StoRIES

STORAGE RESEARCH INFRASTRUCTURE ECO-SYSTEM

2nd IREP Call Documentation - User

StoRIES IREP1 Post Research Questionnaire (with satisfaction survey): User (s)

After the StoRIES International Research Exchange Programme stay, users are required to submit a post research questionnaire. This should be done within 4 weeks after the Access is completed unless otherwise agreed. The Post Research Questionnaire form will be given to the User(s) by the Project Management. The report contains sections related to the work performed, the main results and observations that were achieved.

[redacted]

Summary questionnaire for users who have been granted International Research Exchange Programme (IREP) under the StoRIES project Horizon 2020 IREP scheme.

General information about the project	
Project title (as used in application)	Towards Material Acceleration Platforms for Energy Storage Materials Within Circular Economies
Project acronym (max 15 characters)	RE-MAP
StoRIES IREP RI(s) accessed	*University of Toronto, M5G 1Z5*
Keywords (up to ten, free text)	Material acceleration platforms, self-driving labs recycling, battery slurry optimization
Arrival date (in town where RI located)	05.01.2025
Departure date (from town where RI located)	11.02.2025
Starting date of access (first day at RI)	07.01.2025
Finishing date of access (last day at RI)	07.02.2025
Number of days not using the RI (during the above period)	4

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Reason for not using RI those days (describe)	I did not work on Sundays. I was very happy to be granted access to AC on Weekends to use the time as efficient as possible.
Number of days using the RI	28
Number of users granted access (group size)	1
Comments	
User	
User group leader or sole applicant (user group member 1)	
First name	[redacted]
Last name	
Affiliation / Employer	Fraunhofer-Institut für Silicatforschung ISC
Country of Employer	Germany
E-mail	[redacted]
Comments	
User group member 2	
First name	
Last name	
Affiliation / Employer	
Country of Employer	
E-mail	
Comments	
User group member 3	
First name	
Last name	
Affiliation / Employer	
Country of Employer	
E-mail	
Comments	
User group member 4	
First name	
Last name	
Affiliation / Employer	

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Country of Employer	
E-mail	
Comments	
Please insert more fields if your groups had more than four members.	

Access Summary Report - work performed and initial results

Brief description of the objectives of your project (up to 200 words)

[Please describe short the main objectives of your project]

The main objectives of this project include investigating the concept of input characterization within the framework of Bayesian optimization, with the goal of automating the generation of physical process models that establish a relationship between measured inputs and outputs. This work serves as a foundation for applying Bayesian optimization techniques in recycling applications. Additionally, experiments are being designed to prepare for optimization activities related to battery slurry processes.

Activities performed (up to 600 words)

[Please summarise the work carried you (steps taken, instrumentation used, techniques employed, data sources consulted etc.)]

The work conducted in this project focused on optimizing battery slurry through various experimental and software development steps. Initially, feasible proxy measurements for battery slurry optimization were identified using a pipetting robot, with specific attention given to viscosity optimization and the analysis of particle size and shape distributions via microscopy.

In the experimental preparations, a prototype for viscosity measurements was constructed using a pressure sensor connected to a motorized syringe. The pressure time-series data collected during aspiration and dispensing movements showed promising results in preliminary tests. It was later discovered that the Opentrons Flex robot already had a pressure sensor integrated into its pipettes, providing an efficient means for high-throughput viscosity measurements. Additionally, the available microscopy hardware, specifically the openflexure microscope, was explored for measuring particle size distributions. While improvements in lighting conditions were made, it was concluded that the full integration of particle size and shape analysis would require more time than was available for the project.

For software development, a program was created to interact with the pressure sensors of the Opentrons Flex robot, which were not easily accessible through the standard API but were available via the manufacturer's open-source GitHub repository. Three methods for extracting liquid viscosities using the pressure sensor were developed and compared: pressure-decay-time fits, pressure-integral, and time-dependent derivative evaluations. The pressure-integral method emerged as the most reliable, while the time-dependent derivative evaluations provided insights into the non-Newtonian behavior of the liquids, which is relevant for battery slurries used in slot-die casting.

Furthermore, software was developed for automatic model-building utilizing the Python library ax-platform. This involved implementing optimization strategies tailored for recycling, where the optimization procedure was divided into a model-building phase and a production optimization phase. In the model-building phase, pseudo input samples were repeatedly processed to generate

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a surrogate model. In the subsequent production optimization phase, this process model was utilized under fixed boundary conditions to expedite convergence towards optimal solutions while minimizing the number of optimization iterations, which is critical given the high costs associated with production processes.
Calibration curves for viscosity extraction were generated using viscosity standards ranging from 1 mPas to 1000 mPas. A physical demonstration of Bayesian optimization with an automatically generated surrogate model was also integrated into the experiments. The Opentrons pipetting robot was employed to prepare 30 samples of soap mixed with water impurities. Each sample was mixed to extract a process model for the dilution of impure water, and multiple production optimization runs demonstrated a tendency to converge significantly faster than traditional optimization methods.
The integration of a first physical demo-experiment aimed at demonstrating Bayesian Optimization involved the use of an Opentrons pipetting robot to prepare 30 samples of soap with water impurity. Each sample was subsequently mixed with water to extract a process model for the dilution of impure water. During the experiment, multiple production optimization runs were conducted that included an input characterization measurement. These runs demonstrated a notable tendency to converge much faster compared to initiating the optimization process from scratch.

Scientific results (up to 800 words)

[Summarise the (initial) outcomes of your study at the RI(s).]

The scientific results obtained from this project provide insights into the feasibility of using an Opentrons pipetting robot for various applications involving viscous liquids and particle size analysis.
Firstly, the transport of viscous liquid samples using the Opentrons pipetting robot has been confirmed as feasible. However, it is important to note that the dosing accuracy significantly decreases for liquids with viscosities exceeding 100 mPas. This decline in accuracy is primarily due to two factors: the tendency of viscous liquids to stick to the pipette tips and the extended aspiration and dispensing times required when using tips with smaller diameters. These challenges highlight the limitations of the pipetting robot when handling higher viscosity materials, necessitating careful consideration when designing protocols for liquid handling in such contexts.
Furthermore, the feasibility of conducting viscosity-proxy measurements with the Opentrons pipetting robot has been demonstrated at both the hardware communication level and the physics or data evaluation level. A comparison of multiple evaluation methods was undertaken; among these, the method of time-integrated pressure readings emerged as particularly robust. This method proved effective for accurately assessing viscosities. In contrast, derivative evaluation methods provided useful insights into the non-Newtonian behavior of the liquids tested, indicating their potential application in characterizing complex fluid dynamics. For additional details on these methods, refer to the project's documentation in the following GitHub issue: https://github.com/AccelerationConsortium/ac-training-lab/issues/141.
In terms of mixing capabilities, the standard “mix” method available in the Opentrons API has shown to have limited effectiveness for viscous liquids. This method relies solely on repeated aspiration and dispensing cycles, which can be counterproductive when dealing with higher viscosity liquids. In such cases, the movement of the liquid tends to be significantly delayed compared to the movement of the pipette plunger, leading to inefficient mixing. To overcome this limitation, a self-implemented “stir” routine was developed, which leverages the robot's ability to

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perform freely programmed Cartesian movements. This custom routine allows for mechanical stirring of the liquid within a well plate, resulting in better mixing outcomes. The optimal mixing results were achieved by strategically alternating between the “mix” and “stir” actions, illustrating the need for adaptive approaches when working with viscous materials.
Additionally, the project explored the feasibility of extracting particle size distributions from NMC particle dispersions using an openflexure microscope. Satisfactory image quality was obtained by employing a dropcasting technique, in which dispersed particles were placed on an object carrier and analyzed in transmission lighting mode. This approach yielded contrast-rich images, and the flat surface of the object carrier minimized defocusing during small-distance navigation. However, a challenge arose due to the design of the openflexure microscope, where the objective lens is mounted below the sample. This configuration required the samples to face downward, complicating the handling process, which involved dropcasting, flipping the samples, and managing an object carrier storage system.
Due to time constraints, the potential acquisition of a long-working-distance objective lens was not tested. Such a lens could enhance the ability to analyze samples without necessitating the flipping of the object carrier, thereby simplifying the process. Future work should consider this route, as dropcasting is a straightforward procedure that can be efficiently executed using the pipetting robot. The Opentrons gripper could also facilitate the transportation of object carriers within specially designed trays, further streamlining the workflow.

In its software-related part the project focused on recycling-related optimization, involving the development of algorithms and practical demonstrations to enhance process efficiency. A key goal was to establish an experimental setup that facilitates two critical phases: model-building and production optimization.
In the model-building phase, the objective was to automatically generate a process model that incorporates input characterization. This model aims to predict outputs based on specific measurements of the input materials. By systematically analyzing the relationship between input parameters and resultant outputs, the developed model serves as a valuable tool for understanding the dynamics of the recycling process.
The subsequent phase, termed production optimization, leverages the process model to optimize the same type of process using fixed but unknown input materials. The intention here is to achieve this optimization with as few iterations as possible, thereby enhancing efficiency and reducing resource expenditure during the production process. This aspect is especially crucial in recycling applications, where (input) materials can vary significantly in their properties and, therefore, behavior.
To implement these phases experimentally, the Opentrons pipetting robot was employed to automate the creation of a soap-mixing model. In this setup, various water-soap mixtures, which served as impure inputs, were characterized by their measured viscosities. By systematically mixing these soap-water combinations, the aim was to develop a comprehensive process model that accurately maps the relationship from input parameters—specifically, input soap viscosity and the fraction of impure soap—to the corresponding output viscosity.
Furthermore, this developed model was utilized to optimize the mixture fraction for a specified target viscosity. To illustrate this process, a manually mixed random soap-water solution, representing a potential recycling material, was used as input. By applying the previously built surrogate model, the optimization procedure aimed to determine the ideal proportions of soap and water necessary to achieve the desired viscosity, thereby demonstrating the experimental application of the presented concept.

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Overall, the outcomes of this project underscore the potential of combining automated processes with advanced modeling techniques to enhance recycling operations. The integration of the Opentrons pipetting robot not only streamlined the experimental setup but also facilitated a deeper understanding of the relationships between input materials and desired outputs, ultimately contributing to more efficient recycling practices.
Interpretation of the results (up to 400 words)
[Describe the main achievements during your stay at the site(s)] The primary achievement of this project was the successful implementation and prototypic experimental demonstration of a concept for utilizing Bayesian Optimization in the automated generation and application of surrogate models for recycling purposes. The integration of these concepts into battery slurry optimization was explored, with the identification of target-value driven optimization of liquid mixing processes as a feasible first experimental implementation. Software was developed to enable the use of pressure sensors in an Opentrons pipetting robot for retrieving viscosity proxy values without the need for additional instrumentation. An algorithm for the automatic generation of a liquid surrogate model, specifically for the toy model of mixing of soap and water, was integrated and executed in an example of the "model building phase". Initial experiments utilizing the surrogate model, along with input characterization measurements, demonstrated rapid convergence towards target values, achieving good matches in less than ten iterations during the presumed costly "production phase". This distinction between the model-building phase (laboratory activities) and the production phase (operational activities) will be beneficial for future research involving material acceleration platforms in the context of recycling. Additionally, further steps toward the actual optimization of battery slurries were identified, such as the measurement of particle size distributions through microscopy, though these remain to be implemented in subsequent activities.
Difficulties during the IREP related work (up to 250 words)
[List problems and issues you had, completing out your research project: Did you get access to all the necessary equipment, facilities, databases, etc.? If not, please specify the problems that occurred and list equipment the was not working or accessible.] • Retarded access to labs located at NRC (Toronto, Mississauga facility). This, on the other hand made valuable common activities and exchange with researchers at the Acceleration Consortium (Toronto, Downtown) possible. • Period of 1 Month limited the experimental activities to first examples. For publication-grade results, 3-5 Months would have been needed. Then, however, this stay would not have taken place in the first place. Compared to a 1-week stay at a conference, this 1-month period allowed for deep exchange and collaborative work with Acceleration Consortium and NRC researchers.

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Intended publications
[Explain where and how you expect to publish the outcomes of your project work. Include also anything already published (What and where?)] All activities are documented in publicly available git-issues connected to the git-repository of the AC-training lab: • "Autonomous battery slurry optimization system": https://github.com/AccelerationConsortium/ac-training-lab/issues/141 • "Test out optical microscopy on some dropcast battery slurry samples": https://github.com/AccelerationConsortium/ac-training-lab/issues/142 • "Implement optimization algortihm operating with changing measured boundary conditions (-> recycling)": https://github.com/AccelerationConsortium/ac-training-lab/issues/164 • "Improve Open Flexure Microscope imaging" https://github.com/AccelerationConsortium/ac-training-lab/issues/173 Developed source-code is publicly available at: https://github.com/MatPopp/STORIES_MP A collection of images / videos is planned to be published on youtube.
Data management
[Describe the further usage and storage of project data] • *Preliminary data of calibration curves for viscosity-measurements with the opentrons flex are available within the ipython notebooks within* https://github.com/MatPopp/STORIES_MP • *Source-codes are shared as described above.*
Conclusions / additional comments
[Provide any other comments you might have on your work]
Did you complete the EC user questionnaire available at https://ec.europa.eu/eusurvey/runner/RIsurveyUSERS ? (necessary)
☒ *Yes* ☐ *No*

Feedback – HSE, Ethics and Satisfaction

Please rate on a scale from 1 (excellent) to 5 (poor). Feel free to provide additional comments

	1 (excellent)	2	3 (neutral)	4	5 (poor)
Practical information on how to apply for IREP and the overall application process	☐	☐	☒	☐	☐

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When first submitting the application-form online I did not get any response if the submission worked out on a technical level (no submission notice). Communication worked out much better via E-mail, as soon as contact was established.	
Information provided, once your project was accepted, on how to proceed	1 (excellent) ☒ / 2 ☐ / 3 (neutral) ☐ / 4 ☐ / 5 (poor) ☐
[Comment]	
Support received at the site(s) regarding technical/scientific matters and logistics	Have you got sufficient support from the RI staff during the project? If not, please, specify the problems. ☒ *Yes* ☐ *No*
[Please specify any problems]	
RI extension / upgrades required	In your opinion, is the RI needed to be upgraded? If yes, please give an explanation. ☒ *Yes* ☐ *No*
Both the AC training lab, as well as the facilities at NRC Mississauga are still being built up. It was very interesting to be part of this phase. Scientific output will be facilitated, once all hardware is purchased and built up.	
Problems with local regulations	Have you had any problems with regulations of the visited RI owner (HSE, lab working hours, etc.)? If yes, please, specify ☐ *Yes* ☒ *No*
[Please specify]	
Health and safety issues	Did you encounter any health or safety issue during your research? Please provide details. ☐ *Yes* ☒ *No*
[Please provide details]	
Environment & Ethics	Did your research involve the use of elements that may cause harm to the environment, to animals or plants? Please provide details. ☐ *Yes* ☒ *No*
[Please provide details]	
Environment & Ethics	Did your research deal with endangered fauna and/or flora and/or protected areas? Please provide details. ☐ *Yes* ☒ *No*
[Please provide details]	

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Environment & Ethics	Did your research involve the use of elements that may cause harm to humans, including research staff? Please provide details. ☐ *Yes* ☒ *No*
[Please provide details]	
Environment & Ethics – Dual use	Does your research have the potential for military applications? Please provide details. ☐ *Yes* ☒ *No*
[Please provide details]	
Environment & Ethics – Misuse	Does your research have the potential for malevolent /criminal/terrorist abuse? Please provide details. ☐ *Yes* ☒ *No*
[Please provide details]	
Environmental issues	Were any potentially dangerous substances (materials / gases etc.) released into the environment (atmosphere, water, or land)? Please provide details. ☐ *Yes* ☒ *No*
[Please provide details]	
Ethics issues	Are there any other ethics issues that should be taken into consideration? Please specify ☐ *Yes* ☒ *No*
[Please provide details]	

Overall impression of communication and interaction after finishing your IREP related work

1 (excellent)	2	3 (neutral)	4	5 (poor)
☒	☐	☐	☐	☐

No problems, immediate responses to emails.

Suggestions for other international and EU facilities not included in StoRIES which you would use for your research

[Please provide suggestions for facilities, describe measurement / experiments you would like to perform]

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Suggestions how StoRIES can improve future IREP programme, how to make the IREP more impactful on the energy storage technologies and how to enable the achievement of high TRL levels.
The fraction of time spent on the application process + documentation was high compared to the time spent during the actual research activities.

Feedback – Pro-active Innovation Support	
Awareness	Did you know about the pro-active innovation support of StoRIES. ☐ Yes ☒ No
[Please specify how you learned about the pro-active innovation support]	
Personal experience	Have you taken advantage of or benefited from the pro-active innovation support? ☐ Yes ☒ No
[Please provide details]	
Information/service provided by the pro-active innovation support?	1 (excellent) ☐ · 2 ☐ · 3 (neutral) ☐ · 4 ☐ · 5 (poor) ☐
I don't know what is meant by this? Could you provide a link / desciption?	

I declare that the above provided information and especially that information on the number of days visited the RI is correct.

☒ *I have read the StoRIES privacy policy for participation in the StoRIES Accesses and consent to participation and the associated data processing.*

[redacted]

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