An understanding-based methodology for setting commercialisation objectives for an innovation project - a case study for platform chemicals from paper sludge

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At the very early stages of a project seeking to bring an innovative process and its associated product into the market, it is usually true that there is limited available data coupled with plentiful sources of uncertainty. The project team and business face two problems: how to decide whether to commit valuable resources to continued development; and how to set objectives for a continued development programme that will drive towards commercial viability.

The available data at the early stage considered here will typically take the form of lab-scale demonstrations using the proposed route and technology to generate the desired product. This is also generally common for a wider range of new product introductions (NPI), where the process route and technology are not necessarily seen as 'innovative' but nonetheless present challenges in the transition to commercial production.

This paper explores the role of improving process understanding in identifying and mitigating the risks encountered in scale-up and commercialisation. A methodology for systematic capture and development of process understanding to set objectives for continuing project development is described using the Industrial Strategy Challenge Fund supported LevWave project as a case study.

Capturing Process Understanding

The goal in capturing the current state of process understanding is to explore and define the underlying science of the phenomena being exploited by a process, and to identify the gaps in that knowledge. A critical aspect is thinking about the whole process, to understand the impacts of what is happening in one process task on preceding and following steps. Whole-process thinking also requires linking directly process understanding to the business objectives. The original 'BRITEST' (Best Route Innovative Technology Evaluation and Selection Techniques) collaborative project[1] developed a collection of process understanding tools initially aimed at improving communication between chemists and chemical engineers, but broadening in utility to a much wider range of disciplines with time. The tools have become a proven, effective means of interrogating chemical, physical and biotechnological processes. Several of the tools are used in the methodology now described.

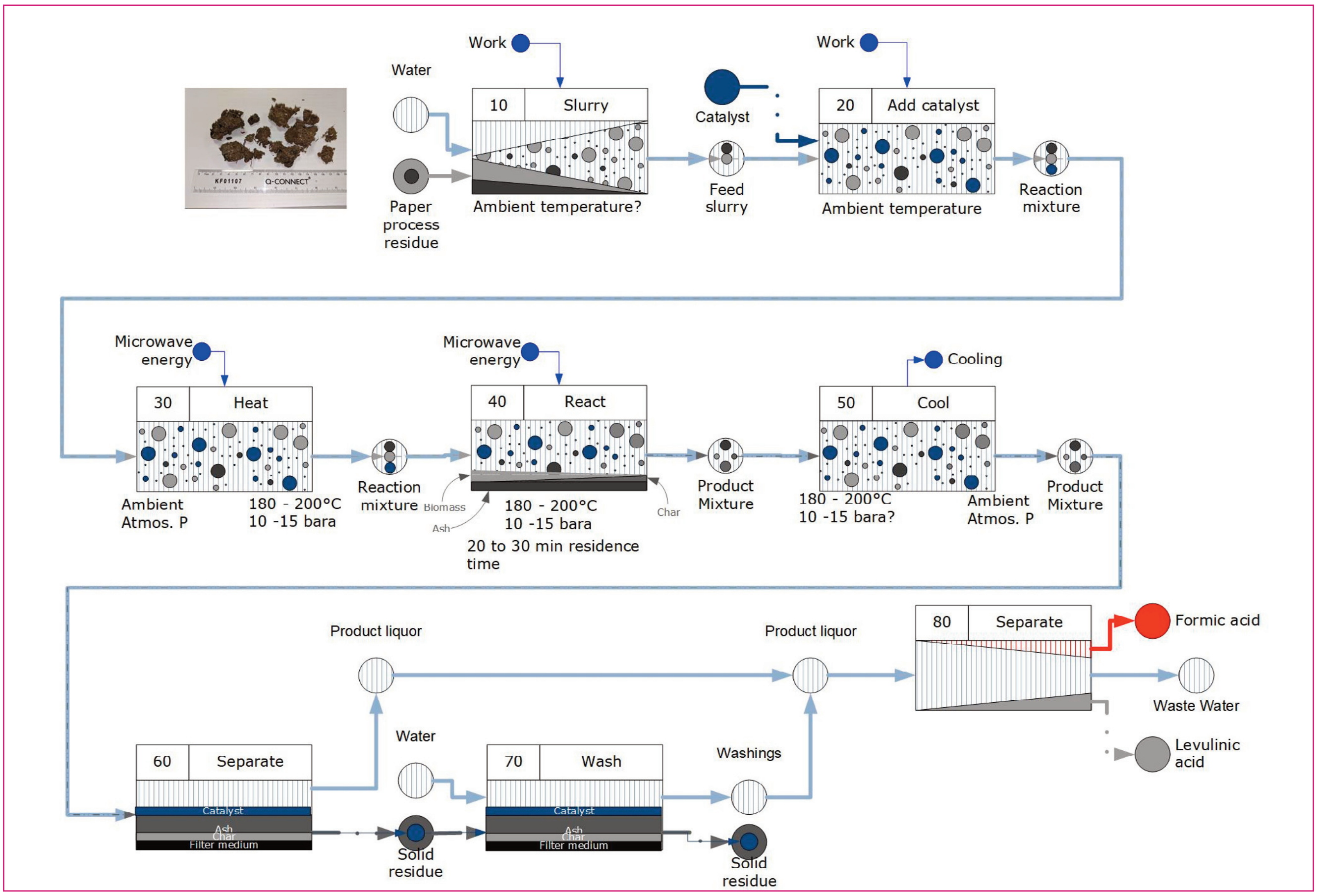

Figure 1: Original Process Definition Diagram (PDD) for LevWave.

Case Study

The LevWave project[2] considered the feasibility of producing the platform chemical, levulinic acid, from a paper recycling by-product stream[3] using a solid-state catalyst and microwave heating. The process is envisaged as continuous, with the paper sludge passing through a reactor within which it is heated by microwave radiation. A solid-state catalyst promotes the reactions that convert cellulose into levulinic acid through the intermediates glucose and 5-hydroxymethylfurfural.

Methodology

Develop the Whole Process Concept

The starting point is to develop and describe the whole process concept. The aim should be to describe the desired experiences that all process materials, molecules, or particles need to undergo to obtain the required product. One way of doing this is to create a Process Definition Diagram (PDD), a form of State Task Network.[4] A well-constructed PDD is independent of scale, equipment, and mode of operation (i.e., batch or continuous). This is possible because it considers the required new state of an entity after each task or experience, and the chain of these experiences required to transform the feed materials into desired products. Conducting several tasks in the same equipment becomes a design decision which inevitably involves some compromise in the desired experience which needs to be tested against the business drivers. It is this focus that differentiates a PDD from a classic equipment related Process Flow Diagram (PFD).[5]

The original PDD for LevWave is shown in *Figure 1*. The experimental programmes at Manchester Metropolitan University (MMU) and Drochaid Research Services (DRS) were focussed on understanding tasks 30 and 40. The PDD shown provides a qualitative description of the required processing experience for all the process materials in a complete LevWave process, in terms of material and energy balances, and phases present, including whether they are dispersed or continuous. The visually rich presentation highlights the salient features of the process in a form that can be easily assimilated by someone not involved in the project.

Develop Qualitative Mechanistic Models

From the whole process description developed it will be necessary to identify what are considered to be the critical tasks within the state-task network. Subsequent stages of the methodology will describe how visual annotations can help identify 'hot-spots' in terms of (for example) sustainability, safety, operability and scale-up; however, an initial prioritisation can be made using the information currently at hand. The project team can then 'home-in' on the task, exploring the relationships between the physical and chemical aspects of processing, and considering the effects of spatial and temporal variability upon how the task will be performed.

Rich Pictures, and Rich Cartoons when the passage of time is considered, are very useful for exploring these questions to help build a deeper level of process understanding for individual tasks in the PDD. *Figure 2* describes as a Rich Cartoon the mixing, heat and mass transfer phenomena occurring in the original lab microwave experiments for LevWave at MMU. The limited mixing of the solids in the reaction tubes is clear. The design intent for scale-up is to use a tubular microwave plug flow reactor and the very different conditions for the same three phenomena are apparent in the corresponding Rich Picture, *Figure 3*. Comparison of such figures can generally guide a project team's thinking towards key challenges in moving from the lab to pilot and commercial scale operations. In this case it is evident that reaction kinetic and performance data from the lab experimental set-up is not directly related to the scaled up situation. Careful interpretation of the existing results, and attention to experimental design, will be required in any follow-up work intended to support scaling-up the process. This leads to the need for a wider

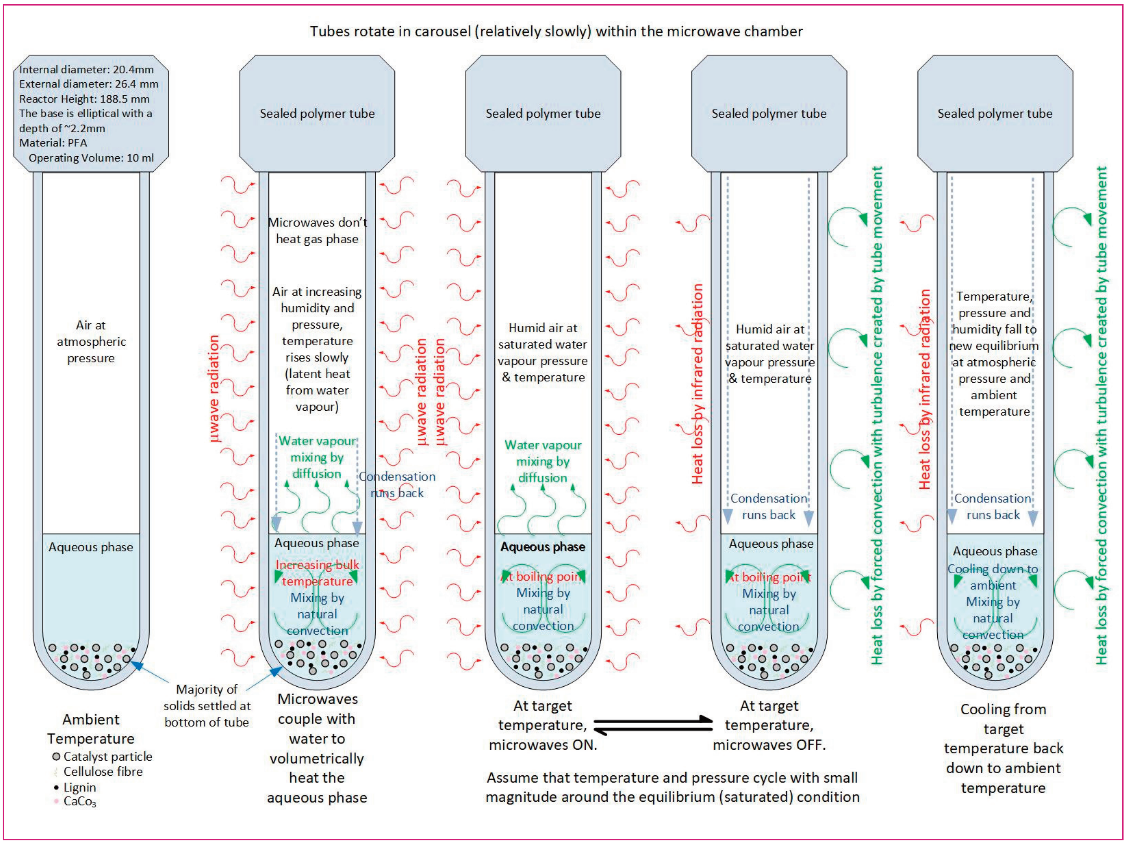

Figure 2: Rich Cartoon for the original lab microwave experiments at MMU.

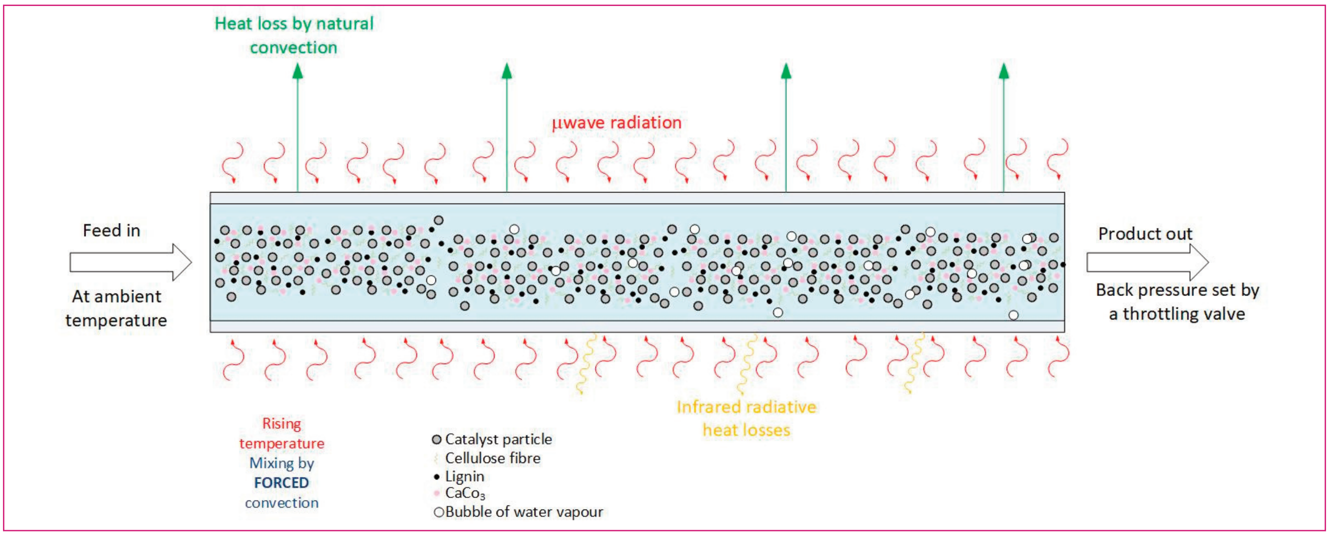

Figure 3 Rich Picture for microwave plug flow/tubular reactor proposed for scaling-up LevWave.

consideration of scale-up risk.

Formal analysis, such as FMEA, could be used to identify scale-up risks and define mitigation strategies. This and related approaches can be and are used by some organisations to assess the technological risks associated with a project but are exhaustive and time-consuming. At the very early stages of development, a 'short-cut' method is compatible with the limited extent of process knowledge the associated high uncertainty and may be more suitable and sufficient. Such a method is now proposed.

Preliminary Techno-economic Assessment

Techno-economic assessment (TEA) is a standard tool for assessing commercial feasibility. It usually accompanies detailed process design and market research and be used as a key data source for making the decision whether to commit significant capital investment. The Levwave project has explored the potential advantages of carrying out a preliminary TEA at a very early stage of a project when uncertainty is high and detailed quantified information is unavailable.

A tool such as Britest Limited's Process Information Summary Map (PrISM) can be useful to capture the outline process and possible costs[6] as a starting point for a preliminary TEA. PrISM captures a simple, semi-quantitative model of the proposed process and is easily extended in both directions along the supply chain.

By applying basic process engineering skills, with some additional data (mostly physical properties), simple heat and mass balances for the main process steps can be compiled. These provide the basis for rough sizing and costing calculations for the main equipment items and variable costs for the process materials. This is sufficient to give an outline of the cashflows and hence profitability (or otherwise) of the proposed process. Doing this with unoptimised lab-scale results would be expected to depict a very pessimistic picture for commercial viability. Analysis of the results will readily reveal how the more significant costs arise in the model.

The task therefore is to use the simple TEA model with some 'what-if' scenarios for reducing the costs and moving the proposal towards profitability. Successful scenarios become objectives to be achieved by the development team, guiding the development programme forwards.

For LevWave, six development objectives were identified in this way:

- Reduce catalyst to solids ratio (decreases cost of catalyst). Efficiently recover and re-use catalyst (decreases cost of catalyst).
- Significantly increase slurry concentration (reduces equipment size, energy costs, and improves technical feasibility).
- Significantly improved yield of levulinic acid from cellulose (increases revenue).
- Use recovered water from LA separation for slurrying (enhances sustainability).
- Recovery of heat from the slurry leaving the reactor to heat the slurry entering the reactor (reduces energy costs, improves sustainability and technical feasibility).

The impact of these scenarios on the TEA model for LevWave is illustrated in *Figure 4*.

Valuable guidance for project management and for stage gate decisions can thus be obtained at the earliest stages of a project because relative comparisons are being employed. This approach should not be extended into later stages of when significant capital expenditure decisions are required, and a more absolute model result is essential.

Prompt	Tolerable impact	Review/check impact	Significant impact
Cost of goods	C	C	C
Water usage	W	W	W
Energy usage	E	E	E
Pollution	P	P	P
Materials usage efficiency	M	M	M
Time to process	T	T	T
Quality	Q	Q	Q

Table 1: Sustainability prompts used for LevWave.

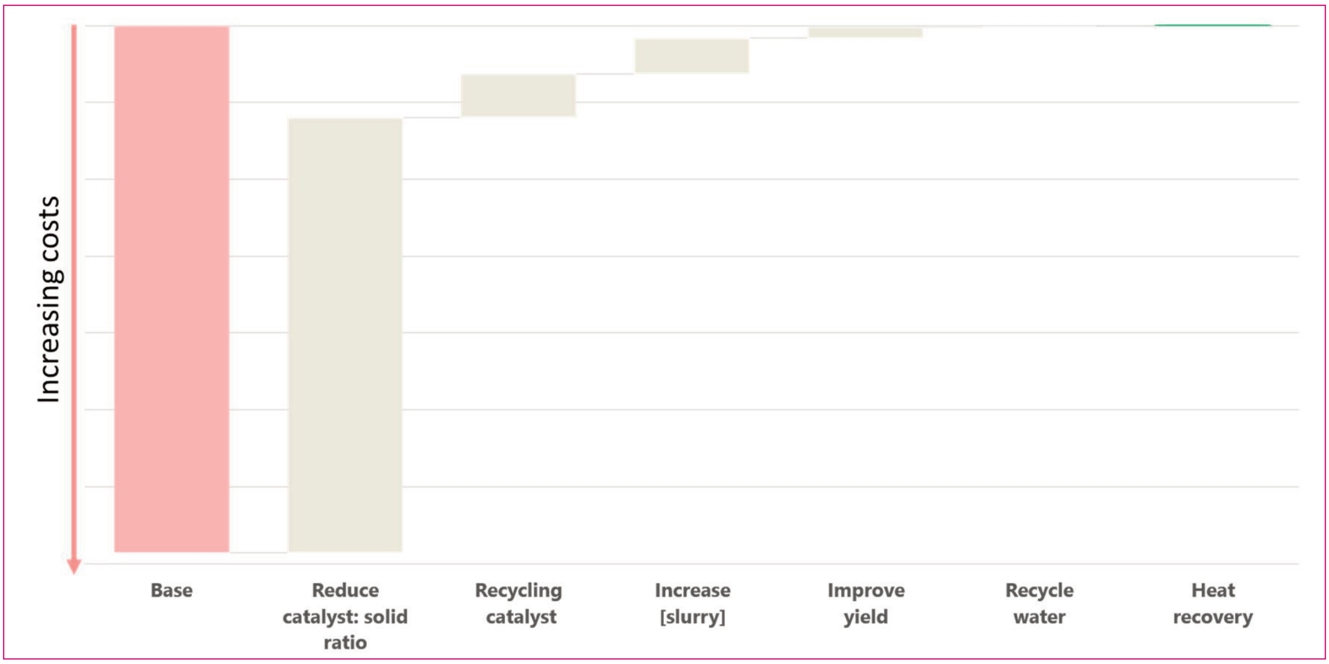

Figure 4: Waterfall diagram showing the impact of development options on the preliminary TEA model for Levwave.

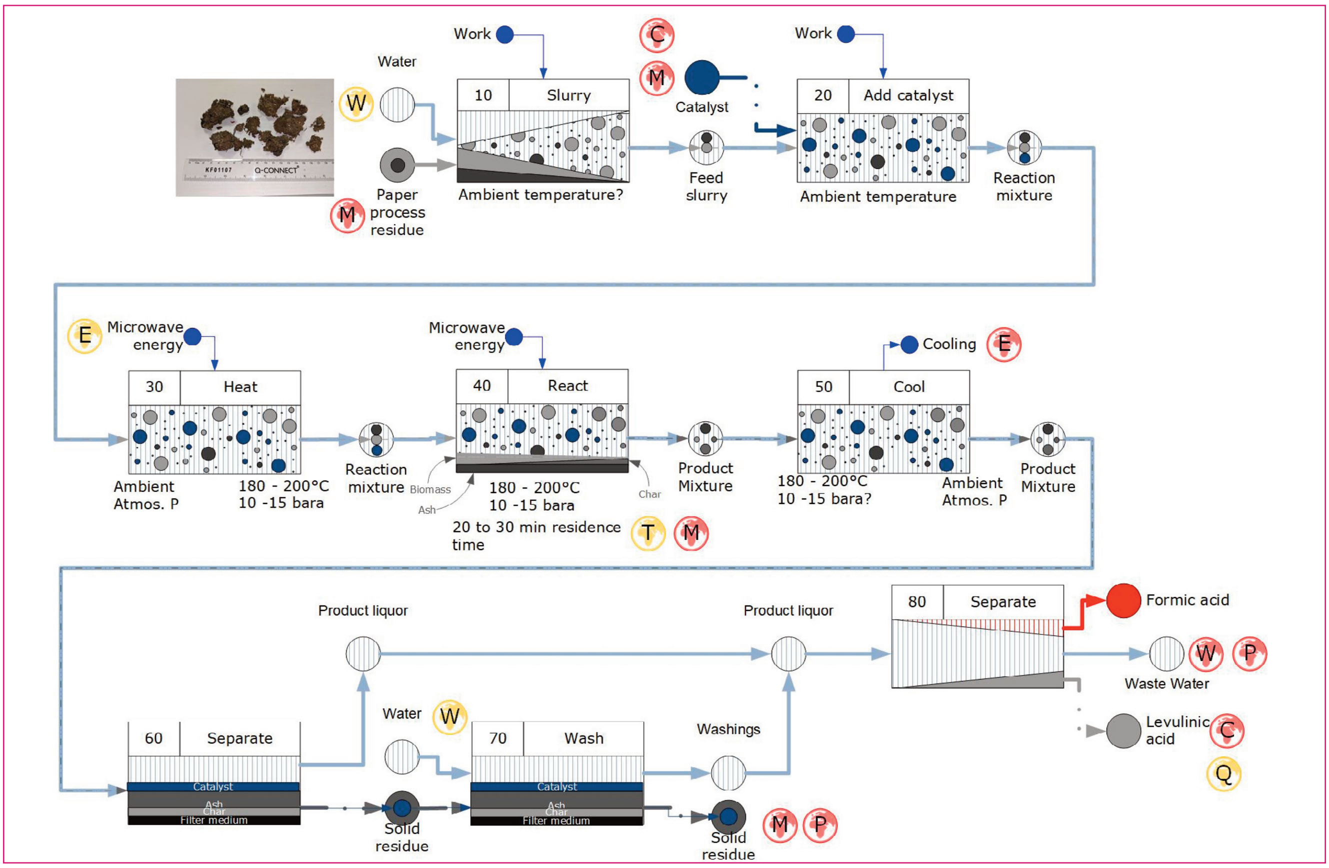

Figure 5: LevWave PDD after applying sustainability prompts.

Sustainability Assessment

So-called "sustainability prompts" may be used to highlight potential process re-design for sustainability opportunities simply and visually. This adapts and extends a similar approach that was developed in-house with one of Britest's members in 2013. This in turn was inspired by the Fundación Entorno's Eco-Compass tool[7] and Arup's Drivers of Change cards[8]. The original Eco-Compass defined six key sustainability indicators. The current sustainability prompts (*Table 1*) are a modified set based originally on an internal case study developed with Britest members.[9] Colour coding has been added signifying the sustainability impact of each prompt: tolerable (green), review/check (amber), and significant (red). This was found helpful in enabling a rapid assessment and comparison of different process sustainability improvement options. The list only covers the two: environmental and economic 'Pillars of Sustainability'. The social pillar is not included because it is more relevant at the whole enterprise level rather than when considering tasks in

Initial Design		After Re-Design	
Task 10: Slurry			
W	Water is introduced into the process to slurry the paper sludge. In the original lab work for LevWave the slurry concentration is quite dilute at 2.5 % w/w. Can the process be operated at a high slurry concentration to reduce the water demand?	W	The entire water demand for slurrying the paper sludge can be met by recycling the water separated in task 80. High purity water is not required here so no additional treatment before re-use should be necessary. After making this change the impact can be considered as tolerable.
M	The paper sludge has a low cellulose content. A large proportion of the feed material will pass through the process unchanged and well need a suitable disposal route once recovered at the end of the process. Could other feedstock give better mass intensity?	M	Improving the yield at reaction mitigates the mass intensity with respect to the paper sludge. However, there will still be a large proportion of the feed material which will pass through the process unchanged and well need a suitable disposal route once recovered at the end of the process. Could other feedstocks give better mass intensity?
Task 20: Add catalyst			
C M	Both tags reflect different aspects of a single factor. In the lab work the catalyst loading is very high with more catalyst (w/w basis) used than paper sludge. This is a concern in terms of mass intensity and even more so in terms of cost of goods since in the lab trials to date, there has been no re-use of catalyst (single use).	C M	Recovery and recycling of the catalyst and substantial reduction in catalyst loading give significant mitigation to both these factors and justify reduction to a tolerable impact.
Task 30: Heat			
E	There is substantial temperature change from ambient to 180°C at this point. The energy demand for this change is fixed thermodynamically but there is scope to consider where the energy required comes from.	E	Most of the heating required can be achieved by cross-exchange with the slurry exiting the reactor. This reduces the impact to tolerable.

Table 2: Sustainability prompts for LevWave (portion).

a manufacturing process.[10]

To use the prompts the PDD in *Figure 1* was simply marked up with appropriately coloured symbols to obtain *Figure 5*. Accompanying notes to record the thinking underlying the placement of each prompt are valuable (*Table 2*).

The sustainability assessment can then be responded to with suitable changes to the process. Assuming the development objectives identified from the TEA can be successfully applied, the PDD can be revised and the sustainability prompts re-applied to assess the improvement achieved. In the case of LevWave all significant impacts were reduced to either tolerable or review/check level and some of the review/check impacts were reduced to tolerable. An Eco-Compass approach (*Figure 6*) may be used to indicate the progress made towards process sustainability through the improvements identified using a semi-quantitative metric for scaling scores derived from the changes reported in the Key Process Indicators (KPIs) in the TEA.

Scale-Up Risk Assessment

A 'short-cut' technological risk assessment can be quickly and easily carried out (in a procedurally similar fashion to the sustainability prompts above) by tagging the tasks in the whole process PDD with scale-up risk factors and impacts taken from *Table 3*. At an early stage of process development, firm decisions on equipment selection will not have been made (and the PDD itself is focused on the experience of the process materials, independent of scale and equipment). The risk factors are experientially derived but have proven to be robust over a range of different and unrelated processes, including the LevWave case study. The current operating scale for LevWave has been limited to ≈10g scale as small batches at MMU and as slightly larger batches at DRS. Scale-up risks have been assessed for continuous processing of 30Ktpa of paper sludge. The scale up concept considered was a microwave-heated, tubular plug flow reactor processing the paper sludge slurry/catalyst mixture.

Scale-Up Risk Register

From the annotations tagged on the PDD in *Table 4*: Risk register for LevWave process scale-up (portion), a scale-up risk register can be compiled (see *Table 4*). Each identified risk is given

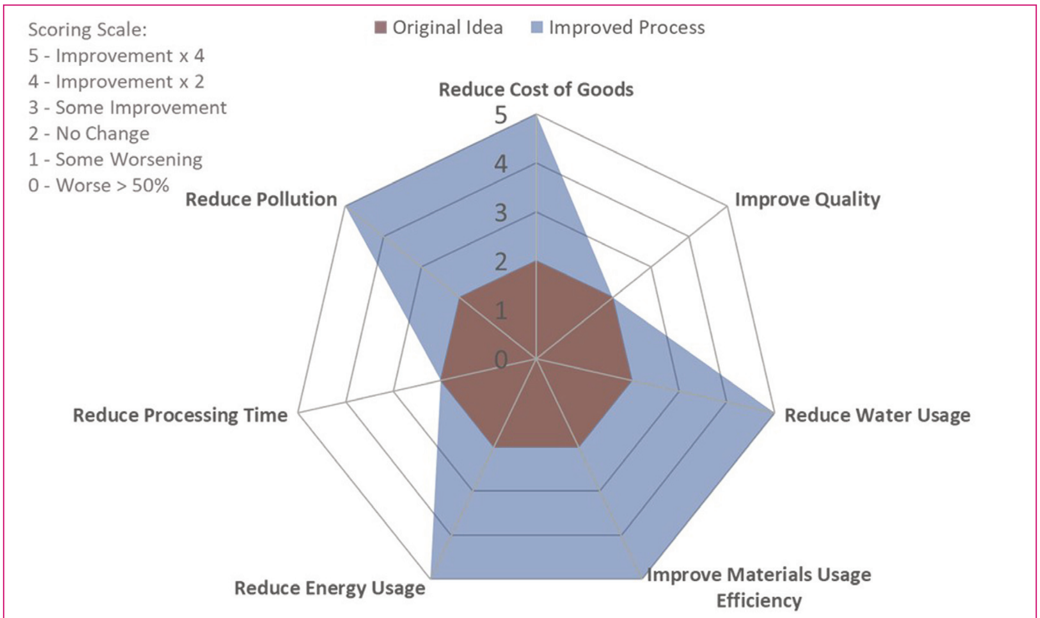

Figure 6: Eco-compass before and after responding to sustainability prompts.

a unique number. The risk is described, and the impact or impacts noted. Each described risk is then scored to assign a priority or importance to mitigating the risk for scale-up to proceed successfully. As a project progresses, the risk register can be used to record the decreasing risk as understanding increases and mitigation strategies are implemented. Again, plotting the evolving priority scores in a compass plot can provide a valuable and highly visual reporting tool.

Conclusion

A methodology has been proposed and exemplified using an ISCF fast start project, LevWave, as a case study. Whilst the methodology has been demonstrated in the context of an innovation project, similar concerns face any NPI project team, and there is every reason to believe the approach will have equivalent value in other situations.

The methodology provides project managers with a systematic tool for planning development objectives in the ongoing programme, together with support for decision making at stage gates based on the state of the project team's process understanding at the time of assessment. It guides objective setting to address three key aspects of commercial viability: techno-economics, sustainability, and scale-up risk.

Based around the Britest process understand tools, the methodology is designed to be simple, easily communicated and as non-onerous in time and effort as possible. The last point is particularly important for an early-stage project, perhaps with as-yet undemonstrated business value. Other techniques for capturing process understanding can be substituted but compromise in terms of simplicity and efficiency may result.

Tag	Risk factor	Rationale
1	Moving solids	Solids handling can always cause challenges when changing scale
2	Multiple interacting phases	Rule of thumb: complexity increases in proportion to [no. interacting phases]^1.6 (J. Atherton)Some physical forms are particularly challenging, especially solids (*e.g.* sticky or large particles)
3	Human engagement	Any task requiring operator or other human engagement could introduce variability
4	Heat/energy introduction/ removal/change	Potential challenges will depend on the magnitude and rate required
5	Sensitive chemistry	Are any transformations operating in a tight window with respect to driving forces?
6	Chemical incompatibility/ unintended reactions	Impact of unintended chemistry can increase on scale-up
7	Extremes of specification	Is the process sensitive to the grade/source of materials used?
Tag	**Type**	**Impact Prompt**
Q	Product	Quality
Y		Yield
O	Process	Operability
S		Safety & Environment

Table 3: Risk factor and Impact prompts for scale-up risk assessment.

Risk Number	Task Number	Task Description	Risk / opportunity and triggers	Impact	Priority / Risk	Reasoning
4	20 - 40	Slurry transport	Slurry pumping risk of blockages	O	2	Fibrous slurry handling
5	20	Catalyst addition	Feeding/Dosing requirement for catalyst	O	2	Little information on physical form and properties, handled wet.
6	20	Catalyst addition	Mixing & dispersal requirement for catalyst	O, Q, Y	2	Gaps in data on requirements
7	20	Catalyst addition	Variation in catalyst quality if recycled	Q, Y	4	Re-use of catalyst has not been attempted in work to date.
8	20	Catalyst addition	Additional process complexity if additional catalysis proves to be required	O, Q, Y	2	Lab work suggests possible advantages from a two-component catalyst system
9	30	Heating	Excessive power demand for microwave technology	O	3	Heat recovery from Cooling step is a valid mitigation
10	30	Reaction	Complex, multi-phase, multi-step reaction	Y, Q	4	Significant gaps in knowledge, especially around physical chemistry

Table 4: Risk register for LevWave process scale-up (portion).

Acknowledgements

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References

1. https://www.britest.co.uk/about_us/history/ Checked July 2022
2. G. Hurst, I. Brangeli, M. Peeters, S. Tedesco,
Solid residue and by-product yields from acid-catalysed conversion of poplar wood to levulinic acid, Chemical Papers, (2019) https://doi.org/10.1007/s11696-019-01013-3
3. Confederation of Paper Industries, Process Wastes from the Paper Industry, March 2021
4. Wall K., Sharratt P.N., Sadr-Kazemi, N, Borland J.N., *Org Proc Res & Dev*, **5(4)** (2001).
5. Ibid.
6. McLachlan et al., J Bioprocess Biotech **7(2)**, (2017).
7. Links to websites for Eco-Compass tool and Fundación Entorno seem to be no longer active. Eco-Compass is not the same as the Horizon 2020 project of the same name. A description can be found in Tambovceva, Assessment model of environmental management: a case study of construction enterprises in Latvia, *Economics and Management*: 2010. 15 pp.799-806.
8. https://www.arup.com/perspectives/publications/research/section/ drivers-of-change
9. Internal Britest document: Driver Prompts Methodology — Case Study, 2015
10. https://www.investopedia.com/articles/investing/100515/three-pillars-corporate-sustainability.asp Checked July 2022