



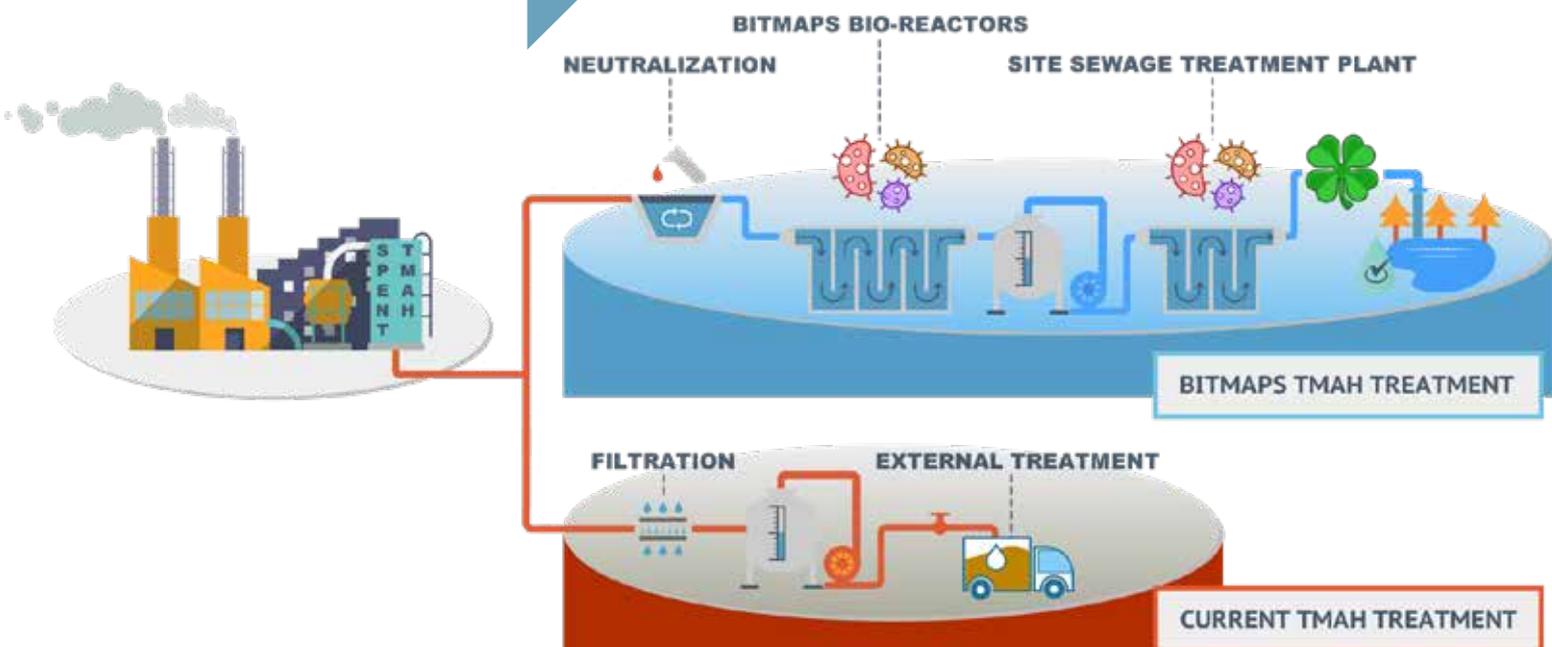
LIFE BITMAPS



This Project receives funding from the European Union Life Program Under Grant Agreement N. LIFE 15 ENV/IT 000332

LAYMAN'S REPORT

Pilot technology for aerobic Biodegradation of spent TMAH Photoresist solution in Semiconductor industries



PROJECT SUMMARY

Scientific tests carried out operating a pilot plant installed at a semiconductor facility, have demonstrated the technical and economic feasibility as well as the environmental sustainability of an innovative process for the simultaneous treatment of three spent solutions with high concentration of pollutants (i.e. tetramethyl ammonium hydroxide (TMAH), Ammonium fluoride, acids mix) typically resulting from the production processes adopted by semiconductor industry. In particular, advanced treatments of industrial wastewaters containing TMAH represent a key challenge for the European E&S (Electronic and Semiconductor) industry that must be improved to safeguard surface and groundwater quality and reduce waste disposal activities.

BITMAPS project ultimately aims to apply the achieved outcomes to the E&S industry and to other “downstream” industry sectors utilizing the same hazardous chemicals difficult to remove or degrade. In particular; the achieved results allow to eliminate the expensive waste disposal for TMAH solutions (hereafter TMAH/PR where PR stands for photoresist) by adopting an integrated chemical-biological process interconnected with the pre-existing wastewater plants at the industrial site capable to transform the pollutants into nitrogen and carbon dioxide with a significant reduction of the environmental impacts.

PROJECT GOALS

Develop a sustainable technology, interconnected with the industry wastewater treatment plants, having the capability to provide an effective abatement of TMAH at the final discharge through aerobic biodegradation and simultaneous chemical-physical treatment of other wastewater streams resulting from the semiconductor manufacturing processes.

Facts about BITMAPS project

- A recent market report show that the global Tetramethyl Ammonium Hydroxide market size will increase to 1180 Million US\$ by 2025, from 950 Million US\$ in 2018, at a CAGR of 3.2% during the forecast period
- Worldwide production of TMAH as a pure substance increased from 190386 MT in 2014 to 252089 MT in 2018.
- GHS (Echa source) classifies TMAH as Hazardous to the aquatic environment (long-term) with
 - Hazard category: Aquatic Chronic 2
 - Hazard statement: H411: Toxic to aquatic life with long lasting effect
- The Istituto Superiore di Sanità (ISS) (“National Institute of Health” acting as technical-scientific body under the supervision of the Italian Ministry of Health) in 2013 recommended to target a value of 0.4 mg/L in sewage and a value of 0.2 mg/L in water bodies as discharge limits.
- For the sole semiconductor sector about 40 facilities operating in EU using TMAH. They are many more considering other industry sectors.

PROJECT BACKGROUND

AND THE ENVIRONMENTAL PROBLEM TARGETED MARKET

A large amount of developer is used by the manufacturing of electronics & semiconductors (E&S) industries. A high alkaline matter is the main ingredient of developer: TMAH solutions (CAS NUMBER 75-59-2) are usually used. This substance is a colourless to light yellow solution and due to its highly alkalinity and toxic properties, there were several accidents of poisoning and deaths even for exposures with diluted formulations.

The ecotoxicity of TMAH is reported by Echa website (Registration dossier (<https://echa.europa.eu/it/registration-dossier/-/registered-dossier/10763/2/1>) which identifies it as "Hazardous to the aquatic life with long lasting effects" (H411). Additionally, no emission limit value (i.e. concentration at discharge) has been set for wastewater containing TMAH by EU regulation. However, the Directive 2010/75/EU on industrial emissions and the EU Water Framework Directive 2000/60/EC (Annex VIII) provides the list of polluting substances that affects the water quality: among those, "Substances which have an unfavourable influence on the oxygen balance (and can be measured using parameters such as BOD, COD, etc)" and "Substances which contribute to eutrophication (in particular, nitrates and phosphates)" and TMAH falls under both categories.

As foreseen by the EU Water Framework Directive 2000/60/EC and considering a value for NOEC (No Observed Effect Concentration) to prevent chronic toxic effects on invertebrates (*Ceriodaphnia dubia*, *Daphnia magna*) equal to 0.02 mg/L, the Istituto Superiore di Sanità (ISS) - "National Institute of Health" which is the technical-scientific body under the supervision of the Italian Ministry of Health - in 2013 recommended to target a value of 0.4 mg/L in sewage and a value of 0.2 mg/L in water bodies as discharge limits.

Regional Agency for the Environment Protection of Abruzzo region has transposed the technical note of ISS and recommended LFoundry (semiconductor industry) to investigate viable solutions for reducing the TMAH concentration on its plant. The problem is clearly of European relevance, in fact many industries synthesize and use TMAH for several industrial applications. Advanced treatment of this kind of industrial wastewaters process solutions represent a key challenge for the European E&S industry that must be improved for safeguard of surface and groundwater quality. Worldwide production of TMAH as a pure substance increased from 190,386 MT in 2014 to 252,089 MT in 2018.

Just to mention some applications, TMAH is used as developer and silicon etchant in the electronics and semiconductor industry; metal manufacturing as aqueous cleaners; plastic manufacturing as additives; resource and extraction of gas; in a variety of products and industries as raw materials (e.g. in cosmetics, chemical manufacturing, production of metals, etc); as oxidation agent; and Ink, toner, and colorant products.

Taking into account the size of E&S European industrial sector, it can be estimated that **more 250,000 tons /year** of diluted TMAH solutions are used and more than 2mln tons/year by the E&S Worldwide sector. Hence, a very relevant amount of wastewater containing TMAH is generated worldwide only considering the E&S industry.

Considering the above depicted scenario, the aim of the LIFE BITMPAS has been to investigate a technical and economic approach to treat TMAH solutions generated by LFoundry in order to meet the ISS and recommendation; moreover other scope has been to develop a technology that can be applied by other E&S companies or other industries that synthesize and use the same hazardous pollutant.



BITMAPS PILOT PLANT

BITMAPS CONSORTIUM

AND APPLICATION TO LIFE PROGRAM

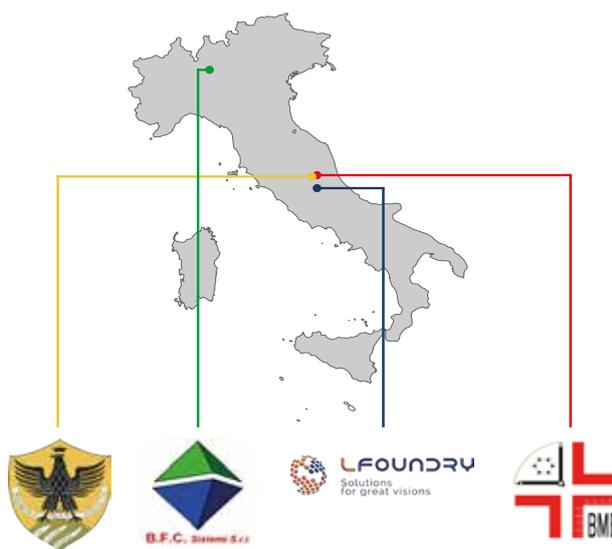
Considering the above scenario, the originating purpose of LIFE BITMAPS is that to define a technical and sustainable technology to treat TMAH/PR solutions generated by LFoundry in order to meet the ISS requirements. On the other side the project provides a solution which might be applied by the semiconductor and Electronic industrial sector and other industries which use or synthesize TMAH. The project has been carried out through a Consortium composed by the following partners operating in Italy:

LFoundry S.r.l. semiconductor site based in Avezzano (AQ) (Coordinating beneficiary)

B.F.C. Sistemi S.r.l. based in Busnago (MB) design a built chemicals plants (beneficiary)

UNIVAQ – University of L'Aquila (beneficiary)

BME Biomaterials & Engineering S.r.l. Spin-off from L'Aquila University (beneficiary)



The demonstration activity was originated by a previous cooperation between UNIVAQ and LFoundry that participated to another European Project (Silver) with similar purposes of LIFE BITMAPS but limited to a lab scale research. The biodegradability of TMAH was demonstrated during the lab scale activity. Based on the encouraging results, it has been decided to set up a new consortium to check the technical and economic feasibility of the process in order to demonstrate at industrial scale the feasibility/sustainability for TMAH treatment through an aerobic biodegradation and finally to propose the technology as BAT for the treatment of wastewater coming from E&S industries.

OBJECTIVES

The LIFE BITMAPS project pursues the construction and operation of a pilot plant to demonstrate a new and never-scaled process for the treatment of effluents from electronics and semiconductor manufacturing.

The project contributes to the implementation of the European Union's Water Framework Directive (WFD) by introducing more efficient treatment technologies that help reduce TMAH pollution at source. By recycling wastewater, it is also demonstrated the application in practice of the circular economy priority of water reuse and savings in industrial processes. Moreover, in proposing a more efficient, effective and innovative solution for industrial wastewater treatment, the project is also contributed to one of the priority areas of the European Innovation Partnership (EIP) on Water.

The project's specific objectives are to:

- Design, construction and validation of a semi-industrial pilot plant enabling the treatment of spent photoresist-tetramethylammonium hydroxide (PR/TMAH), and other

mixed solutions (ammonium fluoride and acid mixtures internally named "SEZ and BOE") generated by the semiconductor manufacturing processes.

- Demonstrate, at industrial scale, the feasibility/sustainability for TMAH treatment through an aerobic biodegradation by adapted microorganisms selected during the R&D phase.
- Prove the cost sustainability of the process, in a LCC perspective, also taking into account the actual annual operating costs for the disposal of concentrated TMAH and the other waste solutions.
- Pave the way for replication and transfer of the results to E&S Sector.
- Propose new quality standards for wastewater discharged helping policy makers to fill the regulatory gaps on TMAH emissions.

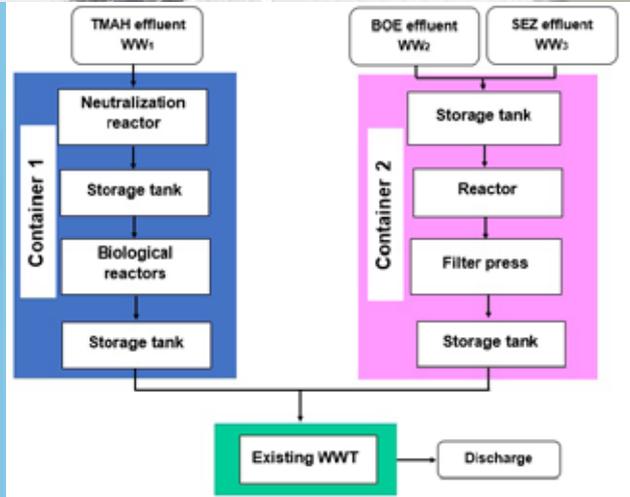
PROJECT ACTIVITIES

ADOPTED TECHNOLOGIES AND PILOT PLANT CONSTRUCTION

Besides the TMAH treatment with an aerobic biodegradation, it has been also decided to verify the possibility to treat some selected spent solutions (BOE and SEZ) having high concentration of pollutants (i.e. fluorides, inorganic and organic acid) by chemical processes with the aim to reduce their toxicity and to manage the streams sending them to the wastewater treatment plant already operating on the industrial site in LFoundry. After a laboratory scale optimization, it has been defined the technical solution including mass and energy consumptions to treat the three effluents. The solution adopted for TMAH/PR wastewater has been a biological degradation carried out by adapted microorganism collected from activated sludge of the biological plant of the site.

For the other two effluents (BOE and SEZ) the solutions adopted have been chemical treatments by addition of precipitating agents. After treatment, the effluents can be safely sent to biological wastewater plant operating at the industrial scale.

The Life Bitmaps pilot plant has been built in two containers installed at the Lfoundry site. After a design optimization for the processes, the first treatment section has been realized with three reactors in series having a capacity of 1.1 m³ aimed to achieve an aerobic biodegradation TMAH/PR (WW1) within the Container 1. Instead the section for the treatment of BOE and SEZ (WW2 and WW3) is composed by a chemical reactor and a filter press placed in the Container 2. The first treatment line works in continuous mode with a maximum capacity of TMAH/PR effluent of 25 L/h, instead the other section works in batch mode treating WW2 or WW3 streams on alternate basis with a maximum capacity of 180 L/batch.



Biological reactor ↑



Filtration system →



← Chemical reactor

Scrubber system for emission abatement ↓



OUTCOMES FROM THE EXPERIMENTAL STAGE

BIOLOGICAL TREATMENT FOR TMAH PHOTORESIST SOLUTION DEGRADATION

Initially the biological reactors have been inoculated by active sludge from the existing biological wastewater plant operating at the industrial site. Therefore TMAH/PR effluent has been fed varying the flow rate (5-20 L/h) with the aim to stress and investigate either the response of the microorganisms and of the whole system as well.

More than 50 wastewater samples have been collected during the pilot tests generating about 800 chemical analysis for the following monitored parameters:

- Chemical oxygen demand (COD)
- pH of the suspensions inside the biological reactors
- Total suspended solids (TSS)
- Tetramethylammonium hydroxide (TMAH) and its intermediates of degradation (Dimethylamine, DMA; ammonium ions, NH_4 ; nitrate, NO_3)

Moreover, a biological characterization of the samples has been carried out in order to track the transformations and select the bacteria population capable of adapting to TMAH. Reactor R101, R102 and R103 have shown the proportions in terms of bacteria communities for each operative condition (i.e. varying the treatment flow rates). Syntrophic biodegradation of tetramethylammonium is driven by different families of gram-negative Bacteria all of them methylotrophs, providing specific features and resistant characteristics.

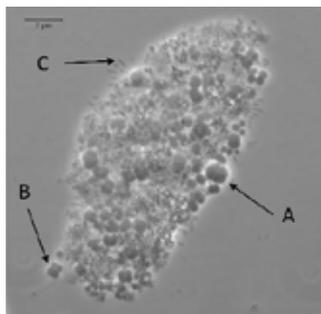
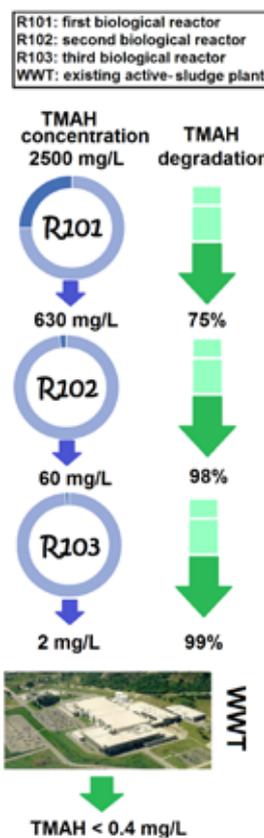


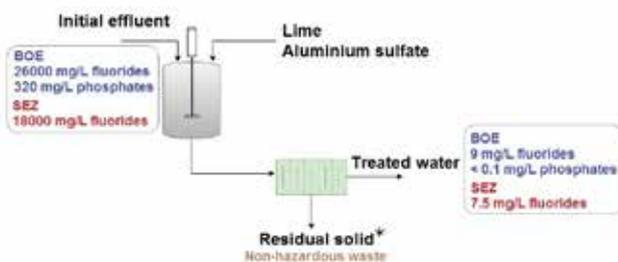
Photo realized on Leika2000 microscope of a microbial aggregate grow on TMA, where there are recognizable different bacterial shapes: A: Sarcinas B: Cocci C: Rods

The following figure shows that a 99% degradation has been obtained through the proposed treatment executed within the Container 1 with a process time of 192 hours. After that the treated effluent has been sent to the operating active sludge plant at the facility (featured by an additional process time of 36 hours) observing a further degradation for TMAH at its final discharge.



Chemical treatment for SEZ and BOE Pilot plant tests period: June 2018 - December 2018

As regard the treatment of two streams WW2 (BOE) and WW3 (SEZ) by adopting the process drawn in the attached diagram, a total abatement of fluorides and phosphates more than 99% of the initial concentration has been obtained.

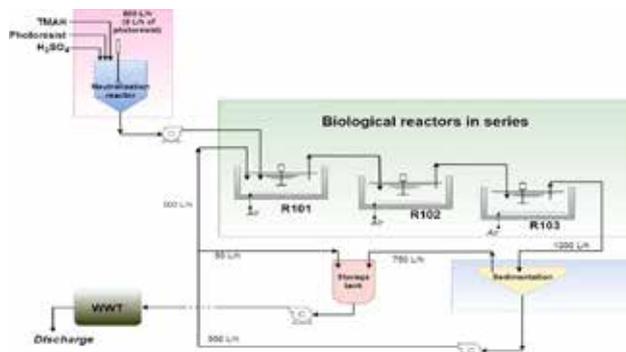


Flowsheet for the treatment of BOE and SEZ effluents (*) Under investigation for possible recovery as fiberglass, ceramic, glass and aluminium metallurgy

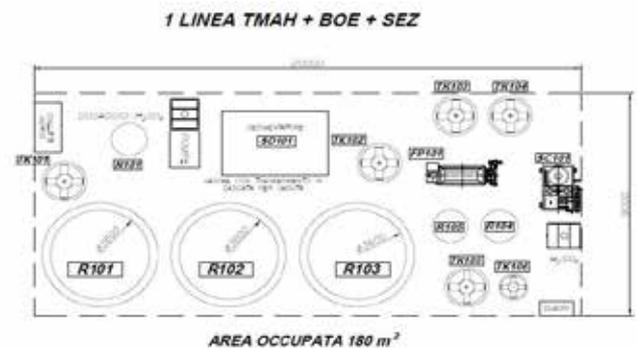
BUSINESS CASE CONFIGURATION FOR THE PROPOSED TECHNOLOGY

Several options have been tested in order to define the optimal configuration of the full-scale plant for biodegradation of TMAH/PR solution. Assuming a flowrate of 800 L/h it has been confirmed the technical solution proposed with the pilot plant adopting three bioreactors in series but with partial recirculation of bacteria in R101 (see the following scheme).

Reactors have a total volume of 89.52 m³ (volume reduction of 90% compared to the option adopting a single reactor). This line works in continuous mode and this is designed to treat a total amount of TMAH/PR wastewater equal to 6336 ton/y (a basis of 330 days per year).



Flowsheet for the treatment of TMAH effluent.



Layout of the industrial Life Bitmaps plant

Instead for BOE and SEZ the operations are in batch and the total amounts of effluent treated are 435 ton/y and 145 ton/y respectively.

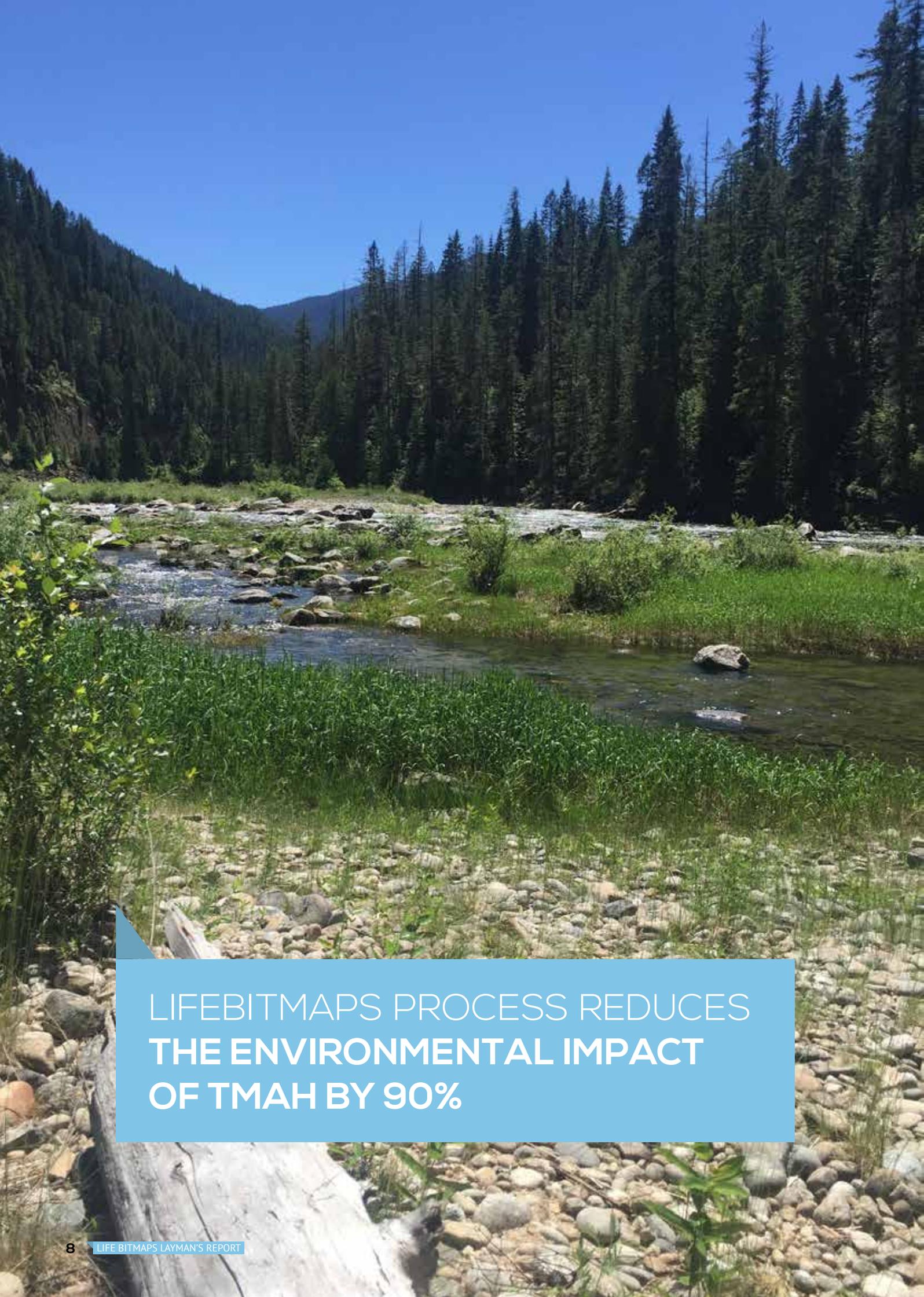
Operating Expense (OPEX) contributions for the three lines of treatment are shown in the following Table. For the treatment of TMAH/PR solution, power consumption is the main cost item, while for BOE the disposal cost of the residual solid and the personnel cost are the masters instead for SEZ the main cost is the one associated to personnel cost followed by the disposal cost of the resulted waste.

Life Bitmaps processes reduce significantly the actual costs (treatment to concentrate and disposal for TMAH/PR and just disposal for BOE and SEZ).

Input	BOE	SEZ
Initial effluent, kg	100	100
Aluminium sulfate, kg	3	3
Lime (16%), kg	90	145
Output		
Residual solid – Non-hazardous waste, kg	17	22
Treated water, kg	176	226

	TMAH	BOE	SEZ
Actual option cost (€/m³)	46	254	582
Bitmaps cost (€/m³)	15	110	207
Raw material (%)	4	16	9
Personnel cost (%)	21	41	67
Disposal cost (%)	0	41	23
Energy (%)	75	2	1

Comparison between the current and the innovative options (OPEX)



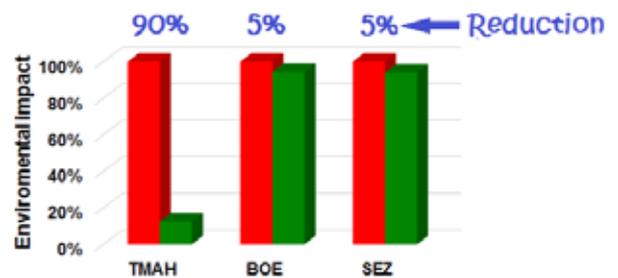
LIFEBITMAPS PROCESS REDUCES
**THE ENVIRONMENTAL IMPACT
OF TMAH BY 90%**

ENVIRONMENTAL BENEFITS

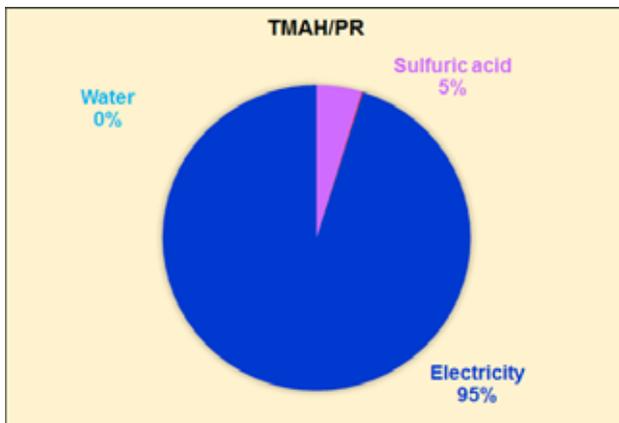
The Environmental benefits of the Project have been evaluated taking into account the mass and energy balances computed for the full scale facility through a life cycle impact assessment (LCIA).

The study has been carried out by the thinkstep GaBi software-System and Database for Life Cycle Engineering, used for the production processes of energy and raw materials and the quantification of the environmental impact of the treatments, following the recommendations of ISO 14040 and 14044:2006 norms.

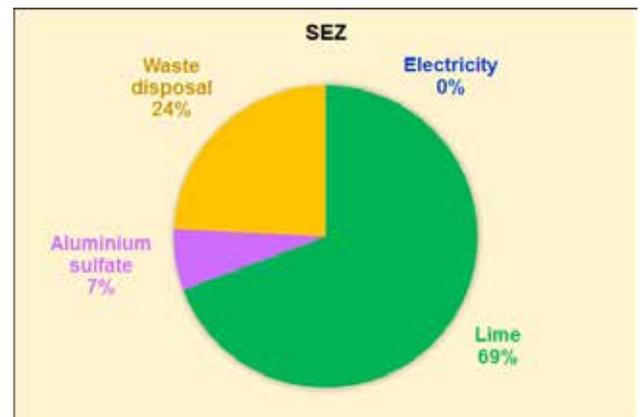
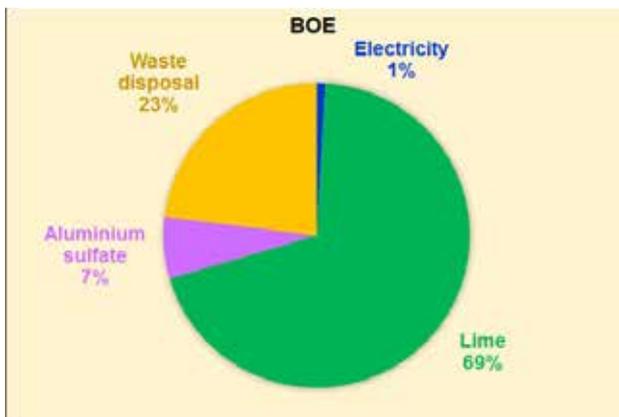
LCIA study includes both the classification and characterization steps and the normalization and weighting phases. The following Figure compares BITMAPS proposed processes and the current options in terms of total environmental loads assuming as reference (100%) the actual disposal and treatment (for TMAH/PR only) approach.



COMPARISON IN TERMS OF ENVIRONMENTAL IMPACT AMONG THE CURRENT DISPOSAL TECHNOLOGY AND BITMAPS



The following pie charts report for each treatment line the process contributions on the normalized and weighted results using as functional unit the annual production of wastewater for each line (WW1, WW2, WW3). For TMAH/PR solution treatment, the electricity consumption represents the main contribution on the whole environmental load (higher than 90%). On the other hand, lime for the precipitation causes around the 70% of the impact associated to the BOE and SEZ treatment.



From the adopted methodology, the resulting main impact categories are respectively: Climate change, ionizing radiation and resources use, mineral and metals.

COMMUNICATION AND DISSEMINATION

SEVERAL COMMUNICATION AND NETWORKING ACTIVITIES HAVE BEEN DONE IN ORDER TO DISSEMINATE THE RESULTS AND TRANSFER THE KNOWLEDGE GATHERED TO END USERS AND STAKEHOLDERS.

WEBSITE

www.lifebitmaps.eu includes the updates on research activities to which, since it was created until the end of 2019, more than 2400 different visitors from 83 countries have accessed, for more than 5000 sessions for a total of almost 11500 pages displayed.

WORKSHOPS

LFoundry Srl, Avezzano, September, 2020 - Final conference and pilot plant visit.

University of L'Aquila, May 31, 2017 - Sinergie tra ricerca e industria. Publication of the workshop on University Socials (Facebook page, web page) and on local newspaper.

Rome July 24th, 2017 - Presentation of the Project at the Final Conference of the Photolife Project ("Process and automated pilot plant for simultaneous and integral recycling of different kinds of photovoltaic panels").

L'Aquila September 29th, 2017 - Exhibition "Street Science".



PARTICIPATION IN CONFERENCES

9th CISAP, International Conference on Safety & Environment in Process & Power Industry, Settembre 2020, Venezia, Italy. V. Innocenzi, I. De Michelis, M. Prisciandaro, G. Iuliano, F. Vegliò, Safety analysis of industrial wastewater pilot plant for the removal of pollutants from microelectronic industry effluents.

ISER- International Conference on Chemical and Environmental Science (ICES-2019), 2019, Auckland, New Zealand. F. Vegliò, V. Innocenzi, I. De Michelis, S. Zueva, F. Ferella, M. Prisciandaro. Integrated biological and chemical process to treat wastes of electronic industries: the European Project Life Bitmaps.

16th International Conference on Environmental Science and Technology CEST2019, 2019 in Rhodes, Greece. V. Innocenzi, I. De Michelis, N. M. Ippolito, G. Mazziotti Di Celso, M. Prisciandaro, F. Vegliò, Aerobic degradation of tetramethyl ammonium hydroxide (TMAH) from effluents of semiconductor industries: kinetic studies of laboratory and pilot experiments.

Convegno Gricu, Il contributo dell'Ingegneria Chimica Italiana alla sostenibilità globale, 2019 in Palermo, Italy. V. Innocenzi, I. De Michelis, N. M. Ippolito, S. Zueva, M. Prisciandaro, F. Vegliò. Processi a basso impatto ambientale per la degradazione di tetrametil-ammina.

International Conference on Chemical and Biochemical Engineering (ICBE), 2019 in Manila, Philippines. V. Innocenzi, I. De Michelis, S. Zueva, V. Corradini, N. M. Ippolito, F. Vegliò, Biodegradation of TMAH (Tetramethyl ammonium hydroxide) in semiconductor industries (Life Bitmaps project): pilot plant design, preliminary tests and process analysis.

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Eighth World Congress and Expo on Recycling, 2018 in Berlin, Germany. V. Innocenzi, I. De Michelis, F. Tortora, M. Prisciandaro, F. Vegliò, Treatment of wastewater from microelectronic industry: process analysis of a combined process scheme.

Ninth Carbon Dioxide Utilisation Summit, 2017 in Reykjavik, Iceland. M. Saraullo, I. De Michelis, F. Vegliò, LIFE BITMAPS project for the biodegradation of Tetramethylammonium Hydroxide (TMAH) in the wastewaters of semiconductor production: neutralization of waste by CO₂ in lab and pilot scale

EESD2017, International Conference on Energy, Environment, and Sustainable Development, 2017 in Phuket, Thailand. I. De Michelis, A. Di Renzo, M. Saraullo, F. Vegliò, Kinetic study of aerobic degradation of tetramethylammonium hydroxide (TMAH) at room temperature.

Sixth International Conference on Environmental Management, Engineering, Planning and Economics and SECOTOX conference in Thessaloniki, 2017. M. Prisciandaro, V. Innocenzi, I. I. De Michelis, F. Tortora, G. Mazziotti di Celso, F. Vegliò, Treatment of TMAH solutions from microelectronic industry: a combined process scheme. Treatment of wastewater from microelectronic industry: process analysis of a combined process scheme

Conference and school GRICU, 2016 in Anacapri (Napoli, Italy). M. Prisciandaro, F. Tortora, V. Innocenzi, Treatment of microelectronic wastewaters with Meuf: recovery of surfactant.



PUBLICATIONS ON INTERNATIONAL SCIENTIFIC JOURNALS

V. Innocenzi, S. Zueva, M. Prisciandaro, I. De Michelis, A. Di Renzo, G. Mazziotti di Celso, F. Vegliò, Treatment of TMAH solutions from the microelectronics industry: A combined process scheme, *Journal of Water Process Engineering* 31 (2019) 100780

F. Ferella, V. Innocenzi, S. Zueva, V. Corradini, N.M. Ippolito, I.P. Birloaga, I. De Michelis, M. Prisciandaro, F. Vegliò. Aerobic Treatment of Waste Process Solutions from the Semiconductor Industry: From Lab to Pilot Scale, *Sustainability* 11 (2019), 3923

V. Innocenzi, M. Prisciandaro, F. Vegliò. Effect of the Hydrodynamic Cavitation for the Treatment of Industrial Wastewater, *Chemical Engineering Transactions* 67 (2018)

EU ADDED VALUE, IMPACTS AND REPLICATION OPPORTUNITIES

Bitmaps aims to bring an innovative and sustainable technology to the attention of the market thus addressing the goals to the EU sustainability policy and building synergies with the objectives of CSR (Corporate Social Responsibility) policy promoted by EU.

Major benefits in terms of water quality improvement because of much lower concentration of harmful chemicals contained in the wastewater discharged by the E&S industry. This represents a positive impact on the water bodies as well as on the ecosystem in the surrounding territory.

TMAH falls under the categories “Substance which have an influence on the oxygen balance” and “Substance which contribute to eutrophication” according to Directive 2010/75/EU and EU Water Framework Directive 2000/60/EC. BITMAPS demonstrates that a very low concentration value TMAH is achievable at discharge by employing more effective treatment methods thus stimulating EU authorities to set up quality standards and guidelines for wastewater discharged by E&S industry.

Given that the developed process has been patented (application number 102017000003185) in Italy and the patent extension at EU level is in progress, in the light of the demonstrated results, BITMAPS makes available an effective reference to set up a possible standardized technology for TMAH degradation.

Moreover, interaction and initiatives are in full progress with the environmental national technical body as well as regional and national environmental authorities to pursue the recognition of the developed technology as “emerging technology” (according to the definition reported in the Art. 3 of the Directive 2010/75/UE). In case the above recognition would be achieved, the technique will have the opportunity to be selected as BAT (Best Available Technology) within the reference BREF for the treatment of wastewater containing TMAH

deriving from the E&S sectors and similar.

LCA and LCC analysis qualify the process from the technical, environmental and economical perspectives by demonstrating the viability and sustainability of its implementation for the E&S industry and other industries using TMAH.

The success of the project will also contribute to increase the attention to an important European industrial sector (E&S) that annually generates relevant amount of liquid and solid waste. For example, at the coordinating beneficiary site (LFoundry) a total of annual amount of about 7,000 tons of wastewater containing TMAH.

In the semiconductor field there are about forty facilities over the EU and many more worldwide where a similar treatment plant could be installed given the huge amount of wastewater they generate. In addition, European E&S Industries have significant and strict policies regarding environmental performance, so that they can be interested in the improvement of management of spent TMAH by eventually adopting the innovative technology proposed by BITMAPS. Furthermore, other industry sectors using TMAH might find attractive the BITMAPS technology.

Particular emphasis has been dedicated to explore replication opportunities. A specific Business model upon a dedicated market analysis has been arranged in order to carry on the appropriate commercialization initiatives for TMAH treatment technology after the conclusion of BITMAPS project. The Business model includes the supplying of R&D services and construction of industrial TMAH treatment plant. Although no commercial contract has been signed with industrial customers during project duration, there are on-going discussions with different players of E&S sector that could be interested to adopt the BITMAPS technology.



PILOT TECHNOLOGY FOR AEROBIC BIODEGRADATION OF SPENT TMAH PHOTORESIST SOLUTION IN SEMI-CONDUCTOR INDUSTRIES

Project start date: 01/07/2016

Project end date: 30/09/2020

Total Budget: € 1,676,923, co-financed at a rate of 60% by LIFE programme of European Commission

Project ref: LIFE 15 ENV/IT 000332

Beneficiaries:

Coordinating Beneficiary

LFoundry Srl (Avezzano (AQ), Italy)

Associated Beneficiaries

University of L'Aquila UNIVAQ (L'Aquila (AQ), Italy)

BME Biomaterials & Engineering S.R.L (L'Aquila(AQ), Italy)

BFC Sistemi Srl (Busnago (MB), Italy)



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For more information about BITMAPS visit the web site:

WWW.LIFEBITMAPS.EU