Production and characterization of vegetal powders in an open science approach

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Production and characterization of vegetal powders in an open science approach

by

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Summary

Acknowledgments

Special thanks to Agnès DURI, Charlène FABRE, Patrice BUCHE, and Claire MAYER for welcoming me and giving me their enthusiasm and support throughout this internship. I thank you for never ceasing to show confidence in my work and my abilities. This has been a new and different experience for me, working for the first time outside my home country. I have learned so much, and I have grown and acquired new knowledge and professional experience.

I'm very thankful to INRAE for the opportunity they gave me to participate in this interesting project. Furthermore, thanks to the UMR IATE and everyone working there, for welcoming me and helping me. Likewise, I would like to thank Christophe Fernandez et Julien Couteaux from IM2 in Bordeaux for their valuable and essential support in the realization of my internship.

Thanks to Agnès, for always encouraging me to do my best and having the best availability to help. Thanks to Charlène, for dedicating her time to training me with all the types of equipment and measuring devices that I used throughout the internship. Thanks to Patrice, for his mentorship and for helping me reach the best results. Thanks to Claire, even though we never meet in person, she has been an essential part of the project, always showing interest and confidence in my work.

Last but not least, my best appreciation to everyone involved in the interviews, their exchange with this project helped tremendously in its improvement.

1. Introduction

Intending to preserve knowledge and create more accessible and verified information, the Joint Research Unit for Agropolymer Engineering and Emerging Technologies (UMR IATE) proposed the conception of knowledge books (LDC, Livres des Connaissances). An LDC is an electronic hypermedia tool accessible online that gathers information to understand and learn about a specific concept or subject.

The main objective of this internship is to design a prototype of “PowderLib” whose main component is an LDC, related to the production and the characterization of vegetal powders, following an open science approach. That means that Data Papers, which contain experimental data and protocols made by the researchers in UMR IATE are directly accessible from the LDC.

These vegetal powders come from agricultural and wood by-products. They can gain value thanks to dry fractionation processes; the ultra-fine powders obtained have shown promising functionalities in energy and material applications. In UMR IATE, much research in milling, separation, and drying processes has been conducted in the past years, with the aim of further understanding the relationship between the processes and the raw materials. Since these unitary operations consume a lot of energy to obtain the targeted particle size (~15-50 µm) (Figure 1), it is imperative to find new solutions to obtain the same result with a lower carbon footprint. Also, the characterization of these powders is an important variable in the studies, considering the impact their physical and biochemical properties have on the procedures.

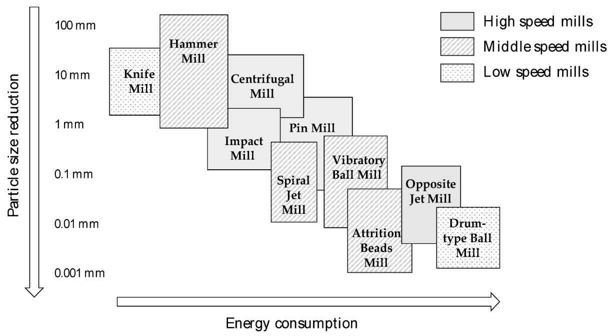


Figure 1. Exemplification of how the energy consumption increases in relation to the particle size reduction (Mayer-Laigle C., Rajaonarivony, R., *et al*, 2018).

This internship is a knowledge engineering project, willing to spread the information to anyone who can be interested in the production of vegetal powders. Four main target groups were identified as potential users of Powderlib; 1) students of master's degrees or engineering, 2) data scientists, 3) company engineers, and 4) experts in the domain of powders. PowderLib’s tools could be useful in many sectors that process powders, including agri-food, pharmaceuticals, composite materials, and biomass energy valorization. A series of interviews with future users were conducted to ensure that the LDC will answer their needs.

Since the objective is to provide information allowing a better understanding of the processes, the interface will be visual and easy to understand. Also, to address requirements expressed by experts in the domain of powders, a few decision trees will be developed with the Capex software tool which is the second component of PowderLib. These decision trees will help in solving powder-quality problems. The navigation between the knowledge book and decision trees facilitates information interlinking. Finally, PowderLib will be written in English to position it in an international scope.

A series of experiments of dry fractionation were conducted with the aim of better understanding the procedures done at UMR. The resulting powders will be characterized by loss of weight, humidity, and particle size and then stored to be used later on in the project Granothèque. This project collects scientific information about vegetal powders. The complete analysis of the results will not be performed throughout this internship.

The internship is foreseen to be conducted from the 21st of March to the 26th of August, this report shows only the progress done until the date of defense. The planning of the internship is shown in Appendix 1. The report is divided into 4 main sections, the state of the art, the methodology followed, results based on use-cases, and conclusions.

2. Structure of the host laboratory

The Joint Research Unit for Agropolymer Engineering and Emerging Technologies (UMR IATE 1208) in Montpellier brings together agents from INRAE (l'Institut National de Recherche pour l'Agriculture, l'Alimentation et l'Environnement), the University of Montpellier, and l'Institut Agro Montpellier. It includes about 90 permanent and 60 non-permanent staff divided into 10 teams (see Appendix 2). This unit studies the processing and exploitation of renewable resources from the forestry and agricultural sectors. Their main expertise is biochemistry, physical chemistry, process engineering, biotechnology, and numerical and computational modeling.

My internship is at the interface of 3 research teams, in which I work in collective to create the project of PowederLib. The PhyProDiv team (Physics of Processes and Divided Materials of Plant Origin) is interested in the controlled generation of plant biomass particles and the characterization of the physical and mechanical behavior within powders, dense suspensions, or filled composites from an experimental and numerical approach. The ICO team (Ingénierie des Connaissances) conceptualizes and develops methods and tools to create knowledge engineering projects, to support collective decision-making in agri-food systems. And the PLANET platform works in the comprehension and studying of the procedures of dry fractionation.

Four contributors participated in the supervision of the internship. Agnès DURI (PhyProDiv) for the characterization of powders and the realization of videos of the experimental protocols. Patrice BUCHE (ICO) is the co-creator of Capex, one of the software used during my internship. Claire MAYER (PhyProDiv) and Charlène FABRE (PLANET) for the milling and separation part. In addition, Christophe FERNANDEZ and Julien COUTEAUX from the I2M team in Bordeaux participated by giving us the training on the informatics programs, especially in the software MakeBook. The project was financed by DataSusFood, a project funded by the French Agency for Research in which ICO is involved.

3. State of the art

3.1 Vegetal powder research

The vegetal powder world includes any powder coming from an organic source. But in the PLANET platform, the lignocellulosic biomass (LB) is the main raw material used in their research. LB is composed of cellulose (~20–60%), hemicelluloses (~10–40%), and lignin

(~10–40%) (Mayer-Laigle, C., Blanc, N., *et al*, 2018). Some examples of LB used in the research are wheat straw, wood, flax fiber, hemp fiber, and hemp hurds.

Usually, this biomass is considered as a waste, but it has been shown to be a great alternative to petroleum-based products. Some of the high-tech applications can be the production of bioethanol and composite material. But in order to exploit all the potential of LB, it is necessary to reduce its size to an ultra-fine powder (Mayer-Laigle C., Rajaonarivony, R., *et al*, 2018). To accomplish that goal it is necessary to undertake extensive milling itineraries that take even hours to achieve the targeted particle size.

The research is based mainly on finding solutions to reduce the time and energy consumption of the procedures of milling, drying, and separation. The present conditions of these procedures affect the overall profitability of the final product (Mayer-Laigle C., Rajaonarivony, R., *et al*, 2018). This can be done by analyzing physicochemical characteristics of the raw materials, type of mechanical stress, the operational parameters, and the type of drying technique (Mayer-Laigle, C., Blanc, N., *et al*, 2018).

Also, the study of the physicochemical characteristics and rheological properties of the raw materials is a key step to fully understand the phenomena occurring during fragmentation processes. Some examples of the analyses performed are particle size distribution, shape, specific surface area, and powder agglomeration (Rajaonarivony, K., *et al.,* 2019).

This type of valuable information coming from the research done at IATE, presented in the form of data papers, is the one that we are most interested in showing in the LDC because it can constitute a guide for the users.

3.2 LDC DOCaMEx as a guide and the Data Papers

DOCaMEx is a tool composed of two software components: an LDC generated with the Make Book tool created by the I2M team (Suciu *et al*., 2021) and Capex, a decision software tool created by the ICO team (Buche *et al*., 2019). DOCaMEx has been applied in the field of AOP French cheeses and its objective was to reduce the loss or distortion of the know-how of this domain.

The structure of an LDC follows a specific organization, relying on two main types of documents: conceptual maps (Cmaps) and knowledge sheets (FC). The conceptual maps graphically represent a set of concepts and the semantic relationships between them. The knowledge sheets provide more detail about the concept by using text and multimedia content such as images, sound, videos, equations, etc.

The data papers published by the PLANET platform are a key element of PowderLib: Milling itineraries dataset for a collection of crop and wood by-products and granulometric properties of the resulting powders (Fabre, C., Buche, P., *et al.,* 2020) and Biocomposites from poly(3-hydroxybutyrate-co-3-hydroxyvalerate) and lignocellulosic fillers: Processes stored in data warehouse structured by an ontology (Munch, M., Buche, P., *et at.,*2022). The data paper by Fabre, C., Buche, P., *et al.,* 2020 shows the milling steps, the equipment used, its operating parameters, and the granulometric properties (D10, D50, D90, span, and specific surface area) of the 19 by-products processed. The data paper by Munch, M., Buche, P., *et at.,*2022 presents 88 formulations to create biocomposites of mixed PHBV and LB powders. It includes the steps and the observations measured (crystallization, melting temperature of biocomposites, water vapor permeability, and mechanical properties.

4. Methodology

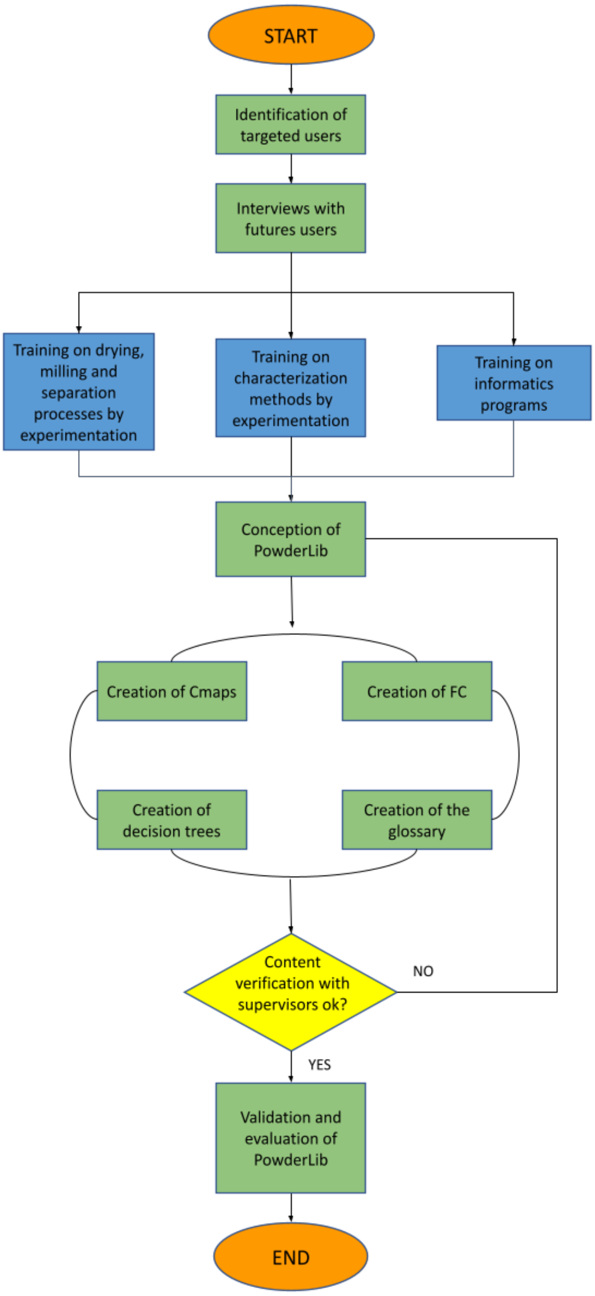


Figure 2. Workflow of the internship

The multidisciplinary methodology which has been designed and followed in this internship is shown in Figure 2. I started with the identification of users that could be interested in PowderLib, and the realization of interviews to determine the possible use cases.

Then, I was formed in the different procedures, methods, and informatics programs that gave me the tools to better understand and conduct this project.

For the conception of PowderLib, I implemented an iterative process in the realization of the software's inter-connected components: conceptual maps (Cmaps), knowledge sheets (FC), decision trees (Capex), and the glossary.

The step of content verification with supervisors was followed all along the internship during weekly meetings.

The validation of the content through use-cases will show if the users are able to solve a specific problem or situation.

The evaluation with cognitive load tests will not be discussed in this report because this step is scheduled after the date of the defense.

4.1 Identification and interviews with future users

The main objective of PowderLib is to create a tool for people who are interested in learning about vegetal powders or finding a solution to problems in powder production. The first task was to identify these users and to visualize roughly in which scenario they can take advantage of this new LDC. The users can be divided into two main categories: the experts of the domain and the non-experts.

The experts in the domain are researchers who could be very interested in finding new articles. The non-experts could be master's degree students, data scientists, and company engineers. The master's degree students can use the website as a reliable resource of information for university classes. The data scientist can have access to the data sets and also to essential information to comprehend the variables of the data paper. The company engineer can use the information to solve different problems in their production line.

So in order to be able to answer the precise needs of the users, a series of interviews were scheduled. This will help to know the perspectives and expectations of the users on the tool developed. This exercise helped settle the type of information and how it will be presented on the platform. The list of interviewees and the topics discussed are presented in Table 1.

Table 1. List of interviewees

Interviewee	**Type of user**	**Main topic discussed**
Melanie MUNCH	Data scientist	What information is important for a data scientist to find in PowderLib to conduct an analysis of a data paper.
Magali WEBER and Caroline BANDU	Domain expert	Glossary: methodology to find scientific definitions.
Leslie LHOMOND	Domain expert	Agroalimentary point of view: durum wheat example.
Thierry RUIZ	Domain expert	Organization and selection of the most important characterization methods.
Luis CASTAÑO	Enterprise engineer	Possible defects and problems encountered through the milling, drying, and separation procedures.

The interview was first composed of a brief presentation of the LDC and the purpose of PowderLib. With the aim of giving the interviewee information to visualize and imagine the extent the project can have. After the presentation of the project, a series of questions were posed to the interviewee to initiate a further discussion about what they considered relevant to include. For example, some questions were:

- What is your opinion about the main topics selected for the home page of PowderLib?
- Is there any topic you would consider adding or modifying? If yes, please specify.
- What is your opinion on the design and presentation of the graphic content?
- Do you find the concept of Cmaps, FCs, and decision trees easy to understand?
- Is it convenient for you that PowderLib is a website?

4.2 Training and experimentation at UMR IATE

With the objective of better understanding the procedures of drying, milling, separation, and powder characterization, I performed a series of experiments. It is a fundamental part of the internship because it helps to correctly understand the principles and the mechanism of the types of equipment. By conducting these experiments it was much easier to imagine the different scenarios that the users could encounter and the potential problems they could have. All the milling procedures were done according to the specification of the PLANET platform.

The raw materials used were rice husk, hemp core, and pine bark, by-products from agriculture and the wood industry. All the raw materials were already pre-milled (size <5cm) and most of the time a drying treatment in an oven was applied, at 60ºC between 12 and 48h. Except for the pine bark which was sun-dried prior to oven-dried.

For the coarse milling, the knife mill Retsch SM300 (Appendix 3) was used. The product passes into the milling chamber where there are double counter knives. In this setup, shear forces and cutting are the main milling mechanisms. For each experiment, a grid of 2 mm was used and fixed to a velocity of 3000 rpm.

For the intermediate milling, the Alpine UPZ100 station (Appendix 4) was used with the hammer configuration. In this case, impact and shear are the main milling mechanisms. This mill needs to be fed with a product with a particle size below 2mm. The ground powders obtained had a particle size of approximately 0.3 mm.

Additionally, two ball mills were used to achieve a much finer particle size: the SWECO (Appendix 5) and the FEMAG (Appendix 6). Both use compression and attrition as the main milling mechanisms. The SWECO uses ceramic pellets as grinding media made with 2 different geometrical shapes (50 %/W spheres and 50 %/W cylinders). The motor produces a vibrational movement that favors the distribution of the product over the entire mass of the media and allows a maximum surface contact (FABRE, C, *et al,* 2017). The FEMAG is a costume-made device designed by the Planet platform. It uses sphere-shaped stainless steel media. The FEMAG works by rotation of a tank which leads to the motion of the media. This mill can achieve particle sizes up to 2 µm.

During and after all the experimentations, the ground powders were characterized. Particle size, moisture content, and weight are measured (Appendix 7). The particle size measurement was performed with the laser particle size analyzer (LD 13 320 XR Beckman Coulter, Appendix 8), which determines the particle size distribution by laser diffraction. Some advantages of using this device are a wide range of analysis (from some mm to nm), the speed of measurement, and more detailed results in comparison to the sieves method (FABRE, C., & MARAVAL, G., 2021). The moisture content was measured with an infrared scale (Precisa XM60, Appendix 9), known as the “rapid” test.

Finally, for each device used, a video was recorded with an explanation of its principles and functionalities. These videos will be added to the LDC in the next steps scheduled after the defense of the internship.

4.3 Training on informatics programs

With the help of the I2M team in Bordeaux who designed Make Book software and the ICO team who designed Capex, we received training regarding the different computer programs needed to create the content of PowderLib. The required software are FreePlan, Capex, Cmap Tool, and Make Book.

Freeplan and Capex are used to create the decision trees. Freeplan is a mind mapping software that helps to create the draft file of a decision tree. In the draft file, the text is associated with nodes that create the link between the different ideas, this procedure creates the branches of the decision tree. When the information in the Freeplane document is correct, the document must be converted into a Markdown (MD) file. Then the MD file can be uploaded to Capex. Capex is the online platform that transforms the MD file into the final decision tree that will be visualized by the users.

Concerning the LDC, for the design and creation of Cmaps, one of the most important steps was to determine which modeling software was the most practical and efficient. The decision was made between PowerPoint, Canva, and Cmap Tool. In Table 2, we can see the pros and cons of these options.

Table 2. Comparison of different modeling software.

Software	**Facility to use**	**Design options**	**Price**	**SVG option**
PowerPoint	+ + +	+ +	Free with the INRAE account	No
Free Canva	+ + +	+ + +	Free	No
Paying Canva	+ + +	+ + + +	119,99 US$ / year (5 users)	Yes
Cmap Tool	+ +	+ +	Free	Yes

The final decision was mainly based on the possibility to transform the final image into a scalable vector graphic (SVG). The SVG format is preferable because it uses vectors to produce the image and this helps retain a better quality of the artwork. Indeed, a simple portable network graphic format (PNG) under 900 pixels can get distorted if PowderLib is viewed on a phone or a tablet. So with this in mind, PowerPoint and the free version of Canva were dismissed. Afterward, even if the paying version of Canva has a much wider design selection, it was not retained as an option because of its price. In conclusion, Cmap Tool is the knowledge modeling toolkit that was selected to create the design of the Cmaps. In the beginning, Cmap Tool was difficult to understand and maneuver, but it became a little bit easier with time. Lastly, the platform that allows putting together all the different inputs is MakeBook. This platform generates the HTML pages of the website. Figure 3. shows the different types of documents that can be uploaded and the logos used for each hyperlink. It has editing tools that are very easy to use:

- Upload, and delete the Cmaps images
- Creates the FCs (Illustration/ Schéma)
- Create, modify, and delete hyperlinks, Cmap to Cmap or Cmap to FC
- Add, modify, and delete keywords
- Add, modify, and delete bibliographic references
- Add, modify, and delete “menus”

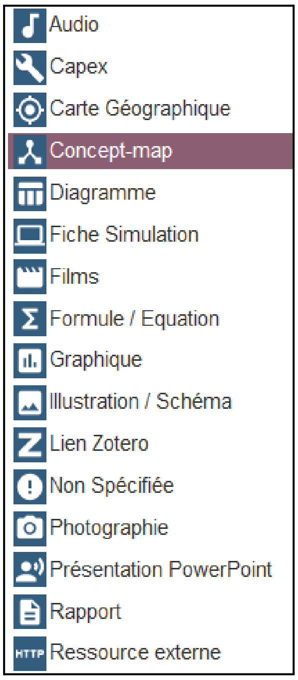


Figure 3. Different types of possible documents to upload/create in MakeBook

4.4 Conception of PowderLib

4.4.1 Creation of Cmaps

The Cmaps developed for this LDC were created by reusing and extending existing templates, such as Figure 4. These templates were provided by the I2M team, in a document including an explanation of the methodology to correctly create a Cmap. As said before, the principle of a Cmap is to use graphics to organize and represent the information. But depending on the domain, it is possible to have a certain freedom to arrange or change the semantic concepts. Nevertheless, it must follow an objective and be consistent according to the targeted audience (Team I2M, 2021).

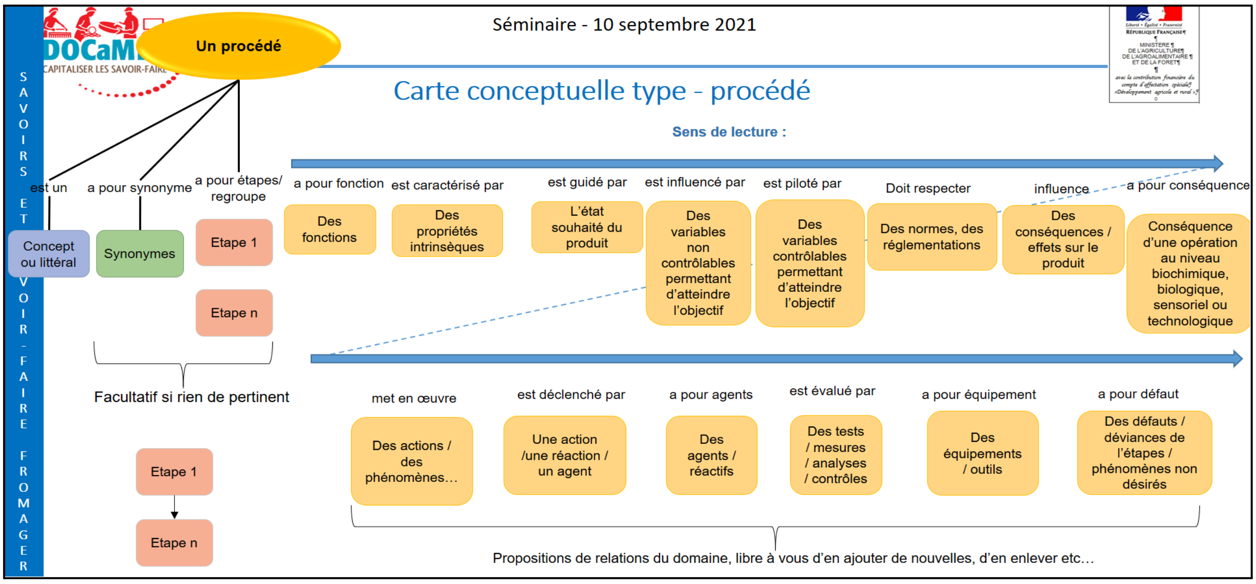


Figure 4. Template of a Cmap from DOCaMEx (Team I2M, 2021).

In this stage, we start to define standard terms that will become keywords defined in a glossary (see section 4.4.4). Also, the information displayed in the Cmaps must be concise and clear, to avoid disorientation and possible cognitive load on the user (Team I2M, 2021).

The steps to create a Cmap are

1. Choose the main concept
2. Write all the sub-concepts that can describe it
3. Organize sub-concepts into categories
4. Define the relevant relationships to relate the main concept to the sub-concepts
5. Carry out the bibliographic research on the selected topic
6. Make a verification of the Cmap with the supervisors

Even though PowerPoint is not going to be the final software to produce the SVG image, it was very useful to make the draft and visualize all the Cmaps in one document. So, the Ppt document will serve as a basis when the project is continued by someone else after the end of my internship (Appendix 10). Indeed all the information for the conception of additional Cmaps is gathered there. Additionally, the ppt was used as a tool to exchange with the supervisors. So the steps to edit a Cmap were to modify the base document in Ppt, modify it in CmapTool, redownload it as SVG, and re-uploaded to MakeBook. This series of steps guaranteed a better organization but with the downside of being very time-consuming. This is because sometimes the corrections appear gradually and some Cmaps could need an edition or correction frequently. For example, some Cmaps were edited and re-uploaded up to 15 different times.

4.4.2 Creation of FCs

The process to create an FC is a little bit different, it is the only type of document that is directly created in MakeBook. This means that the presentation format will be the same for every FC and generated by the software. The FCs are a more detailed explanation of a certain topic, so it is possible to develop a more complex idea. It can be accompanied by images, equations, sounds, or videos. Just as the Cmaps, the FCs need constant editing.

4.4.3 Creation of decision trees

As mentioned before, the decision trees were firstly done in Freeplane. The methodology was to select a problem/default, for example, "obtaining a particle size too large compared to the targeted one". So based on the issue, meetings with Charlene FABRE were scheduled to brainstorm and gather all the possible causes and the solutions to this defect. After having finished the decision tree, the MD document is uploaded to Capex. It is possible to link the decision trees of the Capex site to the elements (FCs or Cmaps) of the MakeBook site. This creates a direct relationship between the two components of PowderLib and the user can navigate between the two websites.

In the decision tree in Capex, the user can navigate between the different possible scenarios until it arrives at a "corrective action" (Figure 5) or a "compensatory action" (Figure 6). Corrective actions will show a "long-term" solution to the problem and a compensatory action will show the "instant" reaction to have in these situations just to solve it in that particular moment of a given experiment.

Figure 5. Logo of a corrective action

Figure 6. Logo of a compensatory action

4.4.4 Creation of the glossary

A glossary was made to help with the standardization of terminology and the ease of understanding of the terms. A selection of keywords was chosen to be defined in English since it is the main language, but the glossary also provides a translation in French and Spanish.

A bibliographic research was conducted to obtain the definitions and the search engine Web of Science was used to get access to scientific papers. The research of the terms wasn't easy, because each definition could be different depending on the context and the field. So it takes not only the time to find a definition, but also to verify if it is the correct one in the context of vegetal powders.

I directly translate into French and Spanish with the support of online software: Reverso and Deepl. Likewise, if relevant, some synonyms could be added to some definitions. A CSV document in Open Office was created as the editing software for the glossary. This type of format helps to preserve the accents used in French and Spanish, unlike Excel which tends to distort the characters.

The final terms will also be used in other projects, such as an ontology of biorefinery. The definitions will help to enrich the existing terms. This BIOREFINERY ontology is available in INRAE dataverse (https://doi.org/10.15454/X2MOWO, version 2.0) and Agroportal (http://agroportal.lirmm.fr/ontologies/BIOREFINERY, version 2.1). It permits vocabulary standardization in order to minimize ambiguities in the datasets published in data papers.

4.5 Evaluation of the prototype

4.5.1 Evaluation of use-cases

In order to evaluate if PowderLib does answer the needs of the users, it is necessary to establish use-cases. These use-cases are scenarios in which we guess a question that a user may ask and try to see if the information does lead to a valid answer. We propose 3 use-cases presented in the results section.

4.5.2 Evaluation tests

The final step and maybe one of the most important is the evaluation with users. This part of the internship will be developed starting the second week of July. This will be done by performing a series of interviews with a questionnaire. The results of the questionnaires will indicate the level of comprehension/retention of the information, the cognitive load, and the disorientation. The steps identify for this task are

1. Presentation of the instructions and the tool PowderLib,
2. Fill in the information sheet,
3. Single choice pre-test,
4. Free navigation on the website (20 min),
5. Cognitive load and disorientation test (Appendix 11),
6. Scenario-based navigation (20 min),
7. Single choice post-test, a second time,
8. Cognitive load and disorientation test, a second time,
9. Review questionnaire,
10. Optional questions.

5. Results

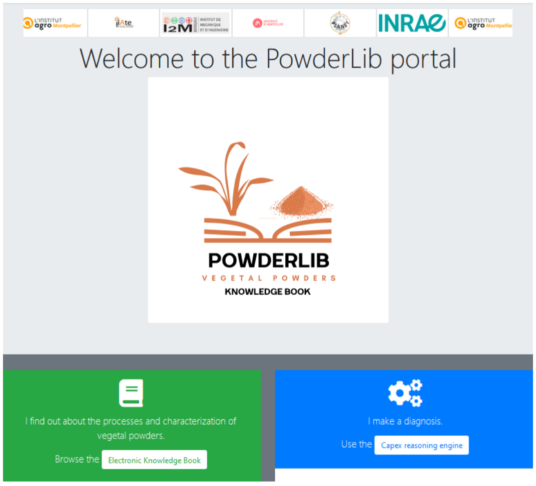


Figure 7. The main page of the portal PowderLib

The access to the prototype of the PowderLib website is available at the next link, https://icotest.iate.inra.fr/powderlib_portail/ (user: testPowderLib, password: secret). In the portal (Figure 7), there is access to both the LDC and Capex.

A total of 14 Cmaps, 12 FCs, 2 audios, 2 Capex decision trees, and the information of 2 data papers were integrated into the site until the date of the defense. In this report, only some of the Cmaps, FCs, and decision trees will be shown according to the 3 use cases priorly defined.

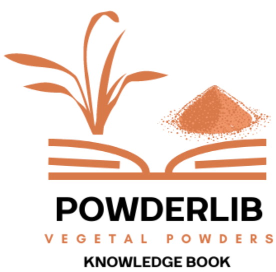


Figure 8. PowderLib logo

Additionally a logo was designed with Canva. This logo shows an open book with a plant to represent the organic biomass and a pyramid of powder. All three components englobe the idea of PowderLib.
The colors and the font were chosen to be simple and easy to read.

5.1 Home page

As mentioned before, one of the first steps was to create the home page (Figure 9). The design, organization, and main topics were based on the homepage of DOCaMEx (section 3.2). The choice of the sub-topics was a complex reflection because the vegetal powder covers a large field. This reflection process and the final selection of sub-topics were coordinated by the supervisors and guided by the resulting discussion with some interviewees.

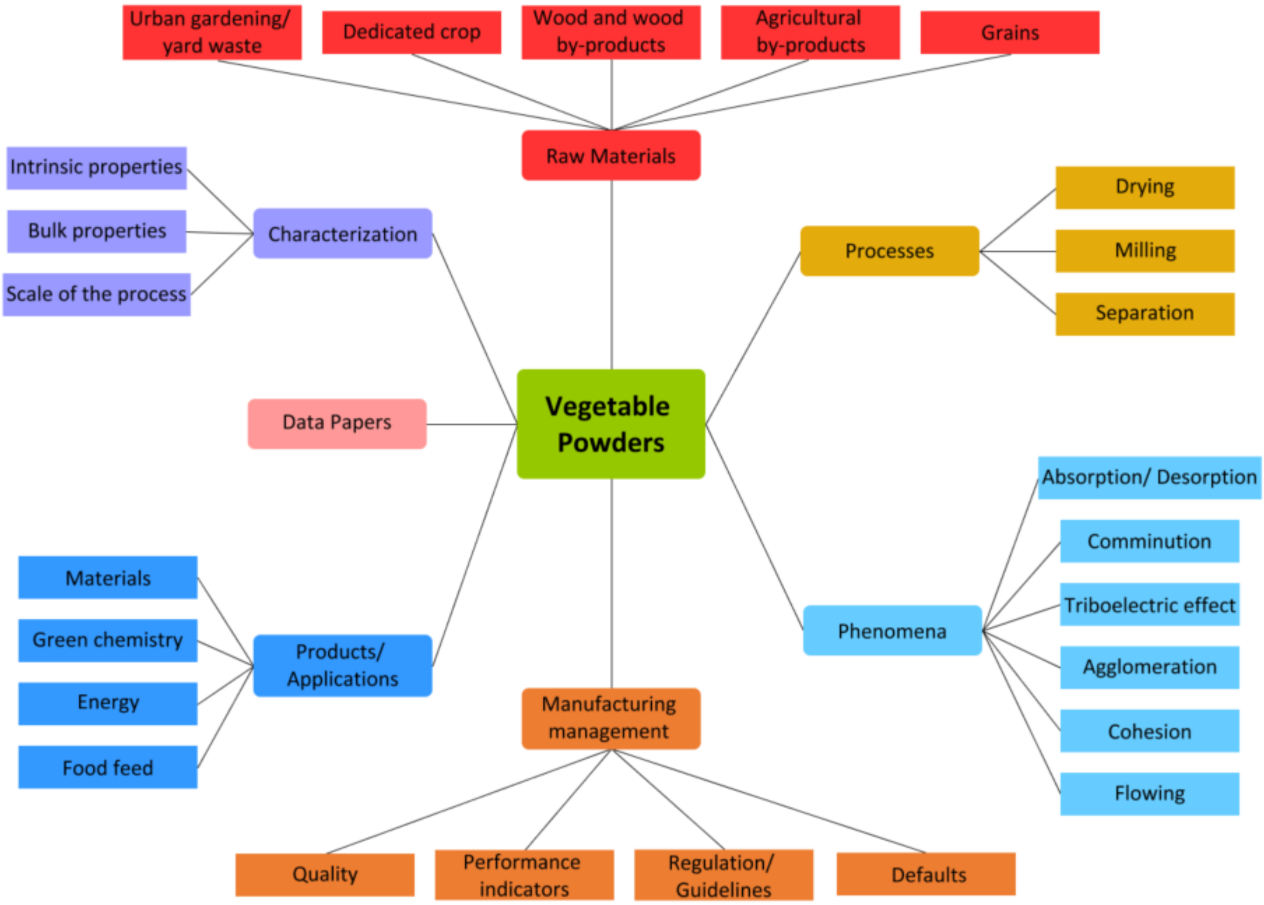


Figure 9. Home page of PowderLib

Because PowderLib deals with the production of vegetal powders, the processes considered to be explained are drying, milling, and separation. The differentiation between a process and a phenomenon was based on the fact that the processes correspond to unit operations in which several phenomena can occur alone or in combination. In addition, phenomena can be induced by the process or on the contrary unwanted. Each phenomenon will be related to one or several processes. At this stage, we selected 6 phenomena: absorption/ desorption (involved in drying), comminution, agglomeration (involved in milling), triboelectric effect, cohesion, and flowing (involved in milling and separation).

The characterization topic is one of the most important ones. A lot of brainstorming and re-structuring was done because it is a complex and vast section. Finally, after several iterative steps, this topic was divided into 3 groups:

1) The intrinsic properties of the powder correspond to the properties measured when there is no interaction between the particles. This category gathers morphology, affinity with water, biochemical, and matter structure properties.

2) The bulk properties of the particles are the result of the combination of several intrinsic properties and/or interactions between particles. This category regroups rheology, mechanical, hydro-textural, and electrostatic properties.

3) The scale of the process gathers properties resulting from the interaction between particles/particles and particles/processes.

The other topics included in PowderLib are displayed on the home page but the Cmaps cannot be all completed during this internship. The raw materials were placed into topics according to the way they are obtained. For the manufacturing management topic, the sub-topics are quality, performance indicators, regulation/ guidelines, and defaults. The products and applications can be extensive, but we focus here on the main application developed by the UMR IATE in the past years: materials, green chemistry, energy, and food feed.

5.2 Use-case: Data Scientist and data papers

Situation: A data scientist enters PowderLib to find more information about a data paper.
Question: How and what are the variables measured?

The open science approach of PowderLib gives the users access to the data papers produced by the Planet platform. Figure 10. shows the resulting Cmap of one of the data papers. Thanks to MakeBook it is possible to define a hyperlink to the DOI, marked with the HTTP (green circle) symbol as shown in Figure 10. so the user has direct access to the article.

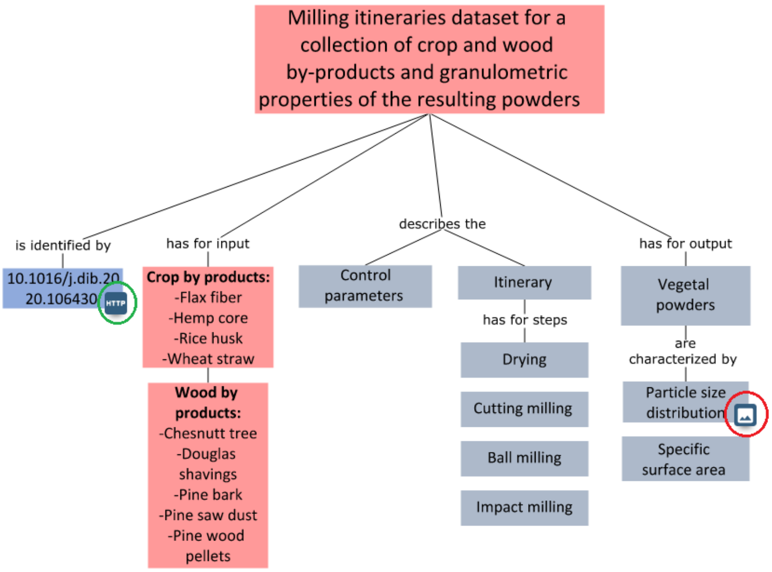


Figure 10. Example of a Cmap of the data paper Milling itineraries

In this use case, the user wants to know the variables measured in the data paper. So in the Cmap, we can easily find this information: particle size distribution and specific surface area. Furthermore, since it is possible to link a Cmap with other Cmaps or FCs to enrich the topic (red circle in Figure 10), the Cmap of this data paper is directly linked with the FC of particle size analysis (Appendix 12). With this utility, the user is able to learn about this variable. According to the interviewee Melanie MUNCH, this is an important feature that will help the data scientist to comprehend the data set that he/she will analyze. The design of the Cmaps patterns associated with data papers is an original contribution of my internship which provides a rapid overview of the data paper's content.

5.3 Use-case: student

Situation: A university student needs to find information about the milling procedure.

Question: What are the parameters we can control for a milling machine? What is comminution?

When the student enters PowderLib, he/she can open the Cmap of milling, shown in Figure 11. The reading sense of the Cmaps always starts from the top to the bottom. For example, “Milling” “is controlled by” “milling parameters”. “Milling” “has as default” “undesired agglomeration of particles”, etc. The most important and basic information about the milling process is shown in the Cmap. In order to enter more in detail about a certain sub-topic in the Cmap, the user can navigate between the different hyperlinks.

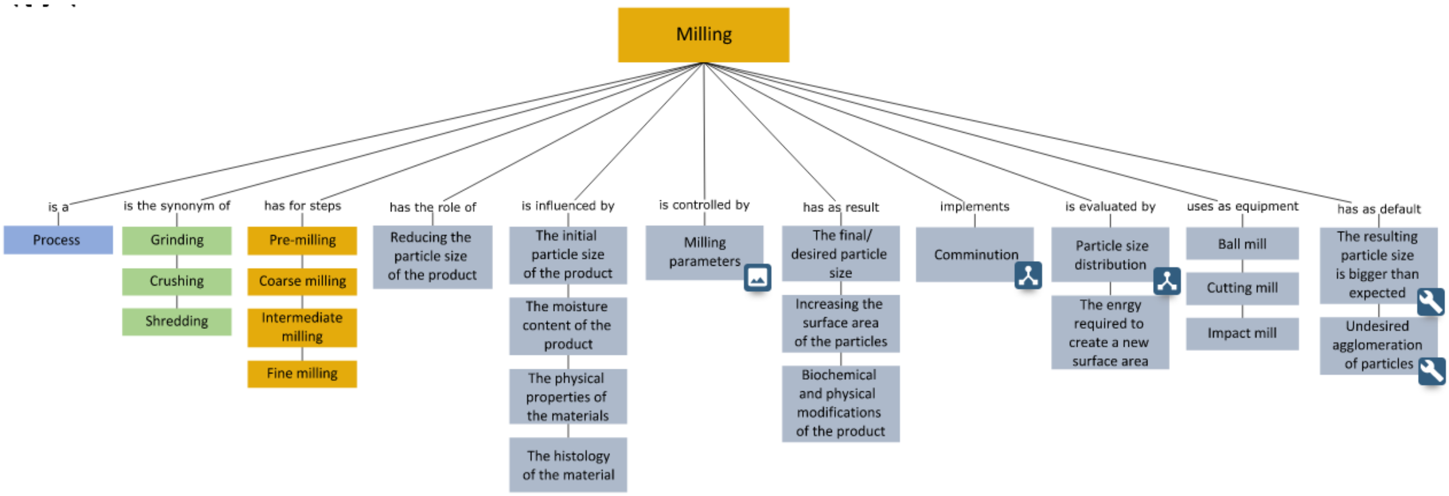
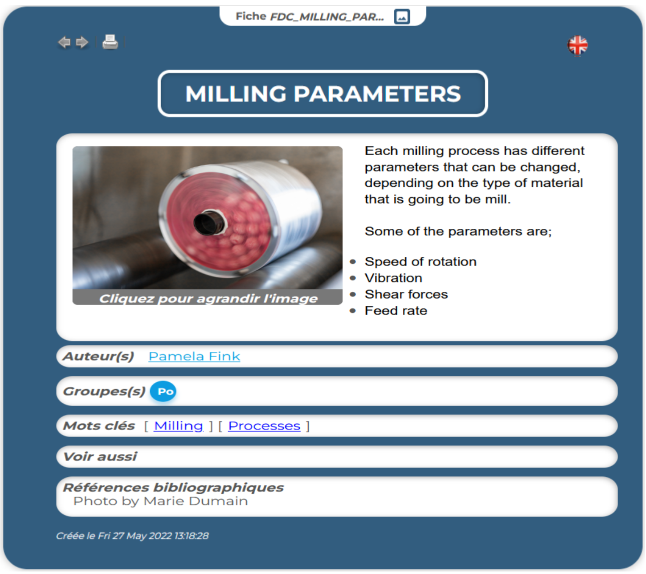


Figure 11. Cmap of milling



For example, next to the text of “milling parameters” he/she can find the logo of the FC as shown in Figure 11. Once he/she clicks on the link, the FC of milling parameters (Figure 12) will open in a pop-up window. In this FC, he/she will find the answer to their first question: speed of rotation, vibration, shear forces, and feed rate.

Some other features of an FC are in the bottom part of Figure 12: the author of the FC, the groups or LDC that is related to that FC, the keywords, and the bibliographic references.

Figure 12. FC of “milling parameters”

Finally, to solve the second question he/she can easily access the glossary through the menu “Glossary” on the left of the website (Appendix 13). There, a list of all the definitions is displayed and he/she will find the definition to comminution (Figure 13). The glossary has 30 keywords, and their definitions are being uploaded to the MakeBook platform. The format used in the CSV document to define each keyword is shown in Appendix 14:

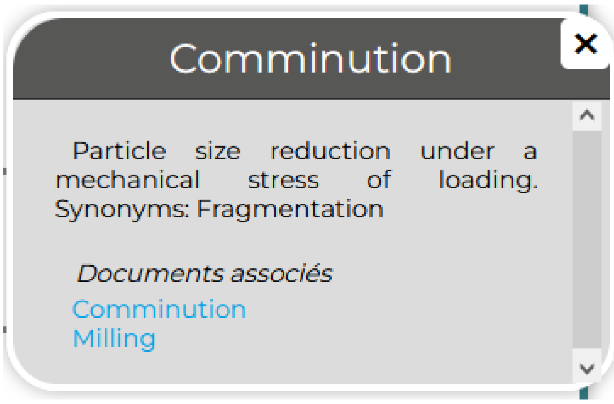


Figure 13. The pop-up window for the definition of Comminution

5.4 Use-case: expert of domain

Situation: A researcher is having problems with the step of coarse milling, the particle size is not getting reduced as it should.
Question: How can I achieve the targeted particle size in coarse milling?

A total of two Capex decision trees were done. Each decision tree will represent a different type of problem and the possible variations it can have. The user can navigate between the possible causes until it finds the one that fits their situation. At the end of each sequence, the user will arrive at a corrective action or a compensatory action.

For example, Figure 14 shows the decision tree for the defect: “Particles too large in comparison to the expected median particle size {coarse grinding, SM100, SM300}”, the user can select between the first two decisions 1) mill setting not suitable, 2) the engine consumes too much energy. If the user selects option 1, another series of options will appear and if the user selects “feed rate too high” the corrective/ compensatory actions will appear.

In this case, one of the corrective actions is to check the noise of the mill (green circle). Two recordings were made to help the user identify the difference between a good motor sound and the sound of a forced motor. This is a perfect example of how Capex and the LDC can be interrelated, the sounds are uploaded through MakeBook (to the LDC) and by doing a simple coding technique, the sounds can be linked to the decision tree. The access to the recordings is shown in Figure 14 in the green circle as two blue squares. With this information, the user can find the solution to their coarse milling problem.

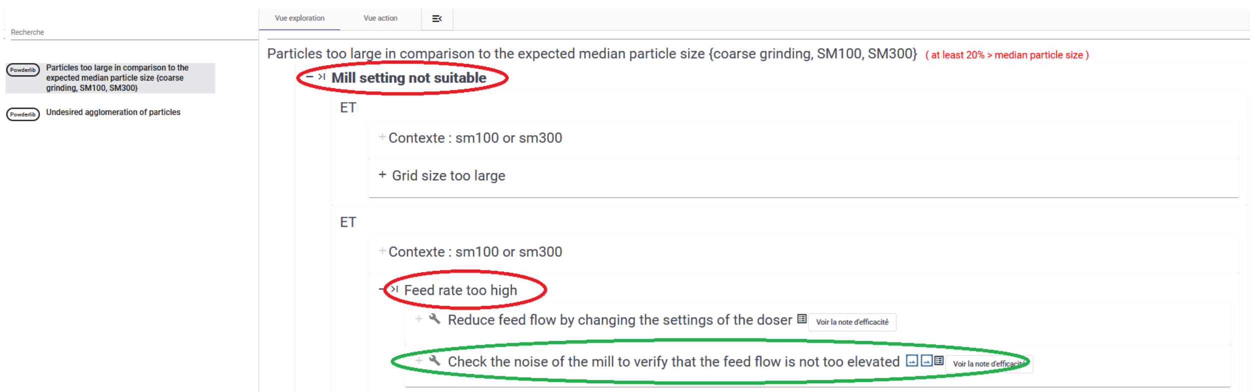


Figure 14. Example of the decision tree “Particles too large in comparison to the expected median particle size {coarse grinding, SM100, SM300}”

6. Conclusion

The main objective was to create a prototype of PowderLib. The conception of an LDC can take several years, because of the amount of information that is necessary to cover. So within the time established for the internship, the resulting prototype is on a good track. Having the websites of the LDC and of Capex is a very tangible result of this objective.

The experimentations with the processes were very interesting. It helped me to gather ideas and to better understand the processes and the know-how of the UMR IATE. There were some minor drawbacks during the experimentations but mainly all of them were accomplished adequately. Finally, the data gathered in this step of the internship will be treated in the Granotheque project which gathers in particular all the data on the powder characterization. Also, the interviews helped a lot to correctly ground the ideas. Knowing what the users expected of PowderLib was essential. I feel that the result would not be the same without this feedback.

As mentioned before, the results shown are just a partial outcome of the internship. There are still a few Cmaps, the evaluation with users, and some powder characterizations that are scheduled to be done in the remaining 2 months of the internship.

The experience at UMR IATE has been very enriching, it was the first time I had the opportunity to work in a research laboratory, especially outside of my home country. This has given me a lot of new skills and knowledge. I am learning a lot about how research is done, the stakes, and the compromise. I appreciate the opportunity to be involved in a realization of a project like this and to collaborate with my supervisors and all the professionals who were involved with the project. With my supervisors, I feel my opinions and contributions are heard and appreciated. I have greatly valued what I have learned from them.

I feel like I am working with my capabilities of responsibility and organization, as this was the most challenging part for me. Also, this internship helped me tremendously with my French and English skills. As well as complimenting my knowledge in the powders field, as I already had experience, this internship gave me a wider perspective.

In the future, I see myself still working on projects involved with the improvement of the environment, but not necessarily in the research field. I feel drawn to working more directly in the industry.

I look forward to the day PowderLib will be finally ready to be shared with the world.

7. Bibliography

- Fabre, C., Buche, P., Rouau, X., & Mayer-Laigle, C. (2020). Milling itineraries dataset for a collection of crop and wood by-products and granulometric properties of the resulting powders. *Data in Brief*, *33*, 106430. https://doi.org/10.1016/j.dib.2020.106430

- Fabre, C., Rajaonarivony, K., & Mayer-Laigle, C. (2017, July). *Instruction, Broyeur SWECO* (No. 1). IATE.

- Suciu, I., Ndiaye A., Baudrit C., Fernandez C., Kondjoyan A., et al.. A digital learning tool based on models and simulators for food engineering (MESTRAL). *Journal of Food Engineering*, Elsevier, 2021, 293, pp.110375.

- Mayer-Laigle, C., Rajaonarivony, R., Blanc, N., & Rouau, X. (2018). Comminution of Dry Lignocellulosic Biomass: Part II. Technologies, Improvement of Milling Performances, and Security Issues. *Bioengineering*, *5*(3), 50. https://doi.org/10.3390/bioengineering5030050

- Mayer-Laigle, C., Blanc, N., Rajaonarivony, R., & Rouau, X. (2018). Comminution of Dry Lignocellulosic Biomass, a Review: Part I. From Fundamental Mechanisms to Milling Behaviour. *Bioengineering*, *5*(2), 41. https://doi.org/10.3390/bioengineering5020041

- Munch, M., Buche, P., Dervaux, S., Breysse, A., Berthet, M. A., David, G., Lammi, S., Rol, F., Viretto, A., & Angellier-Coussy, H. (2022). Biocomposites from poly(3-hydroxybutyrate-co-3-hydroxyvalerate) and lignocellulosic fillers: Processes stored in data warehouse structured by an ontology. *Data in Brief*, *42*, 108191. https://doi.org/10.1016/j.dib.2022.108191

- Buche P., Cuq B., Fortin J., Sipieter C.. Expertise-based decision support for managing food quality in agri-food companies. *Computers and Electronics in Agriculture*, Elsevier, 2019, 163, pp.104843.

- Rajaonarivony, K., Rouau, X., Lampoh, K., Delenne, J. Y., & Mayer-Laigle, C. (2019). Fine Comminution of Pine Bark: How Does Mechanical Loading Influence Particles Properties and Milling Efficiency? *Bioengineering*, *6*(4), 102. https://doi.org/10.3390/bioengineering6040102

- Team I2M. (2021, September 10). *ATELIER création de contenus* [Slides].

8. Glossary

- LDC: Livre de Connaissance; Book of Knowledge
- Data Papers
- UMR: Mixed Research Unit
- Capex: website with arborescences
- Cmaps: Conceptual maps
- FC: Fiches de connaissances; Knowledge sheets

9. List of Tables

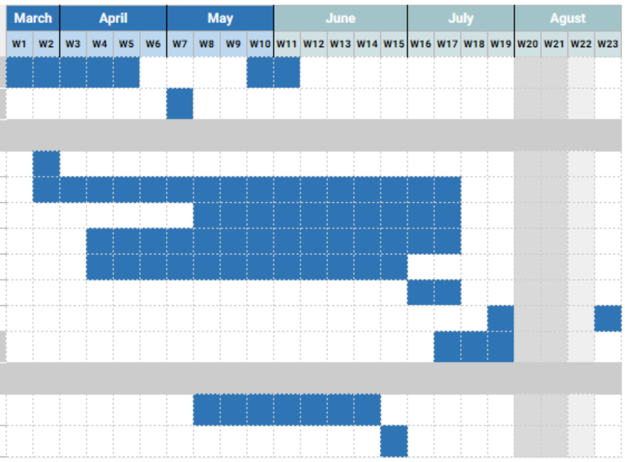
10. List of Figures

11. Appendices

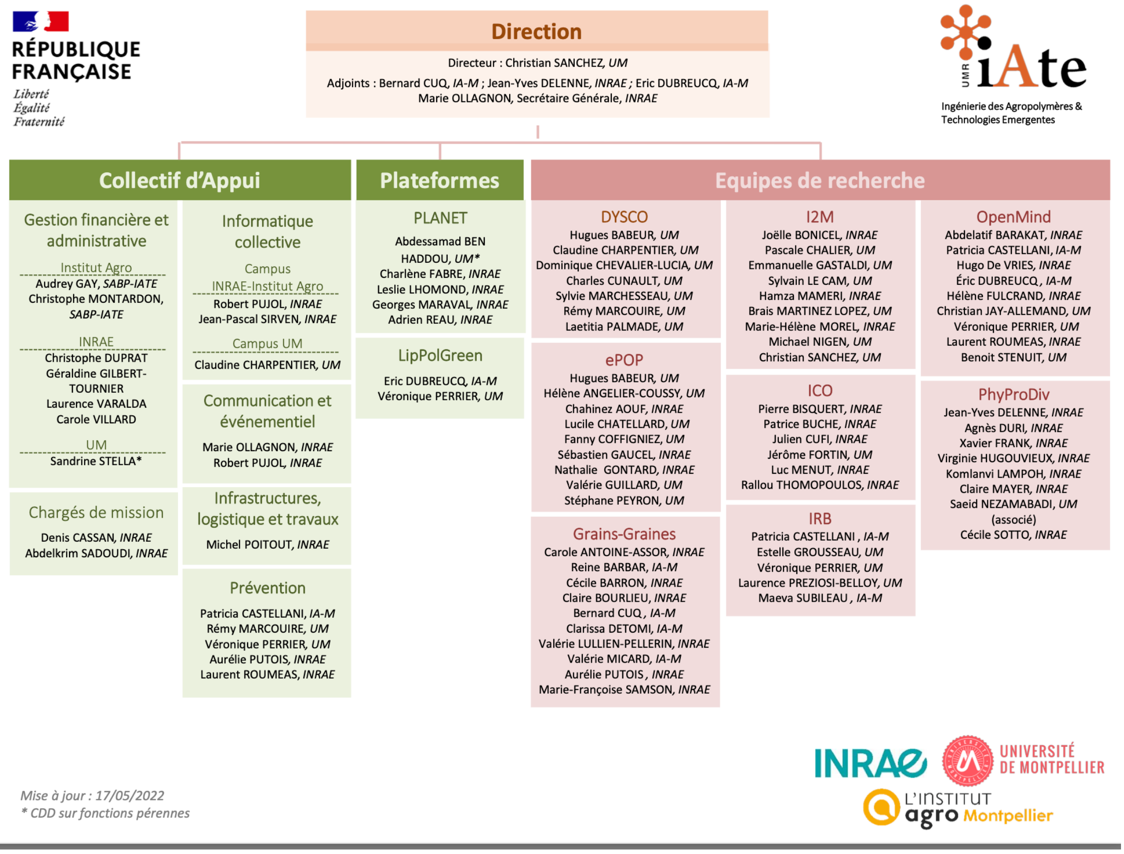
Appendix 1. Diagram GANTT with the planning of the internship.

Production and characterization of vegetable powders in an open science approach		INRAE UMR IATE	
		DATE	21 March 2022 to 26 August 2022

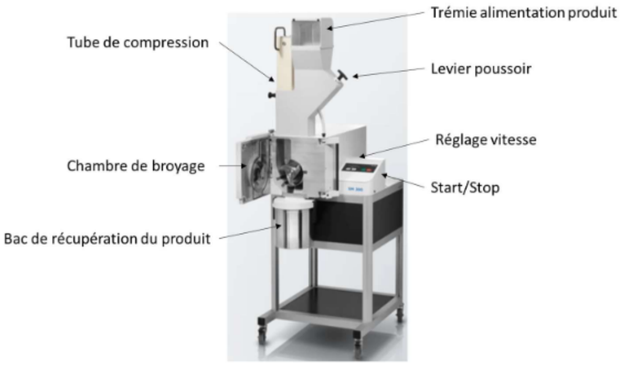
	Title of the task	Starting date	Duration days
1	**Formation and experimentation in UMR IATE**		
2	**Formation in Bordeaux**		
3	**Creation of the prototype of the LDC**		
3.1	Creation of the home page	26/03/22	2
3.2	Creation and content validation of Cmaps and knowledge sheets	26/03/22	30
3.3	Uploading content to the platform	10/05/22	20
3.4	Creation of the glossary	13/04/22	20
3.5	Interviews with possible users	13/04/22	20
3.6	Knowledge homogenization phase	04/07/22	3
3.7	Final revew of the prototype	22/08/22	5
4	**Evaluation of the prototype of the LDC**		
5	**Raport**		
5.1	Creation of the raport	05/03/22	20
5.2	Defense of intenrship	01/07/22	1



Appendix 2. The organizational structure of UMR IATE



Appendix 3. SM 300 mill



Appendix 4. UPZ Alpine mill

Appendix 5. SWECO ball mill

Appendix 6. FEMAG mill

Appendix 7. Results of milling experimentations

Code	Raw material	Steps	Weight	Moist content	Particle size (D50)
RH-29-22-2	Rice husk	Sample preparation	1533 g	9.33%	-
		-Drying at 60 °C for 44h	1424.9 g	2.80%	-
		-Milling in SM300 at 3000 rpm grid 2mm	1416.6 g	2.81%	937 µm
		-Drying at 60 °C for 72h	1395.6 g	2.73%	-
		-Milling in SWECO 30 min			807 µm
		90 min			506 µm
		150 min			349 µm
		225 min			171 µm
		285 min			49.58 µm
		295 min			59.45 µm
		325 min	1368.7 g	2.80%	20.74 µm
RH-30-22-1	Rice husk	Sample preparation	1579.4 g	9.61%	-
		Drying at 60 °C for 30h	1451.9 g	2.84%	-
		Milling in SM300 at 3000 rpm grid 2mm	1438.1 g	3.78%	845 µm
		Drying at 60 °C for 30h	1412 g	3.05%	-
		Sieve RITEC, >400 µm, 200 µm, and <200 µm	400µm: 1215.8 g 200µm: 99.6 g bottom: 48.6 g	8.72%	>400µm: 1027 µm 200µm: 319 µm <200µm: 71.57 µm
RH-21-22-1	Rice husk	Sample preparation	1490.9 g	10.33%	-
		Drying at 60 °C for 72h	1361.5 g	2.70%	-
		Milling in SM300 at 3000 rpm grid 2mm	1346.2 g	2.88%	1154 µm
		Drying at 60 °C for 1 week	1339 g	2.89%	-
		Sieve RITEC, >400 µm, 200 µm, and <200 µm	400µm: 1185 g 200µm: 74 g bottom: 62 g	3.98%	>400µm: 1498 µm 200µm: 412 µm <200µm: 156 µm
PB-12-22-1	Pine bark	Sample preparation	3760.9 g	36.06%	-
		Air drying for 3 days	-	19.53%	-
		Drying at 50 °C for 72h	2117.2 g	5.39%	-
		Milling in SM300 at 3000 rpm grid 2mm	2106.9 g	5.77%	416 µm
		Drying at 60 °C for 30h	2070.7 g	4.27%	-
		Alpine UPZ100 pallet milling grid 0.3 mm	2077.1 g (1) 990 g	4.86%	129 µm
		Division of the sample in two parts (1) and (2)			
PB-22-22-2	Pine bark	Sample preparation (2)	(2) 1087.1 g		-
		Drying at 60 °C for 30h	1070.9 g	4.49%	-
		Milling in SWECO 30 min			41.73 µm
		40 min			34.17 µm
		50 min			29.26 µm
		60 min			23.52 µm
		65 min			22.09 µm
		72 min	1016 g	4.87%	19.73 µm

Appendix 8. Laser particle size analyzer

Appendix 9. Precisa XM60

Appendix 10. Example of the slides in the PowerPoint base document (Drying Cmap and Drying FCs)

FC drying role

Drying is a process consisting of reducing the moisture content of a solid material, from an initial value to a target value. The evaporation of the water is done through the use of heat and airflow.
This loss of water preserves the product and reduces its weight and volume.
Drying is also an important step to facilitate the milling and limit agglomeration phenomena.
By combining different drying methods we can improve the drying efficiency.

Credit Photos : Marie Dumain

FC variability of the product

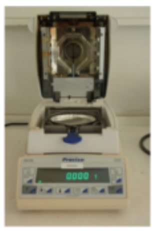
The composition and characteristics of the plant materials differ according to its origins, species, maturity... All these properties have a significant impact on the drying process.
The process parameters of the drying process, should be defined by taking into account:
-The initial moisture content, (the higher it is, the longer it will take to dry the product)
-The shape and size of the particle influencing the geometry and thickness. For a denser or thicker powder bed, the heat can take longer to rich the heart/center.

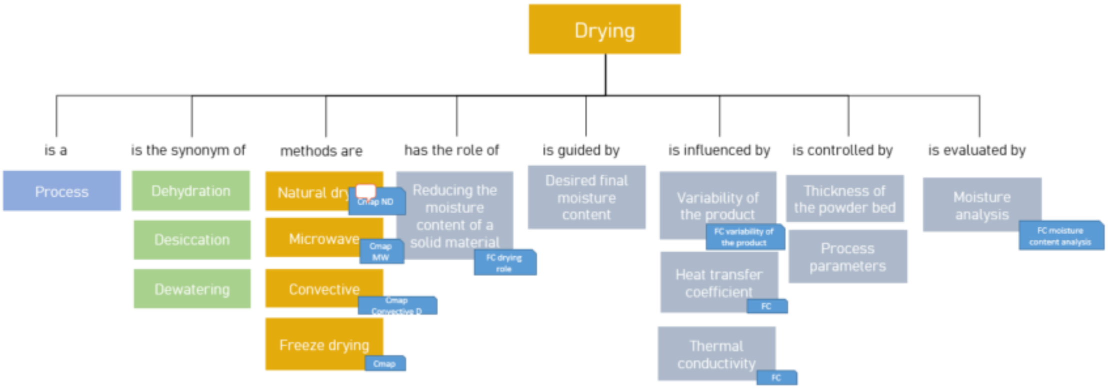
Credit Photos : Marie Dumain

FC moisture content analysis

The moisture content represents the quantity of water in a product, in percentage.
It is a characteristic that influences many properties of the product, such as its weight, density, crushability, etc.
There exist two methods to obtain this measure; loss upon drying and the fast analysis.
Loss upon drying. In this method, a sample is carefully weighed and then placed in an oven to dry until it reaches equilibrium, then weighted again. The loss of weight represents the moisture content of the sample.
There is also the fast method, which heats the sample with higher temperatures using infrared, halogen, or microwaves, giving the result in a few seconds, it works whit the same principle as the loss on dry method

Mermelstein, N. H. (2009, November 1). Measuring Moisture Content & Water Activity. IFT. Retrieved June 1, 2022, from https://www.ift.org/news-and-publications/food-technology-magazine/issues/2009/november/columns/laboratory





Appendix 11. Example of the cognitive load and disorientation test of DOCaMEx

Test de désorientation n°1

Indiquez sur une échelle de 1 à 7, le niveau de difficulté rencontré à :

1) Comprendre les relations entre les différents documents du livre de connaissances

1	2	3	4	5	6	7
☐	☐	☐	☐	☐	☐	☐
Très faible	*Faible*	*Un peu faible*	*Moyen*	*Un peu fort*	*Fort*	*Très fort*

2) Comprendre les relations entre les différents documents de l'outil Docamex

1	2	3	4	5	6	7
☐	☐	☐	☐	☐	☐	☐
Très faible	*Faible*	*Un peu faible*	*Moyen*	*Un peu fort*	*Fort*	*Très fort*

3) Savoir quelle est la prochaine page à visiter

1	2	3	4	5	6	7
☐	☐	☐	☐	☐	☐	☐
Très faible	*Faible*	*Un peu faible*	*Moyen*	*Un peu fort*	*Fort*	*Très fort*

Appendix 12. FC of "particle size analysis"

PARTICLE SIZE ANALYSIS

It is a method of characterization based on the size of the particles, also known as granulometry, it uses statistic measurements of the particles to obtain a median size of the whole mix.

It can be analyzed with sieves or with a laser particle size analyzer (photo).

The sieves are formed by a round-shaped metal rings, with a grid made from wire, metal, plastic or tissue. These grids can have different opening sizes, the openings determine the particle size of the product that is going to be retained and the one that is going to pass through. We can have one or multiple sieves stacked one over the other, starting at the bottom with the one with the smallest opening size of the biggest on top. 100 grams of the product are placed on top of the pile of sieves, it is fundamental the use of circular motions and agitation to help the product go down, in some cases is necessary the use of brushes. The small fine particles pass through the openings and the larger particles are retained. The retained product in each sieve its weighted and the quantity represents the % of particles with the specific size of the sieve.

The laser particle size analyzer is a much more modern equipment which determines the particle size distribution by laser diffraction. Some advantages of using this device are a wide range of analysis (from some mm to nm), the speed of measurement, and more detailed results in comparison to the sieve method. Also, it need only between 5-2 g of sample. This device is able to give another type of measurements like the D10, D50, D90, SPAN, and the specific surface area.

Auteur(s) Pamela Fink

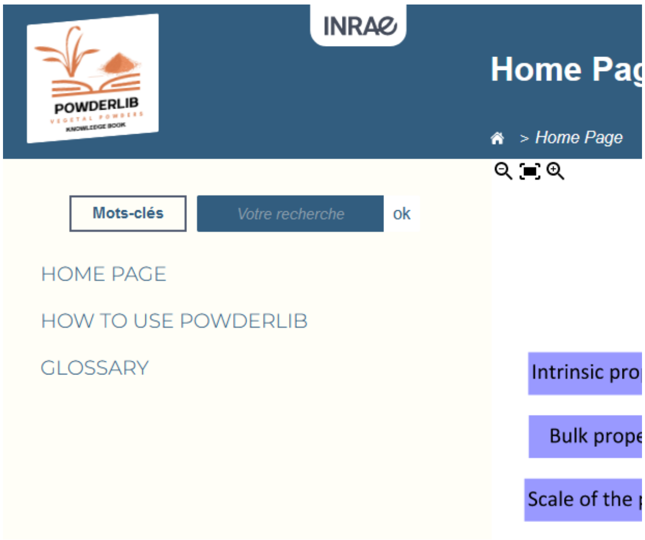
Groupe(s) Po

Mots clés [Milling] [Morphology] [Particle size]

Voir aussi

Références bibliographiques

Appendix 13. Example of the menus, including “Glossary”



Appendix 14. Example of glossary

English	Synonym	EN; scopeNote	Source
Agglomeration	Conglomeration, aggregation, clump	Act of agglomeration, of uniting in one whole. For particles: phenomenon that cluster separate particles in one biggest particle.	Larousse Disctonary/ Doi:10.3390/bioengineering6040102
French	Synonym	FR; scopeNote	
Agglomération	Agglutination	Action d'agglomérer, de réunir en un tout. Pour des particules, phénomène qui réunit en une seule particules des particules qui étaient isolés.	
Spanish	Synonym	ES; scopeNote	
Aglomeración	Conglomerado, agregación, apelmazado	Acto de aglomeración, de unión en un todo. Para partículas : fenómeno que agrupa partículas separadas en una partícula más grande.	

Production et caractérisation de poudres végétales dans une démarche de science ouverte

Résumé : PowderLib est un nouvel outil logiciel proposé pour aider à la diffusion de la recherche scientifique dans le domaine de la production et de la caractérisation des poudres végétales avec une approche de science ouverte. PowderLib est composé de deux logiciels : un livre de connaissances (LDC) et le système Capex. Un LDC, qui est un portail électronique avec des contenus hypermédia, a pour objectif de diffuser les connaissances relatives à une filière ou à une discipline. Capex est un système générique d'aide à la décision facilitant un choix technologique afin d'atteindre une certaine qualité de produit ou de résoudre un défaut dans un processus de transformation. Aujourd'hui, l'intérêt pour les poudres végétales augmente très significativement, avec de nombreux domaines d'application (par exemple, le bioéthanol ou les biomatériaux). Toutefois, le processus d'obtention de ces poudres (séchage, broyage, et séparation) peut présenter des défauts, l'information contenue dans PowderLib pourra aider à les résoudre.

Pendant ce stage, l'objectif était de créer un prototype de PowderLib. Afin d'acquérir les compétences nécessaires pour cela, une série de formations sur les procédures de broyage, de caractérisation des poudres, et de logiciels informatiques a été effectuée. Ensuite, le contenu de PowderLib a été créé en s'appuyant sur des entretiens avec des utilisateurs ciblés, en définissant des cas d'utilisation, en créant des cartes conceptuelles, des fiches de connaissances et en concevant des arbres de décision avec des experts du domaine pour résoudre des problèmes de production. Le prototype PowderLib enfin été évalué pour 3 cas d'utilisation.

Mots clés : Poudres végétales, livre de connaissances, fractionnement par voie sèche, caractérisation des poudres, arbre de décision.

Production and characterization of vegetable powders in an open science approach

Abstract: PowderLib is a new software tool proposed to help with the dissemination of scientific research in the field of production and characterization of vegetal powders with an open science approach. PowderLib is composed of two software components: a knowledge book (LDC) and the Capex system. An LDC is an electronic portal with hypermedia contents and has the objective of spreading knowledge of a specific industry or discipline. Capex is a generic decision support system that helps to choose the right technological action in order to reach a certain product quality or to solve a default in a transformation process. Nowadays innovation in the area of vegetal powders is increasing, with many applications of interest (eg. bioethanol or biomaterials). The process of obtaining such powders (drying, milling, and separation) can present problems, the information in PowderLib will help to solve these issues.

During this internship, the objective was to create a prototype of PowderLib. In order to acquire the necessary skills for this, a series of training on milling procedures, powder characterization, and informatics software were performed. Then the content of PowderLib was created by performing interviews with targeted users, defining use-cases, creating conceptual maps, knowledge sheets, and designing decision trees with domain experts to solve production problems. The final result was the creation and assessment of a prototype of PowderLib which was evaluated by 3 use-cases.

Keywords: Vegetal powders, knowledge book, dry fractionation, powder characterization, decision trees.

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