Identification of Hazards and Generating Inherently Safer Process Options

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In the drive for better chemical processes, there is a need for close collaboration between engineers and chemists. This type of close, multidisciplinary collaboration improves the overall process development effort by identifying potential downstream issues early. However, safety assessments are usually conducted later in the process development stage once the chemistry and basic process steps have been firmed up, for example, in Hazop 1 studies. The benefits of early stage safety intervention are widely acknowledged since the degrees of freedom available to change things is significantly greater during the early stages of process development. This is especially so if we aim to incorporate inherently safer options in the design.

Tools such as reaction maps, driving force tables and process definition diagrams provide an information-rich, common platform for a multidisciplinary team to communicate with each other. These tools are part of the core BRITEST suite of tools and methodologies and they work by building-up process information and understanding in stages. The different tools address different levels of a system under study. For example, reaction or transformation maps (TM) and driving force analysis (DFA) provide an analysis of the chemistry whilst the Process Definition Diagrams (PDD) offers a view at the process level. This intimate structure of the BRITEST tools provides opportunities to study safety issues at various levels and introduce inherently safer design options during process development.

Using these process understanding tools as a basis for safety assessment help to anchor the identification of hazards and their possible solutions within a scientific framework that takes into consideration factors such as chemical and physical transformations, material properties, physics and manipulated parameters. This is achieved using a newly developed tool called the source-pathway-receptor (SPaR) tool. This tool was designed to accept the inputs (chemistry and process information) from the core process understanding tools (i.e. TM, DFA, PDD) and organise them within the source-pathway-receptor model. This model has been commonly used to demonstrate the link between a hazard source and the ultimate impact on receptor(s).

Keywords: inherent safety, Hazop, continuous Grignard process, process understanding

Introduction

Although inherent safety concepts have been in existence for more than 30 years (Kletz 1978; Kletz 1985; Sanders 2003; Hansson 2010) and have been elucidated (Hendershot 1997; Lutz 1997; Kletz 2003; Moore et al. 2008) by a number of researchers, its adoption has been relatively slower when compared with other techniques such as Hazop and quantitative risk assessment (Kletz 1991; Kletz 1996; Kletz 1999).

In practice, inherent safety is often regarded as common sense and its introduction into a process is the result of a well educated, experienced engineer implementing his knowledge of inherent safety concepts into his design. Alternatively, the more systematic method of incorporating inherent safety is via the quantitative, index based approach to evaluating inherently safer process options (Gentile et al. 2003; Gupta et al. 2003; Khan et al. 2005; Rahman et al. 2005; Tugnoli et al. 2007; Leong et al. 2008). Here, various proposed process options are assessed against a metric that provides an inherent safety score for the design. The process option with the best inherent safety scores is then deemed to be the inherently safer option.

However, these approaches do not address the more important issue of systematically analysing and generating inherently safer process options. We are still limited by the quality of the options that we are able to generate. Apart from obvious solutions such as reducing storage or process inventories, the average engineer might not readily generate less obvious process options. Therefore, in order to make inherent safety a more widely accessible and implementable concept in process development, there needs to be tools and methods that encourage engineers to identify opportunities for implementing inherently safer options rather than fitting in add-on control measures.

With this challenge in mind, we have set out to develop a methodology that enables the assessment of safety vis-à-vis process requirements in an integrated, holistic approach. In doing so, we should gain better process understanding and be able to evaluate the trade-offs (if any) when optimising process requirements. This enables us to establish our safety envelopes early in the process development life-cycle. Ultimately, this will lead to embedding inherent safety principles early, during the process development stage, where the flexibility to make significant changes is highest.

Process Understanding Tools

The U.K.-based, not-for-profit consortium BRITEST has, over the past 15 years, jointly developed with its partners a suite of tools and methodologies that takes a conceptual approach toward process innovation. It does this by “developing analytical and procedural guidelines for process development intended to improve coordination among discovery chemistry, process development, and engineering” (Mullin 2007). The BRITEST suite of tools and methodology is geared towards gaining better process understanding. In some situations, the use of BRITEST tools have been able to highlight potential safety related issues. In a case study involving the production of phenol via a diazonium intermediate (Sharratt et al. 2003), an analysis of the reaction scheme, in conjunction with driving force analysis (DFA), was able to suggest opportunities for
Process intensification whilst highlighting the potential safety benefits and reaction hazards. However, in general, the BRITEST tools in their original forms are not able to directly address safety concerns.

Safety assessments are conducted to evaluate process hazards, plant risks and hazards during plant operations. Process understanding tools are commonly used during process development and troubleshooting. It is therefore logical that during these types of process analysis, safety considerations should also be taken into account. An integrated approach where both process and safety factors are discussed using common tools would therefore greatly benefit process design.

Process understanding tools (e.g. BRITEST tools) would be useful for the early identification of potential safety issues during process development and process plant design. They could also provide valuable inputs to support subsequent formal safety assessments (e.g. Hazop). This gives us the opportunity to establish our safety envelopes early in the process development life-cycle. Ultimately, the added process safety understanding could assist us in implementing inherent safety principles at an early stage.

Although tools like the transformation map (TM), Driving Force Analysis (DFA) and Process Definition Diagram (PDD) are useful in gaining an overview of the process, the lack of important aspects related to safety such as hazard, risk, mass and energy flow/balance, inventory (including pre- and post- processing), material properties and material of construction are impediments towards the expanded use of the BRITEST tools in safety. In spite of their limitations, the current suite of BRITEST tools and methodologies still offers possibilities to be used in safety assessments.

**Transformation Map (TM)**

Chemical reactions are the core of chemical processes. Therefore, a thorough understanding of all the chemical and physical transformations in a process is a critical first step in developing processes or troubleshooting. Chemical reactions seldom occur in isolation and processes can involve sequential reactions, side reactions, phase changes and transport phenomena. A reaction or Transformation Map (TM) is therefore used to present the network of physical and chemical transformations graphically (Obenndip et al. 2006). This graphical representation of the physical and chemical transformations provides a multidisciplinary team with a common platform to capture their combined knowledge and then collectively probe for gaps in their process understanding.

**Driving Force Analysis (DFA)**

If TMs are qualitative descriptions of the physical and chemical transformations in a process, the DFAs can be said to represent the mathematics behind the transformation. DFAs are a means of describing the chemical and physical rate processes (i.e. differential equations) in a simple, visual representation that is easily understood by both engineers and non-engineers alike (Sharratt et al. 2003; Obenndip et al. 2005; Obenndip et al. 2006). In doing so, a DFA enables a thorough examination of systems with competing rate processes thus systematically identifying important process parameters and knowledge gaps.

**Process Definition Diagram (PDD)**

The PDD is a graphical, block diagram type, representation of the processing steps involved in converting raw materials into the final product (Wall et al. 2001). It also presents information relating to the material flows into and out of each processing step, waste generated, heat flows and phase changes. It describes a process independently of scale and equipment thus making it eminently useful in early stage process development or in simplifying a complex process during troubleshooting. Its simple representation coupled with rich information content makes it a good platform for multidisciplinary teams to investigate process related issues.

**Methodology Development**

In setting out to develop this new methodology, we have put in place a few design considerations to guide us in the overall development process. Firstly, the new tool and methodology should integrate with the current BRITEST tools and be compatible with current safety assessments used in industry. Secondly, the tool and methodology developed should be easy to use. This means that the average chemist, chemical engineer and safety specialist involved in process development or design could intuitively use the tool and methodology with minimal training. This can be achieved by basing the new methodology on familiar safety concepts (e.g. source-pathway-receptor) and assessment tools (e.g. Hazop). Finally, this methodology should offer a communication platform that is easily accessible across people from the different disciplines associated with process development or design.

The work involved in tool and method development will be described in this section whilst the second major component namely tool testing will be described in the next section on Case Study. The method development work itself consisted of three main parts: (i) Tool screening, (ii) Information mapping, (iii) Methodology and tool design.

**Tool screening**

The development process began with an evaluation of the 14 common process development tools available in BRITEST. The tools were analysed against factors that are important in safety assessments i.e.:


Each of these factors were subsequently evaluated by considering whether the listed factors were:
(A) Currently used by the tool, (B) Currently generated by the tool, (C) Can be modified to incorporate factor(s) and (D) Not suitable.

Based on this analysis, promising candidates were shortlisted and prioritised. The shortlisted tools could then be prioritised and subsequently worked on to enable safety specific assessment and decision making along the lines of hazard identification, risk evaluation and risk elimination/minimisation/control.

This screening exercise showed that three tools namely TM, DFA and PDD have the potential to be used in safety assessments. At the same time, this analysis also showed that by themselves, the three tools are not able to identify hazards and evaluate risk. This observation pointed out the need for modification of these tools and the development of a new tool to enable safety assessments.

Information mapping

After the screening exercise, we shortlisted a few tools for further development. To find out the type of information associated with the tools and how they can support a safety assessment, an information mapping of the shortlisted tools was conducted. The results of this information mapping is shown in Figure 1.

Although the tools, by themselves, cannot be directly used for safety assessments, the information mapping showed that the tools were able to generate important information that were relevant for safety. If this information can be harvested, they would be able to generate safety impacts and scenarios that could then be further assessed.

BRITEST tools basically work by building-up process information and understanding in stages. The different tools address different levels of a system under study. For example, Transformation Maps (TM) and Driving Force Analysis (DFA) provide an analysis of the chemistry and physical transformations in a process. These two tools in their current state are able to provide information relating to possible runaway reactions, side products and waste stream composition. Transformation maps used with DFA to study potentially hazardous undesired reactions would be able to give greater insights into their triggers. If this type of information can be extracted early during the process development stage, they could assist in eliminating or minimising the identified hazards.

The Process Information Summary Map (PriSM) and the Process Definition Diagrams (PDD) offer an overview of the process. In addition to this, the PDD also offers very important information in terms of the number and types of phases present in the process. Together with the mass and energy balances, they can indicate whether mixing and/or accumulation of mass/energy might pose significant safety problems. This innate structure of the tools allows us the opportunity to study safety issues at various levels during different stages of process development.

Methodology and tool design

As seen in the information mapping, we needed a new tool to accept/organise the inputs (safety/hazard information) from the BRITEST tools and to subsequently generate and analyse the corresponding safety impacts. We decided to rely on the commonly used source-pathway-receiver model to organise these inputs. This model demonstrates the link between hazard source and the ultimate impact on receptor(s). Receptors can be in the form of people, physical structures (including equipment) and the environment. The pathway as described by this model indicates how a hazard is transmitted from a source to a receiver.
Conceptually, the information from the modified BRITEST tools will be used to populate the potential sources and pathways. The assessment team will then use this information to ascertain the likely receptors and impact levels. Subsequently, it is envisaged that inherent safety strategies could be infused into generating options for improvements. This should allow us to systematically consider and implement inherent safety strategies at different stages of process development.

In the conceptual framework illustrated in Figure 2, we note that one of the key features is the use of the core BRITEST tools to feed safety related information into the SPaR (source-pathway-receptor) tool. The safety related information extracted from the process and chemistry-level tools can be collated to give an overview of the hazards present in each processing step. This new SPaR tool is based on a modified TE3PO (Transformation, Entities, Properties, Physics, Parameters, Order of Magnitude) table. The TE3PO table is a BRITEST tool that has been designed to collate and analyse transformations related process knowledge. The table is structured to identify the key science/physics associated with the various transformations (e.g. heat transfer), the variables that control the physics (e.g. temperature), and key material properties (e.g. heat capacity). This is an important feature that we want to retain in the new SPaR tool as it will enable us to link potential safety impacts to specific transformations and their associated science.

The PDD is used to conduct a Hazop-like study of possible deviations called a SPaRing session. The new SPaR tool is meant to be used directly with the PDD and it functions firstly as a record of relevant deviations of more/less (+/-) and their consequences that are generated by guidewords (e.g. quantity, temperature, pressure, mixing, holdup) as shown in Figure 3.

Once a significant consequence has been identified using the guidewords, the transformations (physical or chemical) that affects or are affected by the deviations are then linked with the associated properties, physics and parameters. The deviations, entities, transformations, properties, physics and parameters together describe the SOURCE (i.e. hazard) as shown in Figure 4. This helps in relating the consequences to the science that in turn allows us to identify preventive and control measures.
Once the SOURCE has been described, the PATHWAY can then be determined. The PATHWAY is described by the likely routes (e.g. explosion, venting) for the hazard to reach a RECEPTOR. The RECEPTOR of the hazard can be described in terms of the affected people, equipment, buildings, and flora/fauna. Under RECEPTOR, the Order of Magnitude of harm/damage has been included to give a sense of proportion for each of the identified hazards. This also allows for subsequent ranking/prioritisation of ACTIONS if required.

As seen in Figure 4, the final column describes the recommended ACTIONS that can be taken to prevent, control or mitigate against the various hazards. It is here that the facilitator of this assessment when coming-up with the recommended actions can introduce inherent safety principles (i.e. substitution, minimisation, modification and simplification). This would aid the design team to incorporate inherent safety features during the process development stage. Furthermore, the exercise of linking safety impacts to the relevant pieces of science during the SPaRing session should be useful in proposing inherently safer process options as opposed to implementing add-on control measures that are prevalent in Hazops.

**Case Study**

Following the development of the SPaR tool and methodology, we proceeded to test the tools on three processes (Grignard, Sonogashira and Molybdenum chemistries). The tool was used under “live” conditions in order to test their utility under real group dynamics. In addition, several industry members from BRITEST who were assisting in the project also tested the tool within their own companies. In the tests conducted on the tool, we evaluated whether the methodology and tool were:

- a. Easy to use;
- b. Generate reasonable safety outputs relating to hazard identification, evaluation and control;
- c. Communicable to various users in an organisation

One of the processes that the SPaR tool was tested on was the development of a continuous Grignard process. The Grignard chemistry is an important tool in synthetic, organic chemistry for the formation of carbon-carbon bonds. This organometallic chemical reaction involves the formation of Grignard reagents (i.e. alkyl, vinyl, or aryl-magnesium halides) that are subsequently added to a carbonyl group in an aldehyde or ketone. Other similar carbon-carbon bond forming reactions involve lithium (organolithium) and zinc (organozinc). This continuous Grignard process development is similar to the work done previously by Loh et al. (Loh et al. 2012) who worked on a continuous Reformatsky (organozinc) process. The reaction chemistry of this Grignard process is presented in Figure 5 in the form of a Transformation Map.
As in most process development exercises involving BRITEST tools, the TM is converted into a tabular form to carry out a DFA. As shown in previous work (Sharratt et al. 2003; Obenndip et al. 2005; Obenndip et al. 2006), DFAs have proven to be useful in studying the factors that drive unwanted side reactions. Similarly, in this case, one of the key observations was that excess amounts of Grignard reagents relative to pinacol borane tends to drive the formation of an unwanted by product as shown in Figure 5 (S4 MT4). Identification of unwanted side products is very important in safety assessments as it can lead to severe consequences especially if the side products are unstable (e.g. low temperature decomposition) and/or toxic. Furthermore, if such side reactions are highly exothermic, they could trigger runaway reactions.

In addition to working out the TM and DFA, a PDD was also drawn up based on a proposed process flow as shown in Figure 6.

The PDD can be considered as an enhanced block flow diagram of the continuous Grignard process. In addition to the typical information relating to process flow streams, PDDs also captures and presents information relating to phases and heat transfer. As shown in Figure 6, PDDs can therefore be the basis of an intuitive Hazop-like methodology to interrogate a process for the purposes of an early stage safety assessment.

With the PDD of the continuous Grignard process as the basis, the new SPaR tool can now be used together with the process understanding gained from the TM and DFA to conduct a safety assessment we call a SPaRing session. We begin the assessment by choosing an operation block in the PDD. In the illustration given in Figure 7, it focuses on the magnesium activation step with magnesium chosen as the entity (species) studied.
The first stage of the SPaRing session shown in Figure 7 involves the determination of the possible sources of hazards (SOURCE) and is described step-by-step as follows:

a. **Deviation** - As in a Hazop, we introduce a deviation in the process using guidewords and in this case the deviation being considered is a reduction in magnesium quantity (-quantity).

b. **Consequences** - Based on the deviation introduced, the assessment group brainstormed possible consequences that could arise from a reduction in magnesium within the process. One of the consequences identified was the accumulation of unreacted diisobutylaluminium hydride (DIBALH) that would be carried over to the subsequent processing stages. The carryover of DIBALH could in turn lead to an unwanted reaction with water in the quench.

c. **Transformations** – The three transformations of concern associated with the DIBALH carryover were identified as (i) potential unknown reaction between DIBALH and pinacol borane, (ii) reaction of DIBALH with water to release flammable gases (butane, hydrogen) and (iii) precipitation of aluminium salts.

d. **Properties/Physics/Parameters** – For each transformation of concern, the assessment group looked at the associated science in the order given in the table. For example in Figure 7, the blockage concern arising from the precipitation of aluminium salts primarily depends on the solubility of the salts themselves. The table then guides users to identify the physics (or chemistry) associated with the precipitation and in this case, it is a mass transfer related phenomena. The assessment next identifies the parameters that affect precipitation such as temperature, concentration and pH. These identified parameters are very important later in the SPaRing session as it provides users with the options to prevent or control the potential blockage due to aluminium salt precipitation.
The second stage of the SpaRing session shown in Figure 8 involves the determination of the possible PATHWAYS and RECEPTOR and is described as follows:

a. **Pathway** – Each transformation of concern identified will need a pathway to a receptor before it can be ultimately considered as a hazard. If a pathway is not found, the risk associated with the hazard source can be considered to be low. This will help in prioritising the amount of effort and resources used to tackle the hazard source. However, if a plausible pathway can be devised to link the hazard source to receptors then the corresponding risk level will be higher.

b. **Receptor** – Although this tool and methodology can be used very early in the design cycle when scale and process details are not yet available, the receptor column acts to highlight the potential damage or harm that could be caused by a process deviation. In doing so, it helps the design team to assess the impact and magnitude of the hazards posed by the process. Also, by attempting to identify potential receptor(s) and magnitude of impacts, the assessment team will be able to communicate to other members involved in design, the severity of safety impacts. This can then help in prioritising and securing sufficient resources to tackle the identified hazard.

![Diagram of Brainstorming for possible preventive actions guided by inherent safety principles](image)

The third stage of the SpaRing session as shown in Figure 9 covers the preventive and corrective actions (ACTION). The earlier stages that generate hazard related process understanding based on the source-pathway-receptor model ultimately feeds into this final stage where the assessment team makes use of the understanding gained in the two earlier stages to develop solutions. The general principles of inherent safety (substitute, minimise, modify and simplify) are used as prompts to develop solutions by combining suitable prompts with the transformation-linked parameters that have been identified previously. For example, in Figure 9, we can see that by matching the overall process flowrate to the solubility/pH of aluminium salts in the aqueous quench solution, we can prevent the precipitation of salts. This is different from an add-on control approach where perhaps a sensor that triggers an alarm when precipitation is detected might be installed along with a control loop that will increase the quench solution flowrate to dissolve the excess aluminium salt. It is clear that the inherently safer option generated by the SpaR tool is superior to the add-on control measure. Similarly, the other inherent safety prompts of substitute and minimise were able to generate reasonable process options to tackle the other safety issue relating to the release of flammable butane and hydrogen gasses.

### Results and Discussion

The tool and methodology described in the case study is the result of a few rounds of testing. The main purpose of this work is to closely integrate process development/design activities with safety assessments so that process changes could be made early with the greatest degree of freedom available. To ensure a tightly knitted integration with the usual work involved in process development/design, we based this project on state-of-the-art process understanding tools and methodologies from BRITEST. The analysis of the available BRITEST tools indicated that the tools were not in themselves directly capable of carrying out safety assessments. However, the screening and information mapping exercise showed us that the information generated by some of the BRITEST tools would be very useful in safety assessments and if coupled with a customised tool, a safety assessment methodology that incorporates inherent safety principles could be realised.

In view of the realities surrounding safety assessments and process development work, we decided that factors such as ease of use, the ability to generate reasonable safety outputs and simplicity of results communication would be important. Based on the tests that we carried out, the opinion of testers of the tool and methodology are summarised in the Table 1.
Table 1: Summary of feedback obtained from trial users of SPaR

<table>
<thead>
<tr>
<th>Ease of use</th>
<th>Generation of safety outputs</th>
</tr>
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<tbody>
<tr>
<td>Generation of deviations and consequences were easy and intuitive using the</td>
<td>The ability to identify consequences further down the process (domino effect) was very</td>
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<tr>
<td>SPaR tool</td>
<td>helpful as this is not usually done in early stage process development/design</td>
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<tr>
<td>People familiar with BRITEST tools were able to run a SPaRing session with</td>
<td>The use of TM and DFA together with SPaR assisted in systematically identifying potentially</td>
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<tr>
<td>a minimal amount of training</td>
<td>hazardous side reactions that could then be followed up with the necessary adiabatic</td>
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<tr>
<td>PDDs were able to present sufficient details for assessment</td>
<td>calorimetric tests.</td>
</tr>
<tr>
<td>Guidance provided by the use of properties, physics and parameters helps</td>
<td>The identification and linking with transformation, properties, physics and parameters was</td>
</tr>
<tr>
<td>to focus discussions and identify variables that were not immediately</td>
<td>rather difficult and not trivial.</td>
</tr>
<tr>
<td>obvious</td>
<td>The assessment team needed to be multidisciplinary with the members possessing significant</td>
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<tr>
<td></td>
<td>expertise in the various disciplines</td>
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<tr>
<td></td>
<td>Careful generation of relevant parameters linked with transformations of concern helps in</td>
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<td></td>
<td>developing good inherently safer process options</td>
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<tr>
<td></td>
<td>The identification of pathways and receptors helps in prioritising safety impacts based on</td>
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<td></td>
<td>relative risk</td>
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<tr>
<td></td>
<td>The ability to link inherent safety prompts with parameters made it easier to generate</td>
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<tr>
<td></td>
<td>inherently safer design options systematically</td>
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<tr>
<td></td>
<td>The use of TM, DFA, PDD and SPaR allows the presentation of very complex information and</td>
</tr>
<tr>
<td></td>
<td>ideas graphically and systematically thereby aiding communication with other development</td>
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<td></td>
<td>team members or other external parties</td>
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</table>

Overall, the feedback on the use of the SPaR tool was positive. The main drawbacks highlighted by users centre around the need to have the correct expertise within the assessment team. However, this is true for all safety assessments (e.g. Hazop) with the quality of output being highly dependent on the level of experience and expertise of the participants. Another issue highlighted by test users indicated that the entire assessment (TM, DFA, PDD and SPaR) takes a considerable amount of time and effort to complete. However, if we take the view that the use of the other tools (i.e. TM, DFA and PDD) also contributes to other aspects of process development (e.g. process chemistry, process engineering) apart from safety, the effort invested in the entire process would be worthwhile.

Conclusion

The SPaR assessments enable the identification and evaluation of safety impacts during the early stages of process development. State-of-the-art qualitative analysis tools (TM, DFA and PDD) from BRITEST provide the core information (chemistry and process) used in the safety assessment.

With the use of the source-pathway-receptor model as the framework for the safety assessment, we were able to apply a Hazop-like methodology to generate process relevant deviations and interrogate them for possible safety related consequences. Similarly, by identifying possible pathways that link the consequences to receptors, SPaR users were able to identify impacts and qualitatively evaluate relative risk levels.

The guidance provided by the SPaR tool also enabled the assessment to uncover the scientific-based factors that governs and/or affects the consequences and transformations of concern. In particular, this explicit link between science (properties/physics/parameters) and safety impacts provides us with a better understanding of the hazards and the means to control them. This qualitative analysis of the science involved provides a sound basis for brainstorming suitable inherently safer design options to address the various safety related consequences generated from the SPaRing session.

This key relationship observed between the identified parameters (manipulatable process variables) and inherent safety prompts has indicated a potentially new method to systematically generate inherently safer process options. The qualitative approach shown in this study can be the basis of future work that looks into linking typical process parameters with the various inherent safety principles. This could in turn lead to both qualitative and quantitative modelling (e.g. digraphs) of safety impacts that are modified using the inherent safety prompts.

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