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Layman report

Life Clean Up

LIFE16 ENV/ES/000169

INDEX

PROJECT
LIFE
CLEAN UP

LIFE Project
Number LIFE16 ENV/ES/000169.

LIFE CLEAN UP

01

INTRODUCTION AND PROJECT BACKGROUND

Total budget
1,492,412€

7

Associated
Institutions

This document is a summary of the main activities developed in the LIFE CLEAN UP project (LIFE Project Number LIFE16 ENV/ES/000169).

The project started during the second semester of 2017, in October, it came to an end in September 2020, and it was extended until June 2022 after fulfilling the pursued objectives, putting the scientific knowledge at the service of the environment.

The project is the result of close cooperation between Universidad Católica San Antonio de Murcia (UCAM, from Spain) acting as coordinator, University of Bari Aldo Moro (UNIBARI, from Italy), Consiglio Nazionale delle Ricerche - Institute for Physical and Chemical Processes (CNR-IPCF, from Italy), Centro Tecnológico Nacional de la Conserva y la Alimentación (CTC, from Spain) and the private partners involved HIDROGEA, HIDROTEC and REGENERA (all of them from Spain).

The total project budget was 1,492,512 €; nearly 60% (895,506 €) was co-funded by the European Union through the Life Programme as a close-to-market project.

02

WHAT IS **LIFE CLEAN UP?**

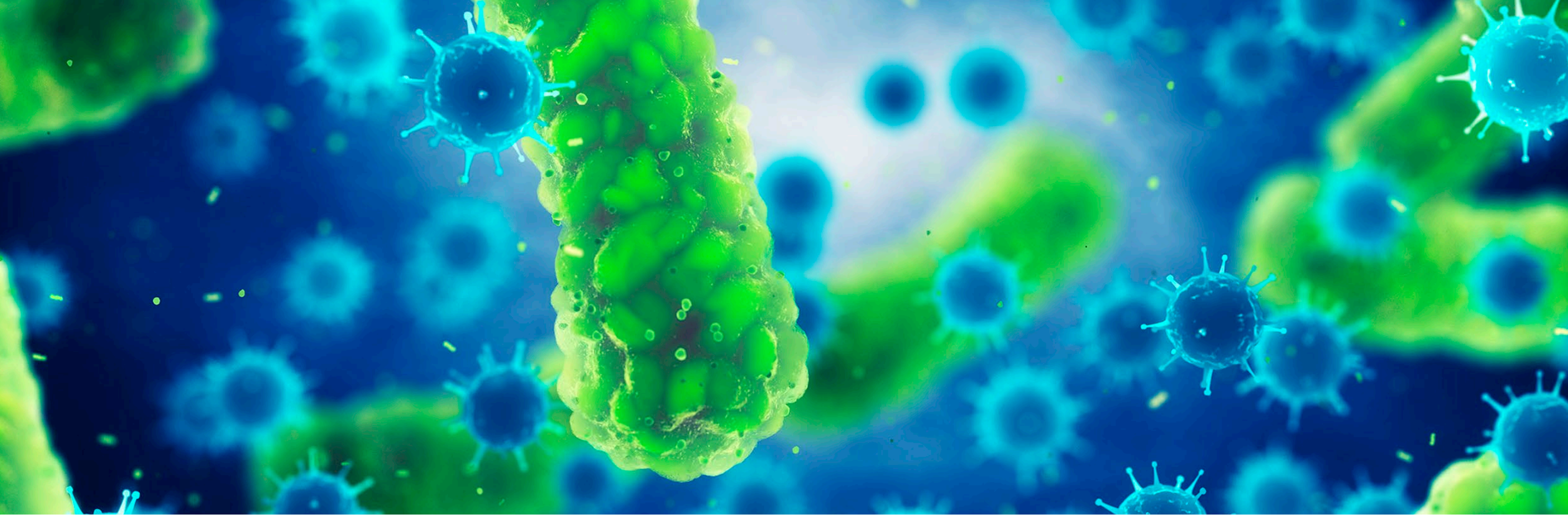
LIFE CLEAN UP was made to improve the management of wastewater depuration by an efficient and environmentally friendly technology, that allows to obtain treated water free of emerging pollutants (EPs).

2.1 THE **PROBLEM**

Water supply is essential for human livelihood, socio-economic development, and ecosystem health, especially in arid or semiarid areas, where water scarcity requires adequate management of water resources. Water scarcity has become one of the main risks to sustainable development in many areas of the world. Thus, in a scenario where water stress and climate change are becoming more and more pronounced, water reuse is presented as a reliable alternative to conventional water resources. But to ensure safe water reuse, a legal framework is required to guarantee safety and a high level of protection for the environment, as well as for human and animal health. To this end, Regulation (EU) 2020/741 of the European Parliament and of the Council of 25 May 2020 establishes minimum water quality and monitoring requirements for reuse as agricultural irrigation water, in the context of integrated water management. This regulation, in addition to microbiological quality indicators (*E. coli, C. perfringens*, spores, and coliphages), considers substances of emerging concern (heavy metals, pesticides, disinfection by-products, pharmaceuticals, etc.) as additional requirements at the level of risk assessment for human health.

This is the case of emerging pollutants. Water contamination by these contaminants poses significant environmental risks. These pollutants come mainly from industry, domestic use (pharmaceuticals, cosmetics, cleaning, etc.), and agriculture.

The removal of these pollutants is problematic because conventional purification methods fail to remove these compounds completely, and because advanced water treatment methods are not feasible due to their high costs. Thus, the low capacity of conventional technologies in EPs removal, together with increasing policies of legal restrictions on effluent discharge and on the reuse of reclaimed water, and environmental awareness, poses a new challenge that requires effective and environmentally sustainable technological responses.



2.2 **OBJETIVE**

In the light of the exposed problem, the main objective of the LIFE CLEAN UP project is the validation of a system for the removal of EPs and pathogenic microorganisms, which are not adequately removed by conventional wastewater treatment systems.

For this proposal, a combination of technologies was employed, that include filtration techniques, adsorption and advanced oxidation processes (AOPs).

Specifically, **the objectives** to be addressed were:

01
Optimize the adsorption capacity of the materials used for different EPs families.

02
Adapt the methodology for the development of polymers and biomaterials for industrial scale-up.

03
Economically and environmentally validation of the technology of the applied advanced oxidation processes (AOPs) such as light pulse, photocatalysis and photosensitizers.

04
Validate and demonstrate a prototype that integrates the proposed technologies (retention by adsorbent materials and destruction by AOPs) on a semi-industrial scale in a WWTP.

05
Validate the process by comparing the results obtained in the plant with those obtained at the laboratory scale.

06
Demonstrate the use of renewable energies for the implementation of the system on an industrial scale.

07
Facilitate replication at industrial scale through feasibility studies and cost- benefit analysis.

08
Enable transfer of the purification system to other industrial sectors.

09
Raise awareness of emerging pollutants, their causes, consequences, and options to minimize their occurrence.

10
Develop a commercial product to be launched after the project is completed.

2.3 DESCRIPTION
OF METHODOLOGICAL SOLUTION

A combination of technologies has been employed to remove EPs from the effluent of WWTPs. Figure 1 shows the experimental scheme of the pilot plant. The system has a first pretreatment stage based on micro and ultrafiltration [1], which ensures the microbiological quality of the water and extends the useful life of the cyclodextrin (CDs)-based polymers, as an efficient material to adsorb a large amount of EPs [2], and which can be reused. At the outlet, the water is subjected to an AOP by photocatalysis (FC) [3] to degrade non-retained pollutants. The desorption product loaded with EPs is accumulated in a reject tank for further treatment by means of a pulsed light system (PL) [4] that destroys the desorbed pollutants.

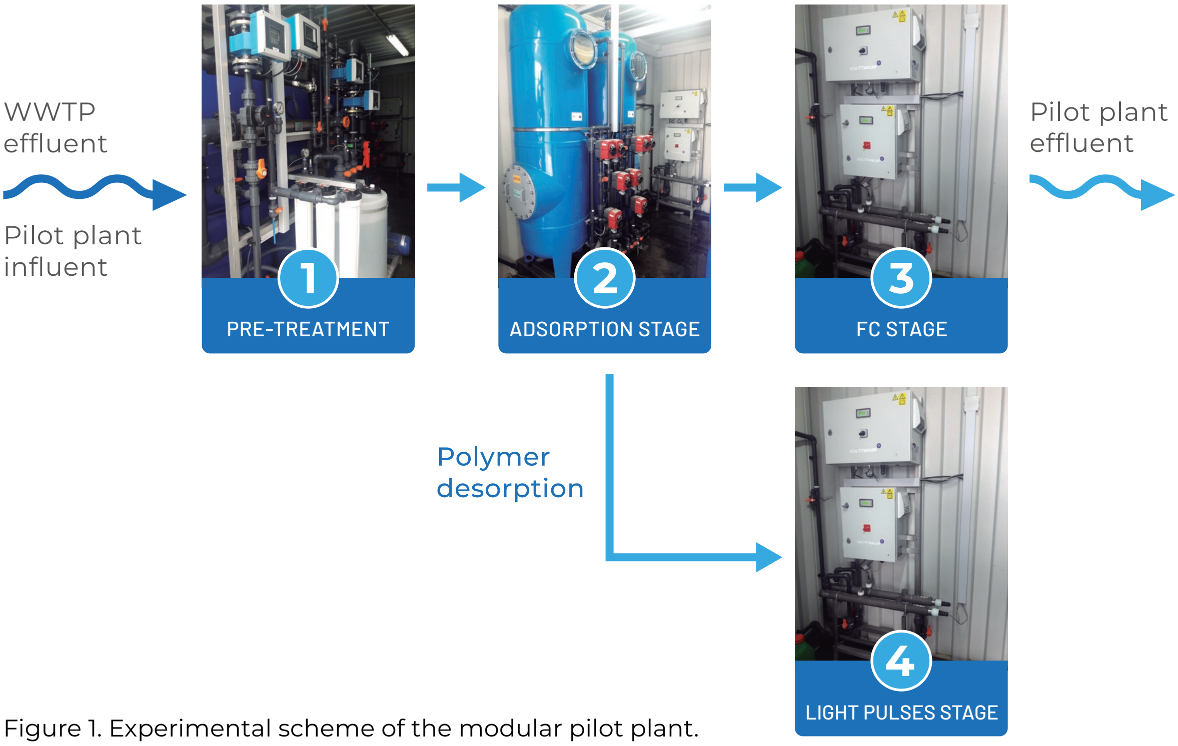
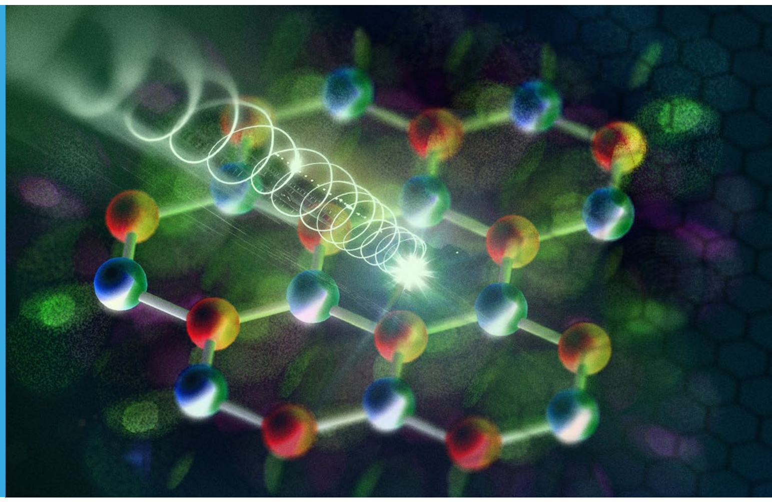
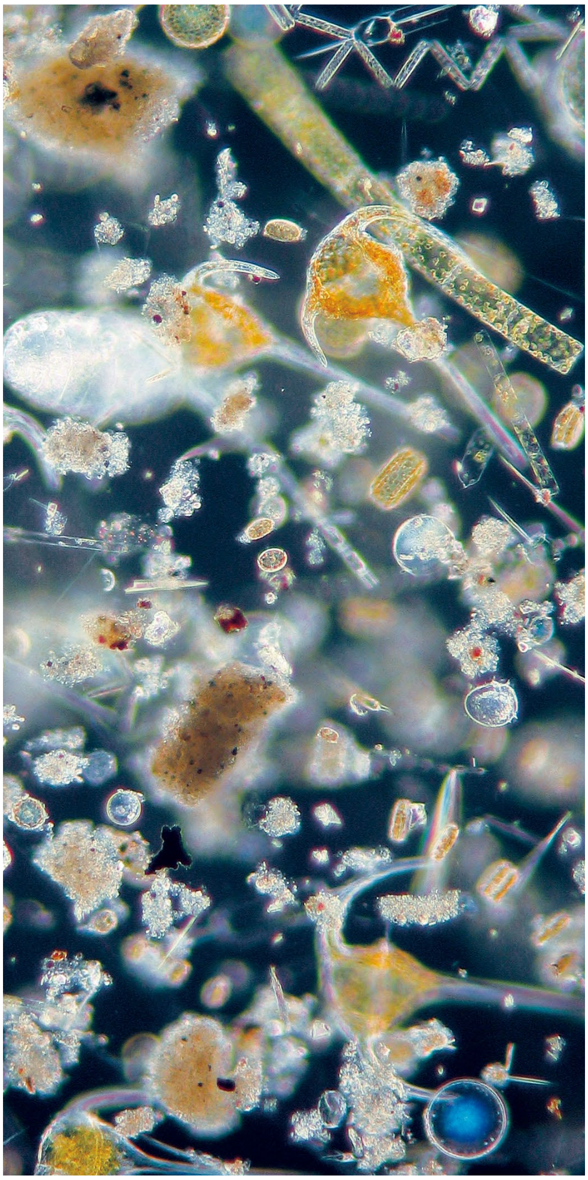


Figure 1. Experimental scheme of the modular pilot plant.

Thanks to this project, a prototype has been built and tested. It has a unit containing adsorbent material inside, which will be reusable, and that is connected with two photocatalysis lamps to degrade the remaining pollutants and with a light pulse equipment that eliminate the EPs trapped in the internal cavity of the cyclodextrin(CDs)-based polymers once it is regenerated.





2.4 TECHNOLOGIES **EMPLOYED**

01. Pretreatment - Phase Filtration

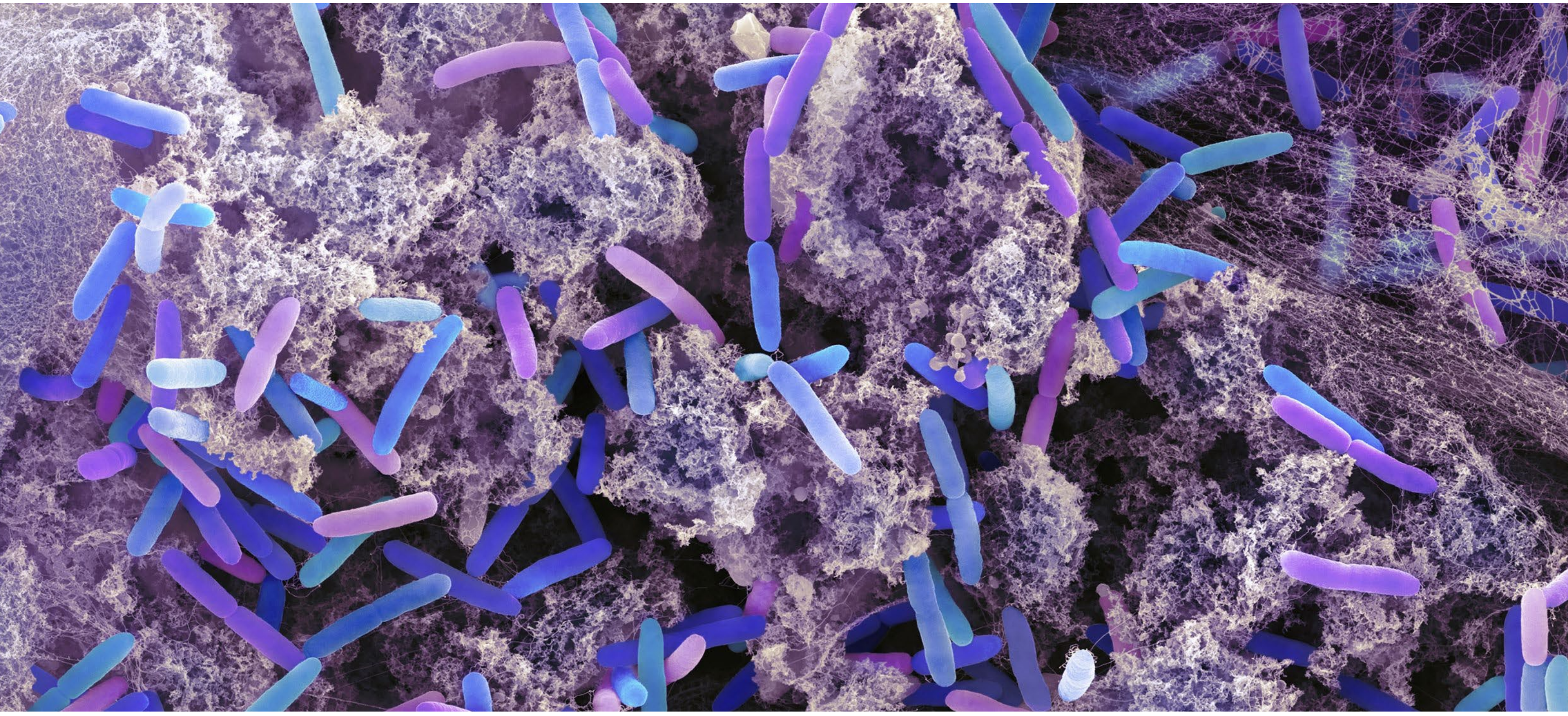
Filtration is a physical method based on the retention and elimination of pollutants depending on the minimum particle size, with a high disinfection capacity. In this case, micro and ultrafiltration membranes have been used as a treatment to improve water quality and as a pretreatment to increase the effectiveness of subsequent treatmet steps.

02. Adsorption phase - Cyclodextrin polymers

CDs are cyclic oligosaccharides consisting of D-glucose units linked by alfa-1,4- glucosidic bonds, obtained from the enzymatic processing of starch. The, alfa, beta and gamma CDs, consisting of six, seven and eight D-glucose units, respectively, and are the most common. They can be cross-linked by polymerizing agents such as epichlorohydrin (EPI) to form an insoluble network of larger size, and with different properties than those of the initial CDs monomer, easily removable from wastewater after the pollutants adsorption, and reusable, since once saturated, the polymer can be regenerated by a chaotropic agent, allowing their use several times.

Indeed, the three-dimensional network formed, confers to the polymer amphiphilic features with both, hydrophilic properties due to the occurrence of glucose units (CDs), mainly hydroxyl groups, and hydrophobic properties, mostly due to the methyl groups and ether bonds of the cross-linking agent, and CD-glyceryl bonds. This is advantageous in the removal of trace levels of pollutants of different nature in complex solutions, versus conventional sorbents like active carbon or ion exchange resins.

In fact, the three-dimensional network enabling the polymer to trap pollutants through additional interactions (external to the inclusion sites), that supplement the sorption capacity considering inclusion complexes alone. Owing to this additional binding, it has been demonstrated that the stability constants of these pollutant complexes are often greater than those of native CD-pollutant interactions, justifying the whole range of sorption results obtained in this project.



OXIDATION

03.

Oxidation phase – Photocatalysis

Photocatalysis is a sustainable technology based on a photochemical reaction under the presence of a light source on the surface of a catalyst or substrate, consisting of a semiconductor material that accelerates the reaction rate. During the process, both oxidation and reduction reactions take place, involving the generation of hydroxyl radicals, with high oxidizing potential.

In this way, most of the pollutants in the water can be removed. TiO_2 is the most widely used photocatalyst for the degradation of organic pollutants due to its low cost, stability, low toxicity, and environmental friendliness.

04.

Regeneration phase and destruction of retained contaminants – Pulsed light

Regeneration of the adsorbent is carried out with a 0.1 M NaCl solution, which does not alter the adsorption capacity of the polymer, allowing easy and fast regeneration, and enabling almost complete recovery of the retained contaminants. This avoids the use of organic solvents or high energy costs, in accordance with the principles of "Green Chemistry"

Pulsed light systems produce a continuous spectrum of light ranging from infrared to UV-C, with the UV-C sub-band being the most effective in the degradation of emerging contaminants.

PL equipment is typically designed and built for the inactivation of microorganisms by working discontinuously.

For this reason, it is noteworthy that, in this project, the pulsed light equipment has been adapted to allow continuous work, by incorporating a special pipe that passes through the treatment chamber, allowing the UV rays to pass through. To date, there is no equipment with these characteristics on the market.

03

MAIN ACTIONS
AND RESULTS

Pollutants destruction

80% pharmaceutical products 100% phytosanitary residues

01.
Characterization of EPs in treated wastewaters in WWTPs (Waste Water Treatment Plants).

The identification of contaminants that were present in the analyzed wastewater on a recurring basis allowed us to focus our attention on the subsequent project's actions. In this way, a list including compounds belonging to different families, paying attention to their danger and incidence in analyzed waters was elaborated, and the analytes included were thoroughly surveyed alongside the project, to apply the most realistic working conditions.

02.
Optimization of the process for obtaining different adsorbent materials of polymeric base

After comparing different absorption materials, all the obtained results in terms of adsorption, desorption, regeneration capacity, contact time and costs, pointed the EPI-CDs Polymer as the one to be used as a model for semi-industrial scaling in the prototype. However, the synthesis of this material at such scale present problems due to the toxicity of the synthesis process. For this reason, a MnCDs-BDE, polymer was finally used in the semi-industrial prototype.

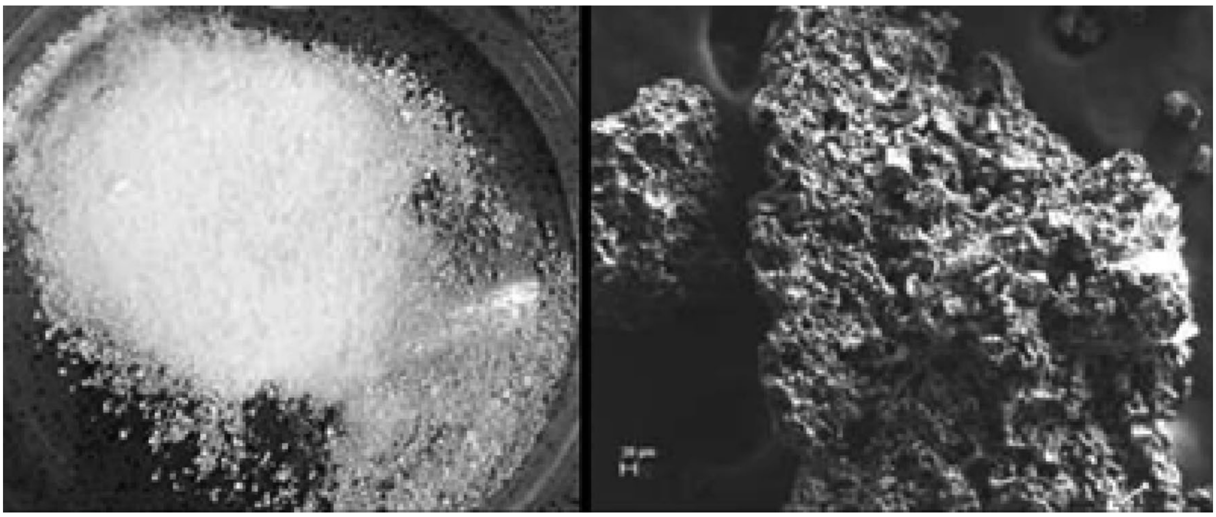


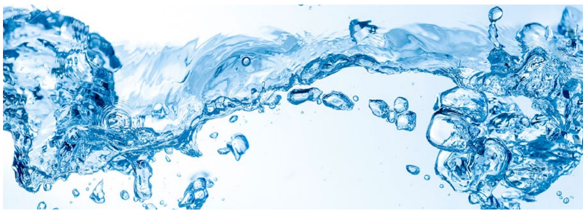
Figure 2. Photographs of epichlorohydrin cyclodextrin polymer.
(A) (left) Dry EPI-CDs polymer. (B) (right) SEM micrography of EPI-CDs polymer

03.
Optimization of innovative Advanced Oxidation Processes

Photocatalysis tests have shown that short treatments for 5 seconds are not effective for most compounds. So, contact times for the semi-industrial prototype vary between 5 and 25 seconds.

The passage of contaminated water through the PL stage increased the degradation rate of EPs.

04.
Preliminary studies and calculations of the prototype at semi-industrial level and installation, commissioning and optimization



Different units of the prototype were designed and sized: frame/container, feed tank, process pump, microfiltration, ultrafiltration, adsorption columns, measure and control instrumentation, electrical installation, piping, valves, photocatalysis, product tank, desorption process pump, desorption tank.

The system consists of two alternative adsorption lines, one active and the other in regeneration/desorption/repose to treat a flow of 3000 L/h. The rest of the equipment installed is common. A submersible pump has been considered for the continuous collection of effluent from the WWTP, so that it is conveyed to a tank for feeding the CLEANUP system. The tank is equipped with minimum and maximum level gauges, so that it activates this pump providing the water necessary for the cleaning process.

Once the effluent is in the feed tank, a centrifugal pump takes the effluent from this tank and pass it through the pre-treatment system, consisting of a microfiltration and ultrafiltration system fitted with ceramic membranes, to prevent unwanted substances from passing through and causing clogging of the adsorbent material.

Once the water has been physically conditioned, and after control of pH and turbidity parameters, it is introduced into one of the two adsorbent material filters.

There are always one in service and the other in the desorption or standby process, so that the process is continuous at all times. At the end of the adsorption process, the water passes through a photocatalysis unit, so that any traces of emerging pollutants that have not been retained by the adsorbent material filter are eliminated by oxidation.

Finally, the water is led to a product water tank, which provides the necessary water storage for the desorption process, backwashing and sponging of the adsorbent material beds.

The water is discharged from the tank through an overflow into the outlet stream of the WWTP, so that the tank always remains full and with moving water.

INNOVATION

The desorption process is carried out when the estimated depletion time for the adsorbent is reached. For this purpose, a membrane pump takes the desorbent solution, at the calculated concentration and flow rate, to introduce it into the cyclodextrin polymer column, in order to produce the displacement of the pollutant towards the desorbent solution, leaving the cyclodextrin polymers with their initial adsorption capacity.

The desorption product loaded with contaminants will be accumulated in a rejection tank for further treatment. This product will be pumped at a suitable rate through a pulsed light system, capable of destroying the contaminants entrained in the desorbent solution. This process has been optimized so that the duration of the process is somewhat shorter than the useful saturation time of the bed, so that the light pulse equipment can be sized in a rational way.

05.
Validation of the technology and feasibility at semi-industrial level

The prototype has been functioning for its validation for one year (2021). The system has an adsorption capacity of around 45% for pharmaceutical products and 100% for phytosanitary residues. The pollutant destruction phase by photocatalysis improves the overall removal performance of pharmaceuticals up to 80%.

In addition, the system allows the complete disinfection of the wastewater, so it can be considered as an effective tertiary treatment that does not require the addition of reagents (which gives it clear advantages over chlorination, the most used disinfection system to date) and also allows the elimination of *C. perfringens* spores, which are highly resistant to the most commonly used treatments in WWTPs such as UV radiation and chlorination. Therefore, from the microbiological point of view, the proposed system allows compliance with the new Regulation (EU) 2020/741, on the minimum requirements that reclaimed water must meet for agricultural reuse.

Also noteworthy is the regeneration and regenerative capacity of the polymer. Considering the reusability tests performed, dissimilar physicochemical characteristics of the inlet water, regeneration step each $200m^3$ of water cleaned, it would be advisable for the treatment of this type of water to change the polymeric material every six months of continuous operation.

Figure 3. External view of the prototype.

VALIDATION

04

PROJECT SUSTAINABILITY **AND REPLICABILITY**

Two Replication sites and five Transfer sites were selected. The results obtained identify the agri-food sector as a target for the initial phase of commercial exploitation and market launch of the LIFE CLEAN UP technology.

For the chosen replication and transfer sites, the presence of the analytes of interest has been studied in order to determine their suitability for the introduction of the proposed solution. For the two replication sites (EMAHSA and SOLVIC) and three of the transfer sites (Citromil, Ecologia Rizzi Arcangelo and Vivolat), an on-site visit has been carried out.

The Life Clean Up commercialization strategy is divided into two phases, a first phase that starts immediately after the end of the project, and with applications based on the favorable results of the transfer study to the agro-industrial sector, and a second phase in the medium term in which the replication in WWTPs is already contemplated.

The target market for the first phase is industrial plants with a maximum treatment flow of 200 m^3/day, for which commercial scenarios such as dairy or agri-food industries are proposed, as well as other industrial sectors such as pharmaceuticals or healthcare. A prior technical and economic feasibility study must be carried out to verify that the LIFE Clean Up technology is viable according to the characteristics of each client.

For the second phase, a modification of the polymer is required to increase the flow treated by the system in order to replicate it in WWTPs. Research is proposed to search and testing of larger surface area cyclodextrin polymers, the reduction of their swelling capacity or even the change in the type of crosslinker or introduction of ionic and cationic groups to improve their versatility, the latter can also reduce manufacturing costs. At this stage, the creation of the Life Clean Up joint venture is foreseen for the commercialization of the developed technology.

05

PROJECT **DISSEMINATION**

Total impressions
171,070

Articles
54

During these 5 years, the Life Clean Up project has carried out dissemination and outreach activities, both to the general public and to scientists in similar areas. Regarding the general public, during this time Life Clean Up appeared in 54 articles (both print and online), 1 documentary in the regional Tv and 3 Radio interviews, and has organized 1 contest on a short movie festival, 1 contest of spots to raise awareness among the general public, 3 guided media visits at EDAR Cabezo Beaza, 2 visits from students with interest common with wastewater and its recycling. The project has also participated in 2 science weeks (a regional event which more than 25,000 participants, focusing on science), 14 networking events achieved and different congresses. Partners have achieved more than 60,000 people, just in the events and congress. Furthermore, the online media, social media, views on our video and the page-views of our website ascend to 171,070.

Figure 4. UCAM researcher, received master´s students to explain how the industrial prototype of the project works and its sustainability.

Figure 5. Media visit to show the final result to Dr. Isabel Fortea, Director General for Scientific Research and Innovation in the Region of Murcia.

Regarding the technical communication of the LIFE CLEAN UP project, several articles have been published in scientific journals (11), some of them of high impact such as “Journal of Hazardous Materials”, “Chemical Engineering Journal”; or “Science of the Total Environment”.

We have participated in ten national and sixteen international conferences and fairs, as well as in six workshops and seminars. A project conference was also organised, with the participation of researchers from other projects and companies related to water management and treatment. Also, five technical visits to the demonstration plant have been organized to encourage and support the replication and transfer of the new treatment system to other locations and sectors.

Figure 6. Dr. Jose Antonio Gabaldón, principal researcher of the project, shows the prototype to the President of the Region of Murcia, Fernando López Miras, during UCAM Science Week.

Figure 7. Project scientists participated in the LIFE AMIA network in 2022.

PROJECT
LIFE
CLEAN UP

(LIFE Project N° LIFE16 ENV/ES/000169).

TRANSFER SITE
CITROMIL
Citrus processing company
Murcia (Spain)

MINOR TRANSFER SITES
Three cheese companies
Visit: 30/03/2022
Barbianco, Gioia de Colle (Italy)

REPLICATION SITE
EMAHSA
WWTP
Huelva (Spain)
Visit: 12/04/2022

REPLICATION SITE
S.O.L.V.I.C.
WWTP
Canosa di Puglia (Italy)
Visit: 30/03/2022

TRANSFER SITE
Ecologica RIZZI
Milk processing company
Gioia de Colle (Italy)
Visit: 30/03/2022

More information about the project:
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