A METHODOLOGICAL APPROACH TO PROCESS INTENSIFICATION

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Process Intensification (PI) is a design philosophy where process plant is designed to match the fundamental requirements of the chemical process and meet business needs. The benefits of applying PI include smaller, inherently safe plant; reduced energy requirement; improved product quality; lower capital cost. This paper describes a methodology that assesses the feasibility for applying PI to a chemical process. Application of the methodology is demonstrated on the design of a continuous, intensified reactor to replace a semi-batch stirred tank reactor. The resulting conceptual PI plant has an inventory three orders of magnitude smaller, eliminates runaway potential, and provides significant economic benefits.

Keywords: Process intensification, static mixer, compact heat exchanger, continuous process

INTRODUCTION

It is likely that the chemical plant of the future will be far smaller than that of today (1). This can be achieved only by a step change in the plant technology used, rather than incremental improvements of existing plant items. The philosophy of size reduction has been in existence for several years under the name of Process Intensification (PI). Smaller equipment can result in reduced capital cost and reduced operating costs, whilst giving improved product quality. Just as important, according to Kletz (2), is that smaller often means inherently safer. Despite these benefits, uptake of PI appears to be low. There are many possible reasons for this. Standard process design and development has stressed the use of batch reactors (3), often with limited available knowledge of reaction kinetics. Lack of awareness of novel technology has to be overcome, from new graduates right through to top level management. Conservatism within the chemical industry may also result in unwillingness to take the risk with novel technology. The challenge of increasing the use of PI lies in promoting a different approach to process development, which should assist in overcoming these barriers.

Current procedures for applying PI technology also need to be considered, as this can tend to be done with an equipment driven approach. Organisations that have developed novel technology will look for applications where a chemical process can be run in their particular equipment. The equipment driven approach can be summed up through the opinion that PI is currently a solution looking for a problem. This situation needs to be reversed so problems look to PI for solutions, known as the process driven approach. Equipment should be chosen to match the process and allow it to run at its optimal rate, resulting in the consideration of a range of intensified equipment where normally only conventional plant would be used. The methodology set out in this paper uses a process driven approach to assess the feasibility for applying PI. It should be stressed that this methodology is not about forcing PI upon situations where it is not really required, but it aims to find the best solution for running a process. Improved understanding of the process as a result of the methodological approach can lead to benefits even if it is shown that full PI is not feasible.

PROCESS INTENSIFICATION (PI)

PI has been categorised as follows by Hannon and King (4):

a) Equipment - reducing the size of a unit operation. Full PI uses novel equipment to reduce size by 2-3 orders of magnitude, improving the process safety. Intensification can also apply to reducing the size of a conventional unit through more efficient operation.
b) Physical - combining two or more operations in one unit. Examples include pumps as mixers and reactive distillation. Compact reactor-heat exchangers, described by Edge *et al* (5), are an example of both equipment and physical intensification.
c) Chemical - improving the reaction scheme. Using different reagents or catalysts can improve yield or speed reactions up. Fast reactions are preferable for PI as they require shorter residence times and lead to smaller equipment.
d) Plant - Size reduction of the entire plant and integration of utilities to save energy and space.

PI can be applied across the whole flowsheet, but for the purpose of this work the focus is on the reactor. Any changes or improvements made here will affect the entire plant. The reacting inventory is often the most dangerous on the plant, as shown by Barton and Nolan (6) in a study of thermal runaway incidents. Reducing this inventory through the use of PI would be a major aspect in improving the safety of the process. Although PI has many benefits, there can be some potential drawbacks, such as lack of flexibility. PI equipment usually has to be tailored to a particular reaction scheme, whereas stirred tanks can run a number of chemical process, increasing plant occupancy and hence perceived value for money. To improve flexibility, a standard framework of feed pipes can be envisaged with interchangeable intensified reactor units to suit different reaction schemes. Not every reaction scheme can be intensified.

The 'S' curve (fig.1) is used to demonstrate how process performance is linked to plant performance. Factors under consideration for plant performance might be the mixing or mass transfer rate or heat transfer capability, while process performance might be yield of desired product, energy efficiency or product quality. Plant performance can be illustrated through the mixing sensitivity of some reaction schemes. For reactions to run at their inherent kinetic rate, the mixing has to be faster than the rate of reaction. If this is not the case, the reaction will be running slower than is theoretically possible, increasing residence time and reducing the process performance as by-products have more opportunity to form. Ideally operation would be close to the top of the S-Curve without moving too far to the right, which would entail excessive costs. PI can be the only means of improving plant performance enough to move up the S-Curve. The interaction between PI and chemistry is also shown. Improving chemistry (for example with a more selective catalyst) can push performance up to a higher S-Curve, but benefits will be lost if operation is lower down this S-Curve.

Removing reactor mechanical limitations to allow reactions to run at their inherent kinetic rate can be achieved by utilising a range of PI technology (7). In-line devices such as static mixers, ejectors (fig.2) and rotor stator mixers have proved to be effective as mixers and reactors, with good plug flow characteristics and mixing intensities up to three orders of magnitude greater than stirred tanks. Exploiting intensified force fields is another approach to intensification. Ramshaw (8) has shown that centrifugal fields can be used for separations, reactions, heat and mass transfer. The centrifugal field within a rotating disk reactor (fig.3) creates thinner, unstable liquid films, improving mass and heat transfer. Ultrasonic, electrostatic and magnetic fields can also be used for process intensification (9).

EXISTING PROCESS DEVELOPMENT AND SAFETY METHODOLOGIES

Hazop (10) is a well established safety methodology which is applied once the plant design is reasonably detailed, giving limited opportunity to intensify or redesign the plant for inherent safety. To gain the maximum benefits, it is necessary to consider safety and PI as early as possible in process development. This requires engineers being involved with development chemists to ensure the right chemical characteristics are being looked for. Several methodologies have been published exploring the inherent safety of a chemical process route (11, 12, 13). These include options to consider novel, intensified technology as a means of achieving inherent safety, though it will be necessary to follow a dedicated PI methodology to determine what this intensified plant might look like.

There can be apparent conflicts between PI and inherent safety methodologies, particularly for fast reactions which are most favourable for reactor PI. Slow reactions are preferred in conventional stirred tanks, particularly for exothermic reactions where the rate of heat generation will be limited. This enables the relatively poor heat removal capability of the stirred tank to cope. Fast, exothermic reactions could be considered as less inherently safe, or even completely undesirable from a conventional plant point of view. Hence, both the chemistry and plant need to be considered together to get a full grasp of inherent safety, as intensified plant can open up new, safe operating windows.

THE PI METHODOLOGY

The methodology sets out structured procedures to follow for considering PI during process development. The overall methodology, known as the framework, consists of a number of protocols detailing the information needed to ensure the potential for PI is fully examined. Figure 4 shows the framework which is formatted to apply to situations where an existing chemical plant is to be replaced or upgraded. Each of the methodological steps is described below.

a) Business Drivers

Determine why it is desirable to change the plant. This step is phrased 'business drivers' as these reasons are normally of an economic nature. Safety, health and environmental concerns are increasingly becoming important factors. Even so, these relate back to business issues as it is preferable to achieve these requirements in the most cost effective manner, or by ensuring costly incidents do not occur. Another major business driver may be to have a higher and more efficient production rate. These drivers are required to set targets for the plant design to meet.

b) Knowledge Elicitation

An understanding of the whole process is required which is gained through the knowledge elicitation stage. The approach is split into separate chemistry and plant audits, though there will be interaction between the two.

The Chemistry Audit examines the whole reaction scheme. The potential to use different solvents, catalysts or operating conditions should be considered. Ideal operating conditions and those conditions that promote byproduct formation should be determined, such as temperature of operation or residence time. Check if the chemical reaction rate is inhibited in any way. Some knowledge of the kinetics and thermodynamics of the reaction is essential.

The Plant Audit examines what the existing plant currently does. The audit should include all physical aspects of the reactor, including mixing and heat transfer capabilities, feed rate and position of feed addition. It is necessary to have a fundamental understanding of the reactor to determine where and how the reaction occurs. If the intention is to run a new chemical reaction scheme in existing equipment, as is the case in many fine and speciality chemicals processes, the equipment should be audited as if it were already running the new process.

c) Examine PI Blockers

Blockers are those properties or conditions of a process which may prevent the application of PI. Many are process blockers to do with the nature of the chemicals themselves, such as the presence of solids. PI equipment often has narrow channels, which large solids would not pass through. Fine solids can be handled. There may be some business blockers which relate to practical problems of running PI plants, such as flexibility or continuous operation versus batch production. Batch production is preferred in some sectors of the chemicals industry, such as pharmaceutical manufacture where there is a requirement for batch identification. Consider whether any identified blockers can be prevented or worked around.

d) Identify Rate Limiting Steps

Rate limiting steps are conditions preventing the overall process running at a faster rate. These may be mechanical limitations such as low heat transfer area, poor mixing or limited supply of feedstock to the reactor from an upstream operation. Chemical rate limiting steps, for example slow kinetics or mass transfer into a solid reactant, may occur. Rate limiting steps and blockers are considered in parallel as there can be common elements, such as slow reactions which are both a PI blocker and a rate limiting step. PI should aim to remove or improve rate limiting steps.

e) Assess PI Viability

The potential for intensifying a process is determined by pulling together the results of the audits, blockers and rate limiting steps into a mid-methodology assessment. This will ensure all the required information has been gathered and properly considered. Even if it is determined that full PI is not possible, it is worth continuing with the methodology as improvements to the conventional plant could be found that partially intensify it.

f) Drivers

Business and process drivers are required to set targets for the plant design to meet. The business drivers identified at the start of the methodology, which are the economic reasons why it is desirable to intensify the process, should be reviewed to keep a clear idea of the overall aims of the project. Process drivers are those characteristics of the chemical reaction scheme that determine the required operating conditions within, and performance of, reactor equipment to allow the process to run at its most efficient rate. A process driver example is the rate of heat release from a reaction determining the heat transfer capability required of the equipment.

g) Initial Concepts

Throughout the methodology, ideas or concepts will occur on how to intensify the process, which will tend to be equipment driven concepts for applying familiar equipment. These ideas should be documented for discussion in the proper manner at the appropriate methodological stage. Accepting an initial concept early on could introduce bias into the rest of the methodology, preventing further, possibly superior, plant concepts being suggested.

h) Generate Design Concepts

A creative problem solving session should be held in which plant concepts are suggested for meeting the process and business drivers. Include the initial concepts in this session. A database of available PI equipment and their capabilities would be useful here so that no possibilities are overlooked, but concepts should not be restricted to plant items already known about. The success of the concepts generation stage depends on thinking laterally to come up with possibly novel solutions to a problem.

i) Select Best Concept

All the concepts suggested must be analysed to study how each of them matches the business and process drivers. There may be factors which limit or rule out the use of a particular piece of equipment, such as it not being available in the required material of construction for corrosion resistance purposes. The best concept must now be chosen. Some economic analysis may be required if there is more than one feasible choice.

j) Laboratory Scale PI Protocols

It will be necessary to prove that the selected concept will work with actual process chemicals. PI laboratory protocols are being designed to demonstrate the performance of continuous, intensified operation without the need for a pilot plant. This will allow the quantification of any potential benefits of intensification, such as improvement in product quality, shorter reactor residence time and lower reacting inventory.

k) Compare With Conventional Plant

List the strong and weak points of the existing and conceptual plant. Showing that the conventional plant is not fully suitable for a process, due to mechanical rate limiting features, could be just as important as showing the benefits achievable by PI when trying to justify its use.

l) Final Choice of Plant

The person or team responsible for making the ultimate choice of plant equipment should have an open mind to the use of PI. This final decision process involves factors currently outside the scope of this methodology, such as the risk of using novel equipment, legislation and lead time to commissioning of plant. A high risk factor and long lead time to commissioning may rule out the use of PI, even if significant financial and operability benefits have been shown to exist.

PI CASE STUDY

The methodological approach will now be illustrated by a feasibility study recently carried out on a fine chemicals nitration process, which generated an intensified plant concept. The process has a multiple sequence of additions of which only the nitration step was initially considered for intensification. It soon became obvious that the whole process could be intensified.

a) Business Drivers

A runaway reaction and explosion occurred in another stirred tank reactor on the production site, emphasising the need for safer equipment. Production needs to be increased. The plant should be relatively cheap to build. Some knowledge of PI does exist within the company and there is a general feeling that continuous, intensified operation is the way forward.

b) Knowledge Elicitation

Chemistry Audit. All of the process steps consist of blending, reaction and heat transfer operations. Reactions in every step are almost instantaneous and some are very exothermic. All reactants are single phase liquid. Solids can exist in the initial stages, though controlling temperature prevents solids formation. The last process stages involve crystallisation, but crystal sizes are small. Byproduct formation for the nitration step at full scale operation (taking 18 hours) is far higher than that in laboratory production tests (taking 4 hours). This shows a PI plant with short residence time could significantly reduce byproduct formation.

Plant Audit. A 13,000 litre glass lined stirred tank (fig.5a) with cooling jacket and coil is currently used. The large reacting inventory is a major safety concern. For the exothermic nitration step, reactant feed is literally dribbled into the reactor over a period of 18 hours to allow the removal of all the heat of reaction. If feed rate was increased for any reason, there is large potential for runaway reaction conditions to occur. Total batch time for all reaction stages is 30 hours. Low heat and mass transport from the reaction zone at the feed pipe exit could promote byproduct formation.

c) PI Blockers

No particular PI blockers exist. Any solid formation can be controlled. Corrosiveness may become an important issue as a glass-lined stirred tank reactor is currently used to resist the operating conditions. Manufacturing intensified equipment in corrosion resistant materials will increase the cost several times over, but the equipment will still be relatively cheap due to its small size.

d) Rate Limiting Steps

As reaction kinetics are fast, the rate limiting steps are all mechanical. Poor mixing in the stirred tank, which restricts heat transport from the reaction zone, then low heat transfer from the vessel combine to cause the very long feed addition and batch time.

e) Assess PI Viability

The process is suitable for PI due to the lack of blockers and fast, single phase liquid reactions.

f) Drivers

Business drivers are improved safety and productivity at low capital cost. Process Drivers are fast kinetics and high heat release, meaning a plant has to deliver intensive mixing and heat transfer.

g) Initial Concepts

Concepts suggested during the project were based upon previous experience, using an equipment driven approach. These included a heat exchanger loop on the existing reactor, which would improve heat removal and reduce batch time, and a compact reactor-heat exchanger.

h) Generate Design Concepts

For the nitration reaction it is desirable to rapidly mix the reactants and then remove the heat as quickly as possible. From these process drivers, a number of concepts were generated in addition to the initial concepts. These possibilities include utilization of existing PI equipment and some more novel solutions involving new arrangements of existing equipment.

i) Select Best Concept

The concept eventually chosen to achieve the drivers is a static mixer followed immediately by a plate and frame heat exchanger (fig.5b). The reaction will take place in the static mixer, with the adiabatic temperature rise limited to an acceptable level by the presence of inert components from upstream stages. Byproducts formation should be significantly reduced due to the short residence time within the reactor. A similar concept is used for the other reaction stages. There are some novel features of the overall plant design that would not have resulted from an equipment driven approach.

j) Laboratory Scale PI Protocols

A requirement before this project can move into the detailed design phase is demonstration of continuous operation as proof of concept. Experimental procedures have been devised to do this.

k) Compare with Conventional Plant

Figure 5 is an approximate scale drawing of the existing reactor and the conceptual intensified nitration reactor, demonstrating the immense size difference. The PI plant will consist of five reactors, but even so, total inventory is three orders of magnitude smaller than the existing stirred tank. Although a full comparison with the conventional plant cannot be completed until the PI protocols are done, a preliminary economic comparison has been made. The product quality achievable, which would be determined by the PI protocols, is important as it could remove the need for a downstream purification stage with all its associated costs. Major points for comparison are:

	Current	PI	Comments
Production	15 tpa	50 tpa	Increased annual sales value of £2 million, based on continuous operation for two weeks per quarter.
Reacting Inventory	13,000 litres	0.2 litres	Full PI plant inventory (including inter-reactor piping) is approximately 15 litres.
Heat transfer	Poor	Good	Current process feed addition is limited by the poor heat transfer. PI reactor runs stoichiometrically.
Operating safety	High runaway potential	Minimal runaway potential	Runaway conditions should not occur in PI reactor, even if cooling fails, as it is designed to operate adiabatically with cooling after each reaction stage.
Capital cost	£100,000 for new reactor	£40,000 for plant	Cost of control system and other associated items will be evaluated in the next design stage.
Plant layout			PI reactors could literally be bolted to a wall and not require building space as the stirred tank does.
Nitration time	18 hours	0.25s for reaction	Total PI nitration time for reacting and cooling is 3 seconds.
Residence time	30 hours batch time	1 minute	Substantially shorter overall residence time limits the opportunity for byproduct formation

Complexity	4 plant elements	11 plant elements	PI plant is more complex with six static mixers and five heat exchangers required to replace the vessel, impeller, cooling coil and cooling jacket.

Other points under consideration include the filtration stage at the end of the process. Currently this is done batchwise. In order to get the maximum benefit out of the PI process, filtration should be continuous. The cost of installing a continuous filtration system will be examined at the next stage of this project. The alternative is using holding tanks to store product until there is enough to operate the batch filtration step. This would still allow the benefits of improved product quality and safer operation of the PI plant to be achieved. Manual intervention and labour required on the PI plant will be greatly reduced compared to the existing plant.

l) Final Choice of Equipment

The company is reviewing market demand for the product and looking into how the plant can be made in such a way that it can be reconfigured for other products, before deciding whether to replace the existing plant or not. PI laboratory protocols will be followed to fully determine the benefits the PI plant would produce before proceeding onto the production of a more detailed design.

CASE STUDY SUMMARY

Application of the PI methodology has been demonstrated on the conceptual design of an intensified plant for a nitration process, with the methodology acting as a checklist to ensure no important aspects were overlooked. The reasons why the existing stirred tank reactor has a long operation time and high byproducts yield have been identified, showing substantial improvements can be made. Following the methodology generated a PI concept with novel aspects that would not have resulted from an equipment driven approach. Comparison and selection procedures have yet to be completed.

CONCLUSIONS

The PI methodology presented in this paper operates as a decision route for assessing the feasibility for intensifying a chemical process. Consequences of applying PI include smaller, inherently safer plant that is cheaper to build and operate. The methodology is tailored for application to existing chemical processes, though it can also be applied to completely new processes. The methodology is not about forcing PI upon situations, but choosing the best possible plant design to achieve the business targets. Ultimately, integration of this PI methodology with inherent safety methodologies has the potential to produce large financial and safety benefits through enabling effective use of PI.

The case study applies the methodology to an existing fine chemicals process, showing there to be substantial benefits achievable through the adoption of PI. The conceptual PI plant has a reacting inventory five orders of magnitude smaller, and total inventory three orders of magnitude smaller than the existing reactor. Capital cost is less than half the price of a new glass-lined stirred tank reactor. Benefits to the company of applying PI will involve safer process operation, improved product quality and increased productivity. A successful application of this individual plant design would give impetus to modernising the whole site, making it a cleaner, safer and more efficient place.

Future work will focus on the laboratory protocols section of the methodology. This involves further development of experimental equipment and procedures to demonstrate intensified, continuous operation. This is a vital part in proving the success of a PI concept and will allow determination of the benefits achievable, without the need for building a continuous pilot plant.

Awareness of PI still has to be raised in some sectors of the chemical industry, though there are signs that many firms are looking towards innovation as a means of gaining a competitive edge and meeting legislation. A change in the way process development is traditionally done will be required for innovation to be properly adopted. This PI methodology provides a mechanism to promote such a change by encouraging PI to be considered where it may normally be overlooked.

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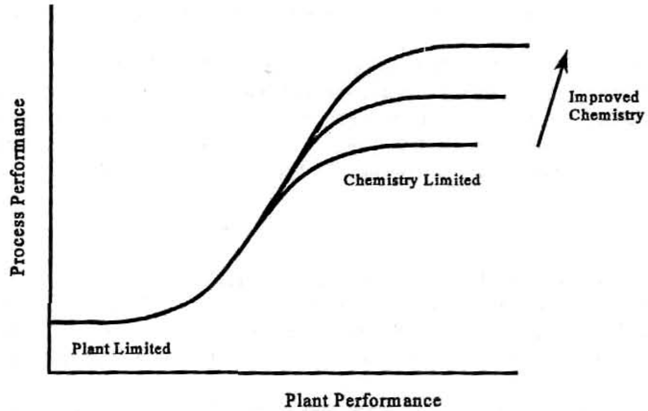

Figure 1. The S-Curve of Process Performance

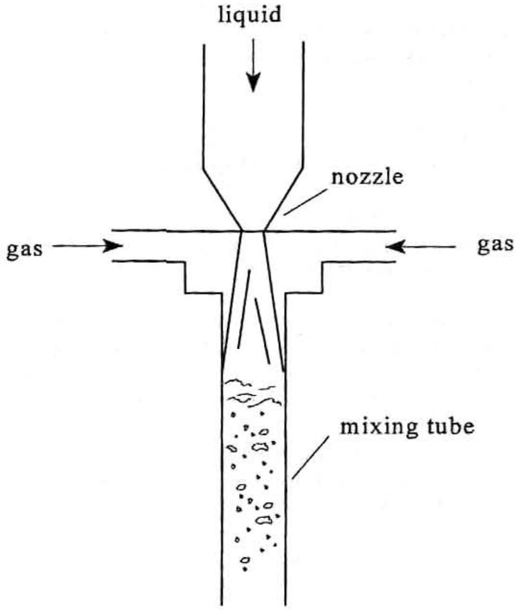

Figure 2. Gas-liquid ejector

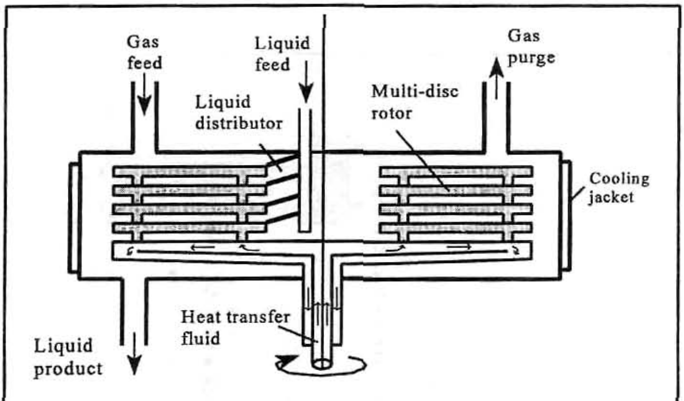

Figure 3. Spinning Disk Reactor

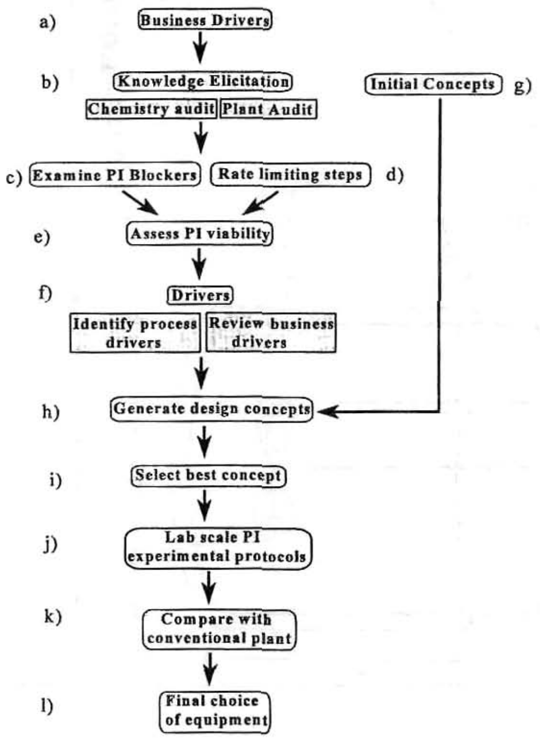

Figure 4. Methodological Approach to PI Applications

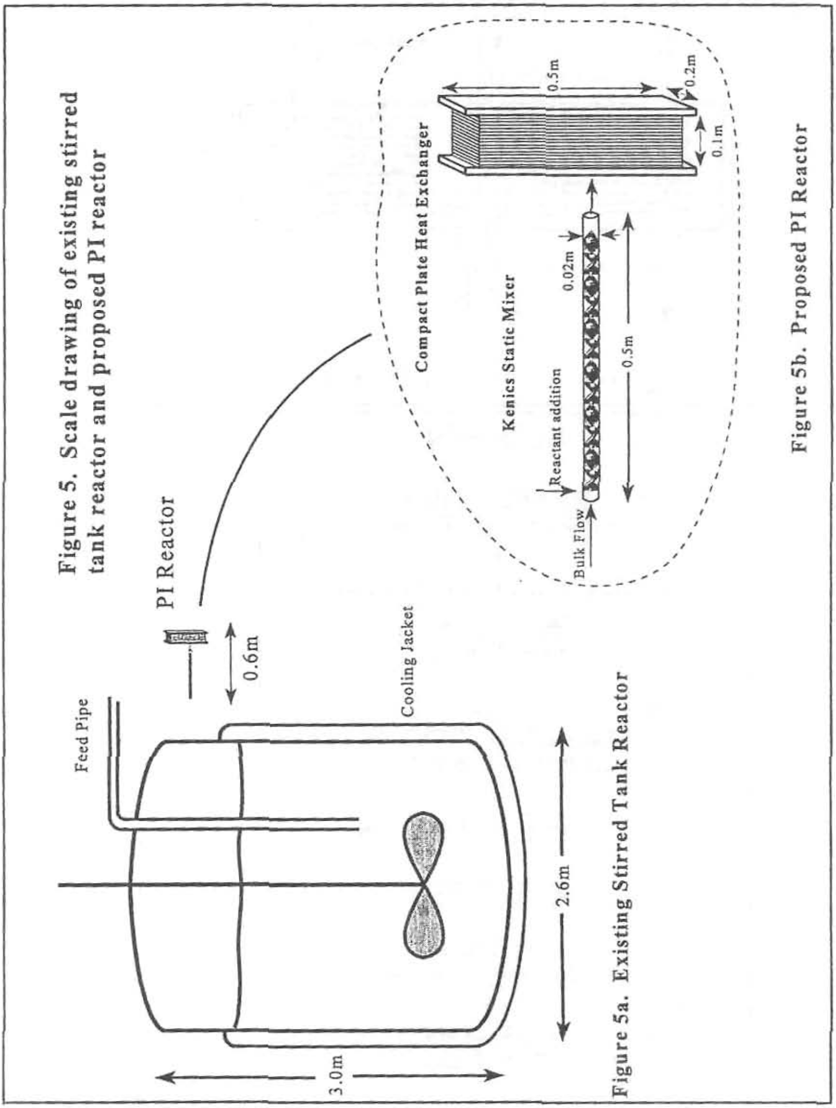

Figure 5. Scale drawing of existing stirred tank reactor and proposed PI reactor

Figure 5a. Existing Stirred Tank Reactor

Figure 5b. Proposed PI Reactor