within the system. Understanding the fractions can also lead to improved techniques for treatment.

4. References

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1.14538.0065 1.14539.0495 1.14679.0495
1.14680.0495 1.14681.0495 1.14682.0495

Spectroquant®

COD solutions A+B

1. Definition

The COD (chemical oxygen demand) expresses the amount of oxygen originating from potassium dichromate that reacts with the oxidizable substances contained in 1 l of water under the working conditions of the specified procedure.

1 mol $K_2Cr_2O_7$ is equivalent to 1.5 mol O_2
Results are expressed as mg/l COD (= mg/l O_2)

2. Method

The water sample is oxidized with a hot sulfuric solution of potassium dichromate, with silver sulfate as the catalyst. Chloride is masked with mercury sulfate. The concentration of unconsumed yellow $Cr_2O_7^{2-}$ ions or, respectively, of green Cr^{3+} ions is then determined photometrically.
The method corresponds to DIN ISO 15705 and is analogous to EPA 410.4, APHA 5220 D, and APHA D1252-06 B.

3. Measuring range and number of determinations

Measuring range (mg/l COD)		4.0 - 40.0	10 - 150	100 - 1500	500 - 10 000
Solution A	Cat. No.	114538	114538	114538	114679
+					
Solution B	Cat. No.	114681	114682	114539	114680
Measuring wavelength (nm)		340	445 or 446	605 or 585	605 or 585
Number of determinations	Solution A	210	210	210	225
	Solution B	170	170	210	275

4. Applications

Organic and inorganic compounds oxidizable by dichromate are measured. Exceptions: some heterocyclic compounds (e.g. pyridine), quaternary nitrogen compounds, and readily volatile hydrocarbons.

Sample material:
Groundwater and surface water
In-process controls
Wastewater

5. Influence of foreign substances

See the package insert of the respective Spectroquant® COD Cell Test (Cat. Nos. 114560, 114540, 114541, 114555).

6. Reagents and auxiliaries

Please note the warnings on the respective bottle!

Store the bottles protected from light!
The solutions are stable up to the date stated on the respective bottle when stored closed at +15 to +25 °C.

Other reagents and accessories:
MQuant™ Chloride Test, Cat. No. 110079,
measuring range 500 - ≥3000 mg/l Cl^-
Potassium hydrogen phthalate CertiPUR®, Cat. No. 102400
Spectroquant® CombiCheck 10, Cat. No. 114676 (for measuring range 10 - 150 mg/l COD)
Spectroquant® CombiCheck 20, Art. 114675 (for measuring range 100 - 1500 mg/l COD)
Spectroquant® CombiCheck 50, Art. 114695 (for measuring range 4.0 - 40.0 mg/l COD)
Spectroquant® CombiCheck 70, Art. 114689 (for measuring range 500 - 10 000 mg/l COD)

Empty cells 16 mm with screw caps (25 pcs), Cat. No. 114724
Dispenser
Pipette for a pipetting volume of 1.0 ml (for measuring range 500 - 10 000 mg/l COD)
Pipette for a pipetting volume of 3.0 ml (for measuring ranges 4.0 - 40.0 mg/l COD, 10 - 150 mg/l COD, and 100 - 1500 mg/l COD)
Thermoreactor

7. Preparation

- Analyze immediately after sampling.
- Homogenize the samples.
- Check the chloride content with the MQuant™ Chloride Test. Samples containing more than 2000 or, respectively, 5000 mg/l Cl^- must be diluted with distilled water **prior to** determining the COD.

8. Procedure

Pipette solution A and solution B into an empty cell (free of scratches and organic impurities!) according to the desired measuring range and mix .
Take care not to exceed the stated volumes!

Measuring range (mg/l COD)		4.0 - 40.0	10 - 150	100 - 1500	500 - 10 000
Solution A	Cat. No.	114538	114538	114538	114679
	Volume	**0.30 ml**	**0.30 ml**	**0.30 ml**	**2.20 ml**
+					
Solution B	Cat. No.	114681	114682	114539	114680
	Volume	**2.85 ml**	**2.85 ml**	**2.30 ml**	**1.80 ml**

Suspend any bottom sediment present in the cell by swirling.

Pretreated sample	3.0 ml [1] or, resp., 1.0 ml [2]	**Carefully** allow to run from the pipette down the inside of the tilted cell onto the reagent (**Wear eye protection! The cell becomes hot!**).

Tightly attach the screw cap to the cell.

In all subsequent steps the cell must be held only by the screw cap!

Vigorously mix the contents of the cell.

Heat the cell at 148 °C in the preheated thermoreactor for 120 min.
Remove the hot cell from the thermoreactor and allow to cool in a test-tube rack.
Do not cool with cold water!

Wait 10 min, swirl the cell, and return to the rack for complete cooling to room temperature (cooling time at least 30 min).

Measure in the photometer.

[1] applies to measuring ranges
4.0 - 40.0 mg/l COD, 10 - 150 mg/l COD, and 100 - 1500 mg/l COD

[2] applies to measuring range 500 - 10 000 mg/l COD

Notes on the measurement:

- **Certain photometers may require a blank** (preparation as per measurement sample, but with distilled water instead of sample).
- For photometric measurement the cells must be clean. Wipe, if necessary, with a clean dry cloth.
- Measurement of turbid solutions yields false readings.
- The measurement value remains stable over a long term.
- Evaluation:
  To determine the COD values from the measurement values obtained (absorbances), prior to the COD measurement a calibration must be performed at the respective measurement wavelength for each batch used. For this purpose it is recommended to use a blank and three freshly prepared potassium hydrogen phthalate standards (application see the website) with the following COD values:

Measuring range (mg/l COD)	4.0 - 40.0	10 - 150	100 - 1500	500 - 10 000
Blank (mg/l COD)	0	0	0	0
Standard 1 (mg/l COD)	10.0	20	200	1000
Standard 2 (mg/l COD)	20.0	75	750	5000
Standard 3 (mg/l COD)	40.0	150	1500	10 000

9. Analytical quality assurance

recommended before each measurement series
The Spectroquant® CombiCheck products given below can be used for this purpose. Each of these articles contains a **standard solution** to check the photometric measurement system (test reagents, measurement device, handling) and the mode of working and also an **addition solution** for determining sample-dependent interferences (matrix effects).
Additional notes see under **www.qa-test-kits.com**.

Measuring range (mg/l COD)	CombiCheck Cat. No.		mg/l COD
4.0 - 40.0	114695	CombiCheck 50	20.0
10 - 150	114676	CombiCheck 10	75.0
100 - 1500	114675	CombiCheck 20	750
500 - 10 000	114689	CombiCheck 70	5000

10. Note

The reagents must not be run off with the wastewater!
Information on disposal can be obtained at www.disposal-test-kits.com.

Merck KGaA, 64271 Darmstadt, Germany,
Tel. +49(0)6151 72-2440
www.analytical-test-kits.com

EMD Millipore Corporation, 290 Concord Road,
Billerica, MA 01821, USA, Tel. +1-978-715-4321