iMERMAID

D3.3: 4DS Pilot equipment for heavy metal removal from demonstration sites

Task 3.3: 4D Scavenger systems for the effective removal and recovery of heavy metals

WP3: Removal and remediation of chemical pollutants via innovative technologies

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ABSTRACT	A demonstrator equipment for selective and efficient heavy metal removal from municipal wastewater was developed. Heart of the demonstrator is a 4D Scavenger technology, in which both chemical functionality and mechanical structure (flow distribution) can be optimized. Development included selection of 4D Scavenger chemistry, optimization of mechanics and particle size, and reactor and system design. Over 90% heavy metal reduction and excellent selectivity were achieved. Performance of fully automatic equipment will be verified in pilot test in SMAT WWTP (Wastewater treatment plant), located in Brandizzo, Turin (iMERMAID, Use Case 3).

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Document Revision Log

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Executive Summary

Aim of this project was to build Weeefiner 4D Scavenger based pilot equipment for selective heavy metal removal from WWTP outlet effluent. Development work included 4DS chemistry selection and its performance optimization in laboratory, 4DS manufacturing, reactor, process and system design including automation, and installation of pilot equipment to container.

Weeefiner 4D Scavenger technology was first successfully demonstrated in laboratory scale to be able to remove heavy metals from WWTP effluent. Several 4D Scavenger material options were tested, and results analysed and compared. Key parameters for 4DS selection were selectivity to target metals (heavy metals) and effective capacity. 4DS-206 was chosen as the most promising material to be tested in the following pilot phase of the project. Performance of selected 4DS (4DS-206) was optimized in laboratory. Scope included optimization of washing solution, its concentration and flow rate.

Performance test results were used as basis of process design of pilot equipment. Pilot equipment was designed, modelled and installed into a shipping container Design included PI -drawing, process automation and 3D modelling whole process. HM Box (by UNIFI) was added to design. Installation included assembly of piping, auxiliary devices (like particle filters before 4DS) and instrumentation. Performance and technical functionality of pilot equipment were successfully verified in Weeefiner premises before pilot tests in SMAT WWTP in Turin.

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Acronyms

4DS	4D Scavenger
Al	Aluminium
BV	Bed Volume = volume of 4D Scavenger filter element
BV/h	Bed volumes in hour. Describes the flow rate through 4DS
Ca	Calcium
Fe	Iron
HM Box	Heavy metals box
IBC	Intermediate Bulk Container
ICP-OES	Inductively coupled plasma atomic emission spectroscopy
K	Potassium
LoQ	Limit of quantification
Mg	Magnesium
Mn	Manganese
Ni	Nickel
PID	Process, Instrumentation and Design.
PVC-U	Unplasticized Polyvinyl Chloride
WWTP	Wastewater treatment plant
Zn	Zinc

1.0 Introduction

1.1 Scope of demonstrator

Scope of the demonstrator is to study and develop 4D Scavenger technology -based solution and equipment, which is suitable for removing and recovering of heavy metals from wastewaters. In the use case discussed here, the focus was on removing heavy metals from WWTP water streams. Furthermore, the project aims to develop 4D Scavenger materials and structures that could selectively target heavy metals focused on this project.

1.1 Technology – 4D Scavenger

Weeefiner's 4D Scavenger® is a sustainable water treatment technology that uses 3D-printed, porous filters to selectively recover or remove valuable or toxic dissolved metals from industrial and municipal wastewater. Designed for flexible use—from small batch processes to large, automated systems—it enables closed-loop production by reducing pollution and the need for virgin raw materials. Applications span mining, manufacturing, and environmental management, helping industries cut costs while improving environmental performance.

The 4D Scavengers can be customized and optimised for specific processes. This involves customizing the 4D Scavenger material as well as the size, structure and the process to fit the specific needs of the application. Another key feature of the 4D Scavengers is the ability to enhance the chemical reaction kinetics of even existing materials – such as ion exchange resins. This leads to several beneficial features such as faster reactions, higher flowrates as well as better quality and products with smaller waste volumes produced during operation.

2.0 Preparation for the pilot tests

2.1 Laboratory phase

During the project, water samples were received from SMAT. Both inlet and outlet samples were sent, but as the project progressed, the focus was shifted towards the outlet samples of the WWTP as the pretreatments necessary to operate with inlet water would have been excessive. These samples were used to test and optimize different 4D Scavenger materials and structures for optimal heavy metal removal. In addition, different process conditions were tested to find out e.g., the optimal residence time and flow rates for specific 4D Scavenger materials.

In general, the laboratory test setup was rather simple. 4D Scavengers were placed in a diameter 25mm PVC-U reactors and chosen samples were pumped through the 4D Scavengers at desired flow rate using peristaltic pump. Samples were then drawn from the inlet and the outlet of the 4D Scavenger. Samples were then analysed for heavy metal concentrations using ICP-OES. After wastewater treatment tests, the 4D Scavengers were washed and regenerated with chosen chemicals by using separate peristaltic pump to pump the chemicals through the 4D Scavenger. Metal concentrations in these streams were also analysed to assess the washing performance of the 4D Scavenger.

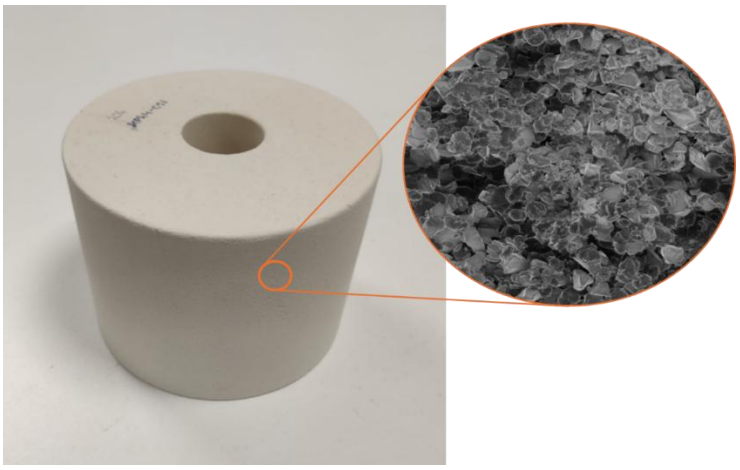

Figure 1: Example of 4D Scavenger types and structures tested during the iMERMAID project

Before continuing to further laboratory tests, different 4D Scavenger materials were tested for heavy metal removal efficiency from samples provided by SMAT (Figure 1). Tables found below show the results obtained for testing 4DS-103, 4DS-104 and 4DS-206 for heavy metal removal. The report does not include all of the tests performed but is rather focused on reporting the notable findings that lead to the specific 4DS scavenger being piloted.

Figure 2: Samples received from SMAT during the iMERMAID project

In Table 1, results show that the 4DS-103 is able to remove most of the heavy metals from the sample feed. Although, a possible challenge observed with the performance is the significant removal of non-heavy metal compounds such as Ca, K and Mg that due to their nature show no ecotoxicity in these concentration levels and therefore are usually not targeted during wastewater treatment. Rather, for the heavy metal process, it would be beneficial to remove as little of these components as possible. This is due to Ca, K and Mg being present at much higher concentrations than the heavy metals and therefore taking up significant amount of 4DS capacity that would preferably be used for heavy metals. Therefore, based on the initial 4DS-103 chemical performance it was deemed not to be optimal material for follow-up tests.

As some of the removal percentages are in essence 100%, the remaining heavy metal concentration is so low that it is below limit of quantification, meaning the lowest possible concentration of the analyte that can be quantified by the method in a reliable way. These results are shown in the tables as < LoQ.

Table 1: Initial laboratory tests results regarding metal removal from SMAT samples with 4D Scavenger S103. BV stands for the bed volume of the 4D Scavenger.

	Al	Ca	Fe	K	Mg	Mn	Ni	Zn
S1	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Inlet	0,064	24,90	0,017	14,24	12,01	0,028	0,006	0,044
Outlet 200BV	0,005	< LoQ	0,003	3,41	0,01	< LoQ	< LoQ	< LoQ
Outlet 400BV	0,004	< LoQ	0,004	7,28	0,04	< LoQ	< LoQ	< LoQ
Outlet 600BV	0,009	< LoQ	0,003	9,11	0,54	< LoQ	< LoQ	< LoQ
Outlet 800BV	0,045	22,16	0,010	14,37	14,13	0,010	0,004	0,021
Outlet 1000BV	0,063	24,91	0,011	13,87	13,91	0,019	0,005	0,039

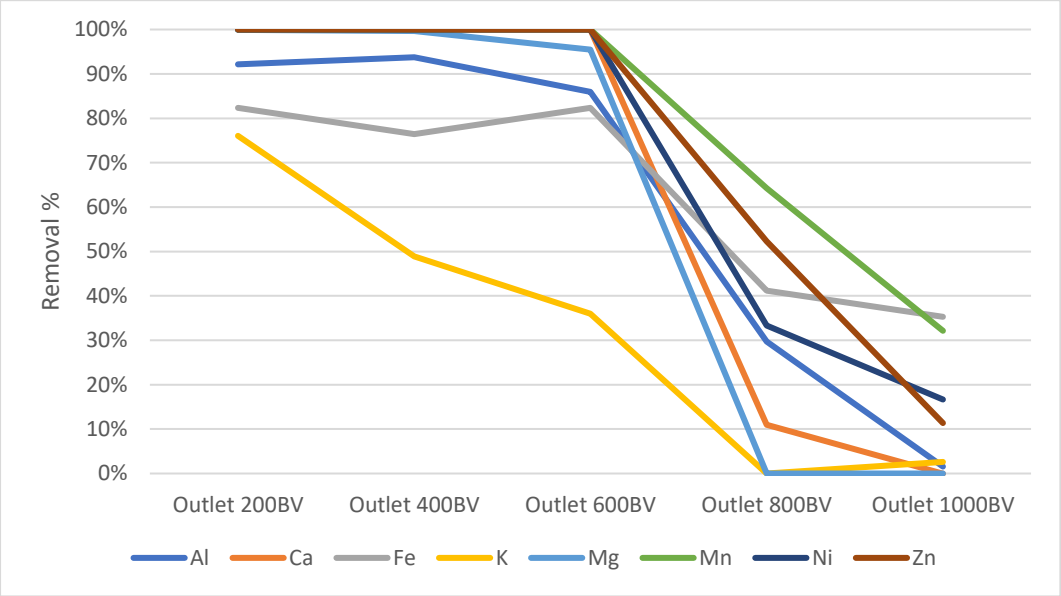

Figure 3: Data from Table 1 represented as metal removal percentages.

Next up, 4DS-104 was tested with the same feed solution. The 4DS-104 possesses almost similar chemical functionality with altered material mesh structure, which can help in tuning the selectivity of the material towards specific metals by controlling the reaction kinetics in play. However, it was noted that with this

specific sample the 4DS-104 shows results rather similar to the results seen from 4DS-103 in Table 1. Therefore, 4DS-104 most likely will not be optimal material to further optimize the process with.

Table 2: Initial laboratory tests results regarding metal removal from SMAT samples with 4D Scavenger S104. BV stands for the bed volume of the 4D Scavenger.

	Al	Ca	Fe	K	Mg	Mn	Ni	Zn
S1	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Inlet	0,064	24,90	0,017	14,24	12,01	0,028	0,006	0,044
Outlet 200BV	0,001	< LoQ	0,001	2,55	0,02	< LoQ	< LoQ	< LoQ
Outlet 400BV	0,002	< LoQ	0,002	6,11	0,01	< LoQ	< LoQ	< LoQ
Outlet 600BV	0,004	< LoQ	0,002	10,99	0,24	< LoQ	< LoQ	< LoQ
Outlet 800BV	0,035	18,16	0,008	14,27	13,43	0,007	0,003	0,015
Outlet 1000BV	0,053	21,91	0,010	14,47	12,10	0,011	0,006	0,029

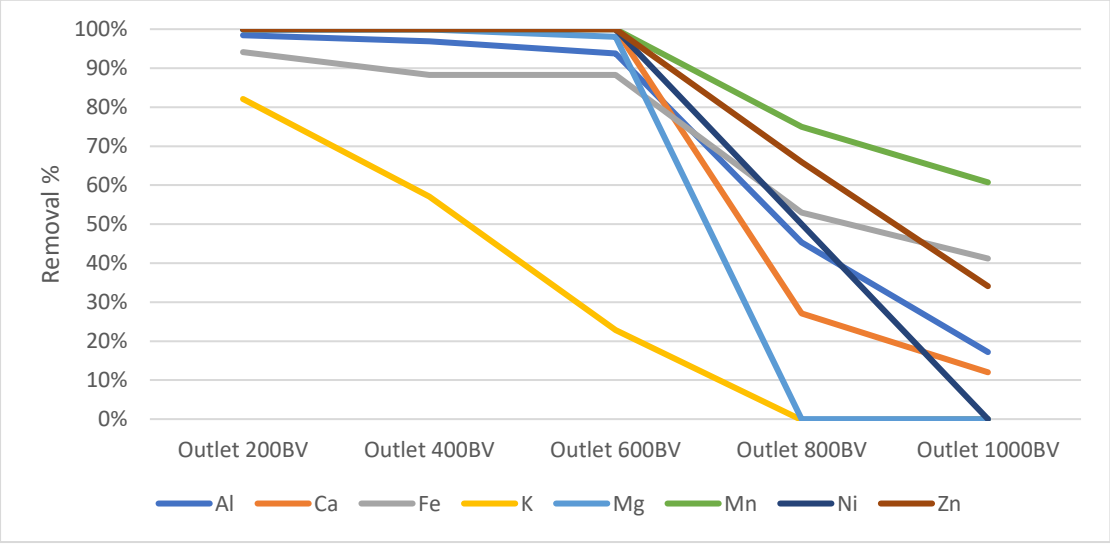

Figure 4: Data from Table 2 represented as metal removal percentages

Next material tested for the more selective heavy metal removal was 4DS-206. Right away it was observed from the results that the 4DS-206 shows greatly reduced up take of Ca, K and Mg. This is evident as the Ca uptake is already rather minimal after 600 BV being treated. Similarly for the Mg the removal can be seen

to drop drastically between 400 and 600 BV. At the same time the heavy metal (Al, Fe, Mn, Ni, Zn) removal efficiency remains at very high level until (and mostly past) 1000 BV. This shows that the 4DS-206 displays great heavy metal selectivity even in the initial tests. Based on this, the 4DS-206 was chosen for more detailed testing.

Table 3: Initial laboratory tests results regarding metal removal from SMAT samples with 4D Scavenger S206. BV stands for the bed volume of the 4D Scavenger.

	Al	Ca	Fe	K	Mg	Mn	Ni	Zn
S1	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Inlet	0,064	24,9	0,017	14,24	12,01	0,028	0,006	0,044
Outlet 200BV	0,004	< LoQ	0,004	7,51	0,03	< LoQ	< LoQ	< LoQ
Outlet 400BV	0,004	5,23	0,001	14,78	4,17	< LoQ	< LoQ	< LoQ
Outlet 600BV	0,003	10,19	0,001	15,40	11,58	< LoQ	< LoQ	< LoQ
Outlet 800BV	0,008	17,26	0,002	14,33	16,49	< LoQ	< LoQ	< LoQ
Outlet 1000BV	0,019	27,86	0,003	14,28	12,51	0,005	< LoQ	0,009

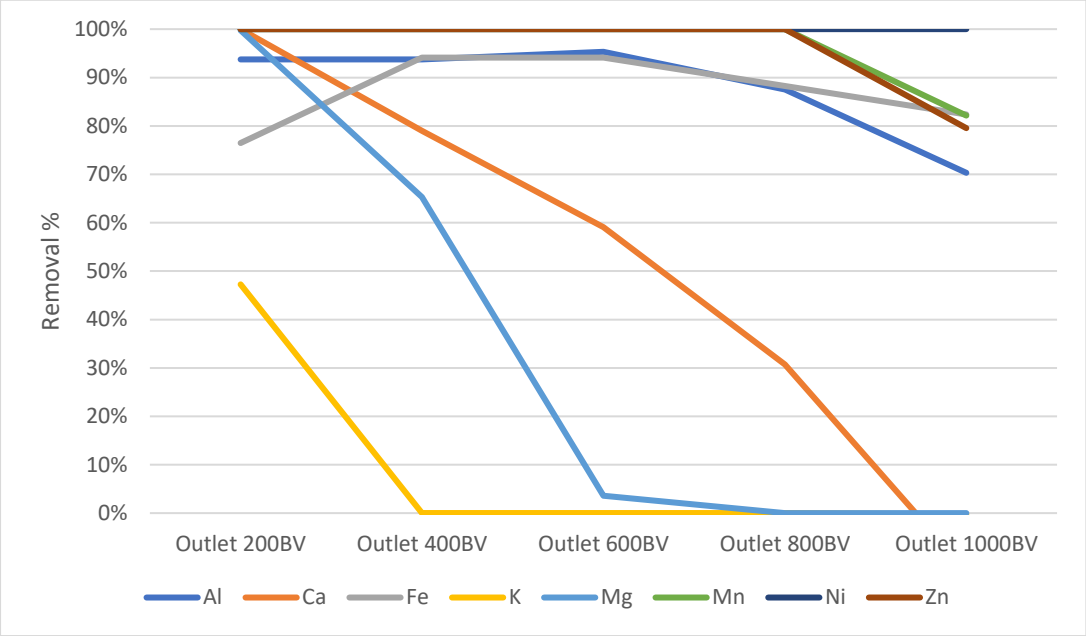

Figure 5: Data from Table 3 represented as metal removal percentages

4DS-206 tests were continued with similar sample to see effects of different process conditions (Table 4). Ability to use higher flowrate of 80 BV/h was tested and the results are seen below in Table 3. Compared to the previous flowrate of 50 BV/h (Table 3), the increase in flowrate could allow for even smaller equipment being needed for the heavy metal removal. The shorter residence time of the wastewater being treated can also in some specific applications increase the selectivity of the material as the components with less affinity will not have adequate time to react with the chosen material. In this case, this was not observed and the tests showed results that very similar to the original results seen in Table 3.

Table 4: Initial laboratory tests results regarding metal removal from SMAT samples with 4D Scavenger S206. BV stands for the bed volume of the 4D Scavenger.

	Al	Ca	Fe	K	Mg	Mn	Ni	Zn
S1	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Inlet	0,064	24,9	0,017	14,24	12,01	0,0286	0,006	0,044
Outlet 200BV	0,011	< LoQ	0,003	9,492	0,03	< LoQ	< LoQ	< LoQ
Outlet 400BV	0,006	3,20	0,006	15,88	8,12	< LoQ	< LoQ	< LoQ
Outlet 600BV	0,007	8,60	0,007	16,23	10,24	< LoQ	< LoQ	< LoQ
Outlet 800BV	0,009	14,85	0,005	14,77	15,13	< LoQ	< LoQ	< LoQ
Outlet 1000BV	0,007	21,02	0,008	14,52	12,91	< LoQ	< LoQ	< LoQ

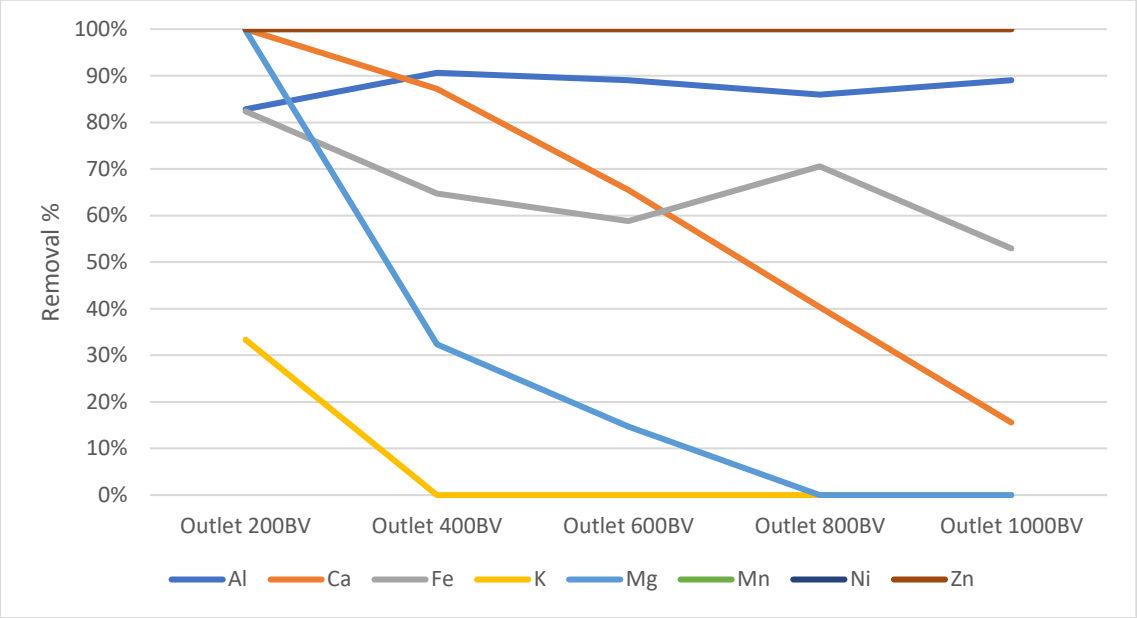

Figure 6: Data from Table 4 represented as metal removal percentages

After more process testing (flowrate, hydraulic residence time, wash chemical amounts) the focus shifted towards scenario where the wastewater being treated would experience sudden spike up in heavy metal concentrations. Although imaginary, the scenario could still be realistic if any of the previous process steps of a WWTP experienced process malfunction, leading to more concentrated wastewater being directed towards the 4D Scavenger. This scenario was tested by spiking the samples received from SMAT with additional zinc to increase the Zn levels up to ~ 0,5 mg/L. Next, the best process parameters obtained from previous tests were employed to see how the 4DS-206 would perform (Table 5).

The results show that even with 10 times the Zn concentration, the 4D Scavenger can remove the target metals efficiently. Additionally, e.g., the Zn removal efficiency can still be seen to remain at very high (~ 99%) level at the 1000 BV mark. This further shows the efficiency of the solution even during a moment of higher heavy metal concentrations at the inlet.

Although, it should be noted that experiments performed in laboratory scale can sometimes lead to results showing better performance than observed in pilot or demonstration scale. This is especially true if the water samples contain components that may precipitate or undergo chemical alteration (oxidation etc) during shipping and storage. Similar effect can be seen if the quality of the water greatly differs during pilot phase and previous laboratory phase. Additional factor that can cause uncertainty with demonstrations focusing on heavy metal removal percentages is if the inlet metal concentrations are extremely small. This is partly due to the fact that increase in uncertainty of analyses correlate with decrease of metal concentrations (the so-called Horwitz trumpet effect).

Table 5: Initial laboratory tests results regarding metal removal from SMAT samples after spiked zinc concentrations with 4D Scavenger S206. BV stands for the bed volume of the 4D Scavenger.

	Al	*Ca*	*Fe*	*K*	*Mg*	*Mn*	*Ni*	*Zn*
S1	*ppm*	*ppm*	*ppm*	*ppm*	*ppm*	*ppm*	*ppm*	*ppm*

Inlet	*0,401*	*21,82*	*0,018*	*14,82*	*11,27*	*0,016*	*0,006*	*0,562*
Outlet 200BV	*0,029*	*0,120*	*< LoQ*	*13,62*	*0,00*	*< LoQ*	*< LoQ*	*< LoQ*
Outlet 400BV	*0,009*	*0,34*	*< LoQ*	*15,67*	*7,37*	*< LoQ*	*< LoQ*	*< LoQ*
Outlet 600BV	*0,017*	*9,45*	*< LoQ*	*14,74*	*15,48*	*< LoQ*	*< LoQ*	*< LoQ*
Outlet 800BV	*0,013*	*17,18*	*< LoQ*	*14,56*	*12,18*	*< LoQ*	*< LoQ*	*0,005*
Outlet 1000BV	*0,040*	*19,68*	*< LoQ*	*14,45*	*11,09*	*< LoQ*	*< LoQ*	*0,007*

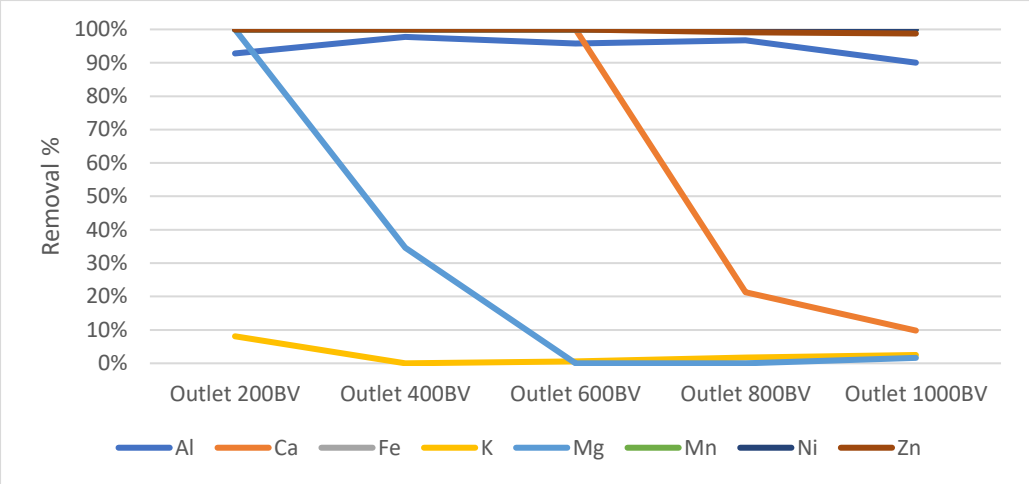

Figure 7: Data from Table 5 represented as metal removal percentages.

In addition to the heavy metal removal testing, the washing process was tested with different chemical concentrations and wash amounts. As an example, washing results from an experiment are shown below. For the washing efficiency, usually the heavy metal washing efficiency was monitored as that is the most relevant performance metric for the washing process. The washing process tests were performed by first conducting the loading of the 4D Scavengers, followed by chosen washing operation. Samples were taken during the washing step to analyse the performance. Below in Table 6, are results from a 5% H2SO4 washing process that shows that the heavy metals monitored in this experiment are effectively washed out between bed volumes 2-4.

Table 6: Wash testing results of 4DS-206 with 5% H2SO4 after heavy metal removal experiment

	Al	***Zn***
	ppm	***ppm***
Wash 1BV	*8,49*	*33,27*
Wash 2BV	*187,50*	*1049,6*
Wash 3BV	*162,73*	*289,17*
Wash 4BV	*15,01*	*22,45*

Wash 5BV	*6,10*	*2,09*
Wash 6BV	*0,41*	*0,55*

In addition to the results shown above, during the laboratory phase the other relevant process parameters were optimized. The choice of washing chemicals (Sulfuric acid and sodium hydroxide) was largely impacted by the fact that these are often the most cost-effective chemicals to deploy and are usually already used in industrial WWTP settings. The laboratory phase optimized the washing process so that it was deemed for 3-5 BV of sulfuric acid and 2-4 BV of sodium hydroxide to be enough to achieve satisfactory process washing result. In addition, it was shown that for this specific application, flowrate of < 50 BV/h should be utilized during the pilot. Although, it should be noted that final flowrate would be chosen on-site depending on if e.g., prefiltration would cause limitations to the usable flowrate.

Overall, the laboratory phase led to 4DS-206 being a clear choice for the piloting phase of the project as it was able to show high metal removal efficiency during small concentrations of heavy metals (original SMAT samples) as well as during higher concentrations (SMAT samples spiked with Zn). In addition, it was noted during laboratory phase that the pilot phase of the project should also include adequate prefiltration as it was observed that SMAT delivered samples often contained some solids that had settled to the bottom of the containers during sampling. Initial laboratory tests also showed that if the solids were directed to the 4DS-206 without prefiltration, some initial signs of clogging of the scavenger structure could be observed.

2.2 Equipment modifications and design

For the pilot project in SMAT's premises, one of the pilot equipment of Weeefiner was modified. The process included process design with modifications to P&ID of the equipment as well as mechanical changes. The final design of the equipment was equipped with 40 dm^3 of the 4DS-206 and with a design flowrate of 1 m^3/h. Pretreatment for the system was chosen to be performed with media (glass particles) filtration to remove any excess solids found in the feed stream. The operation of the pilot unit is automated and can be remotely monitored and controlled. In addition, the equipment was designed to be able to provide samples to the HM Box (controlled by UNIFI) for heavy metal analyses.

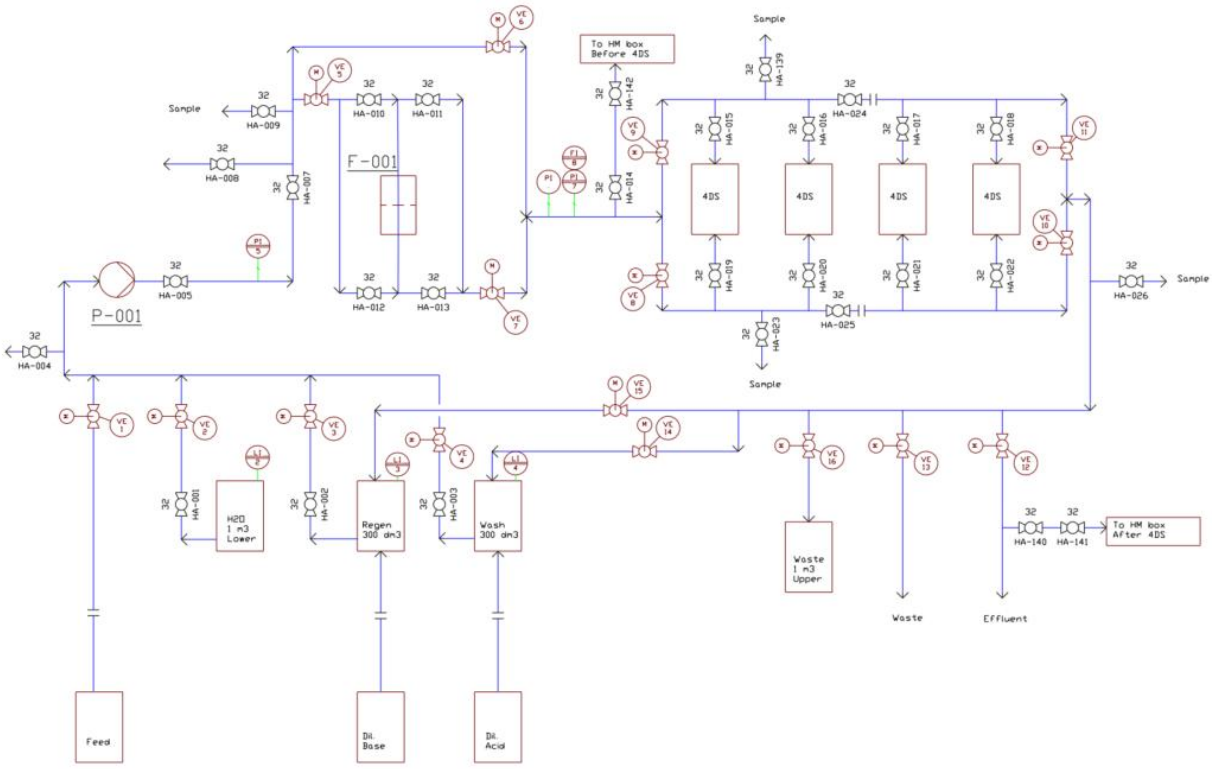

Figure 8: PID draft of the pilot equipment.

During the design phase the 3D model of the equipment was also updated to depict of the equipment better. The rough 3D model of the equipment is shown below in Figure 9. The equipment was equipped with 2x 1000 dm^3 IBCs that could be utilized for interim storage of any waste solutions produced during the pilot. The equipment also included 2x 300 dm^3 tanks for the chemicals that are used during the process (sulfuric acid and sodium hydroxide). In the middle the four 4D Scavenger modules can be seen installed into racks. Before the 4D Scavengers, the equipment also included 60 dm^3 prefilter column (glass particles as filtering media).

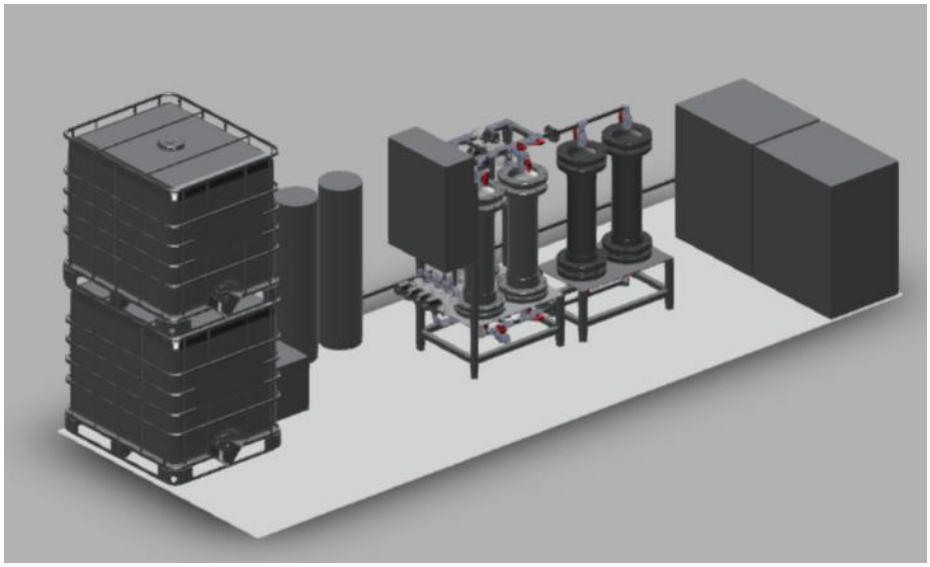

Figure 9: 3D model of the inside of the pilot equipment.

The pilot unit was installed into an insulated 20' shipping container. This allows for two things: containment of the testing unit with adequate ambient control and chemical leakage prevention as well as the easier shipping and commissioning of the pilot unit. After delivery, all that must be done is to connect incoming and outgoing water connections, plug in the electricity and fill up the chemical tanks – after which the equipment should be functional for piloting runs.

Figure 10: Pilot equipment used for the remediation of heavy metals in Brandizzo's WWTP under Use Case 3.

3.0 Conclusions

The 4D Scavenger -based pilot equipment for heavy metal removal development was started in laboratory scale. The focus was first to establish the most promising 4DS material to be further tested. After the 4DS-206 was chosen as the most potential heavy metal selective materials, the process parameters were optimized to get starting point for the future piloting tests.

Initial plan was to use WWTP inlet water, which has higher heavy metal concentrations as reference wastewater in laboratory tests, but its quality was insufficient 4DS. That's why tests were done by using outlet effluent of WWTP. However, scenario of higher metal concentrations in the feed was also successfully tested by spiking the sample with Zn. Overall, the 4DS development in the laboratory phase led to a very promising heavy metal removal process.

Next, in the project, the equipment for the piloting was designed and built. This started off with designing the PID of the equipment, taking into account the specific needs of the project. After the 3D model was drafted, the equipment was installed into a suitable shipping container to allow for it to be safely and easily shipped to location for commissioning.

The Mediterranean Sea and its surrounding regions support a diverse variety of essential socioeconomic activities. It is one of the highly exploited water ways and the influence of anthropogenic activities on its marine habitats and ecosystems has grown significantly since the industrial revolution. Because of this, the Mediterranean Sea basin is very vulnerable to chemical contamination and build-up. To safeguard the Mediterranean Sea basin from contaminants for emerging concerns (CoEC), iMERMAID will integrate, coordinate, and synergize innovative preventive, monitoring, and remediation solutions. iMERMAID will build an evidence-based multidimensional framework that will guide policymaking and transform societal perceptions to reduce CoEC usage, emissions, and pollution. Furthermore, next generation sensor and remediation solutions will be developed within iMERMAID to monitor and remove prioritized chemicals from its source while reducing upstream pollution. iMERMAID builds an ideal interdisciplinary team by bringing together prominent SMEs, researchers, regulators, and innovation professionals who have been essential in improving the knowledge and awareness of CoEC. Beyond state-of-the-art techniques, iMERMAID will strive to strengthen regulations against CoEC, expand economic possibilities and competitiveness, improve the standard of living for EU residents, while preventing the accumulation of chemical pollution in the Mediterranean Sea basin. iMERMAID will empower the efforts to create a zero pollution, contaminant free waters by enabling the Chemical Strategy's goals to become a practical reality.

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