DEPARTMENT OF SURFACE ENGINEERING AND OPTOELECTRONICS F-4

The Department of Surface Engineering conducts interdisciplinary research on designing the surface properties of various materials. We use advanced techniques for surface and thin-film characterization, in particular with our XPS, AES, SIMS and AFM instruments. The scientific activities are focused on surfaces and coatings, gaseous discharges, thermodynamically non-equilibrium plasma and the interaction of reactive plasma species with organic and inorganic materials. Patent applications protect innovative solutions for industry, medicine, biotechnology and agriculture.

Head (since 1. 12. 2021):
Prof. Alenka Vesel

Head (until 30. 11. 2021):
Prof. Miran Mozetič

JSI is a project partner in the H2020-CSA Athena project (https://www.athenaequality.eu/), which aims to remove barriers to the recruitment, retention and career progression of female researchers, lowering gender imbalances in decision-making processes and generating a cultural change needed to avoid gender bias and discriminatory practices through the implementation of Gender Equality Plans (GEPs). To ensure systemic institutional change, the project plans to conduct an assessment of procedures and practices already in place in partner organizations, together with an analysis of the national legislation and policy frameworks. In parallel, it will put in place a participatory process aimed, on one hand, to understand the needs and the preferences of the stakeholders and, on the other, to train them about selected topics related to gender. As a final result, each partner organization will draft and implement its specific GEP.

At the Jožef Stefan Institute, the Athena activities are carried out by 9 research departments and the Director's office. The Department of Surface Engineering participated in the identification of existing gender bias at the organisational level as well as in the assessment of existing national provisions. We were involved in gathering information on gender bias at the JSI by conducting story-telling interviews and discussions in different focus groups (managers, researchers, administration, young researchers and members of the Gender Equality Plan Implementation (GEPI)). Our representative is also a member of the GEPI committee, which is the leading body at the organisation for the preparation and implementation of the GEP. In this role, all the GEPI members have to pass the compulsory education courses given by the coordinator, which will be further disseminated at the JSI. In addition, our department is active in dissemination and communication activities. The news about the project can be found at JSI website (https://www.ijs.si/ijsw/EnakeMoznosti) as well as at Athena website (https://www.athenaequality.eu/).

The major activities of the Department F4 remain scientific research and the development of technological solutions useful for applications in mass production. The surface functionalization of organic materials remains a hot topic of interdisciplinary science, where plasma physics meets surface chemistry, biology, and agriculture. Despite a tremendous commercial potential, the exact mechanisms of surface functionalization upon treatment with molecular fragments on the atomic scale remain unexploited due to the complexity of the interaction. A lack of reliable theories is partially compensated by precisely designed experiments. Gaseous plasma is a source of various radicals and charged particles as well as radiation across a broad range from infrared to vacuum ultraviolet, so it is far too complex for a detailed scientific investigation. The surface functionalization is often accomplished by neutral species such as atoms. We designed an experimental setup useful for the adjustment of the density of neutral oxygen atoms in the ground electronic state in the range from approximately 10^{17} to 10^{22} m^{-3}. The system powered by a radio-frequency (RF) electrodeless discharge is optimized for studying the surface kinetics versus the doses of O atoms. We used polystyrene as a model polymer of a well-defined structure. This polymer has recently been tackled also by theoreticians who identified over ten binding sites of different binding energies even for pristine polystyrene, more for partially oxidized material. We studied the evolution of surface functional groups versus the dose of atoms starting at the dose as small as 10^{19} m^{-2} and found an excellent agreement with the theoretical predictions. The evolution of the functional groups is shown in Figure 1. Considering the peculiarities of XPS, we can conclude that

We confirmed the recent theory of the functionalization of aromatic polyolefins.

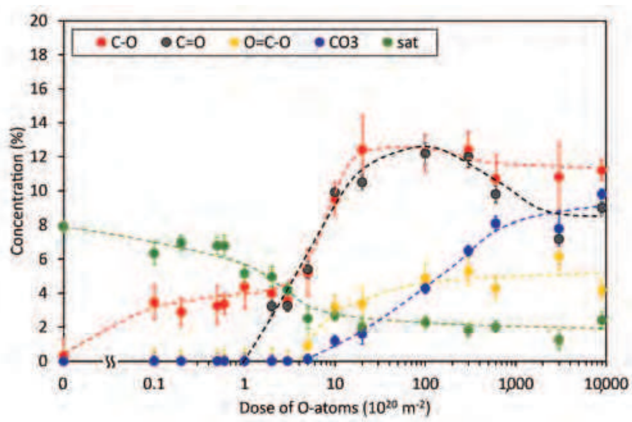

Figure 1: The concentration of surface-functional groups versus the dose of O atoms. Reproduced from [1].

the formation of hydroxyl groups on the pristine polystyrene is a highly probable reaction. The surface saturation with hydroxyl groups is accomplished already at the dose of approximately 10^{19} m^{-2}. Once the surface is saturated with hydroxyl groups, the further treatment causes gradual destruction of the phenyl ring and thus the formation of the surface sites capable of the irreversible binding of oxygen in other functional groups. The concentration of hydroxyl groups starts increasing above the saturation level after receiving the dose of $3x10^{20}$ m^{-2}, which is explained by the etching of the polymer and thus assuming a rich morphology on the scale measured in nanometres. Such a rich morphology causes the virtual surface concentration of oxygen as probed by XPS well above the theoretical limit. Details about the surface kinetics were published in the journal ranked #1 in the scientific niche of "material science, coatings & films" [1].

The functionalization of polymers with polar, oxygen-containing functional groups is beneficial for increasing the surface wettability of all materials. While polystyrene is perhaps the most useful material for studying the evolution of the surface functional groups, the largest technological challenge is the functionalization of biological materials. The kinetics has not been tackled by any group worldwide, probably because of the complexity of the surface interactions. We treated corn seeds in oxygen plasma sustained by inductively coupled RF discharge at various pressures. The absorption of RF power by charged particles in gaseous plasma depends enormously on the gas pressure and forward discharge power because the plasma is sustained in either the E or H mode. The fluxes of some reactive plasma species change by several orders of magnitude at the transition between the modes, so the surface wettability obtained at a certain plasma treatment time depends enormously on the discharge mode. Still, according to the discussion in the previous paragraph, the decisive parameter governing the surface wettability should be the dose of O atoms. We plotted the surface wettability as determined by the sessile drop method versus the doses and actually observed the curve shown in Figure 2. Therefore, we clearly showed that irrespective of the treatment time, gas pressure, or discharge power, the surface wettability follows the general curve. Such an observation has not been reported in scientific literature, so our paper, published in the prominent journal in the niche of agriculture, can be regarded as a pioneering work in the niche of plasma agriculture.

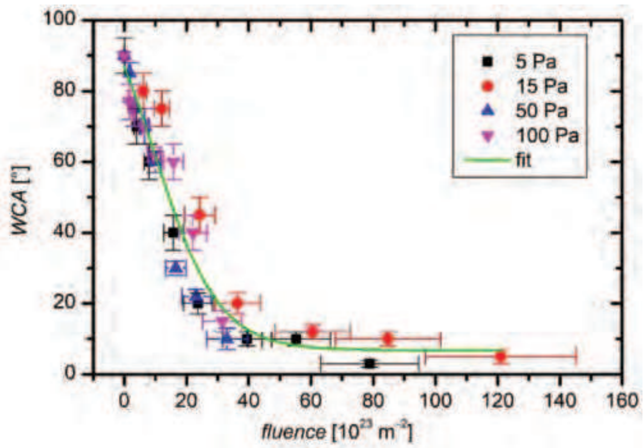

Figure 2: The surface wettability of corn seeds versus the doses of oxygen atoms. Reproduced from [2].

The team developed methods for the rapid inactivation of viruses on respiratory masks.

An appropriate wettability of seeds is useful for immediate water uptake and thus germination of the seeds, even in harsh conditions. The beneficial effect of plasma treatment has been reported by all partners of the COST action CA19110 "Plasma Applications for Smart and Sustainable Agriculture" https://plagri.eu/, where the member of our team, dr. Gregor Primc, serves as the Science Communications Manager. We performed a comprehensive literature survey [3] and found that very few groups worldwide reported on the mass application of the plasma technology and field experiments, so the plasma treatments are still at low technological readiness levels (TRLs). Our group successfully accomplished a large project on upscaling the TRL from 3 to 6. The project entitled "Plasma seed treatment" gathered research groups from 6 partners to develop a prototype of an industrial line for the treatment of about 1 ton of seeds per hour. The coordinator of this project, which was co-funded by European structural funds and the Slovenian Ministry of Agriculture, was the largest Slovenian company for trading seeds, Interkorn Ltd, whose Director is dr. Peter Gselman, and the scientific coordinator was dr. Nina Recek from Department F4. In fact, our department was the only public research organization involved in this project. Other partners were responsible for the construction, automatization, and verification of the prototype lines. We constructed several plasma reactors for the treatment of seeds, and one is shown in Figure 3. The consortium filed several patent applications, which will be published in the future, so we cannot disclose the methods of the invention. This was one of the largest projects involving both industrial research and experimental development in the history of the Jozef Stefan Institute. The research on plasma agriculture was performed by members of several research departments of our institute and the group at the Biotechnical Faculty, University of Ljubljana. The group of Dr. Katarina Vogel-Mikuš revealed the biological aspects of the plasma techniques and supervised Ph.D. students J. Mravlje and P. Starič [4], who will enlighten complex mechanisms of the biological response to non-equilibrium gaseous plasma.

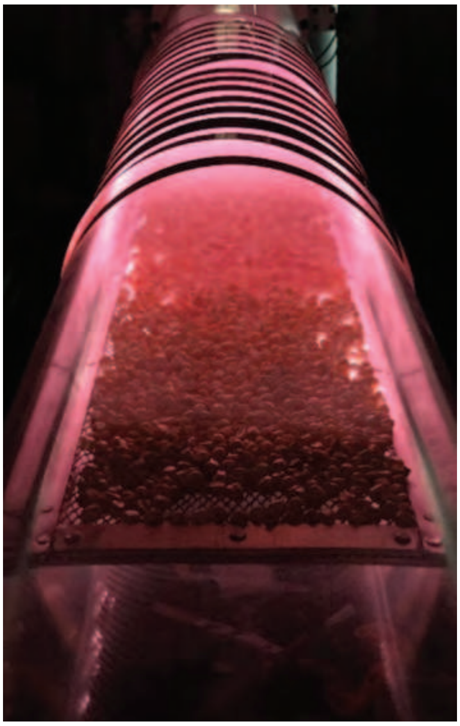

Figure 3: Low-pressure plasma is useful for the decontamination and hydrophilization of seeds before sowing. Reproduced from [2].

The major income of the Department F4 comes from applied research projects that are co-funded by the Slovenian Research Agency (ARRS) and a company that is interested in the commercialization of scientific knowledge. A fruitful collaboration has been established with the company Elvez Ltd., which uses several large plasma reactors in mass production. The typical volume of a plasma reactor is several cubic meters. Currently, it is believed that uniform plasma cannot be sustained with useful parameters in such large industrial reactors for numerous reasons, including the final penetration depth of electromagnetic radiation from the RF-powered electrodes, surface loss of radicals and charged particles, gas-phase oligomerization of the molecular fragments and the formation of dusty plasma. There has been a lack of appropriate methods for measuring the deposition rate of coatings prepared by plasma-enhanced chemical vapor deposition (PECVD). In collaboration with experts from the Laboratory for Electro-Optics and Sensor Systems, University of Maribor, we developed miniature sensors for real-time measurements of the deposition in PECVD systems. The group led by dr. Denis Đonlagić verified the laboratory prototypes of these sensors, and we performed systematic measurements at the premises of Elvez Ltd. company. We used a cylindrical plasma reactor with a diameter and height over 2 m and distributed the sensors at various positions, as shown in the inset of Figure 4. We measured the thickness of the protective coating deposited by the plasma polymerization technique. The lines in Figure 4 exhibit almost optimal linearity due to the careful selection of the adjustable discharge parameters. The slopes of the lines in Figure 4 indicate deposition rates that can vary by an order of magnitude, depending on the position in the industrial reactor for PECVD of polymer-like hydrophobic coatings. Optimization of such industrial reactors is among our key technological challenges for the future [5].

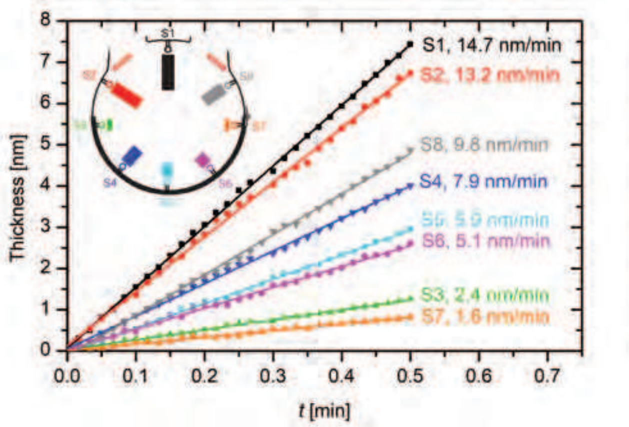

Figure 4: Schematic of the positions of miniature sensors (inset) and the thickness of the deposited films versus deposition time. Reproduced from [5].

The spread of the SARS-CoV-2 virus influenced our scientific and technological activities significantly. We summarized the results on the virus infectivity of various materials and found insignificant differences in survival rates on different materials. Our research group proposed several methods for treating respiratory masks and prepared a patent application on the most promising and feasible technique based on plasma technology (Figure 5 and 6). Meanwhile, we also studied a process for the recycling of single-use personal protective masks. We developed an efficient sterilization procedure. In collaboration with other departments at the Jozef Stefan Institute, we studied the sterilization options using gamma rays and high-energy electrons as well as the relationship between the irradiation, particle removal efficiency, and surface potential on the respirators [6]. The principal investigator in our group was dr. Janez Kovač. He characterized the chemical composition and the changes in static charge for polypropylene fibers from the filter piece of typical FFP3 masks after different types of ionizing sterilization. With our sensitive, non-contact measurements of the surface potential, we found that sterilization with X-rays and e-beam leads to complete removal of the surface charge, which is needed for electrostatic interaction for filtering particles of the virus size. In the next step after sterilization, we successfully recharged the sterilized filter surface to its initial potential. Sterilization and recharging resulted in a complete regeneration of filtering facepiece respirators for protection against Covid-19.

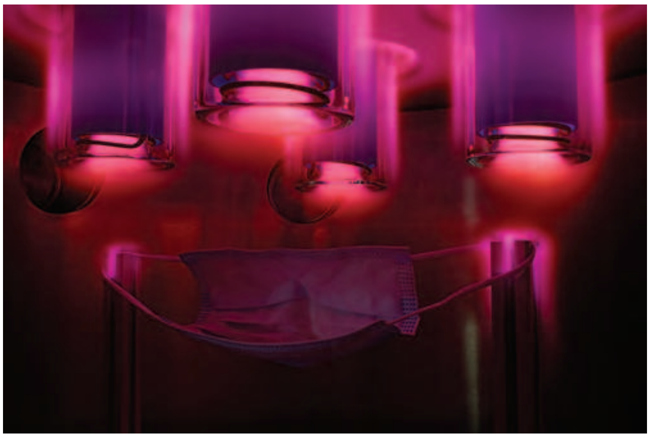

Figure 5: Our plasma technologies make it possible to alter the surface energy of materials for masks against virus infections.

Figure 6: Image shows the dosing of the water drop to the surface of the untreated mask. If the mask is treated the water drop is absorbed.

The F4 department performs basic research within two core-funding research programmes. The activities reported in the text above were performed within the core funding "Thin Film Structures and Plasma Surface Engineering," which is led by dr. Miran Mozetič. Another core funding is for "Fusion Technologies," and the principal investigator in our department is dr. Rok Zaplotnik. Our fusion-oriented scientific activities were focused on the scientific and technological challenges for fusion reactors beyond ITER. The divertor, which is positioned at the bottom of the vacuum vessel of a fusion reactor, controls the exhaust of waste gases (in particular helium) and impurities from the reactor and is subjected to the highest surface heat loads of the tokamak. In ITER, the diverter will be coated with tungsten, but this material has several drawbacks, so tungsten might be omitted in fusion reactors beyond ITER. For example, the next-generation machine is the demonstration power plant DEMO. The final design of the divertor in DEMO is not yet determined. For DEMO and beyond, liquid metal plasma-facing components are also being considered. Liquid tin is believed to be one of the candidates for a liquid metal divertor. Our study focused on hydrogen solubility and deuterium-atom retention in the liquid Sn. An immeasurably low concentration of deuterium was detected in pure tin, but we observed significant retension in the tin-oxide layer, which might be due to the formation of hydroxides. We reported the results of our systematic research in [7]. Apart from the conclusion that the retention of hydrogen isotopes in the liquid-tin divertor free from oxide is unlikely, we also provided a recipe for effectively reducing the tin-oxide layer.

[1] Vesel, Alenka, Zaplotnik, Rok, Mozetič, Miran, Primc, Gregor. Surface modification of PS polymer by oxygen-atom treatment from remote plasma: initial kinetics of functional groups formation. Applied Surface Science, ISSN 0169-4332, 2021, vol. 561, str.15058-1-15058-7, doi: 10.1016/j.apsusc.2021.150058.

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Awards and Appointments

1. Ita Junkar, Metka Benčina, Matic Resnik, Rok Zaplotnik: Best innovation with commercial potential award, Center for Technology Transfer and Innovation at the Jožef Stefan Institute, Novel surface finishing procedures for medical devices, especially vascular stents.

Patents granted

1. Ita Junkar, Rok Zaplotnik, Metka Benčina, Miran Mozetič, Method for treatment medical devices made from nickel-titanium (NiTi) alloys, EP3636294 (B1), European Patent Office, 17. 11. 2021.
2. Gregor Filipič, Kristina Eleršič, Darij Kreuh, Janez Kovač, Uroš Cvelbar, Miran Mozetič, A method of colouring titanium and titanium alloys, GB2530805 (B), Intellectual Property Office, 24. 11. 2021.

INTERNATIONAL PROJECTS

1. COST CA18113; Understanding and Exploiting the Impact of Low pH on Microorganisms
   Dr. Martina Modic
   Cost Association Aisbl
2. COST CA19110; Plasma Applications for Smart and Sustainable Agriculture
   Asst. Prof. Gregor Primc
   Cost Association Aisbl
3. COST CA20114 - PlasTHER; Therapeutical Action of Cold Atmospheric Plasmas
   Asst. Prof. Ita Junkar
   Cost Association Aisbl
4. H2020 - PEGASUS; Plasma Enabled and Graphene Allowed Synthesis of Unique nano Structures
   Prof. Uroš Cvelbar
   European Commission
5. H2020 - ATHENA; Implementing Gender Equality Plans to Unlock Research Potential of RPOs and RFOs in Europe
   Asst. Prof. Ita Junkar
   European Commission
6. H2020 - EUROfusion; Plasma Facing Components-1-IPH-FU, EUROfusion
   Asst. Prof. Rok Zaplotnik
   European Commission
7. H2020 - EUROfusion; JET Campaigns-JET1-FU
   Dr. Aleksander Drenik
   European Commission
8. H2020 - EUROfusion; WPPFC-PEX-FU, WPPFC-PEX-FU, EUROfusion
   Asst. Prof. Rok Zaplotnik
   European Commission
9. Catalytic Probes for Characterization of Hydrogen Plasma
   Asst. Prof. Gregor Primc
   Slovenian Research Agency
10. Control of Chemical Composition of Thin Films by High Resolution Mass Spectrometry

of Secondary Ions
Prof. Janez Kovač
Slovenian Research Agency
11. Advanced Catalysts based on Multilayered Vertically Oriented Graphene Nanostructures
Prof. Alenka Vesel
Slovenian Research Agency
12. Investigation of Helium Retention in Plasma Facing Materials Using Advanced Analytical Methods
Dr. Gregor Filipič
Slovenian Research Agency
13. Characterization of Oxygen Plasma Sustained with Powerful Gaseous Discharges
Prof. Miran Mozetič
Slovenian Research Agency
14. Low Temperature Plasma Diagnostics and its Applications for Seed Treatment
Prof. Miran Mozetič
Slovenian Research Agency
15. Functionalization of Ti-Based Surfaces Using Energy Beams and Plasma for Biomedical Applications
Asst. Prof. Gregor Primc
Slovenian Research Agency
16. Promising Eco-Sterilization of Pathogenic Fungi on Seeds Using Reactive Species in Gaseous Plasma
Prof. Miran Mozetič
Slovenian Research Agency

RESEARCH PROGRAMMES

1. Vacuum technique and materials for electronics
Dr. Vincenc Nemanič
2. Thin film structures and plasma surface engineering
Prof. Miran Mozetič
3. Fusion technologies
Asst. Prof. Rok Zaplotnik

R & D GRANTS AND CONTRACTS

1. Structural and surface properties of fibrous membranes for purification and chromatographic separation of biomacromolecules
Asst. Prof. Ita Junkar
2. Ecologically friendly in-situ synthesis of ZnO nanoparticles for the development of protective textiles
Asst. Prof. Gregor Primc
3. Initial stages in surface functionalization of polymers by plasma radicals
Prof. Janez Kovač
4. Alternative approaches to assuring guality and security of buckwheat grain microbiome
Prof. Miran Mozetič
5. Advanced surface finishing technologies for antibacterial properties of patient specific 3D printed implantable materials
Asst. Prof. Ita Junkar
6. Hybrid and Reengineered Nanocatalysts for New Purification Routes
Prof. Janez Kovač
7. Self-organization of plasma in magnetron sputtering discharges
Prof. Miran Mozetič
8. New startegies for fabrication of biomimetic vascular implants
Asst. Prof. Ita Junkar
9. Innovative procedures for advanced surface properties of medical stainless steel
Dr. Metka Benčina
10. Development of new, environment-friendly approaches for plant and human virus inactivation in waters
Asst. Prof. Gregor Primc
11. Innovative sensors for real-time monitoring of deposition rates in plasma-enhnced chemical vapour deposition (PECVD) systems
Asst. Prof. Rok Zaplotnik
12. Nanoparticle-reinforced new metal matrix composites manufactured by selective laser melting for tooling industry
Prof. Miran Mozetič
13. A Novel High-strength Aluminium Alloy developed for Selective Laser Melting and Lightweight Applications
Prof. Miran Mozetič
14. Innovative configuration of inductively coupled gaseous plasma sources for up-scaling to industrial-size reactors
Prof. Miran Mozetič
15. Carbon nanowalls for future supercapacitors
Prof. Alenka Vesel
16. Selected area functionalization of polymeric components by gaseous plasma
Prof. Miran Mozetič
17. Innovative method for purification of wastewater
Asst. Prof. Gregor Primc
18. Development of safe multifunctional surfaces for catheters to combat biofilms (DemoCat)
Prof. Alenka Vesel
19. Waterborne virus inactivation efficiency of a prototype device combining non-equilibrium plasma and hydrodynamic cavitation
Asst. Prof. Rok Zaplotnik
20. Use of gaseous plasma for higher yields and lower use of antifungal agents in agriculture
Asst. Prof. Ita Junkar
21. Innovative ECO plasma seed treatment (for sowing and for human and animal diet/nutrition
Dr. Nina Recek
Ministry of Education, Science and Sport
22. Method for preparation of bacteriostatic surfaces on 3D printed medical implants
Dr. Matic Resnik
Ministry of Education, Science and Sport
23. Use of gaseous plasma for higher yields and lower use of antifungal agents in agriculture
Asst. Prof. Ita Junkar
Ministry of Agriculture, Forestry and Food
24. Income from Coowners of Invention for Reimbursement of Costs for IP Protection in the Case of EVT140_Mozetič_Carbon Nanowall
Prof. Miran Mozetič
Nagoya University
25. EVT770_Mozetič_CNW2_Reimbursement of the Costs for Patent; Income from Coowners of Invention for Reimbursement of Costs for IP Protection in the Case of EVT770_Mozetič_CNW2
Prof. Miran Mozetič
Nagoya University

NEW CONTRACTS

1. Innovative configuration of inductively coupled gaseous plasma sources for up-scaling to industrial-size reactors
Prof. Miran Mozetič
Vacutech vakuumske zehnologije in sistemi d. o. o.
2. Regulation of mutual relations between the Company and JSI in joint research and development („KET4CleanProduction“)
Asst. Prof. Ita Junkar
Brinox Inženiring d. o. o.
3. Co-finanicing of L-project L2-1834 Carbon nanowalls for future supercapacitors
Prof. Alenka Vesel
Iskra, d. o. o.
4. Innovative sensors for real-time monitoring of deposition rates in plasma-enhanced chemical vapour deposition (PECVD) systems
Asst. Prof. Rok Zaplotnik
Iskra, d. o. o.
5. L-project co-financing: Innovative method for purification of wastewater
Asst. Prof. Gregor Primc
Induktio d. o. o.
6. L-project co-financing: Selected area functionalization of polymeric components by gaseous plasma
Prof. Miran Mozetič
Elvez, d. o. o.

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21. Janez Trtnik

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Original Article

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10. Nina Recek, Alenka Vesel, Rok Zaplotnik, Domen Paul, Gregor Primc, Peter Gselman, Miran Mozetič, "Hydrophilization of corn seeds by non-equilibrium gaseous plasma", *Chemical and biological technologies in agriculture*, 2021, **8**, 32.
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