A purification/regeneration process of spent plating bath based on functionalized magnetic nanoparticles

PureNano project develops a fast and low-cost method for purification of spent plating baths, promoting aspects of circular economy and reuse of secondary raw materials, as plating solutions and metal ions.

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€4.2M Budget

6 Countries

12 Partners



3 Years



AXIA INNOVATION

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### 1. MISSION

Electroplating and electroless plating, are two processes used to improve the corrosion resistance of a material. its strength and hardness and improve its aesthetic appearance. The global sales of the Electroplating industry are estimated to increase with a CAGR of 3.9% over the next ten years, due to the increase in demand from different end-users across the world. Automotive. electrical and electronics. aerospace and defence and jewellery are the main market segments requesting plating industry products due to their longer lifespan and usability.

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One of the challenges of the plating industry is the production of hazardous waste. Every year a total amount of 300.000 tons of hazardous waste is produced per year (an average of 16 tonnes per installation). The plating process is performed by using an appropriate plating bath containing the metal salts to be deposited plus various other chemicals that are fundamental to control the properties and the quality of the coating.

The operation of a plating bath leads to perturbation of the proper concentration of these chemicals due to their consumption, and the production of by-products through parallel reactions or decomposition routes. When the concentration of these species is higher than a certain limit, the baths are not available for further use (spent bath) and should be removed and transferred to recycling centres to recover the metal ions through high cost and environmentally risky methods in order to be re-used.



The PureNano process offers a sustainable, low-risk, economic solution for the purification/ regeneration of the plating bath, by using Magnetic Nanoparticles (MNPs), leading to an extension of up to 10 times of the life of the bath. The PureNano project will develop a system that will allow the purification/regeneration of the bath in -situ, i.e. where the plating process takes place. This will reduce the risk and the costs related to the transport of hazardous waste.

## 2. PARTNERS

PureNano project involves partners from different sectors (7 industrial partners-SMEs, 4 RTDs, 1 Association) that collaborate for the project's purposes and their expertise in different disciplines is fundamental for project's implementation.



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Politecnico di Milano (PoiliMi) is the coordinator of the project leading WP1. PoliMi contributes to the production and characterization of magnetic nanoparticles (MNPs), their functionalization, the set-up of the pilot line, the LCA/LCC analysis and the development of the IPR strategy.

www.polimi.it

Captive Systems is a spin-off company of the Politecnico di Milano and in collaboration with PoliMi has developed the technology of using MNPs for the recovery of the metals and the water treatment. Captive will contribute to all WPs, providing specifications concerning the MNPs, will be responsible for the upscaling of the MNPs production process and will work closely to the other partners for the development of the pilot lines. www.captivesystems.it





RISE is the Research Institutes of Sweden and will focus on formulation and to the formulation and functionalization of MNPs. RISE is leading the task related to the development of targeted coatings and contributes to several WPs. They contribute to the dissemination and IPR protection activities.

www.ri.se

NTUA is the oldest and most prestigious technical university in Greece. They contribute to the characterization and evaluation of the functionalization of the magnetic particles and are involved in the development of a methodology for the recycling and reuse of MNPs. They contribute to the dissemination and IPR protection activities.



### 2. PARTNERS

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Tecnochimica is one of the major suppliers of chemicals and plating baths in Italy. Tecnochimica as a producer of plating baths has proprietary formulations for both electroless and electrolytic baths and contributes to understand the chemistry of the bath and to perform compatibility tests for the MNPs. They contribute to the dissemination and IPR protection activities.

IDENER is a research SME active in the field of Computational Science. IDENER will have the role of designing the purification system which is manufactured by Kampakas and will be installed at the premises of Gaser and Cnano. They contribute to the dissemination and IPR protection activities.

www.idener.es

GROUP KAMPAKAS Kampakas SA is a leading company in the production of wearresistant alloys used in various industries. Within PureNano consortium, Kampakas will give specification regarding the purification systems and will manufacture the units to be installed in the pilot lines of Gaser and Cnano. They contribute to the dissemination and IPR protection activities. www.gmc.gr

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ISQ is an international, independent, private, non-profit, technical, scientific and industrial oriented organization. Within PureNano consortium, ISQ is leading the WP related to Life Cycle Analysis, Life Cycle Costs, and will contribute to the Health and Safety and Safeby-Design activities. ISQ contributes also to the recyclability, circular economy and dissemination activities.

## 2. PARTNERS



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Cnano is a research and commercialization SME focusing on technological product-based solutions, specialized in the field of nanotechnology. Within PureNano consortium, Cnano is responsible for providing the specifications of the chemistry of the plating bath and will provide the pilot-scale facility to test the purification system. Cnano will also contribute to other activities such as the LCA, LCC, dissemination and IPR protection.

Gaser Ossido Duro provides plating stations for electroless nickel plating and electroplating and is the second end user that will provide a pilot-scale facility to test the purification system. Gaser is involved in the development of an in-line apparatus/method for bath regeneration/purification. Gaser will also contribute to the dissemination and IPR protection activities.

www.gruppogaser.com



Axia Innovation offers services specialized in accompanying and supporting companies in all phases of business development and product's commercialization. AXIA is leading WP9 that concerns the dissemination and exploitation activities. AXIA will be responsible for the patent search as well as the IPR handling (e.g. patents, industrial secrets, trademarks) and therefore will be in contact with all the project partners. www.axia-innovation.com

ASFIMET is a no-profit organization, open to all operators of the Surface Finishing market segment. ASFIMET a known and recognized surface finishing association in Italy, is leading the activities related to the standardization activities ensuring the compliance of the materials and processes to the current market status. ASFIMET will also contribute to the dissemination and exploitation activities.

www.galvanotecnica.org



Gaser Ossido Durc



## 3. WORKPLAN

#### WP9: Dissemination

and exploitation (M1-36), Leader: AXIA WP1: Management and coordination (M1-36), Leader: PoliMi

WP8: LCA/LCC (M1-36), Leader: ISQ WP2: Generation of specifications & Requirement (M1-18), Leader: Gaser

WP7: Industrial Implementation Issues (M1-36), Leader: Asfimet WP3: Magnetic NPs development (M5-18), Leader: Captive

WP6: Demonstration activities (M19-36), Leader: Cnano

WP5: Integration of purification system and safe disposal of MNP (M5-36), Leader IDENER WP4: Functionalization of MNPs (M12-25), Leader: RISE

Magnetic Core

μm

**Outer Coating** 

During the first half of the project duration, several objectives have been accomplished with some WPs already completed and others recently launched. Below a short summary of the main achievements for each WPs.

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#### WP2: GENERATION OF SPECIFICATIONS & REQUIREMENTS (M1-18), LEADER: GASER

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The main objectives of the WP2 is to define requirements and specifications of raw materials, purification systems and demonstrator based on endrequirements. The magnetic user nanoparticles (MNPs) are composed of a core made out of a ferromagnetic compound, mainly iron oxides, and they are functionalized with an external coating. The formation of magnetic functionalized nanoparticles is a onestep process, where chemical coprecipitation is combined with in-situ surface functionalization with specific molecules. Captive is able to create different types of coating (anionic, cationic and lipophilic) able to adsorb anions, cations and organic substances. The raw materials used for the MNPs production have been listed by Captive and the main risk associated with their use specified, although none of them



#### **WP3: MAGNETIC NPS DEVELOPMENT** (M5-18), LEADER: CAPTIVE

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The activities within WP3 are focused on the design and modification of the MNPs production, optimization of the nucleation and growth of aggregates and control of the aggregation process of the particles. Captive has developed a pilot plant for the production of magnetic functionalised nanoparticles with a capacity of 50 kg An important characteristic per day. of this pilot is its versatility to produce various coatings, without altering the final syntesis' efficiency and final product quality. Several tests have been already performed and the pilot production plant is able to provide particles with high reproducibility in terms of size distribution. Since the MNPs are produced through a coprecipitation reaction, the

#### **WP4: FUNCTIONALIZATION OF MNPS (M12-25)**, **LEADER: RISE**

The objective of this work package is the development of an appropriate coating for capturing metallic and organic impurities within the plating

#### **4. ADVANCEMENT**

final product will be a dispersion of magnetic particles in water. It has been decided to use the MNPs in a dispersion form in order to comply with Health and Safety regulations. Nevertheless, Captive, in collaboration with PoliMi, investigated three different methods for producing dry MNPs for commericalization purposes. Drying in a vacuum oven and mechanical grinding, freeze-drying and spray drying were tested. Compared to freeze-drying and spray drying, so far the first method did not give satisfying results regarding the morphology and size distribution. The other two have given better results regarding size distribution and morphology but are space demanding and energy-intensive processes. Thus, drying and ball milling will be investigated for producing MNPs with the required properties and activities will continue with MNPs in dispersion form.

bath. This WP started at M12, but some preliminary studies have been already performed, as a preliminary screening of the biopolymers that can be used to produce functionalised MNPs, resistant to low pH conditions as suggested in **WP2**.

#### WP5: INTEGRATION OF PURIFICATION SYSTEM AND SAFE DISPOSAL OF MNP (M5-36), LEADER IDENER

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In this WP a mathematical model will be developed to further demonstrate and validate the PureNano process. Part of this WP is also the development of three methodologies for the disposal/ recycling of the MNPs. The plating bath of the processes under study (electroless Nickel, Zn-Cu electroplating and Nickel electroplating) and their purification systems have been modelled using and EES Python. Furthermore, а multidisciplinary design optimization (MDO) has been carried out, analysing different scenarios of the purification processes to minimize the capital cost and energy demand. The model has been

built using experimental and literature data, therefore the results obtained need to be considered as preliminary. Conceptual and basic engineering of the purification processes has been also undertaken. Process flow diagrams (PFD) and zP&ID have been prepared with the help of the end-users, selecting the real equipment that will be implemented in the purification systems.



Due to the distinct characteristics of the electroplating and electroless processes, the purification processes of the Gaser and CNano pilot lines will be developed according to the needs of each pilot line still keeping the focus on getting an optimization of the streams and achieving an optimal bath purification using the MNPs.



This WP also focuses the on investigation for the safe disposal/ recycling of the used MNPs. The disposal of exhausted MNPs after the treatment process is a critical aspect to consider, but fundamental to be in line with the directives of zero waste treatment. Three main recycling routes have been identified and will be further investigated.

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The first route regards the extraction of precious metals from the surface of MNPs through a low-cost electrolytic process. PoliMi is working the effectiveness of an electrochemical process and further experiments will be performed.

The second route foresees the

use of used MNPs for wastewater treatment. The MNPs after the removal of orthophosphate, still preserve a lipophilic nature that is useful for the treatment of wastewater contaminated with organic compounds. Promising results in terms of reduction of the COD (chemical demand) oxygen obtained from wastewater were contaminated with organic compounds dispersed or in an emulsion.

The third possibility is to include the exhausted MNPs in concrete formulation. First results presented by NTUA have shown that that the cement has good workability and further experiments will be performed to this direction. 9

#### **WP6: DEMONSTRATION** ACTIVITIES (M19-36), **LEADER: CNANO**

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objective of this The WP is to demonstrate and validate the use of functionalized MNPs in a purification process. This WP will demonstrate the effectivity of the purification process in the real operational environment of

#### **WP7: INDUSTRIAL IMPLEMENTATION ISSUES** (M1-36), LEADER: ASFIMET

WP7 focuses on the environmental monitoring, recycling, and cost analysis of the PureNano process. The overall objective is to assess the sustainability of the proposed recycling routes and to follow the current regulations in order to ensure the compliance of final products and assess health and safety risks associated with the PureNano production process. Part of this WP is also the organization of internal and external training activities and workshops. The main achievements of this task include the identification of the chemical substances used



electroless and electroplating facilities of the end-users GASER and Cnano. The work of this WP is going to start in January 2021.

in the PureNano technology, the main hazards associated and a risk assessment of nanomaterials. The same analysis has been performed for the chemical substances used in the Cnano facility. For the risks identified, several control measures were suggested using available literature, namely standards, technical manuals, legislation, scientific articles, project results, and other relevant references. An inventory of the REACH requirements was presented to advise on compliance with the REACH obligations, including nanomaterial issues.

A low-risk priority was obtained from the analysis under the current operational conditions.

#### WP8: LCA/LCC (M1-36), LEADER: ISQ

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This WP aims to evaluate the potential environmental impact of the PureNano process through the Life cycle analysis (LCA) methodology and to perform the Life Cycle Cost Analysis defining the OPEX and CAPEX cost associated with the plating bath purification. The LCA analysis will be of fundamental importance to confirm the sustainability of the PureNano process. The first step of the LCA analysis regards the goal and scope definition, the identification of the functional unit and the boundary conditions. The amount of spent bath treated (kg) has been identified as a functional unit, which means that all the mass and energy flows will be normalized per kg of spent bath treated. The boundary conditions with the relative stages have been also identified and will be further investigated. In order to demonstrate the low environmental impact of the PureNano process, a comparison with two traditional waste treatment process (i.e. Incineration and underground deposit) will be made. The results from the LCA and LCC studies as well as the provided information from the developers of the innovative solution will be used as data for the Eco-efficiency analysis.



#### WP9: DISSEMINATION AND EXPLOITATION (M1-36), LEADER: AXIA

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The purpose of WP9 is to facilitate dissemination and communication activities towards targeted audiences about the possible adaption of the technology to the fields related to magnetic nanoparticles, and study the business potential of innovations, thus laying the ground for exploitation of the project results.

The main objectives of the work package are:

 to disseminate the PureNano results to targeted meetings, workshops, conferences;

• to develop a strategy for the intellectual property right (IPR) protection and to demonstrate the potential of PureNano solution in creating industry business opportunity and to manage the innovations coming from the project through exploitation activities.

The project's social media presence has been the major pillar of the communication strategy and the most direct of its communication measures. In order to raise public awareness about PureNano and promote the

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project's objectives and key messages, social network profiles/pages were created, and AXIA, as an administrator, is managing their activities. For each industrial partner. preliminary а exploitation route has been defined. In particular for each partner, a business model CANVAS was created, and possible end-users identified. The purification system developed in the PureNano project has great market potential and this constitutes a concrete possibility to have it on the market as soon as the project ends.



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### 5. WORKSHOPS AND TRAINING ACTIVITIES

MAGNETO-SPONGES: INNOVATIVE TECHNOLOGY FOR INDUSTRIAL WASTEWATER TREATMENT

ASFIMET in collaboration with Captive organized a training workshop, initially planned to be physical on the 30th of October 2020, in order to showcase the technology and the work done within the PureNano project. Due to the social restrictions as consequence of the COVID-19 pandemic , the exhibition was cancelled and substituted by an online workshop that took place on the 11th of November 2020 with the participation of 25 participants active in the metal finishing and industrial wastewater treatment sector.



MAGNETOSPONGES: LA TECNOLOGIA INNOVATIVA PER IL TRATTAMENTO DELLE ACQUE REFLUE INDUSTRIALI



La rimozione dei metalli pesanti dalle acque reflue industriali è di fondamentale importanza a causa degli effetti nocivi di tali elementi sulla salute umana e sull'ambiente. Per questo motivo, negli utimi decenni, il trattamento dell'acqua per la rimozione dei metalli pesanti ha coinvoto numeroze applicazioni e tecnologi, sebbene siano necessarie utientini ricerche per il loro miglioramento. Nel corso dei verkshop sarà presentato un nuvovo metodo per il trattamento delle acque industriali basato su micro-pugne magnetiche in grado di catturare diversi inquinanti presenti nelle acque reflue. Tale metodo è in fase di miglioramento nell'ambio del progetto europeo PureNano per il trattamento di reflui provenienti dall'industria galvanica.

can industria general. Lintroduzione di mico-puppe magnetiche per la gestione delle acque reflue promuove un tratamento semplice che annulla l'impatto ambientale dei processi idrici, Rovendone il nutilizza, huttre, la possibilità di rigenerare le micor-puppe, suggerisce un tratamento a basso impatto economico, sia per la possibilità di rutilizzare le testes mico-supun, sia per ottenere materie prime secundario.

La partecipazione al webinar è gratuita previa registrazione all'indirizzo: www.galvanotecnica.org/it/eventi/magnetosponges Agli iscritti sarà inviato il link per assistere al workshop e per intervenire nella essesione conclusiva di domande G sisposte.

Per informazioni: A.I.F.M., asfimet@tin.it | tel. 339 8458916





CaptiveS introduced the problem of industrial wastewater treatment from the perspective of the environmental and economic burden and briefly showed up to what point the current technologies treat the problem.

Next, the innovative technology proposed by CaptiveS that is also the core technology of the PureNano project, was presented. Technical details were given and expected and obtained results were illustrated with special focus on the circularity and the economic aspect of the process.

### 5. WORKSHOPS AND TRAINING ACTIVITIES

STAKEHOLDER ENGAGEMENT TRAINING FOR NANOTECHNOLOGY APPLICATIONS

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The wider scope of the stakeholder engagement training was to train the partners on how they can build a connection with the stakeholders in the framework of innovation management. The training was divided in two parts: in the first half, AXIA gave an overview of the benefit and challenges of the engagement process and of the steps to follow to engage in an efficient and focused way targeted stakeholders. The engagement process should start answering the most common questions of Why? How? What? Who? When? It is important indeed to identify the aim, the benefit and motivation that are behind the engagement intention, to tailor the engagement on the base of the con-



tent and condition of the project, to identify the mission of the engagement and the stakeholders that have to be informed, involved, consulted or are the key players. In the second half, an interactive section was organized by AXIA, where the project partners were involved in two exercises to identify the stakeholders to engage for the PureNano project and start to preparing the ground for developing an effective stakeholder engagement plan.

In order to receive the audience's feedback on the activity, an evaluation questionnaire was distributed and positive feedbacks was collected with the suggestion to repeat this kind of section in the future.

#### **6. FUTURE EVENTS**

PureNano partners are planning to attend several conferences/ events in the coming months. Due to the COVID-19 pandemic still ongoing, the attendance to these events can be hindered. In this case a new plan will be developed.

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