FELLOWSHIP SUMMARY REPORTS

Theme 3: Transformational Technologies and Innovation

Sustainable extraction of bioactive compounds from food by-products

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Host institution and collaborator:
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I consent to post this report on the Co-operative Research Programme's website

1. What were the objectives of the research project? Why is the research project important?

The aim of the research project was the analysis and the investigation through the development of a mathematical model of a potentially greener process for the extraction of bioactive compounds using NaDES (Natural Deep Eutectic Solvent) from agricultural residues or food by-products, for food/feed applications. Indeed, NaDES have been deeply investigated in the scientific literature for the extraction of natural products, but there is still a lack of knowledge in terms of both mathematical modelling, and related simulation, of bioactive extraction processes and evaluation of economic sustainability of the industrial-scale process for a potential large-scale implementation. Therefore, the novel aspect of this study, given by UCSC's added value, is the development of a first mathematical model, based on mass and energy balances, matching the lack in the state of the art. The final step of the project is to evaluate the economic feasibility of the selected process of extraction of bioactive compounds from food by-products, taking advantages from the developed and validated mathematical model. However, to achieve this objective, intermediate steps/objective to be pursued were identified:

a) Selection of the bioactive compound extraction process with the highest market potential using both what is reported in scientific literature and what is already known by the AZTI partner, as it is already active in this field.
b) Development of the mathematical model of the extraction process using mass and energy balance equations and its subsequent validation using data from scientific literature. This step is important to highlight criticalities and gaps in the model that can be filled through a dedicated experimental campaign.
c) Development and carrying out an experimental campaign to improve both the knowledge of the selected extraction process and the predictive capabilities of the model developed in the previous point (b).
d) Optimization, using the model developed, of the operating conditions of the extraction process either to reduce the energy consumption of the process or to maximize the recovery yield of the bioactive compounds. Subsequent testing of the optimal operating conditions will be assessed.
e) Economic evaluation of the process based on the optimal operating conditions derived from the previous point.

This research project is important because it could have different potential impacts that could be summarized in the following points:

- The development and the validation of a mathematical model for extraction processes can provide a valuable tool that not only help to better understand the process itself, but also can be used for automated process control (i.e. model predictive control).
- The model developed will allow to evaluate the optimal operating conditions of the extraction process of bioactive compounds, with the aim of making it less impactful from the environmental point of view. Therefore, the model will allow to obtain, after future improvements, the optimal process conditions depending on the raw material used and/or its seasonal variability.
- Finally, the economic evaluation of the extraction process will allow to highlight the critical points (to be addressed in future studies) of the process to make it economically attractive for industries and/or investors. In this way, bringing such processes to an industrial scale level, the food by-products or wastes could be considerably valorised and, then, reduced.

2. Were the objectives of the fellowship achieved?

This research project has achieved some of the goals that were proposed, while others will be implemented in the near future, thus continuing the collaboration between UCSC and AZTI. In particular, following the same list of goals reported above, we can say that:

a) In agreement with the host institution (i.e. AZTI), extraction of polyphenols and reducing sugars from spent coffee grounds using NaDES (Natural Deep Eutectic Solvent) was selected has the “target extraction process”. This extraction process and the starting by-product were selected primarily on the basis of previous studies conducted by AZTI, both in terms of the actual availability of this by-product and the potential application of the recovered products in the market. Therefore, this objective was achieved.
b) The model was developed based on mass and energy balances. In summary, it is a system of differential equations that allows to evaluate the effective extraction of phenolic compounds and reducing sugars from

spent coffee grounds by estimating their concentration in the solvent depending on extraction time, water concentration in the NaDES solutions, and temperature. However, it is fair to point out that the validation of the model was carried out only on the experimental data obtained at AZTI. In fact, there were no papers in the scientific literature dealing precisely with the extraction of polyphenols and reducing sugars from spent coffee grounds with the selected NaDES solvents. Nevertheless, it was still possible to validate the model effectively. It is important to underline that the review of the scientific literature works led to highlighting some relevant aspects and some shortcomings related to the mathematical modelling of the extraction process. Summarizing, the medium-low concentration (in most cases) of bioactive compounds in biomasses/ agricultural residues requires a high-performance extraction to achieve larger yields. Therefore, extraction methods and processing parameters need optimization for each biomass or agricultural residue, polyphenols content, and constituents that can affect extraction kinetics. As already said, to optimize such industrial processes, mathematical models are useful tools for simulating processes' performance, since they allow assessment of several alternatives or operational scenarios in order to define the best processing conditions, without the necessity of carrying out a large number of experimental trials. In the scientific literature it was possible to find two main approaches to model NaDES extraction: (I) Surface Response Methodology modelling and (II) Kinetic extraction models. However, both the approaches present some limitations. The first one has applicability constrained to the experimental range and it only represents studied variables' effect on the process yield, but does not explain the physical mechanisms or pathways of the process. The second (i.e. kinetic extraction models) gives information on the behaviour of the extraction over time, but the other operating conditions (e.g. concentration of water, temperature etc.) are fixed. Moreover, the concentration at the equilibrium of the extracted compounds needs to be known. This confirms that there was a need to develop a new mathematical model that could predict the behaviour of the extraction process not only as a function of time, but also as a function of other parameters such as extraction temperature and concentration of water in the NaDES solution. Therefore, this objective was achieved (see Figure 1).

c) During this project, a new experimental campaign was carried out in order to increase the knowledge of the NaDES extraction process, with particular reference to the evaluation of the process depending on the variation of water concentration in the NaDES solution. Specifically, two additional values of water concentrations were added to those already tested by AZTI in previous works. This was tested for two different NaDES solutions and for different extraction times. In addition, tests were carried out to study the heating rate of the solution since we wanted to see whether faster or slower heating could have an actual influence on the final extraction capacity. This was done with the aim of improving the final predictive capability of the developed model. It is important to point out that, unfortunately, it was necessary to repeat some experimental tests since they seemed to give discordant results, despite being done under the same conditions. After the additional analyses, it could be concluded that some of these results were just outliers, while others were different because there is a high variability of the initial composition of target compounds in the selected by-product (i.e. spent coffee grounds). All this inevitably led to delays in achieving the goals set at the beginning of the project, but at the end also this objective was achieved.

d) Due to what is reported in the previous point, it has only been possible to optimize the process from the point of view of extraction yield regarding polyphenols and reducing sugars. The optimization has been done within the range of operating conditions tested during the experimental campaign. The results have shown, for now, an increase in extraction capacity by increasing the percentage of water concentration in the NaDES solution, without achieving a maximum value. Therefore, it was decided, in agreement with AZTI, to continue the collaboration and further investigate the process through the application of new targeted experimental tests. This will be useful to further optimize the process not only from the yield point of view, but also from the energy point of view. Therefore, this objective was partially achieved.

e) Clearly, due to what reported above, it has not yet been possible to make an initial economic evaluation of the process, as the optimal operating conditions derived from the above points are not complete. This evaluation has only been postponed and it will be carried out as soon as possible. Therefore, this objective has not been yet achieved.

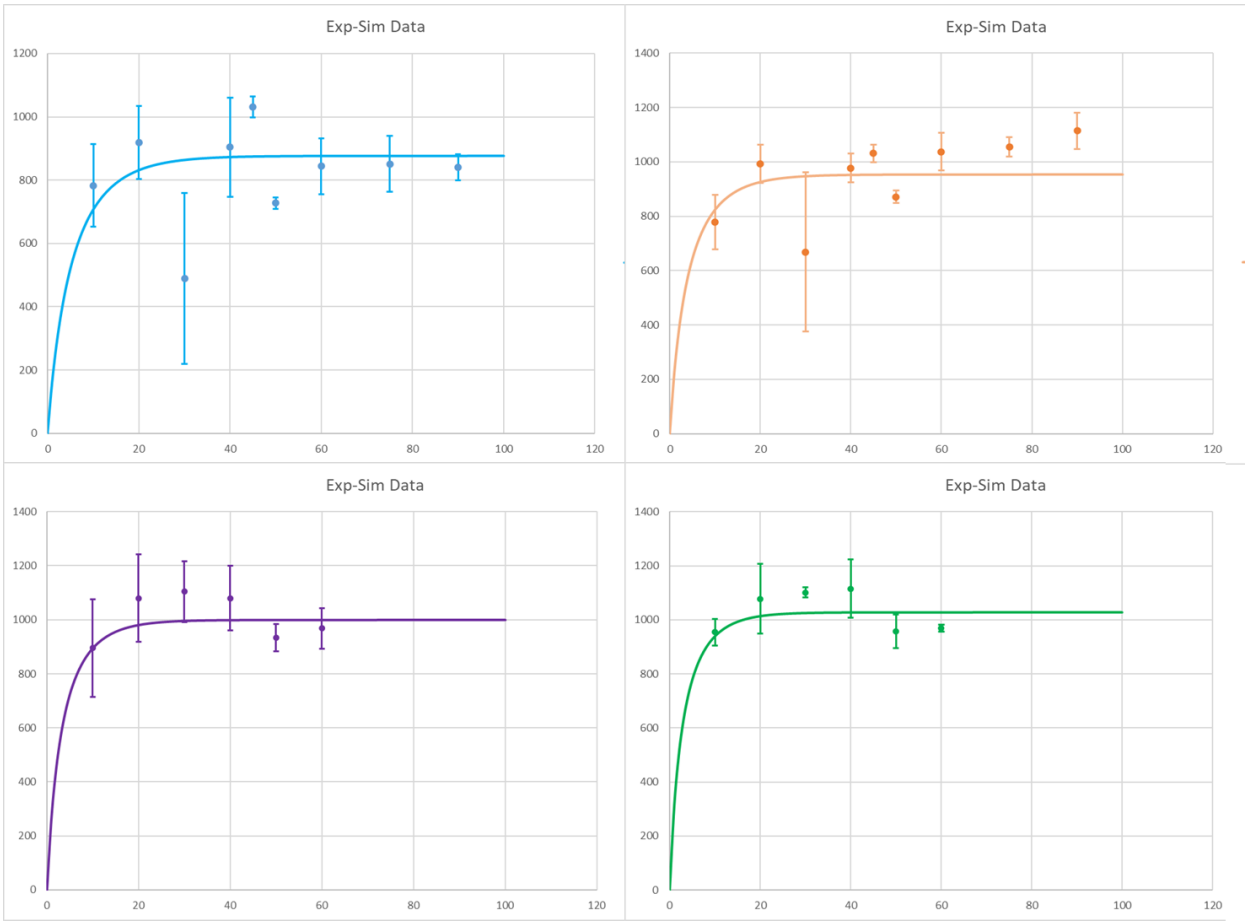

Figure 1 Simulation of the polyphenols extraction process as a function of time. Different colours refer to different concentrations of water in the NaDES solution (Range 20%-70% of water)

3. What were the major achievements of the fellowship? (up to three)

1) Study and experimental analysis of the extraction process behaviour of bioactive compounds and reducing sugars (from coffee grounds) as a function of water concentration in NaDES solution.
2) Development of a mathematical model for the extraction processes of bioactive compounds and reducing sugars using NaDES. This model includes the ability to predict extraction behaviour as a function of extraction time, temperature and water concentration in the solution.
3) Creation of a collaboration between UCSC and AZTI. A first future achievement of this new collaboration will be to verify the economic sustainability of extraction process based on the model developed (see point 2).

4. Will there be any follow-up work?

- Is a publication envisaged? Will this be in a journal or a publication? When will it appear?

At least one publication in a scientific peer-review journal, in cooperation with AZTI, is planned. However, since there have been delays in the completion of the experimental campaign, we are preparing a first draft article, which will have to undergo internal revisions. For this publication, we will focus on the development and validation of the novel proposed model, while we can expect a further article, also in a scientific journal, regarding the economic feasibility study (when it will be done). For the first publication we are thinking to publish it in journals like “Bioresearch Technology” or “Journal of Food Engineering”. Regarding the potential release date, considering the finalization of the work and review time, I could say the work will hopefully come out around April/May 2023.

- Is your fellowship likely to be the start of collaboration between your home institution and your host?

The collaboration between UCSC (my home institution) and AZTI (the host) will certainly continue after this initial collaboration. Proving this, several subsequent steps have been already fixed, including for example:

- Using the already existing data (if enough) to produce a surface response model with two variables (i.e. water concentration in NaDES and extraction time) and compare the predicted optimum extraction time and water composition with the values obtained by model developed in this project.
- To test the capability of the model to predict extraction yield both inside and outside the range of the operating condition tested at experimental level.
- To improve the predictions of extraction yield at different temperatures and different NaDES to food by-product ratios.
- To study and investigate the extraction selectivity.
- To carry out a first economical assessment of the process.

- Is your research likely to result in protected intellectual property, novel products or processes?

It is difficult that this research will be converted into protected intellectual property. In fact, mathematical models, to the best of my knowledge, cannot be considered as patentable. Instead, however, it might be possible that the developed mathematical model of the extraction process will highlight some shortcomings from the point of view of the conditions under which the extraction process is operated, perhaps leading to a reconsideration and modification of the process design towards a better management of it. This last aspect could be patentable. Not least, but this is indirectly, the bioactive compounds extracted by these techniques could be used to make new high-value food products the recipe of which could be patentable.

5. How might the results of your research project be important for helping develop regional, national or international agro-food, fisheries or forestry policies and, or practices, or be beneficial for society?

As highlighted also in the draft of the project, this research proposal does not include the direct involvement of policy makers. However, once the economic viability of the process and its attractiveness in the market in terms of investment are verified, policy makers will be alerted for the development of their national and international agro-food policies. In fact, modelling methodology applied in this project to spent coffee grounds can also be used for other agri-food by-products. Therefore, it will be necessary to develop a targeted policy for the collection and management of these by-products in order to optimize the recovery of bioactive compounds of interest. As already pointed out in this report, the economic sustainability of the process has not yet been done and future studies will be needed to further investigate and improve the process, such as properly assessing the water concentration to maximize the extraction yield. This project may also, after necessary further study, improvement and validation, prove useful to society for several aspects. The first aspect is undoubtedly related to the improvement of the environmental sustainability since both by-products of the agri-food industry would be reused (thus reducing waste and related emissions due to proper disposal), and "natural" solvents (NaDES) are used, which, unlike traditional DES or ionic solvents, can be reused and/or possibly disposed of in a more sustainable way. Second aspect, the products extracted by these processes (i.e., polyphenols, reducing sugars, or even proteins in the future) could become part of recipes for the creation of functional food products that can be healthier and safer for humans. Finally, this project could certainly also bring benefits from the point of view of the economy. Indeed, on one hand new jobs could be created due to the construction of new production facilities, and on the other hand these processes could bring an economic profit where nowadays there is only mandatory and costly processes for waste disposal.

6. How was this research relevant to:

- The objectives of the CRP?

This research has strengthened scientific knowledge in its field and has provided scientific information and results, in particular a novel mathematical model, that can be used in the near future in the framework of the

sustainable re-use of food by-products (e.g. spent coffee grounds as in this research) and agricultural residues to extract high added value products (i.e. bioactive compounds such as polyphenols or reducing sugars) that can be used in the food, feed, cosmetics and pharmaceutical sectors. Although the focus of this research was more related to scientific environment (potential optimization of the energy consumption of the process using the mathematical model), the results obtained, also foreseeing further future studies, may influence the policies for the collection and sorting of both food by-products and agricultural residues. Therefore, the project reached the following objectives of CRP:

- It has contributed to scientific progress, through the development and validation of a model for the extraction processes of bioactive compounds from spent ground coffee. Such model could be also applied, with a proper set of the parameters, to other food by-products or agricultural residues and can be also used in the future for the economic evaluation of the process itself.
- It has promoted international scientific understanding among OECD member countries through a new collaboration between an Italian and a Spanish member.
- It has contributed to a solid knowledge base for future agricultural policy-making, with particular reference to the reuse/disposal of agri-food residues.

- The CRP research theme?

This research can be included in the specific theme "Transformational technologies and innovation" among the three main pillars of CRP set in the context of globalization and climate change. Detailing, this research project has matched the following topics:

- *Digital Technologies*: this topic was covered by the development of the mathematical model using Matlab. Digital technologies have the aim to improve sustainability and productivity, and for linking consumers and producers along the entire supply food chain. In this framework, the mathematical model developed can allow to improve the sustainability of the process after a proper optimization. However, it is important to point out that this project have not covered the entire food supply chain, but the model of the single unit operations and then of the whole extraction process will be the starting point for the digitalization of the whole supply chain. In addition, these models can be used both to understand and predict process behavior and could be also used for advanced and automated process control technologies (i.e. model predictive control). This latter aspect fits well with the scope of digitalization of improving policy design, implementation, measurement, and monitoring of outcomes. Finally, it is important to point out that the approach followed during this research, using a validate first-principles model, matches the aspect related to the increase of capacity to create values in the agri-food system. Indeed, the access to basic connectivity infrastructure (e.g. broadband, telecommunication services) are not strictly necessary, because the model, ones further properly validated, can work off-line both for prediction and control purposes. Therefore, only sporadic connections will be absolutely necessary (e.g. to check for updates of the model used), although constant connection is recommended. Moreover, due to the fact that the model is based on mass and energy balances, it will be needed a lower amount of data to be collected, stored and processed (e.g. using regression tool) compared with the data needed, for instance, for the training of an artificial neural network. However, the presence of a storage data unit could be useful to reprocess old and new data together to further improve the model's prediction and performance even in real time. Finally, regarding the regulatory environment, the data ownership will be of who uses the model as a tool to optimize and/or better control their production plant.
- *Novel waste reduction technologies:* the raw material of the extraction process of this project was spent coffee grounds. Therefore, the extraction of bioactive compounds using NaDES (i.e. green solvent) allows to valorise/reduce the by-products producing high value-added products that can be sold and used in different sectors like food, feed, nutraceutical, pharmaceutical, cosmesis etc. Moreover, the economic assessment that will be done in the near future using the validated model, could allow to highlight the potential criticalities in the process to be optimized or modified in order to make the process itself competitive on the market.
- *Bioproducts and Bioprocesses*: The industrial economic assessment of the bioactive extraction process and the subsequent verification of its sustainability can be a potential solution to the growing demand from the private sector for bio-products derived from biologically based feedstocks and bioprocessed on an

industrial scale to generate high value products. In particular, regarding the extraction process, there is the need for innovation, not in terms of the extraction technologies, but rather in terms of the model than can help optimizing the process. For sure, at the stage of this research, it was not possible to consider the whole supply chain sustainability, but this study could be the base for some future developments in that direction.

- *Innovations in Social Science, Economics and Education:* Regarding this point, the project does not present direct social implications at its conclusions, but, this is a study that wants to be the basis for important future developments. However, it is possible to underline how the innovation brought by this project could be useful both to facilitate the translation of scientific knowledge into results (for instance, the model of the process and the future evaluation of economic sustainability could attract several industrial investors) and to foster the adoption of new practices by stakeholders to achieve productivity growth and the protection of natural capital (e.g., with a better and more conscious waste collection). It is important to develop skills in this field and transfer them to companies and university students.

7. Satisfaction

- Did your fellowship conform to your expectations?

  This fellowship was conformed to my expectations. Indeed, the carried out research produced not only publishable results on a scientific paper, but also potential future developments to fruitfully continue the collaboration with the host institution in the coming years.

- Will the OECD Co-operative Research Programme fellowship increase directly or indirectly your career opportunities?

  This fellowship provided one of the qualifications necessary for the position of Associate Professor in Italy related to expenditure of research period abroad in universities or research centres. In other words, this fellowship will be useful for future career advancement.

- Did you encounter any practical problems?

  I did not encounter any practical problems, and in any case the secretary was always available and quickly responded to any request for clarification

- Please suggest any improvements in the Fellowship Programme.

  This fellowship has already many positive aspects (e.g. the opportunity to create your own research project within defined topic). In any case, I would suggest (in case it is not already there), to create, perhaps directly on the fellowship website, a list of potential "host institutions". In this way, the researcher who intends to participate in the call, can find a host (or the contact of the host) directly from the site (in case the researchers have difficulties in finding a proper host).

8. Advertising the Co-operative Research Programme

- How did you learn about the Co-operative Research Programme?

  I heard about the Co-operative Research Program from a colleague and then looked into it further through the program's webpage.

- What would you suggest to make it more “visible”?

  As first, to make the program more visible, I would suggest creating a social page (e.g. linkedin or facebook) if possible. In this way it might be possible to spread news and research projects carried out (perhaps the most

attractive ones). Another idea might be to reach out to universities/research centers directly each year, begging them to spread the news about the publication of the call for proposals. Indeed, many researchers (like me) would have a lot of interest in participating in programs like this both to create new collaborations and for potential career advancement.

- Are there any issues you would like to record?

  I don't want to record any issues.