



CWPPharma 2

CLEAR WATERS FROM PHARMACEUTICALS

Procurement of and preliminary testing with a technical scale GAC pilot at the Viikinmäki WWTP

GoA2.2: Applying recommendations for planning of API
removal and plant optimization

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Authors/Contributors:

Kuokkanen, Anna (HSY, Helsinki Region Environmental Services Authority)

Korhonen, Kaisa (HSY)

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Introduction

In the CWPharma-project three different methods for separating powdered activated carbon (PAC) were tested in pilot scale at Viikinmäki WWTP: Mecana cloth filter, discfilters and ACTIFO®Carb (sand ballasted clarification). API removal was measured with ACTIFO®Carb using two different PAC dosages and two different contact times. However, in preliminary planning for post treatment for micropollutant removal for the Viikinmäki WWTP and negotiating with the city officials using PAC has turned out problematic, as it would require large above ground storage facilities in an otherwise underground treatment plant and granular activated carbon (GAC) filtration has been deemed as a more feasible option.

This report is part of the Clear Waters from Pharmaceuticals 2 (CWPharma 2) project and funded by the EU's Interreg Baltic Sea Region Programme. It describes the design and preliminary testing with a technical scale GAC pilot.

As part of CWPharma 2, the pilot was designed, procured and preliminary testing was made using one GAC-material and some alterations to the pilot were made. The pilot, test results and modifications made as well as future plans for piloting and for the possible automation of the pilot are described in this report.

The purpose of the GAC pilot is to aid in the hydraulic design of a full scale GAC post treatment step for the Viikinmäki WWTP and enable testing API removal but also hydraulic capacity properties and clogging and backwash requirements of different GAC-materials and possibly combining micropollutant and phosphorus removal.

Pilot design and procurement

The pilot design was made by HSY for a two-column pilot treating 180–400 L/h per column.

API removal can be tested and GAC materials compared in small columns but to assess the hydraulic capacity and backwashing needs, the pilot must be sufficiently large. Also, to be able to make reliable comparison of different GAC materials or filter depths when using real wastewater with varying quality, at least two parallel columns are needed.

The design is presented in Figure 1 and the dimensioning in Table 1.

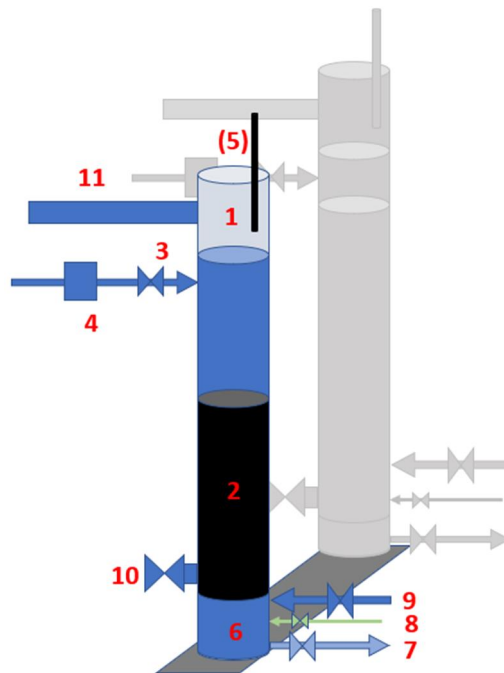


Figure 1. GAC pilot design

The parts of the pilot were (Figure 1):

1. Pilot columns
2. Filter material
3. Manual valve for influent flow control
4. Visual flow measurement
5. (Optional: surface measurement or alarm)
6. Bottom structure
7. Outflow
8. Air for cleaning
9. Backwash water
10. GAC extraction
11. Overflow for washwater and for hydraulic overload situations

Table 1. GAC pilot dimensioning (per column)

Parameter	Unit	Value
Diameter	m	0,25
Surface	m ²	0,05
Filter bed depth	m	3 (varying)
Filter volume	m ³	0,15
Q _{AVE}	L/h	180
Q _{MAX}	L/h	400
Surface load, Q _{AVE}	m/h	3,7
Surface load, Q _{MAX}	m/h	8,0
EBCT*, Q _{AVE}	min	50
EBCT*, Q _{MAX}	min	23

*) empty bed contact time, depends on the filter bed depth used

In the first stage the pilot was to be operated completely manually. All the valves were operated manually, including flow control (Figure 3 b). The influent used was technical water, which is equal to the WWTP effluent from a pressurised network and the air for washing came from the treatment plants pressurised air network. The pilot included a pressure control for air.

Possible future upgrade possibilities were also considered in pilot design, including influent pumping for better flow control, flow measurement and automated control of washing.

The execution of the pilot

Bids for building the pilot were sent to three companies and the choice was made with emphasis on references, the materials used, possible improvements and solutions suggested and schedule. The pilot was purchased from Teollisuuden Vesi Oy.

The pilot was made using plastic and steel components and a protective steel structure (Figures 2 and 3).



a) The pilot on site before the addition of GAC, b) changing of gaskets



a) bottom nozzles, b) flow and air pressure control and connections for washwater and air

Set-up and preliminary testing

The main goal in the preliminary testing was in checking the setup and possible improving of the GAC-pilot by modifications.

The GAC-material

For the setup and preliminary testing of the GAC-pilot, the GAC-material used was from one of HSY's water works. One GAC column was filled with virgin carbon and the other with regenerated carbon. The choice was based on easy access, due to the schedule of testing, and as the main goal was not in evaluating the GAC-material but the pilot setup.

The GAC used was FILTRASORB® 400 from Chemivron Carbon. The effective grain size⁷ was 0,7 mm, which is in the smaller end of typical¹ grain size of 0,5–2,5 mm, and may not be optimal for a full-scale wastewater treatment application from the point of view hydraulic capacity and required backwash frequency.

The regenerated carbon has a slightly higher average grain size.

Observations and modifications made during the preliminary testing

Several small modifications were made during preliminary testing:

The pilot originally had nozzles also on top to prevent loss of GAC during washing, suggested by the pilot manufacturer, but it became soon imminent that nozzles got easily clogged with solids in the wastewater and likely also the smallest carbon particles escaping in the washing in the beginning, creating a risk of pressurizing filter columns. The nozzles were removed, and the pilot column top surface was left open.

The original gaskets in the pilot column were soft and flimsy and difficult to keep in place during joining the components and they were changed to more robust ones.

The flexible effluent hose was lifted upwards to ensure that the water level kept above the filter bed regardless of flow and to better emulate the water levels at a full-scale treatment plant. An additional branch had to be added at the highest location to prevent siphoning. The top level of the hose can be changed if necessary, if for instance running the pilot with a different maximum pressure loss is to be tested, but it will require making a new anti-siphoning connection. With the current setup, the maximum difference in the water levels before and after the filter is 1.7 m.

A pressure control valve was added to the influent as the pressure in the technical water network varied and this caused changes in the inflow.

A scale for the water depth was attached to both filter columns to facilitate recording water levels to be able to assess pilot clogging and hydraulic loss.

Possible future modifications and upgrades

The pressure control valve was not sufficient to ensure a steady inflow and for longer pilot runs and to decrease the need for checking and adjusting the influent flow, the current control should be replaced with influent pumps with accurate flow control, such as membrane pumps or peristaltic pumps.

Even though the filter diameter was quite high, it was observed that sometimes part of the filter bed started rising as a compact cake during washing instead of the GAC particles moving freely. This caused a risk of losing part of the filter bed. Thus, the filter was not left unsupervised during washing. This may be partly due to the GAC used in the preliminary testing, which had a particle size probably too small for wastewater treatment containing solids.

To be able to fully assess clogging and need for washing in different loading situations and to enable long pilot runs, the pilot should ideally be automated, including flow meters, influent

flow control and automated valves for washing and data logging or connection to the treatment plant's automation system. Considering the observed risk of loss of filter material, a sufficiently large basin with overflow for dirty washwater would be needed unless the backwash procedure can be optimized. Adding a rough structure above the filter material (e.g. X-shape) to prevent it from rising as a cake and forcing it to break, should also be tested.

Results

The main goal in the hydraulic testing was to ensure that reliable data from the hydraulic properties and backwash needs could be acquired from the pilot. Also API analysis were made.

Hydraulic testing

According to the carbon manufacturer's brochure⁷, the pressure drop for a backwashed and segregated bed increases with app. 4 cm per m – equivalent to 8 cm for a 2 m filter bed – when the linear velocity increases by 1 m/h.

The observed pressure drops in the pilot, with varying run times after washing, are presented in Figure 4 for both virgin GAC and regenerated GAC.

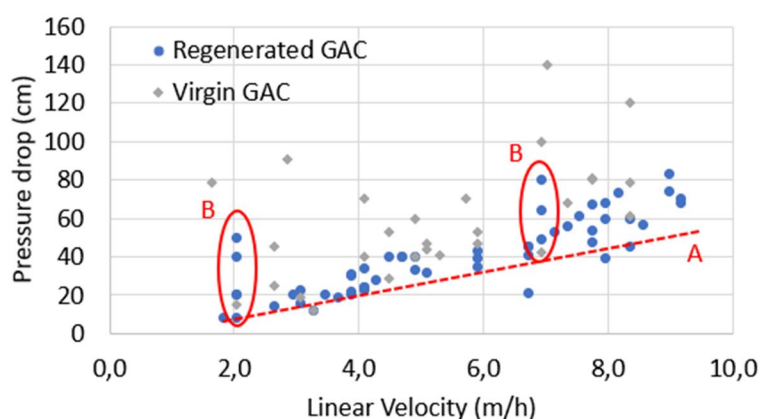


Figure 4. Increasing pressure drops due to A increasing linear velocity and B examples of the effect of clogging caused by wastewater solids.

The increase in the pressure drop with increasing flow for regenerated GAC was close to the values stated by the manufacturer and the effect of solids accumulation could be clearly seen, indicating that the pilot is well suited for evaluating the hydraulic capacity and washing frequency needed for different filter materials. (Figure 4)

The virgin GAC was much more prone to clogging and there is a large variation in results (Figure 4). Also the bed expansion during washing was much higher and small filter particles could be seen moving after the filter bed had settled after the start of expansion (Figure 5). Also, as stated before there was a risk of loss of carbon due to the part of the virgin GAC filter bed sometimes rising as a "cake".



Figure 5. The top of filter beds during washing. Virgin GAC on the left. The virgin GAC filter bed expansion is much higher and the surface appears fuzzy due to small filter particles moving.

API removal

Samples for API analyses were taken during one testing day, using three different influent flows. The influent samples were taken twice.

The average flow at the treatment plant during the testing day was 244 000 m³/d and the previous day (the hydraulic retention time at the treatment plant is roughly on day) 235 000 m³/d, representing dry weather conditions.

The filter bed heights were 2.1 m for new GAC (N) and 2.0 m for regenerated GAC (R). The bed volumes treated before the API sampling were below 500. The influent flows used are presented in Table 2.

Table 2. Influent flows and equivalent EBCTs and hydraulic loads.

Influent flow (L/h)	EBCT [N / R] (min)	Hydraulic load (m/h)
120	50 / 48	2,5
190	32 / 31	3,9
250	25 / 24	5,1

The analyses were ordered from the same laboratory as in CWPharma ACTifloCarb[®] piloting³, referring to the earlier tests, but without separate specifications. After the testing made in CWPharma the laboratory had started doing the analyses themselves instead of ordering them from sub-contractors. The quality of API results was poor, and unfortunately the API tests served mainly as a learning experience on procurement of API tests.

The laboratory was able to quantify only two of the analysed APIs in the influent samples: sotalol 0.041 µg/L and 0.022 µg/L (LOQ 0.007 µg/L) and bisoprolol 0.10 µg/L and 0.15 µg/L (LOQ 0,07 µg/L).

The following APIs were reported to be above the detection limit of 0.04 µg/L but the laboratory was not able to quantify them: bezafibrate, carbamazepine, citalopram, diclofenac, fluoxetine, hydrochlorothiazide, ketoprofen, metoprolol, naproxen, sulfamethoxazole, trimethoprim and warfarin.

These concentrations and detection limits were mainly considerably lower than the concentrations in the samples analysed by the Aarhus university for the Fitness check and the Feasibility study⁹. Not all of the same APIs were analysed.

The following APIs were below their detection limits in all samples: ibuprofen < 0.5 µg/L, metronidazole < 0.06 µg/L, propranolol < 0.07 µg/L, 17a-Ethylestradiol (EE2) < 0.09 µg/L, 17b-estradiol (E2) < 0.05 µg/L, estriol (E3) < 0.08 µg/L and estrone (E1) < 0.09 µg/L.

The concentrations of ibuprofen and propranolol were also low, and hormones were not analysed for the Fitness check and the Feasibility study⁹.

All of the above APIs were below detection limits in all effluent samples, which is consistent with the low influent concentrations and the as the number of treated bed volumes before the API removal test was low. The EBCTs were also high in all tests, so an efficient removal could be expected. In the CWPharma project, API removal using PAC was tested with two retention times: 17 and 34 min and two PAC concentrations 10 and 30 mg/L and several APIs were above their detection limits also with the higher dosage and longer contact time³.

Future plans for testing

There are three special subjects of interest for future testing: non-fossil GAC materials, biochar based GAC and combining API with phosphorus removal. These are all also linked to other research and development projects in HSY.

Setting up also a laboratory scale pilot for rapid small scale column tests to assess the need for replacing or regenerating the GAC material would also be beneficial, as it would enable reaching high values of treated bed volumes in a much shorter time.

Non-fossil GAC materials and biochar based GAC

GAC filtration is used in potable water treatment in HSY for DOC removal to improve the taste and odour of potable water, which is made from surface water, and there are undergoing studies on using a non-fossil GAC. Tests with coconut based GAC and turf based GAC are planned for 2022 and other, e.g. wood based GAC producers in Finland or in Nordic countries with sufficient production capacity are being searched.

Testing the suitability of these same GAC materials in API removal in wastewater treatment is one of the planned future uses for the GAC pilot.

For wastewater one possible GAC material type of special interest would be activated biochar or "sludge char" from digested sludge pyrolysis, originating from HSY's pyrolysis pilot that is operated by the solid waste and biowaste treatment division. However, activating the biochar has not yet been studied and the high iron content may prove it unfeasible.

Combining API removal with phosphorus removal

Combining API removal with phosphorus removal is a point of interest either for replacing a separate phosphorus removal unit process for effluent polishing, which has been planned for Viikinmäki WWTP, or enabling phosphorus recovery at Viikinmäki WWTP.

HSY is piloting the RAVITA™ process based on post precipitation⁸. A second phosphorus removal step is likely to be needed to ensure sufficient effluent quality when the chemical phosphorus removal takes place solely as post-precipitation. Combining API removal with phosphorus removal could enable using the planned phosphorus removal unit process for phosphorus recovery.

Combining API removal with phosphorus removal will be tested with a filter with GAC and sand layers using a precipitation chemical.

Combining e.g. ozonation with GAC?

As according to the bromide analysis made for the Viikinmäki Feasibility study, the wastewater bromide levels were a possible barrier for ozonation, and GAC filtration appeared to be the most suitable process option⁹. However, as there is not yet knowledge on the future requirements for API removal and as some APIs are not adsorbed on GAG, other pilot tests may be beneficial. For example by combining ozonation and GAC, a lower dosage than for ozone alone, producing less bromate might produce a sufficient treatment result.

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