(Projects funded under the Call 2014 onwards must use this format)



# LIFE16 ENV/ES/000169

# **Final Report**

# Covering the project activities from 01/10/2017<sup>1</sup> to 30/06/2022

Reporting Date<sup>2</sup>

# 30/09/2022

LIFE PROJECT NAME or Acronym

# LIFE CLEAN UP

Data Project

Project location:	Murcia, Spain
Project start date:	01/10/2017
Project end date:	30/09/2020 Extension date: 30/06/2022
Total budget:	1,492,512 €
EU contribution:	895,506 €
(%) of eligible costs:	

### **Data Beneficiary**

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<sup>&</sup>lt;sup>1</sup> Project start date

<sup>&</sup>lt;sup>2</sup> Include the reporting date as foreseen in part C2 of Annex II of the Grant Agreement

### This table comprises an essential part of the report and should be filled in before submission

Package completeness and correctness check	
Obligatory elements	✓ or N/A
Technical report	
The correct latest template for the type of project (e.g., traditional) has been followed and all sections have been filled in, in English	✓
In electronic version only	
Index of deliverables with short description annexed, in English In electronic version only	$\checkmark$
Mid-term report: Deliverables due in the reporting period (from project start) annexed	$\checkmark$
<u>Final report</u> : Deliverables not already submitted with the MTR annexed including the Layman's report and after-LIFE plan	
Deliverables in language(s) other than English include a summary in English	
In electronic version only	
Financial report	
The reporting period in the financial report (consolidated financial statement <b>and</b> financial	$\checkmark$
statement of each Individual Beneficiary) is the same as in the technical report with the	
exception of any terminated beneficiary for which the end period should be the date of the	
termination.	
Consolidated Financial Statement with all 5 forms duly filled in and signed and dated	<ul> <li>✓</li> </ul>
<i>Electronically Q-signed or if paper submission signed and dated originals</i> <sup>*</sup> <i>and in electronic version (pdfs of</i>	•
signed sheets + full Excel file)	
Financial Statement(s) of the Coordinating Beneficiary, of each Associated Beneficiary and of each	$\checkmark$
affiliate (if involved), with all forms duly filled in (signed and dated). The Financial Statement(s) of	
Beneficiaries with affiliate(s) include the total cost of each affiliate in 1 line per cost category.	
In electronic version (pdfs of signed sheets + full Excel files) + in the case of the Final report the overall summary	
forms of each beneficiary electronically Q-signed or if paper submission, signed and dated originals*	
Amounts, names and other data (e.g., bank account) are correct and consistent with the	$\checkmark$
Grant Agreement / across the different forms (e.g. figures from the individual statements	
are the same as those reported in the consolidated statement)	
Mid-term report (for all projects except IPs): the threshold for the second pre-financing	N/A
payment has been reached	
Beneficiary's certificate for Durable Goods included (if required, i.e., beneficiaries claiming 100%	N/A
cost for durable goods)	
Electronically Q-signed or if paper submission signed and dated originals* and in electronic version (pdfs of	
signed sheets)	
Certificate on financial statements (if required, i.e., for beneficiaries with EU contribution ≥750,000	N/A
€ in the budget)	
Electronically Q-signed or if paper submission signed original and in electronic version (pdf) Other checks	<u> </u>
	$\checkmark$
Additional information / clarifications and supporting documents requested in previous letters from	•
the Agency (unless already submitted or not yet due) In electronic version only	
This table, page 2 of the Mid-term / Final report, is completed - each tick box is filled in	<ul> <li>✓</li> </ul>
In electronic version only	

 $\ast$  signature by a legal or statutory representative of the beneficiary / affiliate concerned

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AOPs	Advanced Oxidation Processes
ABs	Associated beneficiaries
CTC	Centro Tecnológico de la Conserva
CNR -	Consiglio Nazionale delle Ricerche - Institute for chemical and
IPCF	physical processes
CDs	Cyclodextrins
C2M	Close to Market
СВ	Coordinating beneficiary
EPs	Emerging pollutants
FC	Photocatalysis
PL	Pulsed light
UCAM	Universidad Católica de Murcia
UNIBA	University of Bari
WWTP	Wastewater Treatment Plant

# 2. List of keywords and abbreviations

## 3. Executive Summary

The objective of the final report is to sum up all the actions carried out from the beginning of the project until its end, pointing at the main drawbacks and limitations, identifying variations regarding the project proposal and outlining the continuation of project actions in the future without LIFE funding.

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.

The project began in October 2017 with the main aim to demonstrate the capacity of a semiindustrial prototype to eliminate up to 90% of the emerging pollutants found in treated wastewater. The process of eliminating pollutants consisted of an adsorption material, photocatalysis lamps and a light pulse system. After some complications due to technical problems for scaling up the production of the polymer for the semi-industrial plant (that were worsen because of the pandemic), the project was extended to June 2022 and finally, the demonstrative plant started functioning at the end of 2020 and operated for over a year.

To begin, for the characterization of emerging pollutants present in treated wastewater, samples from different WWTPs were analyzed during a year (2017-2018) taking into account the danger and presence in water and a WWTP was chosen to install the semi-industrial prototype for the treatment process.

Secondly, the evaluation of the best material at laboratory scale (2018) was needed to use it for the adsorption column of the semi-industrial prototype for the elimination of EPs. The ability to eliminate EPs in wastewater was evaluated through absorption and desorption process. The adsorption and desorption process were done in batch as well as in flux in the laboratory prototype used for the characterization and pre-treatment of EPs.

Laboratory results also served to make the designs and calculations for the construction of the semi-industrial demonstrative plant (2018, beginning 2019).

After the characterization of EPs and once the technology was optimized at laboratory scale, the prototype was installed in the WWTP of Cabezo Beaza (Cartagena, Spain) in March 2019, including solar panels to improve the energy efficiency of the plant by using renewable energy.

However, validation tests to be carried out at a semi-industrial scale were stopped due to problems with the adsorbent. Although laboratory tests revealed the convenience of using a EPI-CDs Polymer, this had problems due to its toxicity during its fabrication and it was not possible to find alternatives for producing the amount required. Finally, UNIBA and CNR-IPCF found by the end of 2019 a patented polymer of nanosponges in Italy that, after additional laboratory tests, showed to work well under the project's conditions and showed positive results making it suitable for Clean Up aims. The polymer was ordered in 2020, the year of the outbreak of the pandemic COVD-19 and it was not until September 2020 that the adsorbent was received in the WWTP. Then, the plant was started up with all the units functioning in November 2020 and the running of the semi-industrial plant for wastewater treatment was finally carried out.

A monitoring plan was developed to monitor the progress and validate its technology during 2021. This consisted of the recurrent analysis of EPs in wastewaters sampled in several points of the prototype in the different seasons. Results of the monitoring showed that only phytosanitary were completely eliminated after the adsorption process. These cyclodextrins removed a 77% of EPs, and up to 91% were removed after adding pulses with an Advanced Oxidation Process (AOP).

In early 2022 additional tests were taken to assure a better estimation of some parameters to be more accurate about the polymer capacity. These tests performed in the semi-industrial plant were done to determine the LIFE span of the cyclodextrins and they showed that it can take up longer than 25 tests.

From 2020, the market launch of the project has been also prepared by drafting market and business plans and assessing the feasibility of the technology to be scaled up and transferred. In 2021, partners have worked in the transferability and replication plan by contacting and meeting potential sites in Spain and Italy. Other WWTPs and transfer industries have been visited and water samples analyzed to elaborate study cases.

On the socio-economic side, it was evaluated where to transfer the project and to create an entity with the private partners involved (HIDROGEA, HIDROTEC and REGENERA). It was decided to use the already established semi-industrial prototype to test further improvements of the adsorbent so it may be possible to replicate it at a bigger scale, thus obtaining benefit, and giving the opportunity to research centers to be also part of this investigations. Although results of the market plan suggested the creation of a company as a spin-off of the LIFE CLEAN UP project with its own brand, further the consortium has decided to work separately until having an establish demand on the technology.

During the project, dissemination activities were developed. Firstly, a project video was published and well accepted by the public and the media. Lately, papers were published in scientific journals when obtaining results along the project time, although several communication activities were stopped due to the pandemic. Moreover, the information published in Twitter, Facebook or the Clean Up website was used for raising awareness about

the project in the general public. Reports were also made and uploaded to different digital magazines. Finally, along the public awareness, dissemination activities related to technical part of the project were focused on water management companies, local administrations, companies dedicated to wastewaters and the scientific sector. The dissemination part of the project also involved networking activities with other projects and the organization and participation in events. Due to the pandemic, the main communication activities of the project such as technical and journalist visits to the demonstration plant, the project conference and the networking event have been done in 2021 or has to be adapted to the sanitary restrictions.

# 4. Introduction

#### Description of background, problems and objectives

#### Environmental problem addressed

Emerging pollutants can produce a significant environmental impact. The major source of these EPs is urban wastewater, since they are a sub-product of industrial and agriculture activities and of cosmetics, house cleaning products and most important pharmaceuticals, then, appearing also in effluents of WWTPs. Most of this EPs have a pollution behaviour but have not been yet regulated by European and national legislations whereas they are reaching aquatic and land ecosystems and affect the provision of ecosystem services. Both EPs and their metabolites are toxic substances that can have adverse effects in humans and animal species even when they are in so low concentrations that alter the endocrine system and block or alter hormonal functions.

On the other hand, the content in EPs and other pathogens is a serious issue if treated wastewater is going to be reused for environmental, urban, agriculture, leisure or industrial purposes.

The results of this project have social and scientific-technical repercussions, since a water treatment system for WWTPs that allows the total elimination of EPs resistant to conventional treatments has been validated. This was done thanks to the use of efficient and easy to use adsorbent materials (polymers of cyclodextrins).

#### Description of the technical / methodological solution

The EPs are adsorbed in these new materials and those not retained are degraded through Advanced Oxidation Process. At the same time, pathogenic microorganisms are also destroyed, thus maximizing the efficiency of the process. Thanks to this project, a prototype has been built and tested. It has a unit containing adsorbent material inside, which is 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 polymer once it is regenerated.

#### Outline the hypothesis to be verified by the project

The main hypotheses of this project were to validate the cleaning capacity and effectiveness of treated water system that can eliminate up to 90% of the emerging pollutants and other pathogens that cause great impacts over ecosystems and human health.

The proposed sizing was enough to allow assessing the technical and economic feasibility of the system at industrial level and to prepare the market launch of the system throughout the elaboration of the necessary plans and studies during the project. The running of the plant and the monitoring of its functioning for a whole year has permitted to make the necessary maintenance operations to adjust its functioning and to assess the disinfection power.

#### Expected results and environmental benefits

LIFE CLEAN UP developed a semi-industrial prototype to be installed in a WWTP managed by the company HIDROGEA. During the running of the prototype, retention rates over 90% were expected thanks to the action of polymeric CDs. The installation was compatible with all wastewater depuration systems, what facilitates its implementation and replication. Also, the modular design of the plant permit to offer a customized service for different king of industries. The fact to achieve a better quality of treated wastewater allow to foresee the development of future legislation that will limit the presence of emerging pollutants of different origins in water.

#### **Expected longer term results**

The elimination of EPs from treated wastewater (the main outcome of the project), would contribute to the achievement of the objectives established by the following regulations:

- Water Framework Directive 2000/60/EC (WFD), which, in order to ensure a good state of conservation of water bodies in the EU, establishes (Art.16) "strategies against water pollution" that marks the development of lists of priority substances.
- Decision No. 2455/2001/EC, establishing the First list of priority substances that presented a significant risk to or through the aquatic environment (33 substances, 11 of them hazardous priority and 14 subject to subsequent revision).
- Directive 2008/105/EC, Directive on Priority Substances, relating to environmental quality standards in the field of water policy, modifying the WFD. It reviews the status of the established substances in the previous Decision and establishes the limit concentrations in surface waters for 33 priority substances and 8 other pollutants.
- Directive 2013/39/EU modifies the two previous Directives, in terms of priority substances in the field of water policy, revises the established levels and broadens the list of substances. The contamination of water and soil with pharmaceutical waste is recognized as an emerging environmental problem and the adoption of a strategic approach is urged. Thus, new components of different types (drugs, pesticides, chemical products, etc.) are incorporated, such as dicofol, PFOS, dioxins, Bifan, cibutrin, cypermethrin, etc., the Environmental Quality Standards (EQS) are modified for some and EQS are included in biota (fish) for certain substances. It also establishes the creation of a list of observation of substances on which monitoring data should be collected.
- Wastewater Treatment Directive 91/271/EC. The Report of the European Commission to the European Parliament of 4/03/2016 (8th Report on the Implementation Status and the Program for Implementation of Council Directive 91/271/EEC concerning urban wastewater treatment) marks a continental panorama with deficiencies in the treatment of waters. Undoubtedly, the development of a new technology for wastewater treatment contributes to improving these deficiencies.

Regarding the continuation of the project, LIFE CLEAN UP proposal included a replication and transfer plan to promote the entry into the market of its technological solution, after its validation during the project. With this purpose, the following studies were carried out:

- Technical-economic study to determine the feasibility of a continuous and industrial scale flow treatment plant.
- Technical-economic-legislative study of the different renewable energy generation technologies applicable to the commercial product, in such a way as to supply the plant's own consumption or to pour energy into the electricity grid.
- Cost-benefit analysis for the economic and social evaluation of the industrial scale implementation of the product.
- Case studies to know the transferability of the treatment system in other WWTPs and also in industries that treat the wastewater coming from their industrial process.

However, it has been demonstrated that the entry into the market will be not possible just after the end of the project because further research in the adsorption material to treat major flows is needed. Also because of this reason, although the companies participating in the project (HIDROGEA, HIDROTEC and REGENERA) aimed at the beginning of the project to create a new limited company and this was also the conclusions of the market study and business plan this will not be done in the short term. On the other hand, the After-LIFE Plan schedule the different activities to be developed from now on to continue with the market launch and scalability and transfer plan of the project based on the results of the above-mentioned studies and also because of this, a Stage 0 has been proposed.

# 5. Administrative part

The partnership agreements were signed between UCAM as CB and ABs (REGENERA, CTC, UNIBA, CNR-IPCF, HIDROGEA and HIDROTEC) at the beginning of the project (October 2017), setting the responsibilities of each part and the procedures to be followed for a correct implementation of the project. After this, the coordinating beneficiary UCAM, made the bank transfer of the pre-financing corresponding to each partner.

The project has been structured at two operational levels: technical and administrative/financial. Each associated beneficiary has a contact person to deal with technical and/or financial issues (Table 1).

Partner	Technical contacts	Administrative contacts
UCAM	José Antonio Gabaldón	David Heiser
UNIBA	Pinalysa Cosma	Teresa Lodeserto
REGENERA	Miguel Alegría	Miguel Alegría
	Oscar Amorós	Oscar Amorós
	Tuuli Inkeri	Tuuli Inkeri
	Victor Fabregat	Victor Fabregat
HIDROGEA	Teresa Reyes Serna	Teresa Reyes Serna
	Estefanía Menarguez	Estefanía Menarguez
	Javier Hernández	Javier Hernández
CTC	Miguel Ayuso	Mariano Cerezo
HIDROTEC	Antonio Morte	Blanca Maria Lopez
	Antonio Hernández	
CNR-IPCF	Paola Fini	Annalisa Delre

 Table 1. key people at LIFE CLEAN UP project

On 4th October 2017 took place the Kickoff meeting of the project. Main points addressed were the revision of budget, actions and responsibilities of each partner and the management process to be followed during the project LIFE. Financial issues of the LIFE Programme were explained by UCAM to all the ABs, following the recommendations given in the LIFE16 KOM for coordinating beneficiaries.

The project has established also two types of meetings. In first place, Steering Committee meetings or partners meetings where all partners participate are held, at least, every six months. On the other hand, partners are called for coordination meetings whenever there is some need to prepare actions implementation, speed project's milestones or to prevent further delays.

Partners meetings and coordination meetings (24 from October 2017 to June 2022) are usually organized in the headquarters of UCAM. Due to the movement restrictions due to the pandemic COVID-19 these meetings have been held online and Italian partners have also joined. The agendas, minutes and attendance lists can be found attached to this report.

Apart from these meetings, email communication, Skype meetings or talks are maintained with partners to closely monitor project implementation for both, the financial and technical progress.

The project has received five monitoring visits of the NEEMO external team and all the letters regarding monitoring visits or reports have been already replied unless the letter of the last monitoring meeting celebrated in June 2022 (6th- 7th) whose reply is attached to this Final Report. In this last monitoring meeting of June 2022, the project also received the visit of the project adviser from CINEA.

The communication with the LIFE monitoring team has been very fluent and successful during all the projects. The coordinator has contacted the NEEMO monitor whenever some problem has arisen in order to agree mitigation measures and try to avoid a bigger impact for the project implementation. When the situation has required major changes (project extension) the main objectives to be pursued were analyzed jointly and thanks to the understanding of CINEA regarding all the problems faced, a project amendment was approved. This has enabled the continuation of the project from September 2020 to June 2022 and the possibility to test the built prototype for a year and to extract conclusions that have permitted assess the feasibility of the proposed technology.

Regarding the financial reporting, partners agreed to upload supporting documentation and updated financial reports to a Google Drive folder, although some partners have preferred to send documentation by email. Technical information about monitoring meetings, deliverables and reports delivered to EASME and CINEA are also uploaded in the shared Google Drive.

## 6. Technical part

6.1. Technical progress, per Action

# 6.1.1. Action A1. Characterization of emerging pollutants in treated wastewaters in WWTPs.

✓ Complete action

Foreseen start date: 01/10/2018 Actual start date: 24/10/2017

PLANNED TIMETABLE ACTION A1	2017					2018												
	S	0	Ν	D	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D		
ACTUAL		20	17			2018												
TIMETABLE ACTION A1	S	0	Ν	D	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D		

#### • Participants and responsibilities:

CTC was the action coordinator and responsible for the characterization of EPs in treated wastewaters. HIDROGEA participated by making available for the project 6 of the purification facilities that it manages in the Region of Murcia for the analysis of the water treated in them, in terms of the presence of emerging pollutants and then selecting the most appropriate to locate the pilot plant. HIDROGEA also carried out the physical-chemical characterization of the samples, as well as other analytical parameters such as organic matter, N and P compounds, etc. between others.

#### • What has been done:

A survey of emerging pollutants incidence in treated wastewater from six WWTPs in the Region of Murcia (Mar Menor, La Hoya, La Aljorra, Isla Plana, Cabezo Beaza) has been carried out, in order to select those contaminants that pose a real contamination problem and that are resistant to conventional treatments and to discern which plant was the most suitable to install the semi-industrial prototype. With the selection of these WWTP it is guaranteed to consider different types of wastewaters according to their origin (urban, agricultural, industrial, etc.), since depending on the origin of the water, the contaminant content can be different.

First, each to one of the WWTPs effluent involved were visited, and a 2-year retrospective study of each WWTP was carried out. Then, fact sheets of each WWTP with all the information collected were made.

The technical data sheets include aspects of general characteristics and interesting details of the WWTP, characteristics of the wastewater entering and leaving the treatment plant, and a flow chart that contemplates the type of primary, secondary and tertiary treatment applied to the wastewater. They can be consulted in the deliverable of action A1.

HIDROGEA has taken samples in the different WWTPs, facilitating an aliquot of each sample to CTC for the analysis of EPs and another one to the laboratory of the respective WWTP for the physical-chemical analysis and of organic matter and other analytes. It has been necessary to acquire different laboratory materials and consumables to be able to carry out the analytical determinations (beakers, sample collection boats, etc.) and various laboratory consumables to carry out certain analyses (COD kits, NO3, NH4, between others).

To ensure maximum representativeness of the quality of the water treated, samples have been taken from all the treatment plants during the entire period that the action should last, according to the project proposal, to consider possible seasonal changes.

The samplings have been carried out the following days:

- For Cabezo Beaza, Águilas, Mar Menor Sur, La Aljorra and Isla Plana WWTPs: 24/10/2017 (1), 02/11/2017 (2), 19/01/2018 (3), 12/04/2018 (4), 10/07/2018 (5).
- For La Hoya WWTP: 25/01/2018 (6), 09/05/2018 (7), 17/07/2018 (8).

Then, aliquot of each sample sent by HIDROGEA to CTC was divided into subsamples that were sent to different laboratories, to perform analysis about the content in water samples of pesticide residues (a), pharmacological residues (b) as well as trihalomethane residues (c).

The obtained results were collected in tables to compare the concentrations found for each analyte, the same day in the different WWTPs. An example of these tables showing the results for the samples taken from 5 WWTPs on 24/10/2017 is provided above (

	-	Testitis of sur		om aijjereni v		10,201,	
(µg/L)	DATE SAMPLING / WWTP	WWTP 1	WWTP 2	WWTP 3	WWTP 4	WWTP 5 – MBR	WWTP 5 – SC
Chloroform		2,15	2,45	1,99	1,21	4,16	7,11
Bromodichloro methane		ND	ND	ND	ND	ND	0,46
Chlorodibromo methane		ND	ND	ND	ND	ND	ND
Bromoform	-	ND	ND	ND	ND	ND	ND
		Acetamiprid 0,57	Chlorpyrifos 0,12	Dodine 0,67	Chlorpyrifo s 0,11	Acetamiprid 0,25	Acetamiprid 0,42
Phytosanitary	24/10/2017	Chlorpyrifos 0,48	Dodine 0,32		Dodine 0,52	Chlorpyrifos 0,23	Chlorpyrifo s 0,26
		Dodine 0,29			Methomilo 0,12	Dodine 0,37	Dodine 0,49
		Atenolol 0,22	Atenolol 0,20	Atenolol 0,22	Atenolol 0,22	Atenolol 0,22	Atenolol 0,22
		Carbamazep ine 0,18	Carbamazep ine 0,19	Carbamazep ine 0,18	Carbamaze pine 0,22	Carbamazepin e 0,18	Carbamaze pine 0,18
		Ciproflox acin 0,36	Ketoprofen 0,23	Ciprofloxaci n 0,12	Ciprofloxa cin 0,16	Ciprofloxacin 0,36	Ciprofloxaci n 0,37
Drugs		Ketoprofen 1,28	Diclofenac 0,43	Ketoprofen 1,88	Diclofenac 0,97	Diclofenac 0,76	Ketoprofen 3,45
		Diclofenac 0,88		Diclofenac 0,88	Sulfametox azole 0,19	Sulfametoxazo le 0,15	Diclofenac 0,58
		Sulfametoxa zole 0,15		Sulfametoxa zolee 0,02	Norfloxaci n 0,34	Norfloxacin 0,24	Sulfametox azole 0,19
		Norfloxacin 0,24		,	Trimethopr im 0,15	Trimethoprim 0,01	Norfloxacin 0,48
					Paracetam ol 0,028		Trimethopri m 0,011

Table 2. Results of samples taken from different WWTP on 24/10/2017

Table 2). All the results can be consulted in the deliverable of this action.

Once characterized the treated wastewater in the 6 WWTPs, and selected the list of EPs, the choice of the WWTP where the prototype should be installed was accomplished, according to the following criteria:

- Greater variety of EPs of urban and agricultural origin present in the treated waters.
- Higher concentration of EPs.
- Type of secondary treatment.
- Quality of treated water.
- Presence of tertiary treatments.
- Design of the most appropriate facilities.

Due to the experience accumulated in the exploitation of other pilot plants, the importance of the presence of the following points in the target WWTP was also taken into account:

- Plant personnel: operators, maintenance, analysts.
- Workshop to make repairs quickly.
- Laboratory of analysis sufficiently equipped to perform process control analysis.
- Closeness to HIDROTEC headquarters responsible for the design and construction of the pilot plant to carry out corrective actions shortly when a problem is detected.

Finally, the WWTP selected was the Cabezo Beaza WWTP located in Cartagena, Murcia.

#### • Main problems encountered and solutions provided:

We have not had problems worthy of mention. Like remarkable detail, the analysis of pharmaceutical residues (or drugs), and trihalomethane residues was prepared to detect small concentrations of contaminants in water samples. However, regarding pesticide residue analysis, although lesser amounts were detected, the sensitivity of analytical method of one lab was worse, not reaching the limits of quantification revealed for other lab in the analysis of drug residues. For this reason, extraction methodology was modified, improving in consequence the limit of quantification of pesticide residues, now, in the same range that the used for the analysis of drugs residues.

#### • Achieved results:

The identification of contaminants that were present in the analysed 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, pay attention to their dangerous and incidence in analysed waters was elaborated, and the analytes included were thoroughly surveyed alongside the project, to apply the most realistic working conditions.

Substances that are found in Directive 2013/39 /EU of the European Council of August 24, 2013 as priority substances transcribed into Spanish legislation by Royal Decree 817/2015, (this is the case of Chlorpyrifos, Cypermethrin and the Chloroform), or in Commission Implementing Decision (EU) 2015/495 of 20 March 2015 as substances under observation for follow-up purposes (this is the case of Diclofenac, Imidacloprid and Acetamiprid) have been selected for their recurring presence in analysed waters.

Due to their widespread use, pharmacological substances predominate, both in concentration and in the variety of compounds detected, over the rest of emerging contaminants.

In addition, since other pesticide residues such as the fungicides Penconazol, Carbendazim, or Dodina, or the insecticides Spinosad (A + D) and Propoxur, appear with relative frequency in analysed waters, were also included in the list for further analysis.

Regarding pharmacological compounds, Carbamazepine (antiepileptic), Sulfamethoxazole and Trimethoprim (used together as antibacterial for the treatment of urinary tract infections or pneumonia, among others), Atenolol ( $\beta$ -blocker) and Ciprofloxacin and Norfloxacin (antibiotics belonging to the family of Fluoroquinolones) are common. All of them have been included in our list of emerging pollutants, along with Paracetamol, due to their widespread use in the population, to ensure a selection of compounds that belong to varied families and of diverse origin according to their use.

Based on this knowledge, the WWTP for the installation of the prototype was selected (Cabezo Beaza, Cartagena).

## • Remaining results:

None.

#### • Variations in action:

The development of the action did not suffer variations worthy of mention.

The elaboration of deliverables has not suffered from any delay, the only remarkable note is that, in order to give a comprehensive view of the action, we have made one single report collecting the information of the two deliverables of Action A1:

- List of EPs candidates to be encapsuled in CDs.
- Report about the physic-chemical quality and presence of organic pollution in the treated wastewaters of the WWTPs subject to study.

#### • Action evaluation:

The results obtained allow us to make a complete evaluation of the presence of emerging pollutants in treated wastewaters, regardless of the temporary situation, the origin of the water and the type of treatment carried out in the WWTPs, as the study of these parameters.

ТҮРЕ	Code	Name	Action	Deadline	Responsible	Status
DELIVERABLE	1	List of CEs candidates for encapsulation in cyclodextrins	A1	30/11/2017	CTC	OK 30/11/2017
DELIVERABLE	22	Report on the physical-chemical quality and presence of organic pollutants in the treated water of the WWTPs subject to study.	A1	30/07/2018	CTC	OK 30/07/2018

ТҮРЕ	Code	e Name	Action	n Deadline	Responsible	Status
MILESTONE	E 9	Decision on the WWTP where the pilot test will be carried out	A1	30/07/2018	ALL	OK 30/7/2018

# 6.1.2. A2 Optimization of the process for obtaining different adsorbent materials of polymeric base

#### ✓ Completed action

Foreseen start date: 01/11/2017 Actual start date: 01/11/2017

Foreseen end date: 30/03/2018 Actual end date: 31/10/2020

PLANNED	2017					2018											
TIMETABLE ACTION A2	S	0	Ν	D	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	
ACTUAL		20	17		2018												
TIMETABLE	S	0	Ν	D	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	
ACTION A2																	

PLANNED		2019													2020									
TIMETABLE	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	J	F	Μ	Α	Μ	J	J	Α	S	0		
ACTION A2																								
ACTUAL		2019												2020										
TIMETABLE	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	J	F	Μ	Α	Μ	J	J	Α	S	0		
ACTION A2																								

#### • Participants and responsibilities:

UCAM was responsible for the synthesis and characterization of cyclodextrin polymers. UNIBA was responsible for the synthesis and characterization of hydrogel polymers. CNR-IPCF was responsible for the synthesis and characterization of biomaterials. HIDROTEC was in charge for the construction of the laboratory prototype. HIDROGEA participated in this action by providing treated water from WWTPs that it manages to carry out the laboratory tests with the CDs in discontinuous and continuous, once the laboratory pilot is underway.

• What has been done:

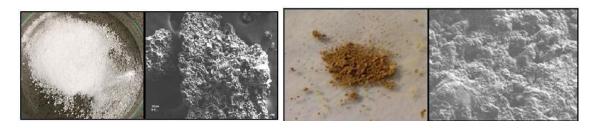
#### Task A2.1 Molecular nanoencapsulation of compounds of different families with CDs

The complexation constants (KC) for complexes between different emerging pollutants (CE) and cyclodextrins (CDs), were calculated by the Phase solubility diagrams or applying the Benesi-Hildebrand treatment. The selection criterion was CE-CDs complexes with higher KC values. Three phase diagrams were made for each CE by triplicate, in order to calculate arithmetic means. Since each laboratory test require almost 3 hours, the task lasted 2 months (November- December 2017).

#### Task A2.2 Optimization of the synthesis of adsorbent materials

a. Optimization and synthesis of CDs (UCAM)

In this task, UCAM evaluated different methods of cyclodextrins polymer synthesis using epichlorohydrin (EPI) and tetrafluoroterephthalonitrile (P) as chemical linkers; obtaining two types of polymers: EPI-CDs and P-CDs. The time needed to obtain EPI-CDs polymer is 24 hours, since the experiment lasts 6 hours but must be drying overnight (Fig. 1). However, to obtain P-CDs polymer 5 days were required, 2 days in agitation and 3 days for drying step. These tests were done for 3 months (November 2017 to January 2018). The use of P-CDs polymer was finally discarded due to the low yield (Fig. 2) and excess of time necessary in its synthesis process. Consequently, following tasks of A2 Action, regarding characterization and assessment were done with EPI-CDs.



*Figure 1. (A) (left) Dry EPI-CDS polymer. (B) (right)SEM micrography of EPI-CDs polymer. Figure 2. (A) (left) Dry P-CDP polymer. (B). (right) SEM micrography of P-CDs polymer* 

#### b. Optimization and synthesis of Hydrogels (UNIBA)

In this preliminary step the <u>polyamidoamine-based hydrogel</u> (PAA) was obtained following the procedure developed by Fiorini et al. Samples were dried either at r.t for 24h, or at 60°C for 6h, obtaining the dry hydrogel materials PAA 24h and PAA 6h, respectively. Results are referred to 100 experiments, in order to evaluate the reproducibility about the formation of the Hydrogel. Experiments were performed from November 2017 to January 2018.

Also, <u>Chitosan</u> (CH) <u>hydrogels</u> (and thus CH films) were synthesized by using two procedures, obtaining CHI and CHII, respectively. The number of tests performed for both procedures were 10. The total cost for obtaining CH II were reduced since a great amount of chitosan powder remains insoluble, and, as a consequence, it could be recovered and potentially used to induce the formation of novel films.

#### c. Optimization and synthesis of bio-adsorbents from agricultural waste (IPCF-CNR)

Five different types of bio adsorbent materials were synthesized:

<u>Olive pomace:</u> The solid waste of olive oil production was provided from local oil industry, Bari, Italy. The time spent for the preparation of the pomace was 8h for the production 100 mg of the adsorbent. The number of tests to obtain these procedures was 150. Experiments were performed from October 2017 to February 2018.

<u>Alginate (with and without TiO2)</u>: The number of test to obtained mAL/CH was about 200. Experiments were performed from October 2017 to February 2018.

<u>Chitosan/olive pomace</u>: Chitosan blended membranes were obtained. The number of tests was about 2000. Experiments were performed from October 2017 to March 2018.

<u>Chitosan/TiO2 and Chitosan/OP/TiO2</u>: The number of tests was 1000. Experiments were performed from October 2017 to February 2018.

<u>Chitosan/Cyclodextrin polymers</u>: CD-EPI-chitosan films were prepared. The number of tests was 2000. Experiments were performed from May 2018 to June 2018. This mixed polymer was synthesized in agreement with the partnership to try to prevent the swelling behaviour showed by the CDs polymers synthesized by UCAM since it was thought that chitosan films could provide a firmer structure to avoid this phenomenon.

#### Task A2.3 Characterization of different materials

These analyses were done to determine the morphology of the polymers synthesized.

a. Characterization of CDs (UCAM)

Once synthesized, the characterization of the following samples was carried out:  $\beta$ -CD,  $\circledast \beta$ -EPI-CD by different methods. The characterization of the polymers was carried out to verify that the polymerization reaction worked correctly between January and February of 2018. The morphologies of the cross-linked adsorbents were investigated by SEM.  $\beta$ -CDs-EPI polymers were characterized for its porous and irregular structure, suitable to entrap the dye molecules. The particle size distribution expressed as the mean volumetric size D<sub>[43]</sub> was higher for the native  $\beta$ -CDs (76 µm). The particle size distribution was estimated by span values, which were 2.8; 5.8 for  $\beta$ -CD and  $\beta$ -CDs-EPI respectively. The characterization of the polymers showed that the polymers were well constructed, and that the polymerization reaction was occurring successfully.

#### b. Characterization of Hydrogel (UNIBA)

The <u>PAA hydrogels</u> were characterized by FTIR-ATR and SEM techniques. The number of tests performed was 200. Experiments were carried out from November 2017 to January 2018. This analysis on the structure and morphology of the material showed that it exhibited a compact and irregular porous surface, suitable for the adsorption of CDs. SEM images of <u>CH films</u> prepared with the procedure I and/or II. Information related to the morphology of CH films was additionally provided by means of AFM analysis. If CH I shows a homogeneous chain distribution, the CH II appears rougher. X-ray patterns collected on both CH I and II show the existence of an amorphous phase for all the investigated samples. The number of tests was 2000. Experiments were performed from November 2017 to April 2018.

#### c. <u>Characterization of Bioadsorbents (UNIBA + CNR-IPCF)</u>

<u>Olive pomace:</u> The raw material was composed by seeds and pulp. From FTIR, XPS, TG analyses arises that the biosorbent matrix consists mainly of cellulose, hemicelluloses and lignin with the presence of amino acids and/or proteins. Lignin can be considered the cementing matrix that holds cellulose and hemicellulose units together. The number of tests was about 500. Experiments were performed from October 2017 to February 2018.

<u>Alginate:</u> The presence of separate alginate micron beads with the absence of clusters were found. The SEM of mAL beads shows as beads properly dehydrated appeared slightly rough with a regular surface in absence of cracks. The number of tests was 250. Experiments were performed from November 2017 to March 2018.

<u>Chitosan/olive pomace</u>: If the alginate-based adsorbents are presented as slightly rough materials, the sample CH/OP based films appear rougher with an irregular surface. The porous structure of such adsorbent shows some irregular structures and cavities on the external surface, important key features for the adsorption of pollutants. These peculiar domains were ascribed to the presence of the blended biomass into chitosan films. It is worth to remember as previously assessed that chitosan films without modification appeared homogeneous and smooth. The number of tests was 800. Experiments were performed from November 2017 to June 2018.

<u>Chitosan/Cyclodextrin polymers</u>: A comparative swelling study of  $\beta$ -EPI/CH and pure chitosan films with 0.5% glycerol was performed, in order to evaluate the effect of the cyclodextrin polymers on the phenomenon. The  $\beta$ -EPI/CH blended film provided similar results: probably, in this case, water molecules interact preferentially with the cyclodextrin polymer, which is more accessible than chitosan moieties but has a lower affinity for water. The number of tests of 1000. Experiments were performed from May 2018 to July 2018.

#### Task A2.4 Assessment of adsorption processes

#### a. Assessment of adsorption processes for CDs (UCAM)

In a first step four EPI polymers were synthetized using the native ( $\Box$ -,  $\beta$ -,  $\gamma$ -) CDs and a combination of them, and their ability to eliminate representative EPs in wastewater was evaluated through experiments of adsorption/desorption in batch to reach the objective since February to September 2018. Based on psychochemical parameters, kinetic models, isotherms and thermodynamic data accomplished to understand the physicochemical process involved in the adsorption of EPs by the four CD-EPI polymers,  $\beta$ -CDs-EPI was selected for showing the best adsorption capacity and lower price, for further studies. While the maximum absorption for some EPs such as propranolol and atenolol are reached in 5 min, others as gemfibrozil require at least 15; also, depending on the structure of the contaminant, certain variability in the retention capacity of the polymer was observed (i.e. 92% and 65% for gemfibrozil and ketoprofen, respectively).

The adsorption tests carried out indicated that follow three stages. 1°: rapid use of the most available sites on  $\beta$ -CD-EPI polymer surface. 2°: EP diffusion from polymer surface to the pores (intraparticle diffusion), stimulating a greater movement of EP molecules from the liquid phase to the polymer. 3°: equilibrium. A fast kinetic has a significant importance, which allows a high efficiency and economy. However, the test showed that  $\beta$ -CD-EPI polymer does not retain with the same efficiency all the EPs (different chemical structures, and charge) and in the same contact time. The useful LIFE of the polymers was evaluated through studies of regeneration with methanol and acetate buffer at different pH values, selecting pH 4.0 as optimum. Despite the regeneration of the polymer shows a low decrease in their effectiveness in encapsulation and release steps, was possible to be reused for 4 cycles. Experiments were performed from December 2017 to March 2018.

#### b. Assessment of adsorption processes for hydrogels (UNIBA)

After a careful study, Polyamidoamine-Based Hydrogel occurred not suitable for the removal of the all the selected pollutants (e.g.: athenolol, trimethoprim, etc.). Different conditions of work were explored, however, always there was absence of significative results, both in batch and "in flow". For example, various experiments were performed by adopting several contact

times between the pollutants and the adsorbent (*i.e.* 1, 2, 4, 6, 8, 24 and 48 h), but there was no removal. The number of tests was 1000. Experiments were performed from November 2017 to March 2018.

#### c. Assessment of adsorption processes for biomaterials (CNR-ICPF, UNIBA)

Since some of the studied EPs appeared recalcitrant (lesser retention values than the initially made goal 90%), for evaluated adsorbents, UNIBA and CNR-IPCF decided to add another natural inorganic product: bentonite, together to the previously synthesized adsorbents to improve EPs removal. In Table 1, a detailed list of the used adsorbents, EPs, adsorption (mg/g) and % of desorption is reported, highlighting in red when removal was greater than 80%. Overall, olive pomace (OP) both in powder (OP) and blended inside a chitosan film (CH/OP), natural and synthetic polymers such as chitosan (CH) and cyclodextrin based polymers (CD-EPI) and the inorganic clay bentonite occurred more suitable. Moreover, the costs associated for each material were calculated.

It is worth to mentioned that the processes occurred time and dose-dependent (amount of EP and adsorbent), as better described in the deliverable D.11-D.15. The number of tests was 2000. Experiments were performed from November 2017 to June 2018.

In order to better assess the adsorption character of  $\beta$ -EPI/CH, the following analyses were performed from May to September 2018:

- Effect of glycerol amount in  $\beta$ -EPI/CH films on the adsorption process (1000 tests).
- CBZ and SMX adsorption onto  $\alpha$ -,  $\beta$  and  $\gamma$ -EPI/CH FILMs (1000 tests).
- Effect of CD-EPI amount in chitosan films on the adsorption (500 tests).

The rigidity of the adsorbent increased along with  $\beta$ -EPI content and the adsorption efficiency of the films increased by incrementing in  $\beta$ -EPI amount. This confirms the hypothesis that not only chitosan, but also cyclodextrin polymers play a key role during the adsorption.

d. <u>Assessment of adsorption process of Mn CDs-BDE, polymer cyclodextrin nanosponges</u> (Cyclodextrins-based polymer, cross-linked with 1-4 butanediol diglyceryl ether)

UNIBA, in synergy with CNR-IPCF, tested the in-batch adsorption properties of the cyclodextrin polymer (nanosponges), obtained by Prof. Francesco Trotta, University of Turin, if in presence of the following EPs: Diclofenac, Furosemide, Carbendazim, Ciprofloxacin, Sulfamethoxazole and Naproxen. Unfortunately, Atenolol and Propoxur were also studied, but they were not retained by the polymer. To evidence the performance of the adopted polymer, a general overview of the adsorption process, detailing the experimental setups, was detailed in the deliverable D-9-11-12-13-14-15.

The polymer exhibited a very high adsorption ability towards the selected pollutants and, if 1g of adsorbent material was used, the removal of EPs was quite complete in 15 minutes. The maximum adsorption capacities were also evaluated for each EP and the polymer showed the highest and lowest capacity for Diclofenac and Carbendazim, respectively. To infer more information about the adsorption process, UNIBA and CNR-IPCF performed a careful investigation by studying the effect of several parameters affecting the process. By adopting as model pollutant Ciprofloxacin (CP), interesting results were obtained. Moreover, the use of salt in solution containing CP, as for example NaCl, hindered the adsorption process favoring the

desorption of the adsorbed pollutant. As a whole, the obtained findings could be applicable to other EPs and, by considering their chemical structures, electrostatic interaction between the polymer and EPs could be taken into account and responsible of the adsorption.

#### Task A2.5 Laboratory prototype for continuous flow assessment

#### a. In flow assessment for CDs through laboratory prototype (UCAM, HIDROTEC)

UCAM performed tests on the laboratory prototype manufactured by HIDROTEC and installed at UCAM. Adsorption tests were carried out to determine the continuous adsorption capacity of the  $\beta$ -CD-EPI polymer. For it, solutions of emerging contaminants are passed through a  $\beta$ -CD-EPI column with a known concentration at a certain speed thus ensuring a previously defined contact time (in batch assays).

The presence of various compounds in the sample alters the adsorption  $\beta$ -CD-EPI, since a decrease in absorption is observed and it is more accentuated in the case of the compound that is most absorbed when it is lonely in water.

After the results obtained in the analytics carried out for each pollutant and viewing the adsorption differences with the different contact times, a working protocol for the semi-industrial prototype is established from 3 to 5 minutes.

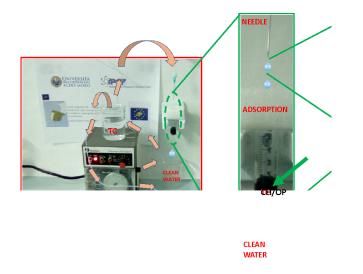
Finally, the continuous desorption capacity of the polymer was determined by passing an acetate buffer pH 4.0 solution through the cyclodextrin polymers columns in different concentrations and volumes determined. The tests carried out for continuous desorption provide results practically similar to those made in batch.

Approximately, 81% desorption of the contaminant adsorbed by the cyclodextrins polymer, is obtained. It is important to note that the adsorption and desorption is very fast.

UCAM has made experiments of adsorption/desorption in continuous to optimize the operational conditions of the semi-industrial prototype since June to September 2018.

#### b. In flow assessment for bio-adsorbent material (UNIBA, CNR-IPCF)

UNIBA has determined an alternative *in flux* experiments (as they do not have laboratory prototype, used an in-house model) using <u>CH/OP</u> as adsorbent and Tetracycline (TC) as model EP (Figure 3). Experiments were performed from March 2018 to June 2018.



*Figure 3. Camera picture of the experimental setup adopted during the influx experiments using ch/op as adsorbent and as model ep. the arrows in the figure indicate the direction of the flux* 

Also, the adsorption and release of TC from water by using <u>Alginate-based beads</u> (mAL/CH) with and without  $TiO_2$  was evaluated from January 2018 to March 2018. Although these outcomes are very interesting, the main drawback related to the release of TC, under these work conditions, is that these results were achieved in the presence of Mg<sup>2+</sup> that broke the polymeric network beads, rendering them not useful for further adsorption processes. Further, the presence of TiO<sub>2</sub> suggests the possibility to photodegrade the adsorbed TC using again the mAL/CH/TiO<sub>2</sub> microbeads.

Regarding In-flux adsorption experiments with  $\beta$ -EPI/CH films, the number of tests was 800. Experiments were performed from May 2018 to September 2018. The adsorption efficiency of the film increases at lower flow rates, due to the longer contact time between the adsorbent and the pollutant. However, even very short contact times allowed over 60% of CBZ removal. Further *in-flux* experiments were performed by using CBZ and SMX (5 mg/L), with a contact time of 2 minutes: surprising results were obtained, since the adsorbents were able to remove over 50% of removal within such a short time.

c. <u>In-flow assessment of the Italian cyclodextrin nanosponges (Mn CDs-BDE polymer)</u> (UCAM-CNR-ICPF, UNIBA)

Since the MN CDs-BDE polymer should adapt to the design characteristics of the semiindustrial prototype, made on the basis of  $\beta$ -CD-EPI polymer, it was necessary to verify the efficiency and behaviour of Mn CDs-BDE, using as evaluation criteria the same parameters that for  $\beta$ -CD-EPI. Briefly, adsorption/desorption test were carried out in continuous way, both in the pilot and semi-industrial prototypes, using ibuprofen as EP model.

In accordance with this, the influence of contact time, dosing of the polymer (700 g and 75 kg for pilot and semi-industrial prototypes respectively, EP concentration, and absorption capacity were evaluated.

Regarding continuous tests, both in the laboratory semi-industrial prototypes, all added ibuprofen was successfully removed at flow times lower than 5 min, which was fixed as

optimum since lower contact times (1 or 3 min.) implies an increase of the flow rate above 1000L/hour, which can lead that the nozzles clogged with the polymer

#### Task A2.6 Assessment of different adsorbent materials

After comparing all the obtained results in terms of adsorption, desorption, regeneration capacity, contact time and costs, the EPI-CDs Polymer is the one that has been used as a model for semi-industrial scaling in the prototype and later industrial in the treatment plant.

In second place, chitosan-based adsorbents are encouraging materials for the removal of the selected EPs from water. The role of TiO2, photosensitizer, sodium alginate and agricultural wastes if mixed inside chitosan hydrogel and/or alone has been studied and suitable conditions of work able to retain a greater number of Eps were established.

The Italian Polymer was also investigated. The obtained results revealed that the sorption process was interesting showing the typical features of the previous studied adsorbent materials rendering it suitable for the European project aim. Details about the adsorbent characterization were also presented by adopting in synergy different techniques such as FTIR-ATR, DSC, TG, SEM, EDX and XRD. The tested pollutants were highly removed from water with high percentage and for example considering the CP case, the 90% of the CP removal was obtained in short time. The sorption process was investigated at several temperature values, indicating that increasing the temperature values, the removal of the CP from water increased. The adsorption was EC and adsorbent amounts dependent; more specifically, increasing the amount of the adsorbent and diluting the CP solution, the pollutant removal efficiency increased. The presence of electrostatic interaction between CP and the polymer were found to be responsible of the affinity between the sorbent and CP. Indeed, the adsorption was largely affected by pH values and presence of salt, i.e., NaCl, in solution. Interestingly, about this concern, the use of NaCl was found to be suitable for the desorption of the adsorbed CP, proposing the reuse of the pollutant and the regeneration of the adsorbent.

However, the use of salt to desorb the pollutant affected the thermal features of the adsorbent, (detected with DSC and TG analyses) but it did not alter largely the polymer adsorption capacity favouring the Ciprofloxacin removal for several cycles of adsorption and desorption. Not surprisingly, the adsorbent morphological, FTIR-ATR and XRD characteristics were retained after its reuse. The mixture of other pollutants was also studied and removed from water showing as the Italian polymer worked well also under these conditions. With respect other materials, the polymer removed the largest number of ECs.

#### • Main problems encountered and solutions provided:

In Murcia, UCAM had delay problems in the supply of pure compounds to perform kinetic studies, which delayed the studies of adsorption and desorption in batch and that influenced the delay in the final design and construction of the laboratory prototype.

Another technical problem was the large-scale production of polymers of CDs, since to extrapolate the synthesis technique used at the laboratory level, a reactor had to be manufactured to perform in flux experiments. This reactor is being also used to synthesize CDs for semi-industrial scale (Figure 4).



Figure 4. Reactor for the synthesis of polymers of CDs.

These two setbacks have supposed the extension of Action A2 from March 2018 (expected end date) to September 2018 (actual end date).

In Bari, the time necessary to perform experiments was not enough to have a comprehensive investigation of a large number of pollutants, so Action A2 in Italy extended from March 2018 (expected end date) to June 2018 (actual end date). For that reason, only some pollutants, selected from compound classes were tested.

Since, UNIBA was mainly involved in the study and characterization of hydrogel, and the latter occurred not performant in the removal of the examined pollutants, the study of UNIBA was focused, in synergy with IPCF-CNR, on the investigation of bio-adsorbents. In order to have the possibility of testing and optimizing the synthesis of the major part of the bio-adsorbents, CNR-IPCF began to work at Action A.2 since October 2017. Further, the Actions A.2.2, A.2.3 and A.2.4 were continued until June 2018, due to some difficulties in additional staff recruitment (for both UNIBA and CNR-IPCF) and the long purchasing procedures, which allow us to have the chemicals, laboratory glassware and plastic ware only after the foreseen end of Action 2. Moreover, due to the Italian procedures for researcher/technician recruitment which requires to freeze all yearly salary money, together with the difficulty in obtaining a fund anticipation for CNR-IPCF, some purchase orders were delayed. In spite of all these difficulties, the Italian partners managed to make a lot of tests using materials already available in CNR-IPCF and UNIBA laboratories or loaned by colleagues and delaying the end of Action A.2 until the end of June.

#### • Achieved results:

Tests of adsorption and desorption in continuous have allowed to optimize the working conditions in the semi-industrial prototype that was installed in the treatment plant. These experiments showed that CDs polymers have a high swelling capacity and behave like a gel so when you work by passing water through them at not too high speed they begin to compress. When the speed of passage increases, the polymer begins to gather until it becomes compacted, forming a block preventing the water from coming out completely. In view of these difficulties and as an alternative solution, tests were performed to work in a fluidized bed.

With the obtained data main design characteristics of the semi-industrial prototype was concluded:

- Fluidized bed.
- Speeds lower than 12 m / h.
- Bed heights of 550 mm and with columns of 1500 mm height and cylindrical.

Operational parameters of the semi-industrial prototype, designed on the basis of the lab results obtained with  $\beta$ -CD-EPI polymer, did not change when substituted by MN CDs-BDE polymer, since similar performances were showed.

#### • Remaining results:

None.

#### • Variations in action:

Tasks A.2.2, A.2.3 and A.2.4 continued until September 2018 in Murcia, due to delay in purchases and in the fabrication of CDs polymers, and until June 2018 in Bari, due to some difficulties in additional staff recruitment and the long purchasing procedures.

Despite this, the objectives of Action A2 have been reached and, overall, the achieved results under laboratory scale conditions suggested the possibility to scale up the approach. Delays for the previous reasons have not made necessary to vary the action.

These variations have not influenced in the start of the related action A4 (design of the semiindustrial prototype), since it has started on time, but they have delayed its finalization until September 2019 when all the parameters of the semi-industrial has been finally optimized. The construction of the semi-industrial prototype (Action B1) has not been delayed for this reason since the units whose parameters were already known were constructed first.

The Actions A.2.2, A.2.3 and A.2.4 were continued until September 2018, due to some difficulties in additional staff recruitment (for both UNIBA and CNR-IPCF) and the long purchasing procedures, which allow us to have the chemicals, laboratory glassware and plasticware only after the foreseen end of Action 2.

The same, the Action A2 was continued by UCAM, UNIBA and CNR-IPCF to test the Italian cyclodextrin nanosponges. So, to overpass the problems related to the use of other tested adsorbents, partners in synergy evaluated, by means of adsorption tests, the performance of the new polymer. To verify the efficiency and behaviour of Mn CDs-BDE polymer (regarding previous  $\beta$ -CD-EPI polymer results), adsorption/desorption preliminary test were carried out in batch, using EPs of different families, to decide whether or not to purchase this polymer on the basis of these preliminary results. In this sense, parameters like contact time, dosing of the polymer, EP concentration, absorption capacity, kinetics and isotherms, were evaluated.

#### • Action evaluation:

Action A2 have been successfully concluded since:

- Different polymer with cyclodextrins have been synthesized and characterized in order to evaluating the capacity of adsorption and desorption of polymeric surfaces in batch and in continuous.

- Information about the adsorption capacity of hydrogel and biosorbents was obtained.
- The synthesized polymers have been capable of eliminating EPs from wastewater in percentages that vary (70-90%) depending on the structure of the same which is a significant environmental benefit for a wastewater treatment plant.
- These polymers improve the capacity of adsorption of pollutants with respect to other more classic, are able to regenerate which allows a reduction in costs.
- The influence of contact time, the dosage of the adsorbent, the concentration of EP, the pH of the initial solution and temperature have been determined. In addition, kinetic models and isotherms were evaluated and thermodynamic data were determined to understand the physical-chemical process involved in the adsorption.
- The conditions of continuous adsorption and desorption measurements in the semiindustrial prototype have been optimized.
- The problems of large-scale manufacture of cyclodextrins to use them at an industrial level have been solved.
- The MN CDs-BDE polymer is easy to large-scale manufacture, remove a great number of EPs like used as model (EPI-CDs), showing higher reusability, and allows the use of alternative washing solutions (like salts) to the acetate buffer
- UNIBA, CNR-IPCF and UCAM have evaluated the performance of the Italian cyclodextrins nanosponge and verified its suitability for replacing  $\beta$ -EPI-CDs polymer in the adsorption columns of the semi-industrial prototype.

This action can be implemented over time because tests with other types of polymer matrices can be carried out, with other types of EPs. At the research level it is important to do experiments with chitosan attached to a  $\beta$ -EPI-CDs polymer since it has given good results in batch. The achieved results under laboratory scale conditions suggested that the use bio adsorbents enable the possibility to scale up the treatment. In the next future, the study of different adsorbents should be taken into account.

Regarding Mn CDs-BDE polymer (positively charged), like used as model (EPI-CDs, negatively charged), remove a great number of EPs and is easy to be synthesized. Also, shows certain advantages over EPI-CDs, with respect to their higher reusability, counter pressures during in flux experiments aren't no required, allows the use of alternative washing solutions (like salts) to the acetate buffer, and her particle size can be tuned. However, is protected under patent.

The results of Action B2 and C1 regarding the treatment capacity of the semi-industrial plant suggest the convenience of keep on researching the possibility of reducing the swelling capacity of the Italian cyclodextrins nanosponge so the treatment flow can be increased. This is an important research line for scaling up the LIFE Clean Up prototype.

ТҮРЕ	Code	Name	Action	Deadline	Responsible	Status
DELIVERABLE	3	Nanoencapsulation report with cyclodextrins from the different EC. Optimal encapsulation conditions	A2	31/12/2017	UCAM	OK 31/12/2017
DELIVERABLE	6	Technical report on the results of optimization in the synthesis of	A2	31/01/2018	CNR-IPCF	OK
DELIVERABLE		bio adsorbents for the retention of ECs	112	51/01/2010		31/01/2018

		Standard procedure for the				OK
DELIVERABLE	7	development of CD polymers with tetrafluoroterephthalonitrile	A2	31/01/2018	UCAM	31/01/2018
		Standard procedure for the				OK
DELIVERABLE	8	development of CD polymers with epichlorohydrin	A2	31/01/2018	UCAM	31/01/2018
		Technical report on optimization				OK
DELIVERABLE	9	results in hydrogel synthesis for EC retention	A2	31/01/2018	UNIBA	21/01/2019
		EC retention				31/01/2018 OK
	11	Report of the desorption capacity	• •	20/02/2010	UCAM,	011
DELIVERABLE	11	(in Bach) of polymeric surfaces	A2	28/02/2018	UNIBA, CNR-IPCF	30/06/2018
		Report on the desorption			UCAM,	OK
DELIVERABLE	12	capacity (continuous) of	A2	28/02/2018	UNIBA,	
		polymeric surfaces			CNR-IPCF	30/09/2018
DELIVERABLE	13	Report of the adsorption capacity (in batch) of the polymeric	A2	28/02/2018	UCAM, UNIBA,	OK
DELIVERABLE	15	surfaces	A2	20/02/2010	CNR-IPCF	30/06/2018
		Report on the adsorption			UCAM,	OK
DELIVERABLE	14	capacity (continuous) of	A2	28/02/2018	UNIBA,	
		polymeric surfaces			CNR-IPCF	30/09/2018
	1.5	Report on the regeneration		20/02/2010		OK
DELIVERABLE	15	capacity and number of useful cycles of the polymeric surfaces	A2	28/02/2018	UCAM	30/09/2018
						30/09/2018 OK
DELIVERABLE	17	Prototyping and parameterization of purification equipment on a	A2	30/03/2018	HIDROTEC	UK
	1,	semi-industrial scale	112	20100/2010		30/09/2018

ТҮРЕ	Code	Name	Action	Deadline	Responsible	Status
MILESTONE	5	Completed lab-scale prototype	A2	31/01/2018	HIDROTEC	OK 25/04/2018 (arrival at UCAM)
MILESTONE	6	First performance tests of different materials in continuous flow	A2	01/03/2018	UCAM, UNIBA, CNR- IPCF	OK14/05/2018

Deliverables have been grouped according to the different materials characterized in order to facilitate the discussion of results. Consequently, UCAM has produced a single report including the following reports for CYCLODEXTRINS:

- D3. Report of nanoencapsulation with cyclodextrins of different EPs. Optimal conditions for encapsulation.
- D7. Normalized procedure for the development of polymers with epichlorohydrin.
- D8. Normalized procedure for the development of polymers with tetrafluorotereftalonitrilo.
- D11. Report on desorption capacity (in batch) of polymeric surfaces.
- D12. Report on desorption capacity (continuous) of polymeric surfaces.
- D13. Report on the adsorption capacity (in batch) of polymeric surfaces.
- D14. Report on the adsorption capacity (continuous) of polymeric surfaces.
- D15. Report on regeneration capacity and number of useful cycles of polymeric surfaces.

This deliverable was updated to include the results obtained with the Italian cyclodextrin nanosponges polymer and finally delivered in May 2022.

UNIBA has produced a single report for HYDROGELS, including:

- D9. Technical report on the results for the optimization of the synthesis of hydrogels for the retention of EPs.
- D11. Report on desorption capacity (in batch) of polymeric surfaces.
- D12. Report on desorption capacity (continuous) of polymeric surfaces.
- D13. Report on the adsorption capacity (in batch) of polymeric surfaces.
- D14. Report on the adsorption capacity (continuous) of polymeric surfaces.

UNIBA and CNR-IPCF has prepared a single report for BIOMATERIALS, including:

- D6. Technical report on the results for the optimization of the synthesis of bio adsorbent materials for the retention of EPs.
- D11. Report on desorption capacity (in batch) of polymeric surfaces.
- D12. Report on desorption capacity (continuous) of polymeric surfaces.
- D13. Report on the adsorption capacity (in batch) of polymeric surfaces.
- D14. Report on the adsorption capacity (continuous) of polymeric surfaces.

## 6.1.3. A3 Optimization of innovative Advanced Oxidation Processes

## ✓ Completed action

Foreseen start date: 01/01/2018 Actual start date: 01/01/2018

Foreseen end date: 30/03/2018 Actual end date: 30/09/2018

PLANNED		20	)17							20	18					
TIMETABLE	S	0	Ν	D	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION A3																
ACTUAL		20	017							20	18					
TIMETABLE	S	0	Ν	D	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION A3																

#### • Participants and responsibilities:

UCAM was responsible for testing pulsed light treatments. CTC was responsible for testing photocatalysis treatments. UNIBA was responsible for testing photo sensibilization with natural molecules for pollutant photodegradation. CNR-IPCF was responsible for testing bio adsorbent/TiO<sub>2</sub> composites for pollutant photodegradation.

#### • What has been done:

After having carried out a study of the characterization of the waters of different sewage treatment plants and having selected the pollutants that are recurrent in them (Action A1), it is

necessary to study how to affect the chosen treatments on the raw materials and their degradation capacity. To do this, in the first place, work must be done at the laboratory level, and these preliminary tests are used to carry out a semi-industrial level scaling of the advanced oxidation technologies that is used later.

#### a. <u>Pulsed light</u>

Two kinds of experiments were carried out, the first was a sequential cyclodextrin-PL process to degrade pharmaceutical compounds and the second a PL and  $PL/H_2O_2$  process to degrade pesticides.

Sequential cyclodextrin-PL process for degradation of pharmaceutical compounds

This experiment required three assays and it was carried out from 20 to 24 November 2017.

The assays were carried out on a mixture of 21 pharmaceutical compounds belonging to six different groups, which was firstly passed through a polymer of a commercial epichlorohydrin cross-linked  $\beta$ -cyclodextrin; samples were collected every five minutes up to 30 minutes, and samples from 15 and 30 minutes were also further treated with PL. Concentrations were determined by liquid chromatography MS/MS spectrometry.

The overall evaluation of the PL efficacy to eliminate the tested pharmaceutical compounds indicates that this technology is able to eliminate 50 % of the global concentration of these compounds. The efficacy is highly variable (Figure 5).

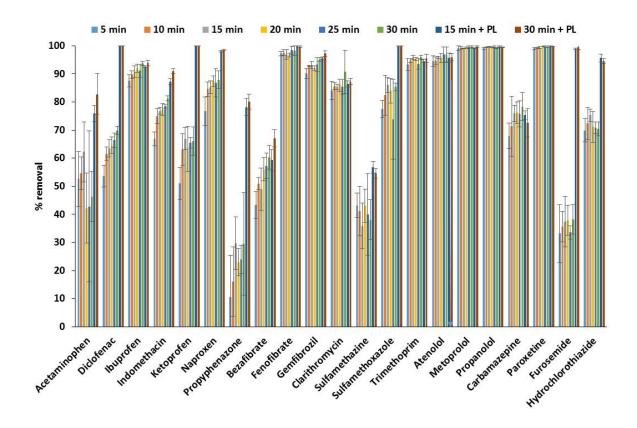


Figure 5. Evaluation of the PL efficacy

The overall evaluation of the sequential process, cyclodextrins plus PL, was very high. Cyclodextrins alone were able to decrease the global concentration of contaminants by 77 %; this value is raised to 91 % by the complementary action of PL.

#### PL and PL/H<sub>2</sub>O<sub>2</sub> process for pesticide degradation

A PL and a PL/ $H_2O_2$  process were tested for the degradation of pesticides in water in a single test. Additionally, a control treatment with only  $H_2O_2$  (no PL) was run before reaching trustable results, four tests were carried out which data was discarded due to quality concerns of the results provided by sub-contracted laboratories, as explained in the section "main problems encountered, and solutions provided".

PL alone was unable to degrade pesticides in water in a significant amount; only two out of eight pesticide concentrations were reduced by more than 50 %. In contrast, the use of PL in combination with  $H_2O_2$  (advanced oxidation process) completely eliminated half of the compounds. The effect of  $H_2O_2$  alone was also remarkable, to the point that two of the compounds completely eliminated by the AOP were indeed absent after  $H_2O_2$  treatment (Figure 6).

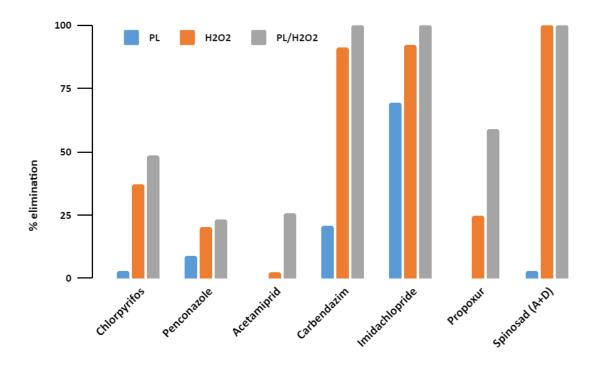


Figure 6. Evaluation of the PL efficacy for pesticide degradation

#### Overall assessment

The results implicate that the project can rely on PL technology in a context of advanced oxidation  $(PL+H_2O_2)$  to achieve the goal of destroying those amounts of emerging pollutants (pesticides and pharmaceutical compounds) that can be eluted from cyclodextrins. In particular, the success in this action serves as basis for action B.1, which deals with the scaling up of the process developed.

#### b. <u>Photocatalysis</u>

To perform the photocatalysis (FC) tests, a photocatalysis equipment was used with  $TiO_2$  as a catalyst. The tests were carried out with different water matrices (drinking water and treated wastewater) and in different volumes, enriched with different concentrations of the selected pollutants, in order to reproduce as much as possible, the actual work effluents that were worked with on a semi-industrial scale in the later phase of the project.

Likewise, it has been worked with and without the addition of hydrogen peroxide  $(H_2O_2)$ , reagent selected because its photolysis generates a high amount of hydroxyl radicals, increasing the oxidative character of the treatment, and the influence of the contact time of the water with the photocatalyst has been studied. Because the pollutants appear in the sewage treatment plants in very low concentrations, we have used similar concentrations in the tests carried out to try to work in real conditions.

In the tests carried out with treated wastewater, water from the station wastewater treatment plant (WWTP) La Aljorra was used. This sewage treatment plant has been selected since the water characterization analyses have revealed that in this WWTP an adequate water purification is achieved, and the output parameters correspond to an approximate average of the parameters obtained by all WWTPs managed by HIDROGEA that participate in the project.

To check the replicability of the results, up to eight tests for each compound have been carried out varying the parameters. The photocatalytic treatment is expressed as contact time. Contact times assessed have been 1, 5, 20, 30 and 40 seconds, in which all the volume of water used for the test goes through the photocatalyst.

The samples were taken in duplicate, the data expressed correspond to the average of the values obtained. Results have been obtained for trihalomethanes, phytosanitary and drugs and can be consulted in the Deliverable of this action A3.

After testing the capacity of the photocatalysis for the elimination of emerging contaminants present in the water, it follows that certain compounds have shown a high resistance to photocatalytic treatment, regardless of the time of treatment administered, while others have been shown to be more sensitive to treatment, so that photocatalysis achieves good degradative performance.

The addition of  $H_2O_2$  to increase the oxidative power of the treatment has been especially effective in those pollutants that *a priori* show a greater resistance to photocatalytic treatment or a dispersion in the results obtained.

Likewise, the implication of photocatalysis on the microbiological activity of water has been studied. The microbiological quality of the treated wastewater conditions the possibility of reuse for agricultural use, so that the quality criteria are based mainly on the sanitary (microbiological) quality of the water.

The following table (Table 3) shows the microbiological results after the photocatalytic treatment:

Table 3. The microbiological results after the photocatalytic treatment

	cfu/mL	cfu/100 mL							
	Aerobics	C. perfringens	Total coliforms	E. coli	Enterococcus				
Initial concentration	14	12	<4	0	0				
25 sec FC	<4	<4	0	0	0				
Initial concentration	900	60	50000	6000	>1000				
25 sec FC	68	0	31	0	<4				
Initial concentration	27000	>1000	760	10	0				
25 sec FC	8	0	0	0	0				

According to the results obtained, the photocatalysis has disinfecting power and improves the microbiological quality of the water by eliminating the low microbiological load that remains after the conventional purification processes, eliminating the spores of *C. perfringens* that, as mentioned above, have a high resistance to disinfection processes, which can cause serious problems.

#### c <u>Photo sensibilization with natural molecules and bio adsorbent/TiO<sub>2</sub> composites</u>

The results obtained by UNIBA and CNR-IPCF in the development of action A3 regards to the photodegradation of Tetracycline (TC), Ketoprofen (Kp) and Diclofenac (DCF), because other examined pollutants did not respond to photocatalytic treatment (Deliverable D16). Among several ways to induce the pollutant oxidation, the synergic use of UV, H<sub>2</sub>O<sub>2</sub>, also in presence of Fe<sup>2+</sup> under Fenton conditions and TiO<sub>2</sub>, was also explored. The number of total tests was 1000. Experiments were performed from January 2018 to September 2018.

Results show that the addition of  $H_2O_2$  to  $TiO_2$  and the combination UV/ $H_2O_2$  improved the TC degradation (times of greater degradation are around 1 hour). In the case of DFC, results indicated different pathways of reaction during the sunlight irradiation, in this case ascribable to the solid-state degradation. The synergistic action of  $TiO_2$  and Rose Bengal (RB) was not observed, and the only use of RB did not exhibit important effects on the degradation process.

#### • Main problems encountered and solutions provided:

In the case of FC, we have not had problems worthy of mention. As highlighted in action A1, the analytical of drugs and trihalomethanes is prepared to detect tiny concentrations of contaminants in water. However, phytosanitary analytics, despite detecting small amounts of contaminants, do not reach the limits of quantification of drug analysis. Since in the tests carried out different concentrations have been used, starting from those detected in the WWTP, in order to reproduce the conditions of real work to the maximum, reason, modifications have been made in the extraction methodology, lowering the limit of quantification to the level of drugs.

Tests for PL technology in continuous mode would have required a PL system that could be operated in that mode which was not included in this stage of the proposal (only for Action B1). This type of equipment does not exist in market (for the semi-industrial prototype, actual equipment was adapted to work in flow as required to the supplier). Alternatively, the PL system available at UCAM was used. This works under a very slow pulse repetition rate (1

pulse per minute) in contrast to the system planned for the project (3 pulses per second), which is 180 times faster. Due to the huge time required to obtain results, only a limited amount of data was possible to collect. Additionally, many tests were wasted because analytical results from sub-contracted laboratories were not trustable. Likewise, some of these laboratories required sample volumes higher than those planned in the project, which also limited the amount of data produced and also precluded the estimation of the variability of results because in many cases samples had to be pooled for analysis.

In Bari, due to the enormous time required to obtain results, only a limited number of experiments was possible to carry out. Thus, only some of the investigated pollutants were studied. Further, it was not possible to study the continuous regime, since the instrumentation present in UNIBA and IPCF-CNR laboratories is not adapted to perform tests under in flow regime.

## • Achieved results:

FC 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. We have seen that when working with wastewater the efficiency decreases as there is competence with other organic compounds. Pollutants of the same family have not shown to follow behaviour patterns when the photocatalytic treatment has been administered, in most cases. A high dispersion has been observed in the data obtained when performing different tests, although most of the pollutants have shown the same tendency in the behaviour after applying the treatment. The addition of H2O2 has effect depending on the drugs; in general, it has an effect on those substances that are reluctant to photocatalysis.

For the PL system, results were collected only under batch conditions for two families of compounds (pharmaceutical and pesticides). Abatement due to CDs is 77%, adding pulses increases up to 91% and at least 55% removal was attained for every compound. The technical specifications for the lamp in the semi-industrial prototype are the same than the one used in UCAM, only the container changes. This lamp works in flow conditions since partners have worked with him to adapt the existing technology to these conditions.

In Italy, among several ways to induce the oxidation of the pollutant, the photodegradation of some of the studied pollutants, i.e., TC, Kp and DCF, was accomplished using UV,  $H_2O_2$  and their synergic use also in presence of Fe<sup>2+</sup> under Fenton conditions and TiO<sub>2</sub> (D.16). The pollutants did not exhibit the same behavior when subject to the photocatalytic treatment. However, these experiences were not applied for the semi-industrial prototype since the adsorbent material was different than the used in Italy.

#### • Remaining results:

None because this action has been completed.

## • Variations in action:

The FC tests have been extended over time in order to obtain as many data as possible to establish the behaviour of the different pollutants when subjected to the treatment. Kinetics studies that were not initially foreseen were included because they are considered essential for the scale up of the technology proposed.

For the PL system, no extra-time was required to finish this action under the aforementioned limitations. Results for degradation of pharmaceutical compounds were obtained from samples obtained in a sequential cyclodextrin-PL treatment, which was not initially planned; this experimental design allowed to make the best profit of sub-contracted (paid) analysis.

Extra-time was required to finish this action in Italy, from January 2018 to September 2018, in order to obtain as much information as possible for comparing the photodegradation behavior of different pollutants.

#### • Action evaluation:

The results obtained for FC comply with the expected objectives according to the project proposal, since we have worked contemplating the different parameters involved, with different families of pollutants, and making as many replicas as possible. The action extended for 6 months in order to make kinetic studies and to test a major number of EPs. This has contributed to get better results for the scaling-up of the prototype. This delay has not affected Action A4 and B1 since the work has been done in parallel.

For the PL system, no results under continuous mode were possible and for a big variety of compound families were possible. However, results were useful for scaling-up the equipment for future actions, although it is foreseen than the optimization of working conditions of the scaled-up system would require more tests than those forecasted. It is worth mentioning that the function of the pulsed light system within the whole system would not necessarily require to work under continuous mode. Additionally, the high variety of emerging contaminants from many different families makes impossible to test all of them, but the project tested 28 different compounds. This could be considered useful to have a good picture of the working conditions necessary for the degradation of other mixtures of compounds.

Unfortunately, the test under continuous mode in Italy were not possible due to the limitations discussed so far. However, the achieved results under laboratory scale conditions suggested that the use of light and AOP enable the possibility to scale up the treatment.

ТҮРЕ	Code	Name	Action	Deadline	Responsible	Status
DELIVERABLE	16	Technical study on the capacity of oxidation technologies for the continuous and/or discontinuous treatment of treated water contaminated by emerging contaminants	A3	30/03/2018	CTC, UCAM, UNIBA, CNR- IPCF	OK 30/09/2018

## 6.1.4. A4 Preliminary studies and calculations of the prototype at semiindustrial level

#### ✓ Completed action

Foreseen start date: 01/02/2018 Actual start date: 01/02/2018

Foreseen end date: 30/06/2018 Actual end date: 30/09/2018

PLANNED TASKS 2017 2018				
	PLANNED	TASKS	2017	2018

TIMETABLE		S	0	Ν	D	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION A4	Task A4.1																
	Task A4.2																
	TASKS		20	)17							20	18					
ACTUAL		S	0	Ν	D	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
TIMETABLE ACTION A4	Task A4.1																
nenonn	Task A4.2																

#### • Participants and responsibilities:

HIDROTEC was responsible for the design and detailed engineering of the prototype, provided Actions A2 and A3 have produced the necessary input. REGENERA was responsible for the energy system design. HIDROGEA participated in this Action by contributing knowledge about the Cabezo Beaza WWTP and providing all the necessary information on the installation and quality of the treated water to carry out the design of the different units.

#### • What has been done:

Once the location of the WWTP is selected, the design of the different units that conformed the pilot plant were developed. After studying the characteristics of this WWTP, partners decided that the installation of a pre-treatment prior to the CD columns is necessary. Although the quality of the water from the WWTP is very good, complying at all times with the established quality standards, the no presence of tertiary treatment implicitly involves a quantity of suspended solids and organic matter that can cause a fouling in the filling material of the columns (CDs) that interferes with the process of elimination of emergent (decrease of the adsorption performance), being necessary a regeneration of the same by dirt, not by accumulation of CEs.

#### Task A4.1 Design engineering and detailed engineering of the prototype at semiindustrial level for water treatment

The activities related to the engineering works of the prototype followed in the project have been:

- a. Functional Specification of the Design: All the functions of the system and the way that they are going to function are described. For this, calculations for sizing the different equipment units and elements that form the plant are done. These calculations can be consulted in the deliverable of action A4.
- b. Design: Elaboration of plans, flux diagrams, electrical schemes, lists of equipment and instruments. Plans of equipment and structures design are attached to this report.
- c. Purchase orders: the equipment and materials needed for the installation are purchased.

The function of the prototype is described as follows:

1. The system consists of two alternative adsorption lines, one active and the other in regeneration / desorption / rest to treat a flow rate of  $3000 \, l/h$ .

- 2. To capture the process water, a WWTP service line is used, taking advantage of the already existing pumping. At the entrance of the prototype there is a motorized solenoid valve whose actuation is determined by the levels installed in the supply tank. The tank is equipped with minimum and maximum levels so that this pump is activated, providing the necessary water for the process.
- 3. 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 an ultrafiltration system with ceramic membranes to prevent unwanted substances from passing through and produce the clogging of the adsorbent material. Once the water has been physically conditioned, and after control of pH parameters and turbidity, it is introduced into one of the two adsorbent material filters.
- 4. There is always a process in service and another in desorption or rest, so that the process is continuous at all times. At the exit of the adsorption process, a photocatalysis equipment is installed so that any traces of contaminants that have not been retained are removed by oxidation.
- 5. Finally, the water is conducted to a product water tank, which provides a storage of water necessary to carry out the processes of rinsing, backwashing and sponging of the beds of adsorbent material. The water is evacuated from the reservoir by an overflow to the WWTP outlet stream, so that it always remains full and with moving water.
- 6. The desorption process is carried out when the estimated time for the clogging has been fulfilled. For this, a membrane pump takes the desorbent at the calculated concentration and flow rate, to introduce it into the polymer column of cyclodextrins in order to produce the displacement of the contaminant towards the solution, leaving the cyclodextrin polymers with their initial adsorption capacity.
- 7. The product of the desorption loaded with contaminants is accumulated in a reject tank for further treatment. This product is pumped at an adequate velocity through a pulsed light system capable of destroying the contaminants it has dragged. This process is optimized so that its duration is somewhat less than the useful time of saturation of the bed; in this way, the light pulses equipment can be dimensioned in a rational way.

Main operational parameters are:

- a. Flow direction: Fluidized bed.
- b. Bed Heights: 500-800 mm.
- c. Operation velocity: 12 m/h.
- d. Operation flow: 3m<sup>3</sup>/h.

# Task A4.2 Design and construction of the subsystem for energy supply of the prototype at semi-industrial level for water treatment by solar energy

When designing and building the photovoltaic solar energy system for the energy supply of the solution, the following steps have been followed:

- a. Study of energy needs
- b. Design of the system adapted to the needs of the semi-industrial prototype
- c. Extrapolation of the design to actual water treatment flows
- d. Definition of the elements of the installation
- e. Design of the monitoring system

The first step was the identification of the consumptions of its components. In order to know the consumption of each component, a key factor to consider is the flow rate. However, in the course of the project this flow has varied. As a result of these previous studies, some conclusions have been reached, that can be used in the final design of the aforementioned energy system.

Following the identification of the consumptions, the operating process was simulated. As a result of the simulations, it is established that the operation and desorption processes do not have to be produced at the same time, so in order to achieve better energy efficiency of the process, a design was chosen that considers the displacement of the loads that separate them in time, thus achieving a lower peak power required.

	Isolated	Connected
Location	Murcia	Murcia
Latitude	38,0 ° N	38,0 ° N
Length	1,2 ° W	1,2 ° W
Altitude	49 m	49 m
Albedo	0,2	0,2
Inclination	25°	25°
Nominal Power	8,45 kWp	5,85 kWp
No. Modules	26	18
Module surface	50,6 m2	35 m2
N° Batteries	16	0
Voltage/Capacity Batteries	<sup>3</sup> 48 V/640 Ah	0

Table 4. Simulation of the process to achieve energy efficiency

These simulations allowed several conclusions since, although the definitive results were obtained, an approximation allowed the design of some of the key aspects of the prototype.

The parts of the energy supply prototype of the semi-industrial equipment are basically the photovoltaic modules and his structure, the inverter, the battery, the energy monitoring device and the electrical installation to connect the equipment.

Another key aspect when carrying out the energetic study of the prototype is the design of the monitoring system, thus being able to adjust the extrapolations to real treatment plants as much as possible.

With the study of the data obtained in the monitoring, the characteristics of the elements of the photovoltaic system were defined. Subsequently the monitoring system included focus on knowing the energy behaviour of the prototype in order to evaluate the following aspects:

- Cost in € of kilowatt-hour generated.
- Energy needed for water treatment in Kw-h / m<sup>3</sup> of processed water.
- Energy cost in € of each m<sup>3</sup> of water treated for different amortization models: 10-15-20 years.
- Renewable coverage ratios of the system load curve for different sizes and energy consumption.

- Avoided greenhouse gas emissions in the LIFE cycle of the future commercial product.

## • Main problems encountered and solutions provided:

Due to the problems found in the tests at laboratory level to work with CDs in downstream direction and to the high probability of dirtiness by microparticles that can exist in the effluents of WWTP, changes have been done in the initial design of the semi-industrial prototype:

- 1. Adsorption columns have been designed with nozzle plates instead of collector arms. The size of the slot of the nozzle is 0.2 mm to avoid leaks of the adsorbent material. It also includes a layer of bracket material in order to avoid clogging. Sight glasses have been also included in the adsorption columns in order to have a visual reference of the adsorbent material during the operation stage.
- 2. The operation flow has decrease to 3 m<sup>3</sup>/h. If the initial operation flow had been maintained (5 m<sup>3</sup>/h), with the new conditions of operation of fluidized bed and velocity lower than 12 m/h, the necessary adsorption columns to maintain the fluidized bed and the optimal contact time should have a minimum height of 2 meters. The final design of these columns has higher total height than the container where the equipment is hosted, so work with this flow is unfeasible.
- 3. The pre-treatment unit works with ultrafiltration by ceramic membranes to have an effluent as clean as possible in order not to interfere with the adsorption process.
- 4. In the desorption process, the buffer compound has been modified, being acetate instead of an alcohol such as methanol. Values obtained in the desorption process with acetate were better than those of methanol. Moreover, we have another side benefits: less pollution and better manipulation.

Due to the fact that the initially estimated flows have been reduced, the consumption of the components has to be re-established. Consequently, the effort used in this activity and its duration has increased.

#### • Achieved results:

- Different units of the prototype 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.
- Simulation of the energy needs of the prototype to work in isolation from the electricity grid.
- Simulation of the energy needs of an emerging pollutant treatment plant with real flow rates in order to operate in a grid-connected manner.
- Installation consumption inventory and process grouping.
- Definition of the elements that make up the photovoltaic system.
- Design of the monitoring system.
- Design of the photovoltaic system based on the simulations carried out.

#### • Remaining results:

None 36

## • Variations in action:

Due to the insufficient capacity of the battery to provide the energy necessary for the prototype to operate at night, the photovoltaic system has been connected to the grid, so that the prototype can persist to operate continuously, being supplied by either PV or grid power, or by both simultaneously

## • Action evaluation:

The development of this action has been successful. Results achieved in laboratory in action A2 and A3 have allowed to better adjust the operational parameters defined and calculated in this action.

TYPE	Code	Name	Action	Deadline	Responsible	Status
DELIVERABLE	19	Execution Project (detailed engineering) of Prototype a semi-industrial scale of an emerging contaminant removal system using adsorbent material and advanced oxidation	A4	30/06/2018	HIDROTEC	OK 30/09/2018
DELIVERABLE	20	Design project and specifications of the generation/storage prototype system.	A4	30/06/2018	REGENERA	OK 30/09/2018
DELIVERABLE	21	Design Project and Prototype calculations on a semi- industrial scale of an emerging contaminant removal system using adsorbent material and advanced oxidation	A4	30/06/2018	HIDROTEC	OK 30/09/2018

Deliverables 19 and 21 has been merged in one single report regarding the detailed and design engineering of the semi-industrial prototype.

## 6.1.5. B1 Construction of the prototype at semi-industrial level for the WWTP

## ✓ Completed action

Foreseen start date: 01/07/2018 Actual start date: 01/07/2018

Foreseen end date: 31/10/2018 Actual end date: 30/04/2019

Between September to December 2020 the adsorption unit was completed with the addition of the polymer.

PLANNED			20	18									201	9					
TIMETABLE	J	J	Α	S	0	Ν	D	E	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION B2																			
ACTUAL					201	18							201	9					

TIM	ETABLE	J	J	Α	S	0	Ν	D	Е	F	Μ	A	Μ	J	J	A	S	0	N	D	
ACT	TON B2																				

PLANNED						20	20					
TIMETABLE ACTION B2	J	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTUAL						20	20					
TIMETABLE	J	F	Μ	A	Μ	J	J	A	S	0	N	D
ACTION B2												

#### • Participants and responsibilities:

HIDROTEC is responsible for the designing and manufacturing of the semi-industrial prototype.

REGENERA is responsible for the design and construction of the energy system of the prototype, which is a photovoltaic installation with batteries.

#### • What has been done:

Once the selection of the equipment that make up the system has been validated, the engineering department begins to develop the manufacturing drawings to start the construction of the semi-industrial prototype.

The following Gannt diagram shows the phases and deadlines used for the construction of the equipment in Hidrotec facilities (Figure 7).

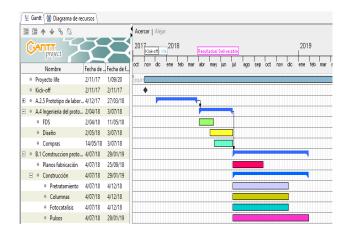


Figure 7. Gannt diagram with phases and deadlines used for the construction of the equipment.

The prototype was designed in 4 distinct phases as follows:

- 1. Pretreatment
- 2. Adsorption
- 3. Photocatalysis

- 4. Desorption/Light pulses.
- 5. Electrical and control panel.

The assembly of the pre-treatment, adsorption and photocatalysis units were carried out from July to December 2018, and the desorption and light pulse equipment was built from December 2018 to February 2019 since the LP equipment did not arrive until January 2019. In the assembly of this phases participated 4 workers of HIDROTEC and another one developed the automatization of the system.

Below we show a sequence of assembly drawings accompanied by different photographs of the manufacturing stages.

Pretreatment Plans

• Photos: Pre-treatment frame/assembly (Figure)



Figure 8. Photos: Pre-treatment frame/assembly

• Adsorption frame/assembly (Figure )



Figure 9. Adsorption frame/assembly photos

Photocatalysis Plans

• Photocatalysis frame/assembly (Figure 10):



Figure 10: photocatalysis frame/assembly photos

Light Pulse Plans

• Light pulse frame/assembly (Figure 11):

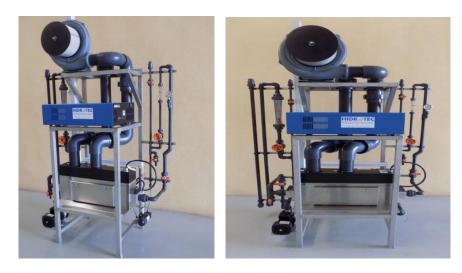


Figure 11. Light pulse frame/assembly.

Finally, the different structures manufactured separately are assembled and connected inside a 20 feet maritime container from February to March 2019 for the final transfer to the Cabezo Beaza WWTP for commissioning.

Container assembly (Figure )



Figure 12. Container assembly photos

Photos: electrical panel (Figure 13):



Figure 13. electrical panel photos

Regarding the energy system of the pilot plant, it consists of a photovoltaic installation. Once the energy demands of the prototype were defined during the study phase, the power and capacity that the photovoltaic and batteries must have been estimated so the system could satisfy these demands, being independent from the electricity grid. The connection diagrams for the equipment were drawn up. At the same time, a search for the necessary equipment is carried out, requesting offers from different suppliers.

When the connections and the equipment to be installed (photovoltaic modules, inverter, charge controllers, batteries, monitoring and electrical system) were defined, the construction at the Cabezo Beaza WWTP began in January 2019, ending in the same month. Once the photovoltaic system has been completed, it was connected to the prototype (Figure ).



Figure 14. Photovoltaic installation

Once all the connections between the equipment were completed, the equipment was configured, and the installation was ready to supply power to the prototype once it was up and operating.

## • Main problems encountered and solutions provided:

During the assembly of the prototype there have been some inconveniences derived from the adaptation of equipment manufactured to work in the laboratory to a system that works continuously.

The equipment acquired had to have the light pulse chamber modified by making an opening on both sides. In this way, the effluent can pass through the pulse chamber at a certain flow rate, applying the pulsed light in continuous mode (Figure ).

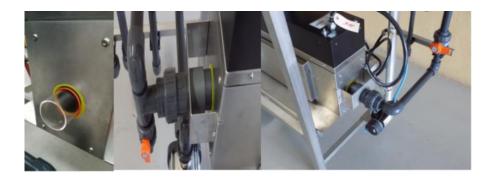


Figure 15. The light pulse chamber modified

Another problem found during the development of this action was the production of the polymer at bigger quantities than those achieved at laboratory scale. During the preliminary actions of LIFE Clean Up (Action A2), the most efficient adsorbent material for the semi-43

industrial prototype was selected. It was concluded that, after comparing all the obtained results in terms of adsorption, desorption, regeneration capacity, contact time and costs, the EPI-CDs Polymer was the one to be used for semi-industrial prototype in the WWTP. However, for the synthesis of this type of polymer, the crosslinker agent is epichlorohydrin which has later presented problems for scaling up the production of the polymer and also for having the amount needed for the semi-industrial prototype (200 kg) due to its toxic character. Neither UCAM nor CTC (as it was foreseen in the proposal) were able to produce the polymer in their facilities, despite HIDROTEC worked in a reactor to do so. Then, the partnership looked for alternatives in order to have enough amount to run the semi-industrial prototype and demonstrate the feasibility of the proposed combination of technologies of LIFE Clean Up (adsorption, photocatalysis and light pulse).

Since no provider was found neither in China or Europe to produce enough amount, IPCF-CNR and UNI BARI proposed to contact an Italian supplier of cyclodextrin polymers (Prof. Francesco Trotta, University of Turin), whose synthesis is under patent. After laboratory tests, the Italian polymer showed much higher adsorption capacities than the UCAM polymers and, at the same time, it exhibited lower counter pressure during in flux experiments (performed in laboratory scale). Also, its synthesis does not involve toxicity, so its production is easier and show more favorable conditions for scaling up and for replicating this solution. During the 4th SCM held on 14 January 2020 in Bari, partners decided to keep on making tests with the Italian polymer to validate its feasibility and calculate in the laboratory prototype the parameters to be scaled up in the semi-industrial one. These analyses were done, and the project ordered 150 kg of polymer to run the prototype, according to laboratory calculations. The polymer finally arrived in September 2020, but the order received did not visually seem like the sample previously received to make the laboratory tests. For this reason, HIDROTEC and HIDROGEA carried out tests to determine the particle size of the polymer received and the swelling capacity of the polymer in contact with water from September to November 2020. It was observed that the polymer did not meet the requested particle size (> 200 microns). So, after the loading of 75kg of polymer in one of the reactors in November 2020 (because it was urgent to start up the plant before the end oy the year), the remaining 75kg were sent to Italy for further synthesis in December 2020. The polymer re-synthesized was received in February 2021.

As for the energy system, when the prototype was ready to operate and the photovoltaic installation was to be started up, this could not be done due to a problem with the installation's control equipment, which prevented the inverters from generating energy. The connections and configuration of the equipment were checked, and the problem was ruled out. After consulting with the manufacturer of the equipment, it was determined that it was defective, and it was replaced with new equipment. Once the defective equipment had been replaced, the photovoltaic installation operated correctly in April 2021.

#### • Achieved results:

- Materials list
- Manufacturing drawings.
- Electrical diagrams
- Control program
- Skid manufacturing
- Assembly of main equipment
- Hydraulic connection of equipment.

- Electrical Connection of equipment.
- Semi-industrial prototype constructed.
- Photovoltaic system constructed
- Energy monitoring system

## • Remaining results:

## N/A

## • Variations in action:

The synthesis of the polymer was subcontracted instead of made by the consortium. This has caused an increase in the external assistance budget line. The purchase was made by UCAM after an internal agreement with all the partners to cede part of their budget.

Due to the insufficient capacity of the battery to provide the energy necessary for the prototype to operate at night, the photovoltaic system has been connected to the grid, so that the prototype can continue to operate continuously, being supplied by either PV or grid power, or by both simultaneously.

## • Action evaluation:

Factory acceptance test. OK 26/03/19

ТҮРЕ	Code	Name	Deadline	Responsible	Status
DELIVERABLE	23	Manual of use of the prototype and flux diagrams	30/10/2018	HIDROTEC	OK 30/10/2018
MILESTONE	10	Prototype system of energy generation/storage built and ready to use	30/10/2018	REGENERA	OK 26/04/2019
MILESTONE	11	Prototype ready to be installed in WWTP of HIDROGEA	30/10/2018	HIDROTEC	March 2019 (without polymer)

## 6.1.6. B2 Installation, commissioning and optimization of the prototype

## ✓ Completed action

Foreseen start date: 01/10/2018 Actual start date: 01/10/2018

Foreseen end date: 31/03/2022 Actual end date: 30/05/2022

PLANNED		201	18							201	9											20	20					
TIMETABLE	S	0	Ν	D	Е	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	J	F	Μ	A	Μ	J	J	А	S	0	Ν	D
ACTION B2																												
ACTUAL	ACTUAL 2018									201	9											20	20					
TIMETABLE	S	0	Ν	D	Ε	F	Μ	A	Μ	J	J	A	S	0	N	D	J	F	M	A	M	J	J	A	S	0	Ν	D

ACTION B2																												
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PLANNED						20	21								20	22		
TIMETABLE	J	F	Μ	A	Μ	J	J	Α	S	0	N	D	J	F	Μ	A	Μ	J
ACTION B2																		
ACTUAL		2021												20	22			
TIMETABLE	J	F	Μ	A	Μ	J	J	А	S	0	Ν	D	J	F	Μ	А	Μ	J
ACTION B2																		

#### • Participants and responsibilities:

HIDROGEA has been responsible for the operation of the plant during a whole year and for its maintenance before and after operation.

HIDROTEC and REGENERA participated in the installation of the pilot plant and energy system, start up and in maintenance tasks when some unit has not worked correctly and has produced a shutdown of the semi-industrial prototype.

UNIBA, UCAM and CNR-IPCF has participated when assessing about the performance of the plant, providing suggestions for improvement. UCAM has also collaborated for the installation and running of the PL System. CTC has also contributed for the installation and running of the plant and has been charge of taking samples for its analysis in Action C1.

## • What has been done:

Before commissioning the semi-industrial prototype at the WWTP, HIDROGEA made site preparation, civil works, installation of a concrete slab, electrical supplies, safe water, service water, sewage and water to feed the pilot plant. These works started in October 2018.

The location within the WWTP responded to the following criteria: the nearest place to power and water supply, having enough space, easy access, the installation does not disturb the WWTP routine works, etc. The container was finally installed in the Northeast area of the WWTP, next to the secondary settlers.

Figure shows the location of the pilot plant, along with the secondary settlers and the chlorination channel, where the treated water intake would be produced to feed the pilot.



Figure 16. Location of the pilot plant in Cabezo Beaza WWTP.

To locate and attach pilot plant units, a concrete slab (Figure , Figure 18). was built to sustain a container of 8Tn, including a perimeter gutter to evacuate water wash-downs (Figure ).



Figure 17 Concrete slab works. Figure 18. Concrete Slab finished



Figure 19. Pilot plant container on the concrete slab.

The pilot plant had two water catchments:

- Wastewater feed catchment Ditch conduction: buried plastic pipe on a narrow trench backfilled to natural ground surface level, from catchment point to concrete slab.
- Service water catchment Ditch conduction: buried plastic pipe on a narrow trench backfilled to natural ground surface level, from catchment point to concrete slab. The industrial water is used for wash-downs, hosing down works, cleaning operations and clean-water inlet of the rotary screen unit. Due to the characteristics of WWTP's industrial water (analytical qualities and flow and line pressure requirements) are high enough to cover the prototype necessities, it is dispensed with drinking water supply.

It was built a siphon pit as a sewage disposal point to collect overflows, purges, etc. The manhole connects with the main WWTP drainpipe which returns the water to the chlorination channel.

There are two buried wire lines:

The first line joins the main electrical panel of the pilot plant with a WWTP distribution board. It is assembled and connected with a TMCB (Thermal magnetic circuit breaker) by a cable section 4x6 (4 wires of 6 mm). The electrical system provides up to 20 A current (AC).

The second line is for the renewable energy grid. This system is composed by three pieces of equipment with energy consumption. These are the photocatalysis, light pulses and the cleansing pump.

The pilot plant was installed in the WWTP "Cabezo Beaza" in March 2019. The control of the operating flow was carried out by means of totalized flow meters, the extraction of data from the SCADA and the export of events and alarms.

REGNERA certified the installation of the photovoltaic modules and structure with the crane, inverter, charge controller and battery into the pilot plant already in the WWTP and its electrical installation, in April 2019. A second photocatalyst was installed in October 2019.

From March 2019 (installation of the semi-industrial plant) to November 2020 (filling of the adsorption columns with the polymer) partners checked the correct function of all units of the pilot plant by circulating water in the circuit, tubes, equipment, etc. and performed tests for each unit separately. Data acquisition measured different variables of the process (pH, flow, temperature, level, etc.). Water samples were analysed in the laboratory of the WWTP by HIDROGEA to carry out the physic-chemical characterization of the treated water in the WWTP which is feeding the pilot plant whereas CTC developed analysis in the pre-treatment and photocatalysis units to know the performance for eliminating EPs and UCAM in the LP equipment.

In 2020, REGENERA also had to work in the improvement of the electrical installation of the pilot plant. The electrical power supply was stablished by a photovoltaic off grid system (10 Kw) and by its connection to the electrical grid (auxiliary power supply).

Once the polymer was entered in the adsorption column, the pilot plant was functioning from December 2020 to December 2021. The two first months were employed for the starting up of the plant and from February to December 2021 it functioned in a continuous mode (Table ).

TEST PERIOD	DATES
Winter period	03/02 - 11/03
Spring period	07/04 - 17/05
Summer period	01/06 - 2/08

Table 5. Summary	of test	period
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Autumn period	01/10 - 17/12
Autumn period (only 1 UV)	13/10 - 17/12

The pilot plant consists of 4 stages: 1) **Pretreatment** to remove organic matter, 2) **Absorption stage** to partial remove emerging pollutants, 3) **Advanced oxidation process** (hereinafter **AOP**) to total remove EPs and, 4) **CD regeneration** by polymer desorption process and **UV light pulse system** to EPs degradation.

The effluent of WWTP feeds the pilot plant. Firstly, an initial pretreatment removes the organic matter and the solids which can provoke a problem in the next stages, by mean of a microfiltration with cartridge filters and ultrafiltration ceramic membrane. The 2nd stage consists of two columns which are filled by Cyclodextrins (herein after CD) polymer, although only one of them operate. The reactor should guarantee more than 10 minutes of contact time wastewater-CD. The 3rd stage ensures EP removal due to a UV reactor made of titanium, which allows the generation of hydroxyl radicals. When the CD are saturated with EP, the polymer must be regenerated by means an EP extraction process. The stream with EC concentrated must be sent to UV light pulse system to degrade the EP extracted. The pilot plant can be operated with a SCADA (Supervisory Control and Data Acquisition) system.

For monitoring the operation of the plant and adjust maintenance parameters, the following aspects have been considered:

- Operational Keys: In the pre-treatment, operation hours (OH), feed flow (FIT01), recirculated flow (FIT02), reject flow (FIT03), pressure before cartridge filters (PI01), pressure after cartridge filters (PI02), ceramic membrane pressure (PTM), feed turbidity (TB01), feed pH (PH01) and feed temperature (TT01). In the absorption, work column (column 1, 2 or both), treated flow (FIT04). In the AOP, operation hours of each lamp, UV dose (kW/m2). In the CD regeneration: power consumption, time of regeneration of reactor and regeneration flow (l/h). The energy parameters registered are power consumption, photovoltaic power supply, grid power supply and battery status.

Based on operational parameters registered, it has been calculated Wh/m3 ratio of the pilot plant and reject flow (%).

- Maintenance keys: frequency of change of the cartridge filters, frequency of chemical cleaning of ceramic membrane and frequency of cleaning of UV lamps.

During the winter season, treated flows were low and operation cycles short due to the fast fouling on the ceramic membrane and the saturation of the cartridge filters. The following operation maintenance were done in coordination between HIDROGEA and HIDROTEC:

- 1. Changing the kind of **cartridge filters** to 25  $\mu$ m.
- 2. Adjusting operation parameters.
- 3. Substituting and cleaning of ceramic membrane (25/03/2021).
- 4. Implement a **disc filter** in the feed on the plant (09/03/2021).

These periodic maintenance operations were needed to achieve continuous work mode with an **average treated flow between 74-250 l/h** for the whole working period. The frequency of the maintenance operations was variable over time and flow, but normally we needed to change the cartridge filters one, twice or more time at week and, at least, do a chemical cleaning of the ceramic filter once a month. About the cleaning of the disc filters and ultraviolet system, it would be carried out once a month and every two or three months respectively, as we can see in the Figure .

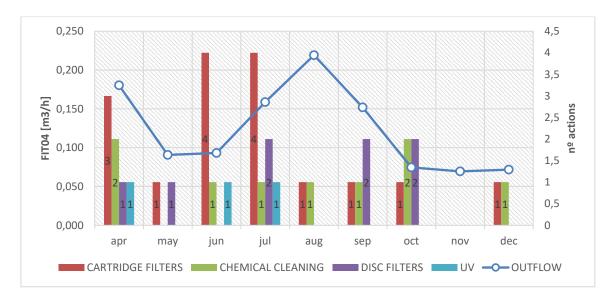


Figure 20. Fit04 related to the maintenance requirements

Because several modifications had to be made to the pilot plant pretreatment, and also to obtain high yields of cyclodextrins, the plant flow rate has been reduced to about 0,25 m3/hr. Therefore, the estimated flow treated in one day would be 6 m3 and per year about 2.190 m3/h.

Regarding the estimated supply to be done by the energy system, it was considered that the photocatalysis has daily consumption of 4000W, the cleansing pump 850W and the light pulses 4000W. The photocatalysis and the light pulses consume energy for around 3 hours/day, 5 days a week, whereas the cleansing pump only consumes 0,5 hours /day, 5 days a week. Thus, the daily consumption of the photocatalysis and the light pulses are 12000 Wh/day and the daily consumption of the cleansing pump 425 Wh/day. The total daily consumption of the prototype is 24425 Wh/day and 8915 kWh/year and 1.7 kWh/m3 (Figure ).

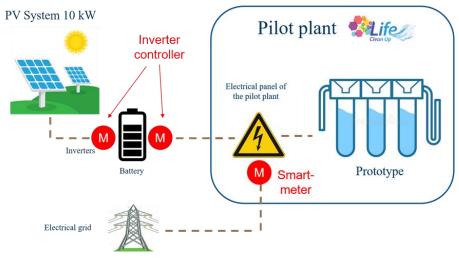


Figure 21. Electrical supply of the pilot plant

## • Main problems encountered and solutions provided:

During the start-up of the pilot plant there was a problem with the connection to the PV off grid system. Due to a malfunction of the battery inverter controller the plant only worked through the supply from the electrical grid. Nevertheless, the equipment was repaired and the PV installation was in operation since 23/04/2021, as well as the meter that registers the energy generation, the storage in battery and the energy consumption of the prototype by the photovoltaic source.

Bad weather and low temperatures since November 2020 resulted in the poor quality of the water at the WWTP (mainly large amount of suspended solids and high COD), causing fouling of cartridge filters in 4 hours, pressure loss, decrease in the flow rate (below 100 L/hour) and fouling of ceramic membranes. The pilot plant could not operate in continue, being the pretreatment the limitation stage. This caused continuous shutdowns in the pilot plant. Samplings (Action C1) were stopped from 11/03/2021 to 07/04/2021 until the plant was operating continuously. Measures taken consisted in the implementation of 3 types of chemical cleaning procedures in order to achieve longer operating cycles, turning out the most effective the combined cleaning. Additionally, standard cleaning times and product concentrations were increased to improve results. The pre-treatment was noticeable improved through changing the kind of cartridge filters, adjusting operation parameters, substituting and cleaning of ceramic membrane, and implementing a disc filter in the feed of the plant. With this modification, the pilot plant increased the operating hours, where 250 l/h was the maximum treatment flow and cartridge filters must be changed with a weekly average frequency. Weekly chemical cleanings and change of cartridges were stablished to get a good performance and alarms were included to support the monitoring and warn of falling flow.

## • Achieved results:

Regarding the objectives of the operational parameters, the physicochemical quality, the microbiological quality and the control of emerging contaminants related and detailed more explicitly in action C1 of Validation of the technology and feasibility at semi-industrial level, the results obtained are the following:

For operational control, we have worked with a peak flow of 0.25 m3/h, obtaining 61% renewable energy from the solar panels and a consumption rate of 1.7 kWh/m3.

Regarding physical-chemical control, we have been able to verify that the pilot plant reduces organic matter through its pretreatment stage.

For the microbiological parameters we can say that 100% is eliminated due to ultrafiltration.

Finally, for emerging contaminants we can confirm that, despite some families of pharmaceutical components that have seen their resistance increased (Azithromycin, Ciprofloxacin, Ofloxacin, Venlafaxine), the rest of the components are 100% eliminated.

## • Remaining results:

None.

## • Variations in action:

The pilot plant was designed for a self-sufficient consumption but has a battery system and a power grid connection for the most unfavorable moments. During the whole operation time of the pilot plant, the photovoltaic power supply was around 61%, while the grid power supply was 39%. The energy system was designed to work with a photocatalysis lamp. To improve the performance, a second ultraviolet lamp was added, so the pilot plant consumptions were increased. Even so, the photovoltaic was able to supply more than 60% of the energy. This allows us to observe that the plant is sustainable for a large part of its operating time, obtaining its energy from the network only when the battery system is discharged or the conditions are the least favorable, thus providing a sustainable solution to its energy consumption.

As for the continuous operating time of the pilot plant, a total of 12 months were expected. Despite the problems in obtaining the CD and in the pretreatment phase, the pilot plant was in operation for 11 months, plus 2 months of start-up.

The estimated flow treated per year is about 2.190 m3 and the estimated impact was 43.000 m3/year. Although the semi-industrial plant is designed such a flow, the swelling conditions of the polymer has not made possible to work with the design flow because the plant has worked countered flow and the speed has been lower.

## • Action evaluation:

The objective of this activity was to install the prototype according to the characteristics of the purification facilities and the quality of the purified water, and to fine-tune all the elements of which it is composed.

At the end of the action, HIDROGEA, coordinated with HIDROTEC, has made the necessary changes in the plant according to the results obtained from the control of operational parameters (water flow and energy consumption), the control of physical and chemical water quality, the control of microbiological quality and the control of EP, in order to maintain the purification efficiency of the whole system and to maintain a continuous operational flow. Although the time of operation of the plant has been one month less than expected, there were necessary two

months of in batch functioning to start up the plant. The main limitations of the plant to work in a continuous mode are known and they have been corrected, so the implementation of the action is considered successful and according to the objectives of the proposal.

TYPE	Cod	e Name	Deadline	Responsible	Status
DELIVERABLE	28	Installation and start up report	31/01/2019	HIDROGEA	OK Updated by 15/07/2021
MILESTONE	12	Installed prototype and working correctly	31/12/2018	HIDROTEC	OK Installed (29/03/2019) but without CDs Start up in January 2021

## 6.1.7. B3 Transferability, replication and sustainability

## ✓ Completed action

Foreseen start date: 01/01/2020 Actual start date: 01/05/2019

Foreseen end date: 30/06/2022 Actual end date: 30/06/2022

PLANNED		2019									2020													
TIMETABLE	Ε	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	J	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION B3																								
ACTUAL		2019										2020												
TIMETABLE	Ε	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	J	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION B3																								

PLANNED		2021										2022							
TIMETABLE	J	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	J	F	Μ	A	Μ	J	
ACTION B3																			
ACTUAL		2021											2022						
TIMETABLE	J	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	J	F	Μ	A	Μ	J	
ACTION B3																			

## • Participants and responsibilities:

REGENERA: Overall coordination of the activities carried out in the Action B3 and the preparation of deliverables. The task has been carried out also with the participation of UCAM, CTNC, CNR-IPCF, UNIBA, HIDROGEA e HIDROTEC, who has supported in the assessment of replication and transfer possibilities. CTC, CNR-IPCF and UNIBA has actively participated in the search and contact with potential replication and transfer entities.

## • What has been done:

The overall objective of action B3 is the development of the transfer and replication plan for the CLEAN UP treatment system. To achieve this objective, the following work has been carried out:

## **B3.1** Transfer and replication plan

This task started on 01/10/2020 and lasted until the very end of the project, consuming buffer time.

It has been very focused in the elaboration of studies to assess the possible replication and scaling up of the plant. One of them is the *Technical, economic and legislative report about renewable technologies implemented in wastewater treatment plants* (*D37. Informe técnico-económico-legislativo de las diferentes tecnologías de generación de energía renovable aplicables en EDARs: requisitos, capacidades, etc. para el futuro sistema comercial*). The purpose of the study was to present the different renewable technologies to obtain energy, which are currently applied in wastewater treatment plants (WWTPs) at three levels: i) technical, ii) economic, and iii) legislative. One of the most important costs in a WWTP is the energy used during the process. Renewable technologies allow generating energy to be used in the mentioned process without using fossil fuels and, at the same time, reducing its associated costs. However, not all these technologies are suitable for every WWTP since it depends on different factors such as location and wastewater volume to be treated. In this way, the aim of the work developed was to present the diverse available renewable technologies as a function of WWTPs requirements to explain the selected one, which is implemented in the LIFE Clean Up Project.

In order to carry out this report, CETENMA has collaborated as a subcontractor. Also, they have done the economic and financial analysis necessary to prepare the market launch of the proposed innovation (techno-economic study of the proposed solution).

Also, during this task REGENERA and the other partners defined the Replication and Transfer Plan and the stages of its market penetration. A complete study has been carried out to assess the feasibility of the Clean Up technology in terms of its implementation in other areas and sectors. For this purpose, the replication and transfer sites indicated in the project's application report were initially contacted. The Replication was carried out in the locations indicated; however, the Transfer sites were modified (Table ). In a second step, the proposed plants were visited (Citromil on 27 January 2022, Solvic, Vivolat and Rizzo Ecologica on 31 March 2022 and EMHASA on 12 April 2022), where information was collected on their processes, in order to compare them with those of LIFE Clean Up. Water analyses were also carried out to assess the existence of the pollutants studied in this project in the water.

Initial Replication sites proposed:	Replication sites studied.
<ul><li>Solvic WWTP (Italy)</li><li>EMAHSA WWTP (Spain).</li></ul>	<ul><li>Solvic WWTP (Italy)</li><li>EMAHSA WWTP (Spain).</li></ul>
Initial Transfer sites proposed:	Transfer sites studied.
- WWTP Comune di Gioia del Colle (Milk sector and Industrial sector, Italy)	<ul><li>Vivolat (Milk sector, Italy)</li><li>Andria (Milk sector, Italy)</li></ul>

Table 6. Replication and Transfer sites

- Destilerías Muñoz Gálvez (Agrofood	- Putignano (Milk sector, Italy)
sector, Spain),	- Citromil (Agrofood sector, Spain)
- Granarolo Group (Milk sector, Italy).	- Rizzi Ecológica Arccángelo (Industrial sector, Italy)

## **B3.2 Market launch**

This task went from 01/10/2020 to 31/06/2022. It complements the previous one by preparing the market launch of the future commercial product. For this purpose, the following studies have been carried out in order to assess the necessary resources and expected revenues for each market. (i) Market study and (ii) Business plan.

Both studies have included the definition of the market volume in different areas and in different pre-identified countries to start the market penetration. The specific market niche and other regulatory issues have also been defined to assess the viability of the business.

The communication strategy for each specific market niche has been defined in order to adapt and optimize the uptake of the business plan by identifying the adoption of the business plan, also identifying key players. In this task, the sales strategy, the market penetration strategy and the marketing plan have been defined.

On the other hand, the business plan also had to take into account conceptual and design deviations from what was initially planned:

- The CD polymer is not self-manufactured but purchased from a supplier.
- A change in the initial plan established in D50 Market penetration study, due to the limitation of the flow treated in the semi-industrial plant, so that in D41 Replication and Transfer, a stage 0 has been added, prior to the 4 stages described in D50.

In order to carry out this task, CETENMA has collaborated as a subcontractor, for elaborating the related deliverables (*D56. Business Plan* and *D50. Market Study*). It is important to note that all deliverables have been reviewed by Neemo's C2M Team.

Regarding the conclusions of the Business Plan and Market Study, it is interesting to mention the regulations related to wastewater reclamation. The *Regulation (EU) 2020/741 on minimum requirements for water reuse*, has been launched by the European Commission to promote the reuse of wastewaters in agriculture and ensure a safe use. It does not establish limits for EPs in treated water, but it establishes the surveillance of some EPs such as personal care products, pharmaceuticals, drugs, etc., in irrigation water, with a view to defining these limits in the near future. This suggest a stronger opportunity for the market launch of the Clean Up technology when the limits are finally set by the EC.

## **B3.3 Industrial scaling up assessment**

REGENERA developed the *Techno-economic study of the proposed solution and analysis of the industrial scale-up of the water treatment system* (D38. Informe de viabilidad técnico-

económico de una planta de tratamiento de aguas depuradas industrial basada en tecnologías de adsorción, fotocatálisis y pulso de luz). The aim of the action has been to describe the results of the technical-economic assessment of *Clean Up* solution. From a technical point of view, the viability of the *Clean Up* system is evaluated being applicated as an advanced treatment system for treated urban wastewater, for the elimination of pathogens and emerging pollutants (EPs), considering the established quality criteria by current regulations. In this sense, it is a technology that has successfully validated at an experimental level, and that offers similar removal performance as the most efficient alternatives available on the market.

- The technical-economic assessment of *Clean Up* solution has been based on the following steps:
  - Estimation of the treatment costs of *Clean Up* system, applied at industrial level. For said estimation, it has been considered both, data obtained in the experimental phase of the project carried out with pilot plant, and general information obtained from various technical bibliographic sources.
  - Estimation of the treatment costs of the most advantageous technological alternative to *Clean Up* system from the technical-economic point of view, applied on an industrial scale, and in the same scenario and conditions as those assumed for the *Clean Up* system. This alternative is based on an ozonation treatment followed by a biological filtration stage (slow sand filters with biofilm formation in upper layers). It has already been validated at technical and economical level and is being implemented on an industrial scale in several European countries, as a way to limit the presence of EPs in WWTP effluents. To carry out the estimation, information from industrial facilities that operate in the same application with the aforementioned technology has been taken.
  - Comparison of the treatment costs of both alternatives, applied in all cases under similar conditions, and assuming similar performance. Treatment costs are considered as the sum of operating and capital costs. By the other hand, for the proper comparison of the costs of both technologies, a similar EPs removal performance has been assumed in both cases. Specifically, yields of EPs removal higher than 80%, figures that have been experimentally validated in each case.

CETENMA contributed to the analysis of industrial scale-up of water treatment system.

## • Main problems encountered and solutions provided:

The problems faced in the implementation of the activities in task B3 are listed below:

- In order to carry out the technical and economic feasibility studies, the results of the laboratory-scale tests (25 cycles) have been used as the useful LIFE period, as explained in D38. It was not possible to determine the useful LIFE for the semi-industrial scale prototype, since after the completion of 7 LIFE cycles, the activity of the polymer was still intact.
- The replication and transfer site visits were delayed due to the COVID-19 pandemic and were finally carried out in March and April 2022.
- The aforementioned has delayed the submission of the following deliverables:
  - □ D38. Technical-economic feasibility report of an IWWTP based on adsorption, photocatalysis and light pulse technologies.
  - □ D41. Report on Replication and Transferability

- D56. Business Plan
  - Achieved results:

## Profitability assessment

Regarding the obtained results, although the treatment costs of the Clean Up system are competitive and are in line with those of most water reclamation treatments, they are higher than those of the proposed alternative. Specifically, they vary between 0.73 and 0.40  $\notin$ /m3 depending on the factors indicated in the previous paragraph, while in the case of ozonation + biological filtration they vary between 0.42 and 0.11  $\notin$ /m3.

Regarding the investment costs, they have been estimated lower in the case of the Clean Up alternative. Specifically, they could vary between 0.19 and 0.05  $\notin$ /m3, while in the ozonation + biological filtration case they could vary between 0.23 and 0.06  $\notin$ /m3, depending on the factors previously pointed out. By the other hand, the operation costs are higher in the case of Clean Up system (0.54 – 0.35  $\notin$ /m3) as compared with the raised alternative (0.19 – 0.04  $\notin$ /m3).

Focussing the analysis on the operational costs of the Clean Up system it is observed that the operational factors with the most influence are the electrical consumption of the photocatalysis stage, the chemicals used in the adsorption stage (cyclodextrins and regenerating solution), and the consumptions associated with micro + ultrafiltration stage. Considering that the proposed alternative has already been demonstrated and optimized, and is being implemented on a full scale, and that on the other hand, the Clean Up solution is of recent development, and therefore its operating costs could be optimized, a comparative study of costs between both technologies is then considered in which the variation in the costs of the Clean Up system is taken into account as a function of these commented factors (Figure ). This analysis has been carried out for each of the considered scenarios, in order to determine in each case, the conditions that should exist for the Clean Up solution to be economically competitive compared to the proposed alternative.

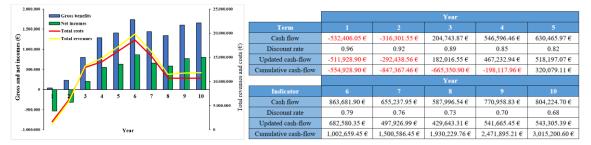


Figure 22. Summary of comparative study of cost between both technologies

## **Replication and Transfer**

In addition to the water treatment industry, the agri-food sector is identified as a potential target for the transfer of Clean Up technology, providing technical and economic factors that support said identification. The transferability objective would be the further treatment of these treated waters until reaching the quality level needed to allow its discharge into water bodies applying conventional technologies, and subsequent reclamation of the water obtained in that first stage through a tertiary treatment system, including Clean Up technology. The economic benefits that can be expected after the implementation of the new technology will come on the one hand, from not paying anymore the fee for discharging treated waters into the sewage network, in those plants that carry out this practice and, on the other, from the sale of reclaimed water. In addition, if said reclaimed water is reused in the company itself in those permitted uses, there will be an additional benefit due to the reduction in the consumption of tap water. All these incomes or savings must provide, compared to the costs of the process, a margin such that the necessary investment is profitable.

Although LIFE Clean Up technology can be applied due to the presence of pollutants, it is extremely important to carry out a personalised technical-economic feasibility study beforehand in order to evaluate the economic sustainability of the process (Figure ).

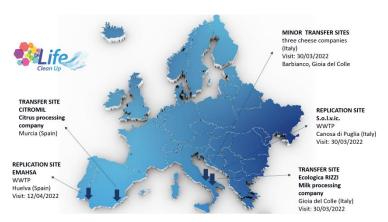


Figure 23. Replication and transfer sites of Clean Up technology

## **Business plan**

The LIFE Clean Up project would allow the creation of a new entity once its technology is validated. This entity would be formed by the private partners: Hidrotec, Hidrogea and Regenera, mandatory. The option is also left for research centres to be part of the company, if their executive body considers it appropriate. Therefore, the company would emerge as a spin-off of the LIFE Clean Up project under its own brand and properly registered, assuming the royalties and rights to third parties.

This new company would place the solution on the market under its own brand, properly registered, and would assume the royalties and other rights to third parties that it must face. The purpose of the company would be to develop a business line whose value proposition to the market is highly advanced products for the removal of pollutants of emerging concern, in different uses of water (industrial, urban, agricultural, reclaimed, drinking, etc.).

## **Market penetration**

The market penetration has been divided into four stages, according D50 Market study, However, in accordance with deliverable D41, Replication and Transfer, a stage 0 has been added due to the limited processing flow rate of the commercial CD used.

Stage 0 has been added due to the limited processing flow rate of the commercial CD used. The following is a summary of the main features of Stage 0 (which is detailed and justified in D42 Replication and Transferability):

Stage 0 consists of a continuation of the research and innovation activities in order to found a modification of the polymer or the adsorbent material that permit the treatment of major flows and enable this way the commercialization of the solution. At the same time, a first commercialisation phase in the industrial sector (agri-food, agricultural, pharmaceutical or sanitary) could be thought but the maximum flow rate of these plants should be similar to the pilot plant. In Stage 0 it is consider using the commercial polymer purchased in the pilot plant and the possibility to design the plant without light pulses to lower investment costs.

## Marketing plan

It has been considered a STP (segmentation, targeting and positioning) marketing model to develop the present business plan. The main targeted market is the companies (private or public) that exploit the WWTPs with a TT (both obtaining and not obtaining reclaimed water) and WWTPs without a TT but interested in expanding its facilities to reclaim water (segmentation and targeting). Whereas, on the other hand, LIFE Clean Up project provides an innovative solution, being a key aspect in the competitive positioning of the present technology.

## • Remaining results:

None

## • Variations in action:

The finalization of the deliverables has been delayed until the end of the project (June 2022) due to the following factors:

- Realization of the replication and transfer site visits in March and April 2022.
- Delay in the results of the water analyses carried out at the replication and transfer sites.
- Establishment of a collaboration plan with the CD polymer supplier.

It should be noted that deliverables D41, D42 and D43 have been unified into one deliverable, since it has been considered that the Replication and Transferability study is much more illustrative and understandable if they are elaborated together. The new deliverable is called D41 Report on Replication and Transferability. The modification of the transfer sites was done as described above and after consultation with the monitoring team. The proposal to merge deliverables D41, D42 and D43 was made during the final visit of the PO and the Monitoring Team on 6 June 2022.

## • Action evaluation:

As a general conclusion to the contents of the deliverables, a realistic approach is shown below, with the following steps to be taken to get the project solution closer to the market:

- 5 deliverables have been developed with the objective of assessing the feasibility of the technology that has been tested in terms of scaling and market launch.

- Fieldwork has been carried out contacting potential companies to be used as transfer sites. The flow rate treated was taken into account in this choice, as this was identified as a key point for the short-term market launch and the future scaling up of the LIFE Clean Up solution in the medium term.
- The technical-economic feasibility study has been carried out, establishing the main indicators of the project, with a favorable result for its market launch.
- It has been identified that the main limitation of the LIFE Clean Up technology at this stage (project completion) is the treated flow rate, which can be set at a maximum of 6 m3/day.
- In line with the previous point, two scenarios are set for the market launch of LIFE Clean Up. One in the short term (i.e., immediately after the end of the project) and the other in the medium term, since actions are needed to improve the product. The first corresponds to Stage 0 described above and the second to Stages 1-4.

#### Market launch with the current status of the LIFE Clean Up product:

- The target market is industrial plants with a maximum treatment flow of 6 m3/day, where LIFE Clean Up technology is known to work perfectly, as it replicates the characteristics of the pilot plant. For this purpose, and in accordance with the results of D41 Report on Replication and Transfer, commercial scenarios such as dairy or agrifood industries are proposed, as well as other industrial sectors such as pharmaceuticals or healthcare (always complying with the premise of the flow rate set as a limit).
- As mentioned above, the Business Plan contemplates the creation of a joint venture, however, the exploitation at Stage 0 would be carried out separately on demand and the synthesis of the polymer by the joint venture (through licensing of the existing patent) is not contemplated, but through direct purchase from the manufacturer. In this way, Regenera would be the company in charge of engineering and adapting the energy supply with the aim of increasing the sustainability and energy autonomy of the process. Hidrotec would be the supplier and installer of all the technology, and Hidrogea would be in charge of commissioning the plant.
- EXERCOM and the University of Turin (suppliers of the patent and the production process of the cyclodextrin polymer) has been contacted for commercial purposes. As mentioned above, direct purchase would be made at Stage 0, with the supplier guaranteeing a maximum quantity of 1000 kg/month of cyclodextrin, with a price negotiated in accordance with the quantity purchased annually, and more economical than the purchase price within the framework of the project.
- In this first phase of commercialization, the introduction of a pre-treatment before the water input to the plant and also the possibility of designing the plant without the light pulses is envisaged (Figure ).

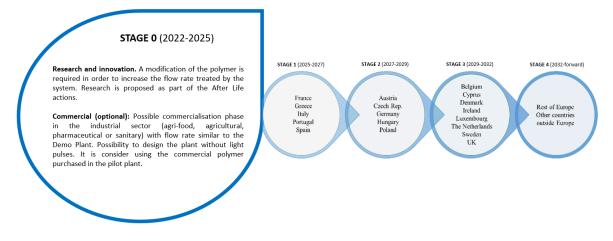


Figure 24. Different stages of commercialization

Technical requirements to scale up the LIFE Clean Up technology to Stage 1-4 for application in WWTPs.

- A modification of the polymer is required in order to increase the flow rate treated by the system. Research is proposed as part of Stage 0 and of the After-LIFE actions, consisting of the search and testing of more permeable cyclodextrins, the reduction of the holistic hydration capacity of the polymer developed or even the change in the morphology of the monomers, the latter can also reduce manufacturing costs.
- For the above, implementation and participation in new innovation projects by the LIFE Clean Up consortium and/or between the consortium and Prof. Trotta is envisaged.

For Stage 1-4, the establishment of the LIFE Clean Up Joint Venture for the commercialization of the developed technology is envisaged. A patent licensing agreement would be made with the Italian spin-off owning the patent to enable the joint venture to synthesize the polymer for specific applications to be detailed in the patent transfer agreement, including water treatment. If at any time during Stage 0 the demand of 12,000 kg/year is exceeded, the process of setting up the Joint Venture and patent licensing would also be initiated.

TYPE	Code	Name	Deadline	Responsible	Status
DELIVERABLE	37	Technical -economic- legislative report on the different renewable energy generation technologies applicable in WWTPs: requirements, capacities, etc. for the future trading system	31/12/2020	REGENERA	OK 31/12/2020
DELIVERABLE	38	Technical-economic feasibility report of an industrial treated water treatment plant based on adsorption, photocatalysis and light pulse technologies	31/12/2021	REGENERA	OK 30/06/2022

DELIVERABLE	41	Case study on technical feasibility in another industria sector	131/03/2022	REGENERA	OK 30/06/2022
DELIVERABLE	42	Case study on technical feasibility in WWTP Italy	31/03/2022	REGENERA	OK 30/06/2022
DELIVERABLE	43	Case study on technical feasibility in WWTP Huelva	31/03/2022	REGENERA	OK 30/06/2022
DELIVERABLE	56	Business plan	31/03/2022	REGENERA	OK 30/06/2022
DELIVERABLE	50	Market study	30/09/2020	REGENERA	OK 31/01/2021

# 6.1.8. C1 Validation of the technology and feasibility at semi-industrial level

## ✓ Completed action

Foreseen start date: 01/01/2019 Actual start date: 01/06/2020

Foreseen end date: 30/06/2022 Actual end date: 30/06/2022

PLANNED		2019										2020												
TIMETABLE	Ε	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	J	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION C1																								
ACTUAL		2019										2020												
TIMETABLE	Ε	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D	J	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION C1																								

PLANNED TIMETABLE ACTION C1		2021											2022						
	J	F	Μ	A	Μ	J	J	A	S	0	N	D	J	F	Μ	А	Μ	J	
ACTUAL		2021											2022						
TIMETABLE ACTION C1	J	F	Μ	Α	Μ	J	J	A	S	0	Ν	D	J	F	Μ	А	Μ	J	

## • Participants and responsibilities:

HIDROGEA is the coordinator of this action and has been in charge, together with HIDROTEC and REGENERA, of evaluating the performance and operation of the prototype on a semiindustrial scale. CTC has carried out analytical tests for the characterization of the treated water, evaluating the removal performance of emerging pollutants and the disinfection capacity of the system. UCAM, together with UNIBARI and CNR-IPCF, has evaluated the specific performance of the adsorbent material, and assessed the capacity of the light pulses.

## • What has been done:

Once the technology was optimised at laboratory scale (A2 and A3), and the prototype (B2) installed and commissioned, validation tests were carried out on a semi-industrial scale on the proposed technologies.

## Task C1.1 Assessment of effectiveness and performance of the proposed process

As explained in Action B2 and can be seen in Figure , the effluent of WWTP feeds the pilot plant, a first pre-treatment (1) removes the organic matter and the solids by mean of a microfiltration with cartridge filters and ultrafiltration ceramic membrane. Then, the wastewater passes through a column of CDs polymer (2) and the photocatalysis unit (3). In the PL System, the EPs concentrated in the stream after CDs regeneration are degraded (4).

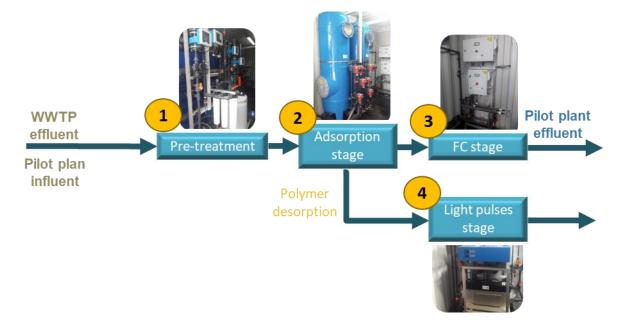


Figure 25. Emerging pollutants extraction process

Analytical controls have been performed in wastewater at the entrance and output of filtration pre-treatment (points 1 and 2), at the exit of the reactor with adsorbent material (point 3) and at the output of photocatalysis treatment (pilot plant effluent).

Similarly, to assess the capability of the light pulse system, the input and output solutions to the equipment have been analysed.

The validation study has been carried out for a full year in 2021, with a total of 36 sampling (each of them with 4 sampling points, which is equivalent to 144 samples), and the results have been evaluated for seasonal period.

The analytical parameters were pH, turbidity, conductivity (C), Suspended Solids (SS), Chemical Oxygen Demand (COD) Biological Oxygen Demand (BOD5), Trihalomethanes (THM), Escherichia coli, Clostridium perfringens and Clostridium perfringens spores. EC analysed were phytosanitary pollutants (more than 350 active substances) and drugs (32 analysed pollutants) selected in A1 task.

Physic-chemical parameters such as pH or conductivity show no significant variation of values throughout the process. In relation to the organic matter present in the inlet water to the 63

prototype, membrane filtration slightly reduces COD and suspended solids, thus improving the quality of the water for its subsequent passage through the adsorption tanks. Membrane filtration also significantly reduces the turbidity of the initial water.

The presence and behaviour of different families of emerging pollutants has been assessed. In the case of phytosanitary compounds detected in the influent sample, that corresponds to the effluent leaving the WWTP, all detected traces have been eliminated after passing through the CD stage, regardless of the family and the season, as shown in the following graph corresponding to the samples taken in the winter and summer period (Figure ).

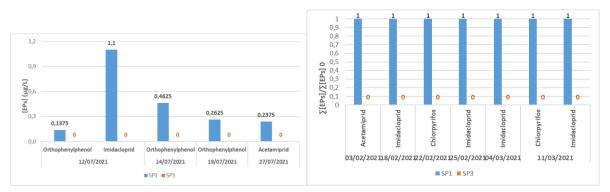


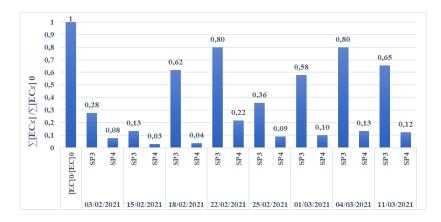
Figure 26. Graph corresponding to the samples taken in the winter and summer period.

Note: SP1 corresponds to the influent; SP3 corresponds to the outlet CD rector.

Regarding pharmaceutical compounds, some of them are recurrent in the influent (4-Aminoantipyrine, Carbamazepine, Ciprofloxacin, Diclofenac, Ketoprofen, Ofloxacin, Venlafaxine). The rest of the drugs are not detected or only appear in a specific sample.

Some compounds, like Ofloxacin or Venlafaxine, have obtained low removal efficiency in the pilot plant, with 32% and 18,5% respectively. The rest of pharmaceutical compounds detected have been successfully removed, with overall elimination rates of 92% for Diclofenac, or 87% for Ketoprofen, for example.

This dispersion in the elimination performance depending on the compound means that the average elimination obtained in the case of pharmaceutical compounds decreases in many cases due to those compounds that show high resistance, as shown in the following graphs corresponding to the elimination of drugs in the winter and summer periods (Figure ).



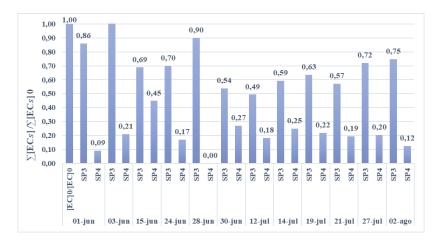


Figure 27. graphs corresponding to the elimination of drugs in the winter and summer periods

*Note*: The removal efficiency of drugs has been calculated as a global value and per substance. The overall EC key is a resistance ratio, which evaluates the amount of EC not removed in the different stages of the pilot plant, specifically in SP3 and SP4. SP1 corresponds to the influent; SP3 corresponds to the outlet CD rector; SP4 corresponds to the outlet prototype (after photocatalysis).

Considering the removal rates at the three most sampled stations:

- In winter, it has been obtained a global removal performance (from resistance ratio) of 47,3% after CD reactor and 90% after AOP to pharmaceutical compounds and a 100% after CD reactor to phytosanitary compounds.
- In spring, it has been obtained a global removal performance (from resistance ratio) of 30,6% after CD reactor and 70,6% after AOP to pharmaceutical compounds, being worse results than the first sampling. However, global removal performance to phytosanitary compounds is 100% after CD reactor.
- In summer, it has been obtained a global removal performance (from resistance ratio) of 29,7% after CD reactor and 73,5% after AOP to pharmaceutical compounds. The overall removal efficiency of the phytosanitary compounds is 100% after the CD reactor.

In terms of disinfection, microbiological parameters (*E. coli*, *C. perfringens* and *C. perfringens* spores) are removed in the pre-treatment step by the ceramic ultrafiltration membrane ( $0.4 \mu m$ ), and for the following sampling points the values are maintained at <10 cfu/100 mL. In any case and given that photocatalysis has a disinfection capacity (which was demonstrated at laboratory scale, A3), wastewater disinfection is enhanced by this last treatment step.

Further details on the results of the monitoring program can be found in the *Deliverable 45*. *Analysis of the level of compliance with the reference indicators.* 

## Assessment of the performance of the pulsed light system

A pulsed light system designed for solid sample treatment was adapted to treat wastewater continuously and fitted to the rest of the plant. The technical evaluation of the system allowed, in a first instance, to know that it was capable to degrade emerging pollutants. Its efficacy varied widely depending on the compound as it is typical of any of these processes, in which

pollutants have very different chemical structures and consequently, diverse photolysis susceptibilities.

The pulsed light system was tested on January 26 2022, once the pilot plant produced the raw material necessary for it. This means, the pulsed light system was used to degrade pollutants desorbed from the cyclodextrin polymer. As can be seen in the next figure (Figure ), which shows results of three tests, a global percentage of reduction of 34% was reached, which even though promising, was not considered fully satisfactory. Therefore, the possibility that increasing residence time would yield higher degradation percentages was considered. Given the fact that the pump that feeds the pulsed light system was already used at its lowest pumping rate, recycling the wastewater was chosen as a feasible option.

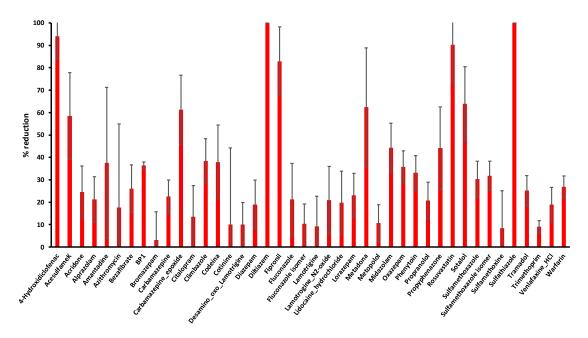


Figure 28. Overall result of the effectiveness of the light pulses in removing desorbed pollutants (n=3).

To this, tests were carried out in the pilot plant in May 2022 in three different weeks using ibuprofen as target compound. This pollutant was fed to the cyclodextrin polymer and once this column was regenerated, the water containing ibuprofen plus the desorbing agent (NaCl) was treated with pulsed light under recirculation. These tests were carried out by collecting outlet streams in a bucket and pumping them back to the reactor through an on-purpose adaptation; a real-LIFE scenario application would require a pump capable to work at lower flows or adapting a return pipe for recirculation. As it can be appreciated in the following figure

(Figure ), the implemented recirculation system indeed increased pollutant degradation, which was 19 % in the first cycle and 73 % after three cycles.

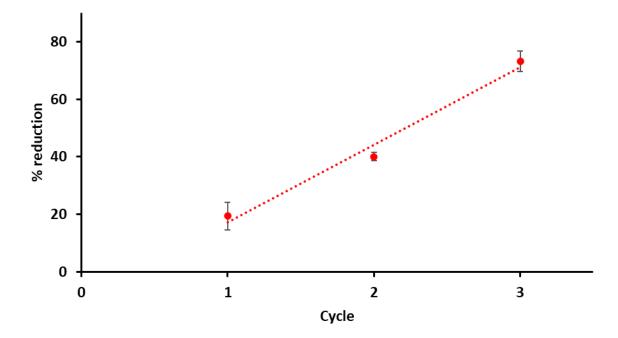


Figure 29. Pulsed light elimination percentage of ibuprofen in doped WWT water desorbed with NaCl from a cyclodextrin column.

Another possibility, alternative or complementary to that before mentioned, is the addition of hydrogen peroxide in order to create an advanced oxidation process, which effectivity was demonstrated in laboratory test; this would require the inclusion of a tank for this reagent and a dosing pump. While a 100 % degradation of all compounds would be ideal but hard to reach, higher degradation rates than those achieved seem feasible.

## Task C1.2 Evaluation of the quality of the material in relation to the time and number of cycles used

Firstly, the working capacity of adsorbent material was evaluated considering two working cycles (winter and spring). The pilot plant (Tank 1) was launched in late 2020 when 75 kg of CD-BDE polymer was introduced in the reactor (to ensure at least 10 min of contact time) and has been operated until the end of the project. The first working period (February to March 2021) required short cycle operation and low water flows, since cartridge filters were saturated in 4 hours, and a fast fouling of the ceramic membrane was observed. This drawback was consequence of particle distribution of polymer particles (24% smaller than 200µm, an initial requirement of the pilot plant). Some modification was carried out to improve the pre-treatment stage, also implementing a disc filter in the feed on the plant, allowing to increase the operation hours, flow (up to 250 L/h) and one-week replacement of cartridges frequency. For this period, the pilot plant worked in continuous flow for 431 hours (18 days), where 114 L/h (46.13 m3) were treated, removing 100% of pesticide residues and 47.3% of pharmaceutical compounds (73.7% of total EPs).

In the spring period (April-May 2021) the pilot plant worked in continuous flow for 638 hours (27 days), where 156 L/h (84.15 m3) were treated, removing 100% of pesticide residues and 30.6% of pharmaceutical compounds (65.3% of total EPs).

Since in spring the pilot plant has had a workload of 33% more than in winter, EPs removal efficiency significantly decreased (73.7% in winter vs. 65.3% in spring), changing Tank 1 by Tank 2 (filled with 30 kg of CD-BDE polymer fulfilling 200µm particle size criterion) for the third sampling period (June to 2nd of August), working 474 hours (20 days), were 146.5 L/h, (63.61 m3) were treated, removing 100% of pesticide residues and 29.7% of pharmaceutical compounds (64.9% of total EPs), whereas in the last working period (Autumn 2021) Tank 2. Worked 844 hours (35 days), were 107.9 L/h, (137.53 m3) were treated, removing 100% of pesticide residues and 38% of pharmaceutical compounds (69% of total EPs).

In summary:

- The adsorbent material reduces the pollutant load of the sewage treatment plant effluent by more than 65%.
- CD reactor 1 worked during 45 days in two periods (winter and spring), cleaning 130.28 m3 of water.
- CD reactor 2 worked during 55 days in two periods (summer and autumn), cleaning 201.14 m3 of water.

In addition, the polymer reusability (seven cycles) was also evaluated using the Tank 1. For that, a 600 L tank was filled with tap water, adding NaCl to obtain a 0.1 M solution (scheme followed first four cycles, *Figure*). After that, the saline solution was introduced in the CD tank, recirculating with a desorption flow of 1,200 L/h from the outlet of the tank, up to the point of entry, for 30 min. From this moment (initial time), the samples were taken in triplicate (at selected times upon test), and the concentration of EPs in water samples were determined by Chromatography.

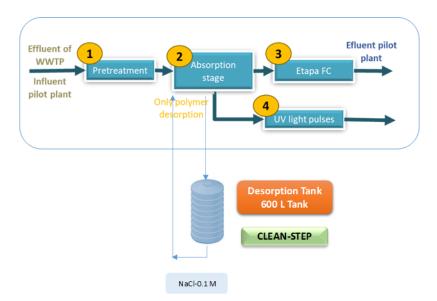


Figure 30. Assay design and sample points for reusability tests

The first desorption assay was carried out in July 2021. In this assay (see Figure 27), samples of cleaning solution were taken at the adsorption tank exit every 15 min up to 105 min total assay time. All EPs determined (4-aminoantipirin, cyprofloxacin, diclofenac, ketoprofen, ofloxacin, sulfamethoxazole and venlafaxine), showed a maximum desorption concentration at 15 min, remaining constant (asymptotic behaviour), during all the times set. Since these results recommend using a polymer cleaning period of no more than 20 minutes, the second,

third and four desorption assays, carried out January 2022, samples were taken at the adsorption tank exit at 20 min (see Figure 34). From a total of 62 EPs detected in the outlet of the rejection tank, 43 of them were quantified by chromatography (inside the linear range protocol assay), from  $6.12 \,\mu$ g/L to  $6.96 \,\mu$ g/L, being, therefore, a partial indicator of the capacity of the polymer to remove EPs.

Three additional polymer desorption tests were carried out in May 2022 to evaluate the LIFE time of the adsorbent material, doping the effluent with a known quantity of ibuprofen, as EP model, not previously detected in the effluents of our WWTP. For that, the same procedure described above for the previous desorption tests was applied. As can be seen in the Figure , the sewage effluent was doped with 2 mg/L of ibuprofen (EP) in the 1,000 L tank, and was subjected to a retention cycle in the cyclodextrin tank, followed by a desorption process.

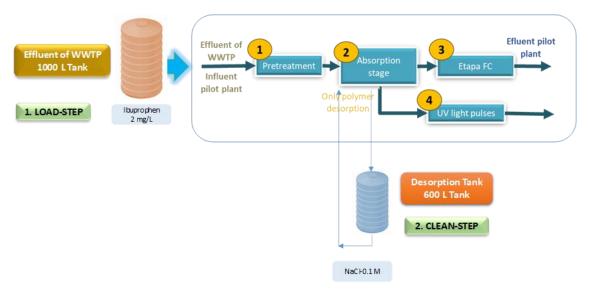


Figure 31. Assay design and sample points for reusability tests with ibuprofen

Ibuprofen was quantified at points 1 and 2, however, at point 3, at the outlet of the polymer tank, it was not detected in any of the samples analysed (0–20 minutes). The levels of ibuprofen quantified were  $1.81 \pm 0.07$ ,  $1.91 \pm 0.03$  and  $1.94 \pm 0.02$  mg/L (test 1, test 2 and test 3, respectively), ensuring complete clean-up of the polymer, since the fortification level was 2 mg/L.

In summary:

- The polymer cleaning step was stablished in 20 min (including 30 min previous conditioning step for 30 min).
- After cleaning step, the polymer adsorption capacity is fully re-established, based on test carried out with EP model ibuprofen.
- The polymer efficacy it is conditioned by the physicochemical characteristics of the inlet water, especially in relation to suspended solids and organic matter, which influences the workflow and cleaning stages.
- Due the non-homogeneity in the physic-chemical parameters of water influent to the semi-industrial prototype, a regeneration step is almost recommended each 200 m3 of water cleaned, since the adsorbent material is fully regenerated without losses in adsorption efficacy.

The tests carried out, taking into account the type of water treated, have not made it
possible to determine the useful LIFE of the adsorbent material used, since it has not
been possible to saturate it, which reveals its great robustness.

#### Task C1.3 Socio-economic assessment

The assessment of the socio-economic impact of the project has been subcontracted by CTC. Results are integrated in the *Deliverable 46*. *Study of socioeconomic benefits and LCA of the developed technology*, together with the results of the subtask C1.4.

The socioeconomic study can be addressed from two different aspects:

- 1. Social and technical-scientific: The LIFE Clean Up project provides an innovative and non-existent technology nowadays, being utterly required to remove the EPs from the wastewater. This removal lets obtaining reclaimed water of high quality and, thus, benefiting both the environment and human being health.
- 2. Social and economic: The developed technology can be used in other business sectors, which can improve its spread across the world. In addition, this fact would cause a larger internationalization of the involved entities: new markets and consolidating those markets where they are present.

The main goal of the Clean Up technology is to remove the EPs to release the wastewater into the water bodies or to reclaim it. Therefore, the WWTPs are the main beneficiaries of the Clean Up technology, together with the new WWTPs to be built. The solution can directly be sold to the private companies, transferred, or implemented by a consortium in order to gain the public contract, beside the public entities which are completely in charge of the urban wastewater treatment service. However, for urban wastewater treatment there is currently a constraint that may delay or hinder its market options: treatment capacity in terms of volume.

This is why thought has been given to other target sectors where the technology can be transferred or replicated. Pharmaceuticals are one of the most important compounds of the EPs. For this reason, the pharmaceutical sector has been identified as the ideal one to be reached by Clean Up technology. Similarly, this technology would be appropriate in specific urban sectors such as hospitals or nursing homes, whose wastewater contain a high concentration of pharmaceutical compounds and/or their metabolites, which to date are usually discharged into the network.

As in the case of pharmaceuticals, any other chemical sector (dyeing companies, soaps, detergents or other cleaning products sector, pesticides, fertilizers and other agrochemicals sector, etc.) can be a potential customer for Clean Up technology, as they generate waste effluents with specific emerging compounds of concern. In any case, the proposed technology composed of different treatment modules (pre-treatment, adsorption phase, oxidation phase) allows the adaptation of the treatment according to the characteristics of the wastewater generated, which makes it a very versatile and functional technology.

#### Task C1.4 LIFE Cycle Assessment

The objective of the LCA study is to determine the environmental impact linked to the LIFE Clean Up technology. For this study, HIDROGEA subcontracted externally the LCA, which

was drafted between October 2021 and June 2022. The LCA is included in the *Deliverable 46*. *Study of socioeconomic benefits and LCA of the developed technology*.

Since the LIFE Clean Up solution is considered as an extension of the current wastewater treatment plant (WWTP), the LCA was performed after the secondary treatment, including the disinfection process.

There are two technologies widely spread to remove the EPs: ozonation and activated carbon. Therefore, the *Clean Up* solution is compared to these ones. The assessment was performed under the ISO 14040 and 14044 standards (v.2016). The evaluation of the environmental impacts was achieved using the GaBi software (v.9.5). The three wastewater treatment scenarios were presented in schematic diagrams. The  $\beta$ -CD manufacturing and the wastewater treatment were also considered.

The construction, maintenance and dismantling of facilities at the end of the LIFE has been observed to have a negligible impact compared to the overall system, so these processes are not included within the system boundaries. The functional unit was set as one cubic meter of secondary-treated wastewater, including the substances in the wastewater (Figure ).

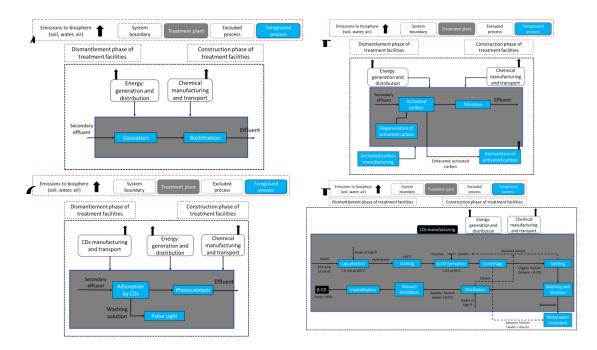


Figure 32. Schematic diagrams of the three wastewater treatment scenarios. Ozonation (A), granulated activated carbon (B) and Clean Up system (C). Additionally, the CDs manufacturing process (D) is shown.

The three technologies have been compared for some impact categories: i) climate change, ii) freshwater eutrophication, iii) human toxicity (carcinogenic and non-carcinogenic effects), iv) acidification, v) ecotoxicity and vi) water scarcity. These impact categories have been selected because they represent water quality, global emissions and human health issues, which are the most frequently included in wastewater related LCA studies.

The Clean Up solution presented the highest value within the climate change impact category: 0.9 kg CO2-eq. This value was 9 and about 3.5 times higher compared to ozonation and activated carbon, respectively. The impact on freshwater eutrophication is much higher for Clean Up technology compared to the other alternatives. This is due to the use of starch to 71

produce  $\beta$ -CDs, whose process (included in GaBi) takes into account the stages: wheat cultivation, harvesting, milling, kneading and cyclone separation. The environmental impacts within the category of human toxicity (cancer) are similar in the cases of activated carbon and Clean Up technologies. However, both technologies present a value between 7 and 12 times higher than that of ozonation. In the human toxicity category (non-carcinogenic), the environmental impact of Clean Up was clearly superior to that of ozonation and activated carbon. In the case of acidification, both Clean Up and activated carbon had a similar environmental impact; ozonation, on the other hand, was much lower compared to them. The Clean Up solution also presented the highest values of environmental loads for both impact categories. The following figure summarizes the impacts of each compared technology (Table ).

					Impact category			
		Climate change (Kg CO2eq)	Freshwater eutrophication (Kg P eq)	Human toxicity (cancer, CTUh)	Human toxicity (non-cancer, CTUh)	Acidification (Mol H <sup>+</sup> eq)	Ecotoxicity (CTUe)	Water scarcity (m <sup>3</sup> eq)
3y	Ozonation	0.10	2.20x10 <sup>-7</sup>	7.30x10 <sup>-11</sup>	3.22x10 <sup>-9</sup>	1.69x10 <sup>-4</sup>	3.70x10 <sup>-3</sup>	1.30x10 <sup>-2</sup>
Technology	Activated carbon	0.24	4.06x10 <sup>-7</sup>	5.10x10 <sup>-10</sup>	1.25x10 <sup>-8</sup>	1.72x10 <sup>-3</sup>	2.26x10 <sup>-2</sup>	1.50x10 <sup>-2</sup>
Ĥ	Clean up	0.90	1.88x10 <sup>-5</sup>	8.87x10 <sup>-10</sup>	4.36x10 <sup>-8</sup>	2.14.x10 <sup>-3</sup>	1.84x10 <sup>-1</sup>	2.63 x10 <sup>-1</sup>

Table 7. Summary of the considered impact categories and values for each technology

## • Main problems encountered and solutions provided:

The main problems encountered in this action lie in the difficulty of response when there are episodes that worsen the quality of the water entering the prototype (treated wastewater leaving the WWTP), for example during episodes of rainfall. This poor water quality leads to rapid saturation of the filtration membranes corresponding to the pre-treatment stage, which means that poor quality water reaches the CD tank, saturating the polymer.

This problem has to be solved at the outlet of the WWTP, and before going on to the treatment in the prototype.

In relation to the treatment capacity of the technology, which is directly related to its market capacity, the main problem identified is that the currently proposed system does not have the capacity to treat large volumes of wastewater, and, in any case, the cost of treatment would be very high.

However, the system is suitable for water treatment in specific sectors, such as pharmaceutical companies, where the volume of water to be treated is smaller. Moreover, as it is a modular system resulting from the combination of different technologies, it can be adapted to the casuistry, which makes it a versatile and functional system.

## • Achieved results:

The results obtained in this task have made it possible to determine the technical feasibility of the proposed solution for its commercialisation and implementation.

The system has an adsorption capacity of around 45% for pharmaceutical products and 100% for phytosanitary products. The pollutant destruction phase by photocatalysis improves the overall removal performance of pharmaceuticals to 80%. At this point we must consider that the performance calculations have been calculated considering some of the most persistent and resistant compounds to treatment technologies cited by the scientific community, such as Venlafaxine or Carbamazepine.

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 200 m3 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.

#### • Remaining results:

None.

#### • Variations in action:

The action was carried out without variations in relation to the monitoring activities proposed.

#### • Action evaluation:

The work done has permitted the assessment of the adsorption capacity of the separated units and the overall system. Also, the LIFE span and regeneration capacity of the polymer was evaluated. The results obtained lead to conclusions about the possibilities of scaling up and transferring the technology that are analyzed in Action B3.

ТҮРЕ	Code	Name	New deadline	Responsible	Status
DELIVERABLE	24	Analysis of the level of compliance with the reference indicators	30/06/2021	HIDROGEA	Included in D45
DELIVERABLE	39	Analysis of the level of compliance with the reference indicators	30/11/2021	HIDROGEA	Included in D45

DELIVERABLE	45	Analysis of the level of compliance with the reference indicators	31/03/2022	HIDROGEA	OK 30/06/2022
DELIVERABLE	46	Study of socioeconomic benefits and LCA of the developed technology	31/03/2022	HIDROGEA	OK 30/06/2022
DELIVERABLE	47	Report on the loss of capacity of different adsorbent materials and the factors that influence it. The useful LIFE of the reactors or filters	31/03/2022	UCAM, UNI BARI, CNR-IPCF	OK 06/06/2022
DELIVERABLE	48	Report of results and validation of the proposed system for the treatment and elimination of polluting organic compounds emerging from treated water	31/03/2022	HIDROGEA	OK 30/06/2022
MILESTONE	15	Beginning of the analytics for the validation of the proposed treatment system	30/09/2020	HIDROGEA	ОК

### 6.1.9. D1 Public awareness and dissemination of results

### ✓ Completed action

Foreseen start date: 01/10/2017 Actual start date: 01/10/2017

Foreseen end date: 30/06/2022 Actual end date: 30/6/2022

PLANNED		20	17	2018	2019	
TIMETABLE	S	0	Ν	D	2010	2019
ACTION D1						
	2017					
ACTUAL		20	17		2018	2010
ACTUAL TIMETABLE	S	20 0	17 N	D	2018	2019

PLANNED		2020							2021	2022									
TIMETABLE	J	F	Μ	A	M	J	J	A	S	0	N	D	2021	J	F	M	A	M	J
ACTION D2																			
ACTUAL						20	20						2021			20	22		
TIMETABLE	J	F	Μ	A	Μ	J	J	A	S	0	N	D	2021	J	F	Μ	A	Μ	J
ACTION D2																			

• Participants and responsibilities:

UCAM has been responsible for coordinating the communication actions and the public dissemination of the project's results. The rest of the partners have contributed to the development of contents and the distribution of the materials produced.

### • What has been done:

The activities included in the proposal and the dissemination plan were carried out between October 2017 and June 2022. The communication materials and tools used to achieve this objective are summarized below.

#### Task D1.1. Communication Plan

In October 2017, UCAM developed the logo and visual materials of the project and templates and scheduled the communication activities of the project, which included:

- Dissemination and training activities.
- The identification and evaluation of the external diffusion activities: fairs, conferences, etc.
- A database of interested agents with contact details.

The tasks related to information management were established, asking all technical and scientific partners to report to UCAM communication team the progresses of the project so they could be posted in the webpage of the project and in the social networks.

#### Task D1.2. Development of the diffusion pack

As part of this task, the project's website (5,175 unique visits) and the profiles on social networks (Facebook and Twitter – 41 and 403 followers) were also created, in January 2018 and October 2017 respectively.

LIFE Clean UP has been featured in 44 articles (both print and online), a documentary on regional TV, and 3 radio interviews. The media monitoring can be seen in Table .

Media	N° of project publications	Area	Scope
7RM	1	Regional	41000
AgriTech Murcia	1	National	No data
Aguas residuales	3	National	600000
COPE	2	Regional	312000
El economista	2	National	26000000
Europa Press	3	National	41700000
Hidrogea	1	National	21400
iAgua	3	National	1200000
La Opinion	3	Regional	67623
La Vanguardia	1	National	22730000
La Verdad	1	Regional	79000

Murcia.com	5	Regional	880000
Nova Cienca	6	National	192000
Popular TV	10	Regional	183230
Retema	2	National	38000
RTVE	1	National	1147000

In addition, the Layman report has been prepared, information brochures on the project (one initial in January 2018 and one final in March 2022) have been distributed among researchers and general public, a board information has been placed in the HIDROGEA WWTP, next to the prototype, and a dissemination video has been made (1070 total views, 327 on YouTube, 743 on Twitter). Other materials have also been produced that have helped spread the project such as t-shirts, stickers or a roll up. Table shows the interaction with these tools.

Type of material	Partner creator of the material	Number of copies edited	N° of copies distributed	Material receiving public	Interaction s /Visits
Website	UCAM	-	-	General public	
Twitter	UCAM	-	-	General public	330 Tweets 403 Followers
Facebook	UCAM	-	-	General public	41 Followers
Brochures	UCAM	2	200	Researchers and general public	-
Information panels	UCAM	2	2	General public	-
Videos	UCAM	1	1	General public	Total views:
Video news	UCAM	3	3	General public	1070
Newsletter	UCAM	4	-	General public	Total views: 156
Layman report	UCAM	1		General public	
T-shirts	UCAM	1	750	General public	-
Informative panel	UCAM	1	1	Researchers and general public	-
Stickers	UCAM	1	210	General public	-
Roll Up	UCAM	1	2	General public	-
Canvas	UCAM	1	1	General public	-

Table 9. Summary of material used for diffusion the project

#### Task D1.3. Organization of communication events

Two info-days have been organized, one at the beginning of the project and another at the end. Guided tours of the demonstration plants, three of them aimed at journalists and others two at students, have also been carried out to raise awareness of the environmental problems caused by emerging pollutants. Table shows a summary of all these events

Events organized									
Date	Responsible	Event	Impact/Attendants						
09/10/2017	UCAM	Launch info-days	20						
31/03/2022	UCAM	Final Info-day	11						
19/07/2018	UCAM	Guided tour with students	14						
31/12/2018	UCAM	Guided media visit at WWTP Cabezo Beaza	41,000*						
02/06/2022	$I = I = \Delta \Lambda I$	Guided media visit with journalist at WWTP Cabezo Beaza	1,165,323*						
27/05/2022	UCAM	Guided tour with students	8						

Table 10. Summary of the events attended

\*Average audience on the broadcasting day

In addition, other event not included in the proposal have been organizes, the Table contains them.

Table 11. Summary of	of events n	not included in	the proposal
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EVENT	DATE	WHERE	ORGANIZING PARTNER	IMPACT
"La Luciérnaga Fundida" competition	09/06/2018	Murcia	UCAM	200
Science week	26/10/2018	Murcia	UCAM	25000
'LIFE Clean Up' short film competition	01/04/2019	Online	UCAM	30
5th Research and Doctoral Conference "Ciencia sin fronteras"	04/06/2019	Murcia	UCAM	40
Science week	08/11/2019	Murcia	UCAM	25000

#### • Main problems encountered and solutions provided:

The main problem we had was the appearance of COVID 19. This milestone delayed all actions and had a direct impact on the dissemination actions. We changed the deadlines of the projects and had to freeze the newsletter until the final results of the different deliverables were achieved. In addition, the scout visits had to be changed to other audiences due to the restrictions caused by COVID19. Despite this, we continued to disseminate the project activities, mainly with online actions and we made some visits to small groups to compensate for the scouts' visit.

• Achieved results:

During these 5 years, we developed every action included in the proposal and the dissemination plan that we carried out. In this time LIFE Clean Up has appeared in 54 articles (both print and online), 1 documentary in the regional Tv, 3 Radio's interviews, 1 contest on a short movie festival, 1 contest of spots to raise awareness among the general public, 2 science weeks (a regional event which more than 25.000 participants, focusing on science), 3 guided media visits at WWTP Cabezo Beaza, 2 visits from students with interest common with the wastewater and the recycling of it. These activities sum with the 14 networking events achieved and the different congresses where our project members participated. Project 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 pageviews of our website ascend to 171.070 interactions.

- Awareness raising has been developed among all project partners through the above mentioned activities (Events, press releases, brochures, video, Info-days, newsletters, radio interviews, TV interviews and visits) This action was led by UCAM with the collaboration of all partners.
- Media and website (Twitter, Facebook, YouTube, https://www.LIFEcleanup.eu), the latter realized and updated thanks to UCAM. This action was led by UCAM with the collaboration of all partners.
- Behavioral change has been developed by the partner Hidrogea through surveys. This action was led by Hidrogea with the support of the research partners.
- Different networking have been carried out between the different partners, mainly UCAM and CTC have been the attendants to these events. This action was led by UCAM and CTC with the collaboration of all partners.

Table shows the achieved results.

			E	xpected results	Final results
		Number of individuals reached in public activities		15,066	17,966
Communicatio n, dissemination, awareness	Awareness raising	Number of individuals reached by media	107,000	8,000 UCAM Press release 28,000 Other NPs Presse release 25,000 Twitter 29,000 Facebook	171.070
rising		Number of entities/made aware	2,000 entities received project information		4,000 entities receive project information
	Website	Number of visits		2,500	5,175
	Behavioural change	Number of entities/individuals changing behaviour		15	20

#### Table 12. Summary of archived results

### • Remaining results:

All actions have been carried out.

#### • Variations in action:

None

#### • Action evaluation:

Two visits were made to Cabezo Beaza WWTP, instead of 10 foreseen. This has been the only activity that has not met the targeted number. Unfortunately, due to the COVID 19 pandemic situation we were not able to plan as many visits as we would have liked. Despite this situation, it is considered that communication activities have reached a great audience.

ТҮРЕ	Code	Name	Deadline	Responsible	Status
MILESTONE	3	Launch info-days	31/12/2017	UCAM	OK 09/10/2017
MILESTONE	4	Creation of profiles in social networks	31/01/2018	UCAM	OK October 2017
MILESTONE	7	Launch of the first newsletter	30/03/2018	UCAM	OK April 2018
MILESTONE	8	First radio interview	30/03/2018	UCAM	OK 12/05/2020
DELIVERABLE	2	Communication plan	31/12/2017	UCAM	OK November 2017
DELIVERABLE	4	First article in the media	31/12/2017	UCAM	OK 09/10/2017
DELIVERABLE	5	Project website	31/01/2018	UCAM	OK January 2018
DELIVERABLE	10	Initial brochure (500)	31/01/2018	UCAM	OK November 2017
DELIVERABLE	25	Second article in the media	31/12/2018	UCAM	OK March 2018
DELIVERABLE	27	Information board	31/12/2018	UCAM	OK October 2020
DELIVERABLE	32	Project video	30/06/2019	UCAM	OK July 2019
DELIVERABLE	36	Third article in the media	31/12/2019	UCAM	OK May 2019
DELIVERABLE	33	Final brochure (500)	31/12/2021	UCAM	OK March 2022
DELIVERABLE	49	Layman report	31/03/2022	UCAM	OK 30/06/2022
DELIVERABLE	53	Fourth article in the media	31/03/2022	UCAM	OK May 2021
MILESTONE	13	First guided tour with journalists	31/12/2018	UCAM	OK July 2018
MILESTONE	14	Start of visits to the HIDROGEA WWTP demonstration plant (20)	31/12/2020	UCAM, HIDROGEA	OK May 2022
MILESTONE	17	Second guided tour with journalists	28/02/2022	UCAM	OK June 2022
MILESTONE	22	Final Info-days	31/03/2022	UCAM	OK June 2022

# 6.1.10. D2 Technical dissemination of the project

### ✓ Completed action

Foreseen start date: 01/06/2019 Actual start date: 01/11/2017

Foreseen end date: 30/06/2022 Actual end date: 30/06/2022

PLANNED	2017			2018	2019												
TIMETABLE	S	0	N	D	2010	E	F	Μ	A	Μ	J	J	A	S	0	N	D
ACTION D2																	
ACTUAL	2017			2018						20	19						
TIMETABLE	S	0	Ν	D	2018	E	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION D2																	

PLANNED		2020								2021	2022								
TIMETABLE	J	F	М	A	Μ	J	J	A	S	0	Ν	D	2021	J	F	Μ	A	Μ	J
ACTION D2																			
ACTUAL		2020							2021	2022									
TIMETABLE	J	F	Μ	A	Μ	J	J	A	S	0	Ν	D	2021		F	Μ	A	Μ	J
ACTION D2																			

### • Participants and responsibilities:

CTC has been responsible for this action and has counted with the participation of the rest of the partners, attending national and international conferences and technical events in general for the communication and transfer of the project results.

### • What has been done:

Communication activities aimed at a technical audience were carried out to raise awareness of the innovative advantages of the *Clean Up* treatment system.

The target audience for these activities was:

- Integral water cycle management companies
- Companies dedicated to wastewater treatment and engineering companies dedicated to providing technical solutions for such treatment.
- Local administrations responsible for water management.
- Scientific sector dedicated to research and application of new technologies for water treatment.

Various types of activities have been carried out, which can be summarized as follows:

### Task D2.1. Development of technical & scientific publications:

80

A technical guide on the implementation of Clean Up treatment solutions has been developed (D40).

Several articles (10) have been published in technical journals: CTC Alimentación (D26 & D34), Retema (D51), Newsletter of ISEKI-Food Association (IFA) and FuturENVIRO (D52)

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".

Advertisements (4) have also been placed in various magazines and newspapers.

All the articles, magazines and newspapers in which the project has achieved, can be consulted in the Table .

	Articles, magazines and newspapers							
Date	Responsible	Name	Impact/distribution					
02/2018	CTNC	<b>RETEMA</b> (D51) <u>https://www.LIFEcleanup.eu/downloadable-</u> <u>descargables</u> <u>https://www.retema.es/revistas/enero-febrero-xwyTT</u>	18,000 readers from Spain and Latin America					
06/05/2018	CTNC	'La Verdad' journal	No data					
01/2019 01/2020 07/2020 01/2021 01/2022 07/2022	CTNC	CTC Alimentación 2 Articles (D26 & D34) 4 Advertisement https://www.LIFEcleanup.eu/downloadable- descargables	700 subscribers + 600 download					
03/2019 12/2019 06/2022	CTNC	Newsletter of <b>ISEKI-Food Association</b> (IFA) 3 Articles	More than 10.000 subscribers					
07/2019	UNIBA, CNR, UCAM	Science of the Total Environment https://doi.org/10.1016/j.scitotenv.2019.133620	IF 7.963					
08/2019	UNIBA, CNR, UCAM	Environmental Technology & Innovation https://doi.org/10.1016/j.eti.2019.100454	IF 5.263					
10/2019	UNIBA, CNR	Biomolecules http://doi.org/10.3390/biom9100571	IF 4.694					
11/2019	UNIBA, CNR	Materials http://doi.org/10.3390/ma12233810	IF 3.623					
04/2020	UNIBA, CNR	Environmental Technology & Innovation https://doi.org/10.1016/j.eti.2020.100812	IF 5.263					
05/2020	UNIBA, CNR	Colloids and Surfaces A	IF 4.539					

Table 13. Summary of articles, magazines and newspapers

		https://doi.org/10.1016/j.colsurfa.2020.125060	
05/2020	UCAM, UNIBA, CNR, CTNC	Journal of Hazardous Materials (D35) https://doi.org/10.1016/j.jhazmat.2020.123504	IF 10.588
11/2020	UNIBA, CNR, UCAM	Journal of Environmental Science and Health, Part A https://doi.org/10.1080/10934529.2020.1853985	IF 2.32
01/2021	CNR, UNIBA	Chemical Engineering Journal https://doi.org/10.1016/j.cej.2021.128514	IF 13.273
05/2021 04/2022	CTNC	Newspaper <b>murcia.com</b> (2) <u>https://www.murcia.com/region/noticias/2021/05/03-el-proyecto-LIFE-clean-up-concluye-a-escala-piloto-que-la-eliminacion-de-contaminantes-emergentes-escasp</u>	Región of Murcia
05/2021	CTNC	FuturENVIRO Magazine (D52) https://futurenviro.es/digital-versions/2021- 04/42/index.html	7,000 copies of the printed version. Digital version distribution to >100,000 e-mail addresses worldwide
05/2021	CTNC	Book of Abstracts <b>International Symposium</b> on Food Technologies https://prod5.assets-cdn.io/event/5847/assets/8378586182- 27cda9d408.pdf	590 people + online
11/2021	UCAM, UNIBA, CNR	Chemosphere https://doi.org/10.1016/j.chemosphere.2021.131238	IF 7.086
01/2022	UCAM, UNIBA, CNR	Springer Link - <b>International Journal of</b> <b>Environmental Science and Technology</b> <u>https://link.springer.com/article/10.1007/s13762-</u> 021-03895-x	IF 2.86
03/2022	CTNC	Technical Guide (D40) https://www.LIFEcleanup.eu/downloadable- descargables	300 copies + online
04/2022	CTNC	Digital newspaper <b>murciaempresarial</b> <u>https://murciaempresarial.com/el-proyecto-LIFE-</u> <u>clean-up-concluye-a-escala-industrial-que-la-</u> <u>eliminacion-de-contaminantes-emergentes-esta-por-</u> <u>encima-del-90-asegurando-la-calidad-</u> <u>microbiologica-de-las-aguas/</u>	Region of Murcia

#### Task D2.2. Networking with other projects:

Information has been shared with other projects, exploring future options for cooperation and synergies in relation to wastewater treatment and the removal of pollutants of emerging concern.

Networking actions (Milestone 16) have been developed at two levels: online networking actions and face-to-face networking actions.

22 different European projects related to wastewater treatment have been contacted, 16 of them from the LIFE call.

Table shows a summary of networking activities.

	Networking activities							
Date	Responsible	Name	Type of action	Attendees				
18/06/2018	UCAM		ORAL SPEECH	15 people				
19/06/2018 21/06/2021	UCAM	THE LIFE PERSEN	ORAL SPEECH (2)	15 people 30 people				
12/02/2019	UCAM	EMPORE	ORAL SPEECH	40 people				
28/06/2021	CTNC	SOLIEVA	ORAL SPEECH	25 people				
04/11/2021	CTNC		ORAL SPEECH	30 people from 7 countries				
17/05/2022	CTNC	Gamia	ORAL SPEECH	More than 100 people				
1-2/06/2022	CTNC	Networking CLEAN UP event (online) 16 projects https://ctnc.es/cursos/LIFE-clean-up-networking junio/ https://www.LIFEcleanup.eu/blog/2022/5/23/LI networking-days		Day 1: 49 people Day 2: 45 people				

Table 14. Summary of Networking activities

#### Task D2.3. Participation and organization of events:

We have participated in ten national (Milestone 18) and sixteen international (Milestone 23) conferences and fairs, as well as in six workshops and seminars.

A project conference (Milestone 20) has been organized, with the participation of researchers from other projects related to water management and treatment, as well as organizations such as the Entidad de Saneamiento y Depuración de Aguas Residuales de la Región de Murcia (ESAMUR).

Similarly, five technical visits to the demonstration plant (Milestone 21) have been organized to encourage and support the replication and transfer of the new treatment system to other locations and sectors.

All these events can be consulted in more detail in the table below (Table ).

	Participation and organization of events						
Date	Responsible	Name	Type of action	Attendees			
08/05/18	CTNC	<b>CIBUS 2018 Fair</b> in Parma, Workshop: EU smart projects: innovative results in the agrifood sector	Oral speech	30 people from 7 countries			
17-20/06/18	CTNC, UCAM	<b>9th International Congress of Chemistry of the ANQUE</b> , San Pedro del Pinatar, Spain	Oral speech Round table Poster	400 people			
26-28/06/18		<b>II XLVI Congresso della Divisione di Chimica physics</b> , Bolonia, Italy	Oral speech Abstract (2)	50 people			
13/07/18	UCAM	Nursing Week 2018 "Investigacao Week in Enfermagem", Oporto, Portugal	Oral speech	50 people			
24-26/07/18		<b>3rd International Conference on Applied mineralogy and Advanced Materials</b> (MMS), Bari, Italy	Oral speech	30 people			
25-28/09/18	UCAM	<b>IFT-EFFoST 2018</b> International nonthermal Proccesing Workshop", Sorrento, Italy	Poster	100 people			
22/11/18		XIV Technical Conference on water purification in the region of Murcia (ESAMUR), Lorca, Murcia	Oral speech	350 people			
26-29/11/18	HIDROGEA	<b>14th National Environmental Congress</b> (CONAMA 2018), Madrid, Spain	Poster Article	8700 people			
09/05/19	CTNC, UCAM	6 <sup>th</sup> Management of the Integral Water Cycle and the Environment Conference (UCAM), Spain https://eventos.ucam.edu/nuevos-desafios-en-la-gestion-del-agua	Oral speech	50 people			
14-15/05/19	CTNC	9 <sup>th</sup> International Symposium on Food Technologies, Murcia, Spain	Oral speech Poster	650 people from 16 countries			
2-6/06/19	UNIBA	14 <sup>th</sup> International Symposium on Macrocyclic and Supramolecular Chemistry, Lecce, Italy	Oral speech	50 people			
1-4/07/19	CNR, UNIBA	<b>XLVII Congresso Nazionale di Chimica Fisica</b> , Roma, Italy	Oral speech (2) Poster (2)	50 people			
28- 30/08/19	UNIBA	Chemistry meets Industry and Society (CIS2019), Salerno, Italy https://www.soc.chim.it/sites/default/files/chimind/pdf/2019_6_3 6_ca.pdf	Oral speech (2)	200 people			
02/10/19	CTNC	<b>EFIAQUA</b> , International trade fair for efficient water management	Working meetings	15 people			

11-13/12/19	CTNC, UCAM	5 <sup>th</sup> EU Water Innovation Conference 2019, Zaragoza, Spain	Oral speech Poster (2)	750 people from all European countries
16/12/19	UCAM	<b>Demo Day – The zone of hope challenge</b> , Barcelona, Spain	Oral speech Poster	100 people
26/02/20	CTNC	Worshop of the Erasmus Agrifood project, Tarimas, Turkey	Oral speech	12 people
26/11/20	CTNC	Webinar – <b>WORKSHOP OF FOOD INDUSTRY</b> https://www.sftt.info/index-26-11-20.html	Oral speech	133 people
27/11/20	UNIBA, CNR	La Notte dei ricercatori (Night of the Research)	Video	70 people
26/05/21	CTNC	1st INTERNATIONAL DIGITAL DESALATION AND REUSE CONGRESS (AEDyR) https://aedyr.com/ultimas-innovaciones-id-desalacion- reutilizacion-agua-presentadas-sesion-5-i-congreso-digital/	Oral speech Article	1000 people
17-19/05/21	CNR, UNIBA, CTNC	X International Symposium on Food Technologies https://murciafood2021.b2match.io/	Oral speech Poster	590 people from 39 countries
1-3/09/21	CTNC	XIII Agrifood Economics Congress	Informar ion stand	150 people
14-23/09/21	UNIBA, CNR	XXVII Congresso Nazionale della Società Chimica Italiana. La chimica guida lo sviluppo sostenibile.		60 people
24/09/21	UNIBA, CNR	European Researchers' Night 2021	Informar ion stand	300 people
07-08/10/21	CTNC	10TH INTERNATIONAL SYMPOSIUM Euro-Aliment 2021, Galați, Romania	Oral speech	15 people
08/10/21	CTNC	Technical workshop with Uruguay's water technology centre	Oral speech	20 people
05/11/21	CTNC	International conference – biotechnologies-present and perspectives, Suceava, Romania https://fiajournal.usv.ro/conference2021/	Oral speech Abstract	50 people from 10 countries
28-30/03/22	UNIBA	2 <sup>nd</sup> Edition International Conference Materials Science And Engineering (MATERIALS 2022)	Oral speech	50 people
6-8/04/22	CTNC	III Symposium Ibérico de Ingeniería Hortícola 2022 Smart Farming, Cartagena, España https://sibih22.com/_files/_event/_61258/_editorFiles/file/ Guia del congresista SIBIH22_Actualizada.pdf	Poster Technica l guide	300 people
10-12/04/22	UNIBA, CNR	7 <sup>th</sup> International Conference on Environmental Pollution, Treatment and Protection (ICEPTP'22)	Oral speech	150 people from 30 countries

10-12/04/22	UNIBA	7 <sup>th</sup> World Congress on Civil, Structural, and Environmental Engineering (CSEE'22)	Paper	60 people
27/04/22	UCAM,	Project Conference, in CTNC facilities (M20) https://ctnc.es/cursos/tratamiento-y-regeneracion-de-aguas- residuales-lineas-de-idi/	Oral speech (2)	30 people
13-17/06/22	CNID	<b>20th International Cyclodextrin Symposium</b> , Giardini Naxos, Italy.	Oral speech	200 people
		33 events		14,765

#### • Main problems encountered and solutions provided:

No specific problems worth mentioning have been found.

The main difficulty encountered in the last two years has been caused by the COVID pandemic situation. This has made some dissemination activities more difficult, especially due to mobility restrictions and cancellation of events, which has only led to a lower number of technical visits than initially planned.

In any case, the objectives set for this activity have been met satisfactorily and effectively.

#### • Achieved results:

These activities (around 340 activities) have made it possible to reach a technical target audience of more than 155,400 people, with a high impact on the target sectors of the integral water cycle, as well as key actors in the sustainable development of water (researchers, administrations, companies, etc.) (Table ).

	Summary		
Expected results	Achieved Results	Number of actions	Impact
Development of technical & scientific	Dissemination through articles, magazines and newspapers:	28	
publications	Technical articles	10	> 136,890
	Scientific articles	11	JCR: 6.988
	<ul> <li>Advertisement</li> </ul>	4	Difficult to estimate
	<ul> <li>Abstract/brief news</li> </ul>	3	Difficult to estimate
Networking with other projects	Dissemination in Training courses	3	41 people
	Dissemination to bodies/companies related to water management	8	61 people
	Dissemination through Networking activities	8	> 350 people
Participation and organisation of events	Dissemination in congresses, conferences and technical workshops	33	14,800 people

	Project Conference, with the participation of 10 related R&D&I projects	1	30 people
Technical visits to the demonstration plant (10)	5 visits has taken place	5	20 people
	Other results: • Network broadcasting • Dissemination in media	>250 3	3,219 people <i>Difficult to estimate</i>
	TOTAL	>339	> 155,400 people

#### • Remaining results:

None.

#### • Variations in action:

The only aspect to highlight is that, due to the aforementioned pandemic situation, the networking action proposed in Brussels had to be organized online for two days.

In addition, other technical dissemination actions not considered in the proposal have been carried out, in particular dissemination in networks and media. In addition to disseminating results on the project website and its social networks, dissemination actions have been carried out on the website and social networks of the CTNC, as well as other partners such as REGENERA.

Similarly, three dissemination actions have been carried out in the media, such as the Spanish radio stations "Onda Cero" and "Cope".

#### • Action evaluation:

This activity has been satisfactorily carried out, since all the objectives set, and the desired impact have been met.

TYPE	Code	Name	New deadline	Responsible	Status
MILESTONE	16	Start of online networking with related projects		CTC	OK 18/06/2018
MILESTONE	18	Participation in national conference	31/12/2019	CTC	OK 26/11/2018
DELIVERABLE	35	"Bioresorcurce technology" articlein "Journal of Hazardous Materials"	31/17/7010	CTC	OK 01/05/2020
DELIVERABLE	26	First article in CTC Food	31/12/2018	CTC	OK 01/01/2019

DELIVERABLE	34	Second article in CTC Food	31/12/2019	CTC	OK 01/01/2020
DELIVERABLE	40	Technical guide	31/12/2021	CTC	OK 01/03/2022
DELIVERABLE	51	Article in "ARPA News" Article in "RETEMA"	30/09/2020	CTC	OK 01/02/2018
DELIVERABLE	52	Article in "FuturENVIRO"	30/09/2021	CTC	OK 01/05/2021
MILESTONE	20	Project conference	30/11/2021	CTC	OK 27/04/2022
MILESTONE	21	Technical visits to the demonstration site in the WWTP facilities of Hidrogea (10)	31/03/2021	HIDROGEA	OK 16/06/2022
MILESTONE	23	Participation in international conference	30/08/2020	CTC	OK 17/06/2018

# 6.1.11. E1 Project management

### ✓ Completed action

Foreseen start date: 01/10/2017 Actual start date: 01/10/2017

Foreseen end date: 30/06/2022 Actual end date: 30/06/2022

PLANNED		20	17		2018	018 2019		2020										
TIMETABLE	S	0	Ν	D	2010	2017	J	F	Μ	А	Μ	J	J	Α	S	0	Ν	D
ACTION E1																		
ACTUAL		20	17		2018	2019					2	020						
TIMETABLE	S	0	Ν	D	2010	2017	J	F	Μ	Α	Μ	J	J	Α	S	0	Ν	D
ACTION E1																		

PLANNED		2021							2022									
TIMETABLE	J	F	Μ	Α	Μ	J	J	Α	S	0	N	D	J	F	Μ	Α	Μ	J
ACTION E1																		
						20	21								20	22		
ACTUAL						20	41								20			
ACTUAL TIMETABLE ACTION E1	J	F	Μ	A	Μ	J	J	A	S	0	N	D	J	F	M	A	Μ	J

### • Participants and responsibilities:

UCAM was the coordinator responsible for the activities related to project management. Each partner was responsible for notifying the coordinator any circumstance that may jeopardize the

project implementation, for the technical reporting of actions under their responsibility and for the financial justification of their expenditures.

#### • What has been done:

#### Task E1.1 Project management by UCAM

As it was explained in section 5 (administrative part), the project is structured at two levels, one administrative/financial and one technical, having nominated persons in each partner organization.

UCAM monitors and asses the project performance, preventing deviations, managing problems and ensuring the high-quality performance of the project.

Templates and models for project documents have been also developed and provided to project's partners.

Pending tasks are checked by using meetings minutes and through email and telephone communication with partners.

UCAM works with the rest of partners in the financial implementation of the project by evaluating expenditures and supporting documentation whenever is required. Main tasks as project coordinator over this period have been:

- Preparation of partner contracts to fix rights and responsibilities for each partner as well as fix reporting deadlines and transfer of funds.
- Set up of a shared project management platform.
- Preparation of reporting templates.
- Preparation of financial reporting guidelines.
- Continuous monthly reporting about the project progress to NEEMO.
- Coordination activities with the partners to prepare the info days and the meeting with regional policymakers
- Supervision of the internal reporting periods and documentation.
- Coordination with the partnership regarding the indications and feedback from NEEMO and EASME/CINEA
- Extraordinary teleconference meetings with the international partners regarding the financial justification.
- Continuous email and phone contact with all partners regarding technical and administrative reporting.
- Uploaded the data for the LIFE KPI database.

The Grant Agreement has been amended two times during the project implementation. The first one was initiated by EASME, and it was approved on September 14<sup>th</sup> 2018. This amendment contained modifications regarding personnel costs, VAT eligibility and the certificate on the financial statement. The second one for the extension of the project until June 2022 was requested the 4th of August 2020 and approved September 16th 2020.

#### Task E1.2 Project monitoring

Project partners have met regularly in partners or coordination meetings in order to review, coordinate and prepare activities. These meetings have been normally held in UCAM's facilities. Partners make a revision of the activities, deliverables and milestones due to the date of the meeting and discuss the main setbacks or limitations that may arise from technical or financial constraints. Conclusions and tasks derived from the meeting are recorded in a minute that is emailed to partners to get their approval and have in mind next steps agreed.

During the project, the partnership has held 24 meetings where 5 were full consortium meetings and 19 coordination meetings, and 5 monitoring visits with NEEMO external monitoring team (Table ).

	Coordination and Consortium Me	etings
04/10/2017	Coordination Meeting	MURCIA
02/11/2017	Kick-Off // Consortium Meeting	MURCIA
20/02/2018	Coordination Meeting	MURCIA
22/05/2018	Consortium Meeting	MURCIA
04/07/2018	Coordination Meeting	MURCIA
25/07/2018	Coordination Meeting	MURCIA
15/11/2018	Coordination Meeting	MURCIA
29/01/2019	Coordination Meeting	MURCIA
25/02/2019	Coordination Meeting	MURCIA
28/03/2019	Coordination Meeting	MURCIA
02/05/2019	Coordination Meeting	MURCIA
07/05/2019	Coordination Meeting	MURCIA
24/09/2019	Coordination Meeting	MURCIA
14/10/2019	Coordination Meeting	MURCIA
14/01/2020	Consortium Meeting	BARI
04/09/2020	Coordination Meeting	ONLINE
30/10/2020	Coordination Meeting	ONLINE
12/03/2021	Consortium Meeting	ONLINE

Table 17. Coordination and consortium meeting

15/07/2021	Coordination Meeting	MURCIA	
13/12/2021	Coordination Meeting	MURCIA	
29/03/2022	Consortium Meeting	BARI	
31/05/2022	Coordination Meeting	ONLINE	
23/06/2022	Coordination Meeting	ONLINE	
08/07/2022	Coordination Meeting	ONLINE	

EASME/CINEA/NEEMO Monitoring Meetings						
07/03/2018	First Monitoring Visit	Received letter: 25/04/2018				
09-10/05/2019	Second Monitoring Visit	Received letter: 26/06/2019				
10/06/2020	Third Monitoring Visit (online)	Received letter: 28/07/2020				
07/05/2021	Fourth Monitoring Visit (online)	Received letter: 01/10/2021				
06-07/06/2022	Final Monitoring Visit					

Apart from these meetings, email communication was maintained on a regular basis to keep partners updated about technical and communication activities, to coordinate actions and to share information coming from CINEA and NEEMO.

Regarding the financial reporting, partners agreed to upload supporting documentation and updated financial reports to a shared Google Drive folder. However, some partners have been sending the information by email to the coordinator and it is uploaded afterwards to have an easy access in case it is required for verification purposes. A financial review is made at least every three months, provided that no NEEMO monitoring visits or project meetings are organized. In these cases, project expenditures are updated for this purpose.

#### Task E1.3 After-LIFE Plan

This deliverable has been produced in the last stage of the project, in collaboration between UCAM and REGENERA who has entered the main information regarding the future activities for the market launch of the project and the possibilities for transferring the technology to other sectors. The After-LIFE Plan summarizes the role that each partner will play after the end of the project and scheduled activities during the next 5 years.

#### • Main problems encountered and solutions provided:

Technical problems for the scaling up of results from laboratory to semi-industrial level (difficulties in the production of the polymer) produced changes in the duration of the actions.

The partnership worked together to find solutions (search of suppliers) and the project could finally continue.

### • Achieved results:

The project coordinator has overcome unexpected barriers that came out during the project in a successful way. Although the activities and results foreseen has suffered from variations related to the project proposal, it has been finally ended and the main actions have been implemented.

### • Remaining results:

None.

### • Variations in action:

The main variation was the project extension requested to enlarge the project until June 2022.

### • Action evaluation:

E.1 has achieved the objectives set out in the initial proposal with a reasonable cost-efficiency. Project management and monitoring has been successful during this period. Coordination meetings allow a close collaboration among the partnership to deal with technical and administrative issues that may affect the project implementation. Expenditures related to the project are updated in a timely manner to monitor the financial implementation of the project in a satisfactory way.

TYPE	Code	Name	Deadline	Responsible	Status
MILESTONE	1	Project start	01/10/2017	UCAM	OK 01/10/2017
MILESTONE	2	Kick off meeting	30/11/2017	UCAM	OK 02/11/2017
DELIVERABLE	18	Initial evaluation report	30/06/2018	UCAM	OK 30/06/2018
DELIVERABLE	30	Midterm report	15/12/2018	UCAM	OK 20/12/2019
DELIVERABLE	31	Interim evaluation report	30/06/2019	UCAM	OK 14/03/2019
DELIVERABLE	44	Final evaluation report	31/03/2022	UCAM	OK 13/05/2022
DELIVERABLE	55	After-LIFE Plan	31/03/2022	UCAM	OK 30/06/2022
MILESTONE	24	Project end	30/06/2022	UCAM	OK 30/06/2022
DELIVERABLE	57	Final report	30/09/2022	UCAM	OK 30/09/2022

### 6.2. Main deviations, problems and corrective actions implemented

The main problem experienced during the project has been of **technical character** since the most innovative aspect of the proposed system has not worked as foreseen. The fact of not been able of producing the first tested polymer of CDs internally by partners' means and neither finding a solution for the externalization of this task, produced a delay in the implementation of project actions. Thanks to the transnational cooperation, a solution was found by changing the initial adsorbent material, so this unit of the semi-industrial plant used a polymer of nanosponges instead of the beta-polymer EPI-CDs. But the initial delay was worsened by the outbreak of the pandemic COVID-19 that difficulted international orders, purchase of materials and the production of the polymer.

On the other hand, another difficulty related to the use of CDs has been the extraordinary swelling capacity of this material that has involved changes in the initial treatment capacity that was expected for the semi-industrial pilot plant. Operation conditions have been changed to ensure the feasibility of the prototype: the operational flow decreased from 5 m<sup>3</sup>/h to 3 m<sup>3</sup>/h and velocity was lower than 12 m/h to permit working in fluidized bed and having an optimal contact time. The operation of the prototype under these conditions for a whole year has allowed assessing the cleaning capacity of the proposed combination of technologies (CDs, photocatalysis and PL) but it has made difficult the possibility of the system to be implemented at WWTP level have been undermined and final project results suggest further investigation in the polymer for being able to go further in this direction. Since the scaling up of the whole system cannot be suggested by the moment, the project has put effort on the transfer of the technologies to those industries that have a similar characterization of treated water (e.g.: they have phytosanitary compounds and drugs) but output flows are smaller than those treated by WWTP.

# 6.3. Evaluation of Project Implementation

#### **Methodology applied**

The project methodology has consisted in the preparation of the semi-industrial plant by means of laboratory activities to find the best adsorbent material and define operational and engineering details. This approach is necessary to consider all the elements that may affect the future function of the prototype. When technical problems have arisen, partners have had to come back to this initial stage in order to reassessed the suitability of the new polymer for the whole system. On the other hand, the operational time schedule of the plant for a whole year and passing through all the seasons is also optimal to include all the possible variables that may occur in the start-up and running out of the plant. Finally, the approach followed in replication and transfer activities has been also valid to have a first approximation for the market opportunities of the system in other industrial environments and even in other countries (Italy).

Action	Foreseen in the revised proposal	Achieved	Evaluation
A1	<u>Objectives</u> : Characterization of EPs in treated wastewater from WWTPs	Selection of the Cabezo Beaza WWTP. Selection of EPs from more than 5 families to increase	The WWTP with the best location for plant sampling, validation and maintenance work was selected. In addition, it

Comparison of results achieved against the objectives and expected results foreseen

	Expected results: Characterization of treated wastewater in 6 WWTPs, for the installation of the semi- industrial prototype, selection of 5 different families of EPs with a detailed list.	the reliability of the results obtained.	collects a greater variability of water (of urban, agricultural and industrial origin), which allows the results obtained in action C1 to be as representative and extrapolated as possible. The selection of EPs was successful, since they have been detected in all the samples of following phases (C1).
A2	Objective:Obtention of the most efficient material for the adsorption of EPs to include in the prototype.Expected results: optimization of the synthesis of adsorbent materials, evaluation of % of EPs eliminated and n° of cycles and LIFE span and development of a laboratory prototype.	Selection of the Italian cyclodextrin nanosponges able to remove the largest number of EPs both in batch and in flux mode.	Although different materials were tested, the nanosponges polymer was finally selected to ensure the real application during the industrial scale up. The cost associated to the used material could be potentially lowered by considering its recycling.
A3	Objective: Know operational parameters to work with Advanced Oxidation technologies in the semi- industrial prototype, according to EPs characterized in WWTPs. Expected results: optimization of operational parameters of photocatalyst and light pulses. Assessment of the capacity to treat big water volumes	PHOTOCATALYSIS: Minimum treatment time $\rightarrow 25$ s. PULSED LIGHT: Flux of 96 J/cm <sup>2</sup> achieves 53% of removal rates encompassing factors such as light intensity or treatment time. Unlimited capacity of the photocatalyst depending on the equipment (up to 1000 m <sup>3</sup> /h)	The work carried out on a laboratory scale has made it possible to scale up emerging contaminant oxidation technologies: - Installation of a second photocatalysis unit in the prototype to increase the treatment time (or the flow rate if working in parallel) - Adaptation and development of a light pulse equipment to work with a continuous flow.
A4	Objective: Engineering of the semi-industrial prototype integrating adsorption, AOPs, energy and operational control and renewable energy supply Expected results: Designed and detailed engineering of the semi-industrial prototype building solar panels for energetic necessities. Design	The prototyping and parameterization of a semi- industrial Pilot Plant (deliverable 17) was elaborated on time. Simulations were done to better known the final technical requirements of the solar panels.	The expertise provided by partners (HIDROTEC, REGENERA) and the good collaboration between scientific and technical partners have allow adjusting the engineering parameters of the plant to the results obtained at laboratory level.

	of the demo-plant in the existing WWTP. Integration of SCADA monitoring.		
B1	Objective: Prototype at semi-industrial scale integrated in the WWTP. <u>Expected results:</u> Prototype building. Synthesis of the adsorbent material and installation of the prototype.	Construction of a semi- industrial plant combining adsorbing and AOP technologies.	The construction of the demonstrative plant was timely done, although some equipment such as the photovoltaic panels and the pulse light system was included later. This did not delay the installation of the plant. The adsorbent material was not synthesised by partners but by an external company who poses the patent.
B2	Objective: Start up and running of the prototype to test its performance in real conditions. Expected results: Functioning of the prototype for 12 months non-stop and control of operational and biological parameters.	Starting of the pilot plant $\rightarrow$ 2 months Operation $\rightarrow$ 11 months, Flow $\rightarrow$ 0.25m <sup>3</sup> /h with a consumption rate of 1.7 kWh/m <sup>3</sup> . Ultrafiltration removes 100% of microbiological parameters. The rest of compounds were 100% removed.	The running of the plant has served to validate the combination of technologies for the disinfection of treated wastewaters and to standardize the maintenance procedures that ensure a continuous treatment of water. However, treatment flows were not those proposed initially (5m3/h). The initial flow was reduced to 3 m3/h due to the properties of the cyclodextrins and design restrictions. During the validation phase a maximum flow of 0.25 m3/h was achieved.
B3	Objective: Economic and financial assessments to prepare the market launch. <u>Expected results</u> : Technical feasibility of scaling up the technology at industrial level. Assessment of transferability to other sectors. Preparation of market launch.	<ul> <li>Technical-economic feasibility study (economic indicators, profitability assessment, calculation of costs, etc.)</li> <li>Technical-economic- legislative feasibility study</li> <li>Definition of the business model</li> <li>Industrial exploitation plan</li> <li>Market study</li> </ul>	The action is considered 100% completed. The work done has allowed knowing the possibilities of the project to be replicated/transferred and to contrast the reality with the targets of the proposal.

		<ul> <li>-Identification of target market (application scenarios)</li> <li>-Market penetration study (definition of the different phases)</li> <li>-Definition of sales strategy</li> <li>-Marketing plan</li> <li>-Technical visits to WWTP to study replication and transfer potential.</li> <li>-Study and definition of the replication plan</li> <li>-Study and definition of the transfer plan</li> </ul>	
C1	Objective: Validation of the proposed solution by comparing laboratory results and real conditions operation. Expected results: Adsorption capacity of the polymer, elimination capacity of the pulse light and photocatalysis equipment. Cleaning capacity test. Adsorption capacity after regeneration and modification of the operational para meters.	The overall removal performance of pharmaceuticals is 80% (including the pollutant destruction phase by photocatalysis). Separately: the system has an adsorption capacity of around 45% for pharmaceutical products and 100% for phytosanitary products. The PL produced 34 % removal of EPs in wastewater desorbed from cyclodextrin column. Removal percentage is increased by recirculation.	The technical feasibility of the proposed system has been validated on a semi- industrial scale, in terms of its capacity to remove emerging pollutants and its capacity to disinfect wastewater. PL system could reach the removal mean value of 53 % reached at laboratory scale by recirculation. The system can be considered as an effective tertiary treatment (allows the complete disinfection of the wastewater). In terms of marketing capacity, the system must undergo modifications to increase the treatment capacity in terms of volume.

D1	<u>Objective:</u> Awareness raising during the project's development by the design of materials and communication tools to	Webpage created and maintained since January 2018. Facebook and Twitter	Despite sanitary restrictions, it is considered that the outreach of the project has been great. LIFE Clean
	<ul> <li>involve the general public.</li> <li><u>Expected results</u>: communication designing plan. Development of a webpage, a leaflet, social media accounts, videos and a board at the WWTP.</li> <li>2 Info-days, 2 guided visits with journalists to the demonstration plant and 10 visits for non-technical audience.</li> </ul>	<ul> <li>profiles created and maintained since October 2017.</li> <li>2 leaflets elaborated and distributed.</li> <li>1 video launched.</li> <li>1 board installed at the WWTP.</li> <li>4 newsletters published.</li> <li>2 info-days celebrated.</li> <li>2 Visit for non-technical audience</li> </ul>	Up has appeared in 54 articles (print and online), 1 documentary in the regional TV, 3 Radio's interviews, 1 contest on a short movie festival, 1 contest of spots to raise awareness among the general public, 2 science weeks (a regional event which more than 25.000 participants, focusing on science). Furthermore, the online media, social media, views on our video and the pageviews of our website ascend to 171.070.
D2	Objective: Reach technical audience to disseminate the innovative character and enlarge replication opportunities Expected results: Networking and face-to-face meetings, publication of technical guides, participation in events, project conference and 10 technical visits to the demonstration plant.	Around 340 activities reach a technical target audience of more than 155,400 people.	The objectives set have been met satisfactorily, with a high impact on the target sectors of the integral water cycle, as well as key actors in the sustainable development of water (researchers, administrations, companies, etc.). During the last two years, dissemination actions have been somewhat hampered by the COVID pandemic, as it has meant the cancellation of events, the difficulty in organising workshops and visits to the demonstration plant, etc.

#### Visibility of project results.

For laboratory activities, UCAM, UNIBA and CNR-IPCF achieved the first results already in January 2018. In September 2018, the scientific partners had a whole investigation of the used and studied materials. Later, the results arisen from the use of the Italian cyclodextrin nanosponges appeared evident in June 2020.

For the implementation activities, a whole year was necessary to assess the overall performance of the prototype plant and its operation in real conditions. Conclusions have been gathered by June 2022.

For replication and transfer activities, the initial objectives have been achieved. This is to have feasibility studies about the potential of the prototype to be implemented in other industries. Nonetheless, further commercial agreements could be done but they could be agreed only after the end of the project.

Finally, about the market launch of the system, this has not been produced within the lifetime of the project. Partners have agreed a calendar of actions and responsibilities to work in the business plan of the technology proposed.

#### Importance of the project amendment

The project amendment requested in August 2020 has been key to ensure that all the project actions have been finally implemented. Partners asked for an extension of the project to continue working in the running and monitoring of the pilot plant, replication and transfer and communication activities beyond September 2020 that was the first final date of the project. The project was enlarged until June 2022, and this has permitted to have enough time to finish the planned activities and overcome new difficulties (e.g.: different characteristics of the polymer finally received to the first sample sent, shutdowns of the plant because of high content of organic matter of the treated wastewater, etc.).

#### **Results of the replication efforts**

Between March and April 2022, replication and transfer companies were visited to explain the technology employed in the semi-industrial prototype to deal with emerging pollutants and present the results obtained. Water samples were taken and analysed in 7 sites to know the characterization of the effluent wastewater. All samples contained drug residues in varying quantities and in order of ppb, therefore they would be suitable for the implementation of LIFE Clean Up technology. But conclusions about the feasibility of implementing the technology differs among replication and transfer sites.

For scaling up the technology to other WWTPs, it would be necessary to treat major flows what is not possible nowadays due to the restrictions posed by the polymer and the current design of the plant.

The most feasible solution would be the transfer of the proposed technology to other industrial sectors that produce smaller amounts of wastewaters. For this purpose, commercial scenarios such as dairy or agri-food industries are proposed, as well as other industrial sectors such as pharmaceuticals or healthcare (always complying with the premise of the flow rate set as a limit). The introduction of a pre-treatment before the water input to the plant and also the possibility of designing the plant without the light pulses is envisaged. However, a prior technical and economic feasibility study should be carried out to check that LIFE Clean Up technology is viable with the flow rate proposed, and in accordance with the characteristics of each customer. Within the project, a full feasibility study was made for the Citromil case (citrus company in Murcia, Spain). Results show that the implementation of the proposed treatment system would not be profitable for the company.

#### Effectiveness of the dissemination activities and major drawbacks

The dissemination activities, both those aimed at the general public and those aimed at a technical audience, have been highly effective given the capacity of the participating research centres, which have a high level of experience in the development of dissemination material, both oral (presentations, technical meetings, interviews, audio-visual material) and written 98

(scientific and technical articles, abstracts, advertisements). Their impact has been high thanks to the partners' direct contact with other research centres and groups, as well as with the business sector, public administration (especially regional) and the general public (customers, students, etc.). More than 65,000 individuals have been reached during dissemination and communication activities organized by the project or by other entities. Also, 21 publications about the project have been done, counting articles in scientific journals and specialized magazines.

However, the possibilities of making demonstration visits have been conditioned by the restrictions posed by the pandemic COVID19 in each moment. The WWTP has maintained a very strict policy for the entrance of people out of the company, so the option of making the planned activities in the plant have been reduced to very few months, just at the end of the project.

#### **Policy impact**

• <u>Project achievements.</u>

The main achievement of the project has been to develop a treatment system capable of providing a very important technological response to the problem of treating emerging pollutants, included in Annex II of Regulation EU 2020/741 as additional requirements for risk management, and which allows to ensure the required microbiological quality, thus allowing the safe reuse of reclaimed water. In addition, the technological proposal does not involve the addition of reagents to the water or the generation or addition of disinfection by-products such as trihalomethanes or chlorates. This fact finds its maximum benefit in compliance with the also recent Regulation EU 2020/749 of 4 June 2020, amending Annex III to Regulation (EC) No 396/2005 of the European Parliament and of the Council, as regards maximum residue levels for chlorate in or on certain products.

• <u>Main barriers</u>

At laboratory level, scientific partners (UNIBA and CNR-IPCF) refer that any barriers were undertaken during the work. The limitation for the synthesis of the beta-polymer was overpassed by means of the Italian cyclodextrin nanosponges. Additionally, the latter material, with respect the other, avoided the presence of high counter pressures when the experiments were performed in flux mode.

Regarding the future opportunities of the project for its market launch, one of the main barriers is that some of the EPs found in treated wastewaters are not still regulated so there are not minimum concentrations allowed. If WWTP had the obligation to measure and limit the concentration of recurrent drugs found in treated wastewaters, the convenience of investing in the further development of this system and, especially, to improve the operation capacity of the polymer, would be bigger. Thus, the scale up of the project would be easier.

<u>Policy developments</u>

The importance of the main achievement is even more important since, during the implementation of the project, specifically on 25 May 2020, the EU developed the aforementioned EU 2020/741 Regulation on minimum requirements for water reuse, which in Spain replaces the previous Royal Decree 1620/2007, including various pollutants of emerging concern, including pesticides, pharmaceuticals and disinfection products, among others. Therefore, we can affirm that the project is part of a priority area in the European Union's policies.

Some of these compounds have only previously been considered as priority substances at the observation level in environmental quality standards (EQS), but there is still no specific regulation or legislation governing them.

However, there is a growing awareness of these compounds and their potential impact on the environment and on human health, and it is quite possible that in the near future there will be limitations resulting from their presence in wastewater.

Therefore, it can be considered that the project has been a success in terms of the subject matter addressed. Firstly, because it is framed within the current socio-economic policy that prevails in Spain and Europe, in which the principles of the Circular Economy prevail and, specifically, the aim is to promote the reuse of water (which is reflected in Regulation 2020/741). Secondly, because it allows for the elimination of pollutants that until now have been little known and which are resistant to current treatment systems, and of which there is growing awareness.

Therefore, projects of this nature, which study the magnitude of the problem of these types of pollutants and develop technologies for their elimination, provide tools for legislators to improve the regulation of these pollutants.

• Deliberation of the projects foreseen in the Grant Agreement

The project has positive impacts on biodiversity since it avoids that EPs, and their metabolites reach the aquatic and land ecosystems and its derived negative effects in the provision of ecosystem services. The system has demonstrated to eliminate nearly 80% of the EPs found in treated wastewater so it can guarantee that these flows do not contain dangerous contaminants if they reach natural environments. Moreover, the combination of technologies used does not produce disinfection products that could be also harmful for the ecosystems. This contributes to the vision for 2030 of the Zero Pollution Action Plan of the EU: air, water and soil pollution is reduced to levels no longer harmful to health and natural ecosystems this creating toxic free environment.

The project has built up results also in climate mitigation since the design and function of this prototype to improve water quality has also considered the integration of renewable energies, so the impact on climate due to CO2 emissions has been reduced. The energy system has been formed by 30 photovoltaic modules of 330 V each, inverters, a battery of 13,8 kW and a control panel. By the end of the project, the consumption rate has been of 1.7 kWh/m3, being the power supply by the photovoltaic unit of 61% the total consumption of the plant. LIFE Clean Up is aligned with the climate targets of the EU to reach climate neutrality by 2050 as it has been set by the European Climate Law.

On the other hand, the results obtained have also implications for climate adaptation, especially at local and regional level, so the project boosts a more systemic adaptation, thus supporting the development of the EU Strategy on Adaptation to Climate Change. Water scarcity is currently a reality in regions where it was unimaginable some years ago. The reuse of treated wastewater is a common practice in Member States of South Europe such as Spain, Italy, Greece, Malta and Cyprus and northern Member States like Belgium and Germany, but generally is unexplored. According to past year studies supported by the European Commission, the potential of EU in this area is estimated in the order of 6 billion cubic metres, six times the current volume of treated urban wastewater that is reused annually (about 1 billion cubic metres, which accounts for approximately 2.4% of the treated urban wastewater effluents and less than 0.5% of annual EU freshwater withdrawals). So having into account the fall in water availability as a consequence of climate change and the potential for reusing wastewater, the treatment technology proposed by LIFE Clean Up could be a solution to ensure a secure

supply of water for farm irrigation, industrial uses (cooling, paper mills, etc.) or for urban uses other than drinking (watering of parks and gardens, street cleaning, fighting fires, etc.).

# **6.4.Analysis of benefits**

### 1. Environmental benefits

### a. Direct / quantitative environmental benefits

Quantitatively, the proposed solution is capable of eliminating an average of 80% of pharmaceutical compounds and 100% of phytosanitary products present in treated wastewater, in addition to allowing water disinfection without the input or generation of disinfection by-products, thus considerably reducing the number of pollutants that can enter the environment through irrigation or water discharge.

The prototype has demonstrated the complete elimination of detected phytosanitary contaminants from various families: neonicotinoids (imidacloprid and Acetamiprid), organophosphates (Chlorpyrifos), acylalanines (Metalaxyl) or benzimidazoles (Carbendazim), including other unclassified compounds such as Orthophenylphenol/2-phenylphenol.

Regarding pharmaceutical compounds, elimination yields of approximately 24-100% were observed in the pharmaceutical compounds analysed, obtaining for some compounds not reaching 100% such as Diclofenac or Ketoprofen elimination percentages above 90%, while for other compounds such as Ofloxacin or Venlafaxine rates just around 29% or 43%, respectively. In any case, an average elimination rate of 80% has been obtained for this type of compounds. Regarding EP, phytosanitary compounds detected are totally removed (more than 400 actives substances analysed).

Also, although trihalomethanes have not been detected in the wastewater treated at Cabezo Beaza WWTP, since chlorination disinfection is not used, it should be noted that the prototype has demonstrated the 100% elimination of disinfection by-products such as chloroform, a trihalomethane generated in greater quantities when chlorine reacts with organic matter, which is toxic in nature and is carcinogenic.

The proposed combination of technologies has demonstrated, both at laboratory and semiindustrial scale, compliance with the microbiological water quality required by the current Regulation (EU) 2020/741, thanks to the pre-treatment step. For its validation, the microbiological parameters of E. coli and C. perfringens spores have been taken into account, which are kept below 10 ucf/100 mL from passing through the pretreatment unit onwards. In any case, it is important to note that the advanced oxidation stage by photocatalysis, would ensure the microbiological quality of the water given its disinfection capacity, which has also been previously demonstrated.

Similarly, the filtration stage allows suspended solids to be removed and the organic load of the influent to be reduced, improving the effectiveness of the adsorption stage by means of cyclodextrins, thus extending its useful LIFE. This pre-treatment also significantly reduces the turbidity of the water, which is essential to guarantee the effectiveness of the photochemical treatments (photocatalysis).

Regarding the volume of water treated by the Clean Up prototype, it has been demonstrated that the water quality in the effluent entering to the cleaning system has a great influence in the operation cycles and treated flows. So, the pretreatment unit became essential in the overall performance of the cleaning system. The adjustment of the pretreatment unit (by changing cartridge filters once a week) has permitted to reach flows of 250 l/h. However, this is still far from what it was foreseen by the project proposal (5 m<sup>3</sup>/h) and from the flow design of the

semi-industrial prototype (3  $m^3/h$ ). The treatment flow has been limited by the swelling capacity of the adsorbent polymer.

### b. Qualitative environmental benefits

Improving the quality of wastewater undoubtedly brings environmental benefits.

Water treatment focused on the elimination of emerging pollutants reduces the negative impact of these compounds on health and the environment, especially because: a) it reduces the likelihood of the development of bacterial resistance to antibiotics due to the continuous discharge of antibiotics into the environment; b) it guarantees the food safety of agricultural products irrigated with reclaimed water due to the possible transfer of contaminants to the crop.

Wastewater, as part of an integrated approach to wastewater management, not only saves water but also provides a reliable alternative water supply for a variety of purposes, with agriculture having the greatest potential as the most water intensive economic sector. Thus, agricultural irrigation with reclaimed water contributes to reducing water scarcity across Europe.

In general terms, the treatment and reuse of treated wastewater, in addition to allowing the recovery of wastewater and reducing the environmental impact it may cause, while alleviating water stress, has a lower environmental impact compared to the use of other sources such as water from water transfers or desalination, offering a wide range of environmental, economic and social benefits. Moreover, it allows the LIFE cycle of water to be extended, thus contributing to the preservation of water resources in full compliance with the objectives of the circular economy and also providing solutions to alleviate water scarcity under climate change scenarios.

#### 2. Economic benefits

Due to the treatment restrictions of the adsorbent unit, the upscale of the technology to the industrial level (e.g.: to treat all the treated wastewater produced by a WWTP) is not feasible, so there are still not business opportunities regarding the implementation of the technology in this sector unless the polymer is modified to deal with bigger amounts of water.

The technical and economic feasibility study developed during the project compares the costs of implementing the *Clean Up* technology with state-of-the-art technologies, without having into account the limitations posed by the adsorbent unit regarding the treated flow. Regarding the obtained results, although the treatment costs of the *Clean Up* system are competitive and are in line with those of most water reclamation treatments, they are higher than those of the studied alternative. Specifically, the *Clean Up* system costs vary between 0.73 and 0.40  $\notin$ /m3 depending on the different scenarios, while in the case of ozonation + biological filtration, they vary between 0.42 and 0.11  $\notin$ /m3.

Regarding the investment costs, they have been estimated lower in the case of the *Clean Up* alternative. Specifically, they could vary between 0.19 and  $0.05 \notin m^3$ , while in the ozonation + biological filtration case they could vary between 0.23 and  $0.06 \notin m^3$ , depending on the factors previously pointed out. By the other hand, the operation costs are higher in the case of *Clean Up* system (0.54 – 0.35  $\notin m^3$ ) as compared with the raised alternative (0.19 – 0.04  $\notin m^3$ )

In relation to the cost-benefit of transferring the LIFE Clean Up technology to other sector, a 20-year cost-benefit study has been carried out in Citromil case. It has been observed that a positive cash-flow is not obtained in any case, if  $0.40 \notin/m3$  is set up as selling price for reclaimed water, which is the current market value. So, the implementation of the proposed treatment system in Citromil S.L., would not be profitable for this company. The value of the reclaimed water selling price from which the technology transfer would be profitable according to the defined conditions would be  $1.42 \notin/m3$ . In order to obtain a return period less than 10 102

years, it would be necessary to sell the water above  $1.9 \notin m3$ , and less than 5 years, selling it for approximately more than  $2.7 \notin m3$ .

The project has had an impact on qualified employment since it has allowed creating 2 FTE in the University of Bari and in CNR-IPCF. These jobs are related to research positions developing laboratory activities for analysing adsorbent materials and AOP techniques.

#### 3. Social benefits

Thinking in freshwater as a resource, not every country presents the same quantity of available freshwater, which depends on the climate conditions and transboundary water flows.

When the use of freshwater is above 20% of the renewable resource, that indicates that water resources are under water stress, whereas above 40%, water stress is severe, and the use of freshwater is unsustainable. Italy and Spain present a water exploitation index plus (WEI+) of 15.6% and 23.3%, respectively. Therefore, water reclamation is mandatory in order to alleviate not only the pressure on freshwater sources, but also to fight off water scarcity and anticipate future droughts across Europe, above all taking into account the increase of European population percentage exposed to water stress conditions.

According to the European Environment Agency (EEA), agriculture, forestry, and fishing consumed a total of 65,286 hm3 during 2017, (59% of the water used in Europe), above all caused by Southern European countries. Most of that use takes place in the southern basins where the production of some crop types would not be possible without irrigation. Besides the agricultural areas with intensive irrigation, the popular tourism destinies (island in southern Europe) and large urban agglomeration are the biggest water stress hotspots.

Around 7-8 % of the total agricultural area in Europe is irrigated, reaching 15 % in southern Europe. In addition, Southern Europe uses around 95 % of the total volume of irrigation water at the European level. Crop irrigation is particularly intensive (80 % of total water use in southern Europe) between April and August.

In southern European countries, such as Greece, Italy, Portugal and Spain, a scenario characterized by water stress is therefore settled down, and where agriculture is a fundamental part of economic growth. Therefore, reclaiming wastewater is extremely important in this scenario, and Clean Up technology stands as an alternative to this challenge.

Within the scope of the *Regulation (EU) 2020/741 of the European Parliament and of the Council of 25 May 2020 on minimum requirements for water reuse*, the main objective pursued is to ensure the quality of the water for its correct reuse, which, in turn, will allow increased confidence among users with regard to the safety of reuse practices.

The elimination of emerging contaminants achieved with the Clean Up treatment system, ensuring that these compounds do not enter the environment or, in any case, in very low concentrations, increases confidence in the ecological discharge or in the reuse of these waters for agricultural irrigation not only by consumers, but also by farmers or producers and, thus, by society in general. This, ultimately, allows arid and dry regions such as the Region of Murcia where near 90% of treated wastewater is reused, to ensure the economic growth of the agrifood industry, while guaranteeing the safety and health conditions of society.

Another important aspect in terms of food safety is the safety of the products, which is guaranteed thanks to the complete disinfection of the wastewater achieved with the treatment system, eliminating any pathogenic microorganism that could alter human health.

### 4. Replicability, transferability, cooperation

The policies related to the possibilities to replicate or transfer the Clean Up systems are those regulations related to wastewater reclamation. On one hand, Directive 2013/39/EU includes 45 priority substances for which maximum permissible concentrations in waterbodies are established. This list is extended to 75 substances under observation by means of the annex to Commission Implementing Decision (EU) 2015/495. On the other hand, European Commission is lately promoting the reuse of wastewaters in agriculture, and, in this way, it has developed the Regulation (EU) 2020/741 on minimum requirements for water reuse. Until now, that regulation does not establish limits for EPs in treated water. However, it is established the surveillance of some EPs such as personal care products, pharmaceuticals, drugs, etc., in irrigation water, with a view to defining these limits in the near future. In this way, the vision of Clean Up established by the Business Plan is to provide a technology to be the first option within the wastewater treatment sector to be implemented in order to remove all of the EPs and pathogen contained in wastewater and, at the same time, saving operating costs.

According to the Market Plan, the market to which the product is directed is that of the companies in charge of the construction and exploitation of WWTPs, (private or public) not only in Europe but in the rest of international markets, and mainly in developed countries. The pharmaceutical sector has been identified as the ideal target for the transfer of the technology.

Also, in the Replicability and Transfer Report, the agri-food sector is also identified as a potential target for the transfer of Clean Up technology, providing technical and economic factors that support said identification. But in this report, technical issues are emphasized, especially the necessary modifications in the current treatment facilities, as well as in the Clean Up system itself, to adapt it to this particular scenario, and due to the study of different cases for replication and transfer, the conclusions of the Market Plan are modified accordingly.

The conclusions of the Replicability and Transfer Report delays the 4 commercialization stages for the replication in WWTPs established in the Business Plan, at least until 2025 since a preliminary stage is needed. Consequently, the likelihood of replication just after the end of the project is low. In order to facilitate the full scale up of the technology, it would be necessary further research and innovation work for the modification of the polymer in order to increase the flow rate treated by the system, between 2022 - 2025. In this period, it would be also possible the commercialization in other industrial sectors (agri-food, agricultural, pharmaceutical or sanitary) whenever the targeted industry has a flow rate similar to the demonstration plant. In this scenario it would be possible to build the plant without the light pulse unit, so the investment costs would be lower. The adsorption unit would use the commercial polymer supplied by EXECOM SRL (supplier of the patent and the production process of the cyclodextrin polymer) that would be purchased for that purpose.

Stages 1-4 focus on different countries according to four pillars: number of WWTPs, water exploitation index plus (WEI+), irrigation water demand and chemical status of the water bodies. These factors have been scored to establish the criterium to choose the different countries in each stage. However, to select the first target markets, it was considered only those with a high WEI+ and a high irrigation ratio: the southern European countries. The overall potential penetration rate (maximum percentage of the European WWTPs with tertiary treatment, TT) is 13%, taking into account the four stages.

The Business Plan contemplates the creation of a joint venture, however, the exploitation at Stage 0 (if any installation of plant were to be carried out), will be executed separately on demand and the synthesis of the polymer by the joint venture (through licensing of the existing patent) is not contemplated, but through direct purchase from the manufacturer, as previously said. In this way, Regenera would be the company in charge of engineering and adapting the energy supply with the aim of increasing the sustainability and energy autonomy of the process.

Hidrotec would be the supplier and installer of all the technology, and Hidrogea would be in charge of commissioning the plant.

#### 5. Best Practice lessons

Treated water is a particularly complex system because it consists of components having very different chemical-physical characteristics, the presence and quantity of which depend on the geographical area, the season, and the possible occurrence of unforeseeable phenomena such as extreme weather events.

The great variability of wastewater composition in the first place and the treated water consequently makes the idea of a single type of inadequate Wastewater Treatment Plant (WWTP). WWTP should be designed and manufactured in a versatile and modular way to be adapted to various contexts.

Among the possible additional treatments, an important role is played by the possibility of adding, to the need, some treatments aimed at breaking down organic matter, the presence of which is highly dependent on the origin of the water to be treated, from temperature and other variables.

A high organic content causes a decrease in the effectiveness of subsequent treatments, such as those considered in this project. However, using these treatments routinely for economic and environmental reasons makes no sense.

Furthermore, by focusing on the theme of the project, namely the removal of emerging contaminants by adsorption, another and perhaps more important difficulty is the wide variability of substances that can be present as pollutants in terms of structure (for example, size, presence of a wide variety of functional groups, etc.) and, consequently, with a great diversity of chemical-physical characteristics (solubility, stability, the possibility of ionization, etc.) as well as quantities. In particular, since the project focuses on developing efficient materials suitable for adsorption from aqueous matrices, the large variability of target emerging molecules/contaminants has clearly shown that it is impossible to identify a single highly efficient material, eco-sustainable and economical.

However, the preliminary study revealed the general properties of the adsorbent material. In particular, the working mechanism of the adsorbent material should not depend on pH and temperature (i.e., the removal efficiency should not be affected by seasonal thermal fluctuations). It should be designed to have various types of moieties that can expand its ability to interact with the greatest number of contaminants. Also, rheological characteristics (in the presence of water) must be compatible with the passage of high water flows.

In this regard, the experience gained on the use and recycling of agri-food waste as adsorbent materials has led to the awareness that it is necessary to combine different types of adsorbents to reduce, on the one hand, the use of synthetic adsorbents is more eco-sustainable and economical. But on the other hand, this material would have the advantage of not requiring the payment of patent usage fees.

However, these materials of natural origin leave outstanding problems for their use in this field of application, which make it necessary to deepen their study. It follows that, with the knowledge and methods currently known, cyclodextrin polymer is the most efficient and versatile material. In this context, another important aspect is the need, when needed, to combine the adsorption process with other pollution remediation methods, such as post photocatalytic treatment, which has been shown to increase the efficiency of removal of pollutants from the plant.

Another aspect that emerged during the project was the importance of involving more scientific and technical skills that allowed us to have a greater awareness of the issues associated with scaling up the process.

Moreover, based on the problems that emerged following the transition from the plant on a laboratory scale to the semi-pilot plant, it was found that the scaling up had to be carried out more gradually. This is because the larger amount of adsorbent material needed in the prototype gave unpredictable multiplicative effects such as swelling and phenomena to its associates.

#### 6. Innovation and demonstration value

The obtained results evidence the concreate applicability of the proposed material for industrial application. Moreover, the use of hard conditions of work or organic solvents is avoided according to "Green Chemistry" principles. Indeed, numerous adsorption/desorption cycles have been studied avoiding the disposal of the adsorbent as secondary hazardous waste material. With respect the use of other materials, the selected adsorbent removes a great number of EPs suggesting the possibility to low the associate cost.

Among all types of EPs, pharmaceutical and personal hygiene products are the most considered, as these compounds are of greatest concern in terms of its increasing presence in the water cycle. These are organic compounds, with medium or low biodegradability for which, in general, the biological treatments of WWTPs are not very effective. There are many technologies available for the removal of those kinds of EPs, but the cost-efficiency of each one depends on the concentration of the pollutants to remove. In the case of WWTPs effluents, only a few technologies have been implemented at industrial scale.

The need to implement treatment processes for the elimination of EPs in WWTPs is encouraging many actions throughout Europe, both by public and private initiative. The main objective is to determine the most efficient technology to achieve EPs removal not only at a technical level but also economic and environmental. In this way, some countries such as Switzerland, the Netherlands, Sweden, Germany and Denmark have started to explore the extra end-of-pipe treatment at specific WWTPs with the purpose of addressing only one specific category of micropollutants: pharmaceuticals for human use. These European experiences will also be presented, besides some funded by the European Union in R&D projects like NEPTUNE and TREATEC. Considering all the work carried out all over Europe, the most efficient treatment processes would be both, the ozone (O3) and powdered active carbon (PAC), both with a final filtration stage.

The technology developed by Clean Up Project is focused on improving the quality of the effluents coming from the WWTPs removing the EPs and pathogens. However, the present project will use cyclodextrin polymers as adsorbent material, which can be recovered and reused, combined with 2 different AOPs systems: photocatalysis and light pulses. The advantages showed by the present system lay in the fact that is able to cope with a wider range of EPs than other used technologies. Nevertheless, it is necessary further research to improve the characteristics of the polymer and enable an industrial scaling up of the whole system.

#### 7. Policy implications:

The Region of Murcia is a national, European and world leader in water treatment and purification, and in the reuse of reclaimed wastewater in agriculture, which has been possible thanks to research and investment efforts aimed at adapting WWTPs to new, more efficient and advanced treatments. However, the efforts made are insufficient, in almost all cases, for the removal of pollutants of emerging concern, which continue to pose a challenge for water treatment systems, especially now that European Union policies aim to promote water reuse and the presence of these compounds is beginning to be considered in European regulations.

In this aspect, the project has its greatest advantage since the technological combination developed allows for a non-selective treatment system for contaminants of emerging concern that has been validated for a wide variety of contaminant families, as well as a modular system capable of adapting to treatment needs, also allowing for water disinfection.

The non-selectivity of the technology is especially important considering the constant development, manufacture and synthesis of new compounds that can be considered contaminants of emerging concern, be they of pharmaceutical, pesticide, personal care, etc. origin.

Therefore, technologies such as the one developed in the LIFE Clean Up project will be necessary to comply with the Regulation (EU) 2020/741 of the European Parliament and of the Council of 25 May 2020 on minimum requirements for water reuse, which will be mandatory throughout the EU from 26 June 2023, and will allow the sustainable development of agriculture, especially in those places with greater water stress, such as the countries of the Mediterranean basin.

Similarly, it is important to consider that this system also allows the disinfection of wastewater, even eliminating *C. perfringens* spores, which have been included in Regulation EU 2020/741 as a microbiological indicator of quality for protozoa.

However, our technology presents its greatest barrier in its current treatment capacity, which is limited for large-scale application and for the treatment of large volumes of wastewater, although it is suitable for punctual and specific sources of pollution such as pharmaceuticals, hospitals, chemical synthesis companies, etc

# 7. Key Project-level Indicators

The final values entered in the online KPI database of the LIFE Programme are commented below.

Indicator code	Indicator name	Descriptor	Start project	End project	Updated End Project Value	Beyond 3 years value	Updated Beyond 3 years Value
1.5	Project area/length	Partial reduction of specific pressures/threats affecting the spatial extent of the project in comparison to the present level	0	2486 ha	0,488 km²	11313 ha	0,488 km²

In the case of the **project area**, a new end project value has been provided according to the guidelines to complete this indicator in the Webtool. The first time the target values were completed, estimations referred to the surface of the municipalities where the WWTP could be implemented. At the end of the project, guidelines indicates to consider the surface of the WWTP where the technology is implemented and this is the project area considered at this stage. The Cabezo Beaza WWTP has a surface of 488,000 m<sup>2</sup>.

Indicator code	Indicator name	Descriptor	Start project	End project	Updated End Project Value	New indicator descriptor	Beyond 3 years value	Updated Beyond 3 years Value
1.6	Humans (tobe) influenced by the project	Residents in the project area; (DEPRECATED)	0	14000	15,066	Persons who may have been influenced via dissemination or awareness raising project-actions (reaching)	98000	17,966

In the case of the **indicator 1.6**. the descriptor has been changed according to the webtool indications. The new descriptor is estimated in 15,066 persons since this is the number of people attending to dissemination activities organized by the project or where the project has been communicated. Beyond 3 years an estimated number of additional 2,900 people have been estimated, since this is the average number per year of targeted audience during the project.

Indicator code	Indicator name	Descriptor	Start project	End project	Updated End Project Value	Beyond 3 years value	Updated Beyond 3 years Value	Unit
2.1	Terrestrial extent affected by the pressure or risk addressed	Terrestrial extent affected by the pressure or risk addressed	130 // 4,300	129	4298.925	0	4,300	ha

For the **indicator 2.1**, a new calculation for the terrestrial extent affected is purposed according to the final location of the semi-industrial prototype. At the beginning of the project a different and smaller WWTP was used to estimate this indicator value since it was still not decided where to instal the prototype. Finally, it was installed in a bigger WWTP that provides reused water for a larger extension of crops. Project end value (1.075 ha) has been calculated proportionally to this surface (4,300 ha) and to the highest treatment capacity reached by the pilot plant (6 m<sup>3</sup>/day). Since the prototype will not be functioning in a continuous mode after the end of the project and there is any feasible replication scenario in the next 3 years, this value has been considered the same than at the project start.

Indicator code	Indicator name	Descriptor /New descriptor	Start project	End project	Beyond 3 years value	Unit
2.3.6	Point source pollution	CAS_135410-20-7 – Acetamiprid	9,320.20833	0	9,320.20833	µg/year
		CAS_138261-41-3 – Imidacloprid	200,860	0	200,860	µg/year
		CAS_2921-88-2 – Chlorpyrifos	50,722.222	0	50,722.222	µg/year
		CAS_723-46-6 – Sulfamethoxazol	38,041.6667	0	38,041.6667	µg/year
		CAS_738-70-5 – Trimethoprim	52,751.1111	7,354.7222	52,751.1111	µg/year
		CAS_15307-86-5 – Diclofenac	1,415.15	23.839444	1,415.15	µg/year
		CAS_22204-53-1 – Naproxen	68,475	0	68,475	µg/year

In the case of the **indicator 2.3.6**, the following descriptors corresponding to Emerging Pollutants found in treated wastewaters have been deleted because their presence were not recurrent in the samples analysed during the monitoring period: CAS\_52315-07-8 – Cypermethrin, CAS\_15687-27-1 – Ibuprofen and CAS\_29122-68-7 – Atenolol.

Also, at the beginning of the project, there was no data about the start project values of this pollutants because they are not usually analysed in WWTPs, so they are set with the final report.

The start and final values have been calculated based on the average concentration of each pollutant in the 36 samplings carried out and on the average flow treated by the pilot plant.

To calculate the total volume treated, the volume treated continuously from 3 February to 17 December 202 (317 days) and an average flow rate of 0.12 m3/h were considered. The final volume treated is 913 m3. It should be noted that the first two months of 2021, used for the start-up of the plant, have not been considered. Start Project values are based in the concentration of EPs found in the water samples taken from the outlet of the wastewater plant and before passing through the semi-industrial prototype. End project values are based in the concentration of EPs found in the water samples taken after passing through the adsorption columns and the photocatalysis equipment. Most elimination rates of the EPs found and reported under this indicator are 100% (5 from 7).

The presence of these compounds in wastewater is highly variable and depends on multiple factors such as: seasonality, area of influence of the WWTP (influence of industrial activities, agricultural or livestock activities, purely urban, etc.), regulations about active compounds (prohibition or restriction of use), development of new pesticides or pharmaceuticals, etc., which will mean variability, in turn, between geographical areas.

Since the prototype is not working in continuous mode any longer and there is not a clear vision for the transferability to other industrial sectors, the values beyond 3 years are the same than the initial ones.

Indicator code	Indicator name	Descriptor	Start project	End project	Updated End Project Value	Beyond 3 years value	Updated Beyond 3 years Value	Unit
4.1.3	Renewables production	Other	0	8,700	7,500	60,000	15,319	kWh/year

For the **indicator 4.1.3**, the renewable production during the project has been less than the expected value because the photovoltaic system was not working since the beginning of the prototype operation because it needed reparations after the installation and start-up of the prototype. This means that the photovoltaic system has served the 61% of the energy demand of the pilot plant during its 1 year of operation. The differences in the values beyond 3 years are explained because at the beginning of the project, it was considered the industrial scale-up of the system in a small-sized WWTP and, consequently, the energy demand was supposed to increase. However, this scaling up is not possible because the limited treatment flow of the system, and the renewable energy produced considered is that of the current energy system, which will be cede to HIDROGEA WWTP.

Indicator code	Indicator name	Descriptor	Start project	End project	Updated End Project Value	Beyond 3 years value	Updated Beyond 3 years Value	Unit
10.2.	Involvement of non- governmental	NGO	0	2	1	4	1	number of
	organizations (NGOs) and other	Public body/bodies	0	40	2	50	2	stakeholders involved due to the
	stakeholders in project activities	Private for profit	0	15	15	25	20	project

Regarding the **indicator 10.2**, values differences between established at the beginning of the project and at the end depend on the descriptor. For the number of NGOs involved it was only 1 compared with the 2 expected. This value is not expected to increase in 3 years. The public bodies involved have been only 2 corresponding to regional authorities. Municipalities and the river basin authority have not been involved as it was foreseen initially. This value is not expected to increase in the future. For the private entities involved, the initial value has been reached (15) and correspond to water companies reached through technical dissemination activities and companies involved in replication and transfer activities. For the next 3 years, 5

additional companies to assess possible transfer of the technology have been taken into account.

Indicator code	Indicator name	Descriptor	Start project	End project	Updated End Project Value	Beyond 3 years value	Updated Beyond 3 years Value	Unit
	website	No. of unique						Number of unique
11.1	(mandatory)	visits	0	2500	5175	5000	5500	website visits

The number of unique visits has overcome the value estimated at the beginning of the project. Beyond 3 years, the annual number of visitors is expected to decrease because there are no news about the progress of the project to feed the webpage with contents so frequently, so the total value would not increase so much related to the project end value.

Indicator code	Indicator name	Descriptor	Start project	End project	Updated End Project Value	Beyond 3 years value	Updated Beyond 3 years Value	Unit
		Number of different displayed information created (posters, information boards)	0	2	4	2	4	Number of outcomes (e.g. nr of reports, events, etc)
		Number of events/exhibitions organised	0	6	12	6	12	Number of outcomes (e.g. nr of reports, events, etc)
11.2.	2. Other tools for researching/raising awareness of the general public	Other distinct media products created (e.g. different videos/broadcast/leaflets)	0	1	10	1	10	Number of outcomes (e.g. nr of reports, events, etc)
		Number of articles in print media (e.g. newspaper and magazine articles)	0	4	10	4	11	Number of outcomes (e.g. nr of reports, events, etc)
		Number of different publications made (Journal/conference)	0	5	11	5	13	Number of outcomes (e.g. nr of reports, events, etc)

In the **indicator 11.2** the descriptor Number of Hotline/Information center created was deleted because there was any hotline foreseen during the project. A new descriptor "Number of different publications made (journal/conference)" have been included to better collect the activities implemented during the project, instead of the former "Publications/reports".

All the indicator values at the end of the project have surpassed initial values set. For the next 3 years, it is expected 1 additional article in print media and 2 additional publications in journals or conferences.

Indicator code	Indicator name	Descriptor	Start project	End project	Updated End Project Value	Beyond 3 years value	Updated Beyond 3 years Value	Unit
12.1	Networking	Professionals - experts in the field	0	14	28	20	28	No. of individuals

The number of professionals contacted for networking activities have been higher than the initial value set. By the end of the project 16 projects participated in the networking event organized by LIFE Clean Up on 1-2/06/2022 (with an average of 1 professional per project) and also 6 projects more have been known in networking activities organized by other projects or in LIFE Platform meetings (with an average of 2 professionals per project). In the future this number is not expected to increase but to conserve the relationship already established with professionals working in the water sector.

Indicator code	Indicator name	Descriptor	Start project	End project	Updated End Project Value	Beyond 3 years value	Updated Beyond 3 years Value	Unit
13	Jobs	Jobs	0	2	2	0	0	No. of FTE

The number of jobs correspond to the 2 FTE created in the University of Bari and CNR-IPCF during preliminary activities. However, these positions have not been maintained during all the project duration.

Indicator code	Descriptor	Start project	End project	Updated End Project Value	Beyond 3 years value	Updated Beyond 3 years Value	Unit
14.1	Running cost/operating costs during the project and expected in case of continuation/replication/transfer after the project period	0	1492512	1,558,052.68	8000000	1,574,533.87	€

The indicator 14.1 summarizes the total eligible costs expended during all the project. They have been higher than the proposed budget due to the project extension. The costs beyond 3 years includes the foreseen expenditure made by HIDROGEA for the maintenance of the pilot plant and by UCAM for the periodical sampling of wastewater (+16,481.19€).

Indicator code	Indicator name	Descriptor	Beyond 3 years value	Unit
14.2.2	Operating expenses expected in case of continuation/replication/transfer after the project period	Operating expenses expected in case of continuation/replication/transfer after the project period	16841.19	€

The continuation of the project is based in the waster sampling and analysis made by UCAM and the maintenance of the pilot plant by HIDROGEA. The value beyond 3 years are the expenditures estimated by these partners.

In the initial KPI forecast, the indicator 14.2.1, corresponding to the capital expenditure expected in case of continuation/replication/ transfer after the project period was also included. However, it has been deleted since this is considered 0 because it is not competitive to make any of these activities without a modification of the adsorbent. The amount considered at the beginning of the project was based on a preliminary business plan prepared for the project proposal in case the technology could be replicated just after the end of the project.

Indicator code	Indicator name	Descriptor	Beyond 3 years value	Updated Beyond 3 years Value	Unit
14.3.	Future funding Beneficiary own contribution		910000	16,481.19	€

Although transfer and replication studies have been done in other industrial sectors and geographical areas during the project, the **indicator 14.4.2** and the **indicator 14.4.3** that were included in the KPIs forecast have been deleted because the project is not expected to enter into new sectors or geographical areas in the next 3 years.

Regarding the project indicators collected in the KPI Excel file, those not included in the KPI webtool are commented as follows:

- **Reduction / substitution of dangerous substances Persistent /Bioaccumulative**: the estimated impact expected was 80% of removal rate of several EPs that were expected to be found in wastewaters. As commented in previous sections, although some drugs have resulted reluctant to the adsorption by the CDs polymer, the average elimination rate was 79.4%. Regarding phytosanitary compounds detected, they have been all completely removed. In addition, photocatalysis unit has eliminated further traces of EPs found after the adsorption unit. Consequently, the proposed solution is capable of eliminating an average of 80% of pharmaceutical compounds and 100% of phytosanitary products present in treated wastewater.
- **Improved water quality**: although the treatment capacity of the prototype at semiindustrial level was supposed to be 5 m3/h, and this would have made around 43000 m3/y and 14% the treatment volume of a WWTP for a small population (~14000 inhabitants), finally the treated flow was less. It only represents 0.71% of change having into account the maximum treated flow that has been 250 l/hour.
- **Energy form Renewable Energy Sources**: it has meant an impact of 61% instead of 100% because the photovoltaic system has not been able to supply the total amount of energy demanded by the prototype. The reason is that when starting up the pilot plant, the energy system did not function and had to be repaired, so the solar panels were not providing energy for all the operation time and just after the reparation was finished. The total amount of energy from renewable sources was 7,500 kWh/year. The potential of annual production is however higher than the initially estimated (15,319 kWh/year vs 8700).
- **N. of replication/transfer**: this number has increased from 5 to 7, considering 2 WWTPs, 1 company for treatment of industrial wastewaters, 1 company of citrus fruits and 3 dairy companies.
- **Number of entities/individuals changing behaviour**: the initial value of 15 has increased up to 20, considering the 5 entities that have participated in technical visits, 8 bodies/companies related to the water sector that have received information about the project, and 7 companies participating in transfer/replication activities.
- **Number of individuals reached by the media**: this number has reached 171,070 individuals (above the initial 107,000), considering 160,000 interactions in the project social networks and 11,070 visits to the webpage.