

PROCESS INTEGRATION AND INDUSTRIAL POLLUTION PREVENTION

Merging Theory and Practice in Graduate Education

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Pollution prevention and process integration are highly consistent concepts in chemical manufacturing. Pollution prevention through source reduction, reuse, and recycle of resources is stipulated by law.^[1] Process integration is inherently conservation oriented, as it has the primary goal of maximizing the efficiency of design by minimizing the consumption of materials and energy.^[2] A chemical process in which components are integrated in an efficient system will generally be more economical and will pollute less. Ultimately, our goal in chemical process design should be closed-loop systems with zero emission to the environment, but this goal, of course, has to be balanced against economic factors and other constraints that would normally be encountered in practical situations. This can only come about through the education and training of scientists and engineers who can implement technological advances in designs that integrate regulatory and environmental considerations with the goal of economic development within the constraints of an industrial operation.

A new course, "Process Integration and Industrial Pollution Prevention," was recently developed at the University of Virginia with the assistance of several experts and with the participation of a company sponsor. The course is offered within the university's Environmentally Conscious Chemical Manufacturing (ECCM) Program (supported by a Graduate Research Training grant from the National Science Foundation and by a grant from the Academic Enhancement Program of the University of Virginia), which has both research and educational components. Fundamental research

is conducted in four major areas: development of inorganic catalysts; development of biocatalysts for chemical synthesis; development of alternative solvents; and development of adsorption processes for separations and energy applications. In each area the goal is to provide a foundation for the conception and development of alternative, environmentally benign technologies.

This graduate course in pollution prevention provides a central focus for both the students and the faculty participat-

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ing in the program. It seeks to provide an opportunity for integrating technological developments within the framework of real industrial constraints. Thus, a major component of the course is a project carried out in collaboration with an industrial partner. In addition, the course exposes the students to legal and regulatory issues, integrated design methodologies, hierarchical strategies for pollution prevention, and computational tools for waste minimization.

As a whole, the course is an example of a new approach to graduate education in chemical engineering. It provides an environment where students can *learn while simultaneously solving significant industrial problems*. This, of course, is always done through the research component of doctoral studies, and that must continue to be the main focus. On the other hand, doctoral research is typically long-term, fundamental work, often remote from industrial applications and almost always conducted in ways quite different from those used in industry for the solution of plant problems. In an industrial setting, short time frames, teamwork, and integration of economic considerations are often dominant issues that are not considered within the typical doctoral research activities. An approach where graduate students are immersed for a time within a practical setting is common in many professional graduate schools, such as business, law, and medicine, but is not often found in chemical engineering. This field, however, could also benefit tremendously from such exposure in ways that could provide avenues for successful partnerships between industry and academia.

COURSE RATIONALE

The fundamental premise for this course is shown in Figure 1. An efficient, nonpolluting chemical process is based on technological innovations that are rationally integrated in a system. The "building blocks" (fundamental principles and unit operations) come from research; the "how to assemble"

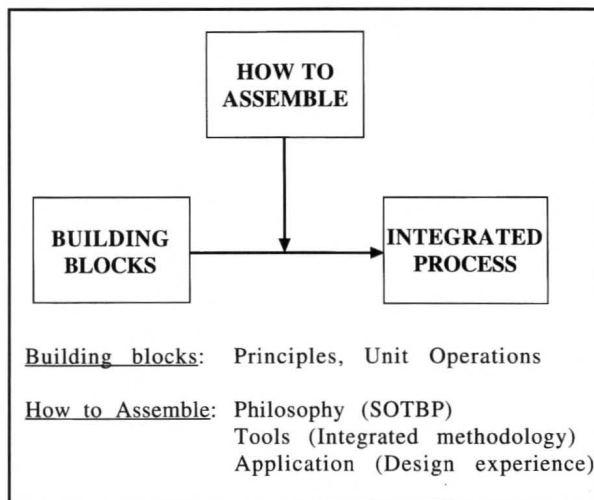


Figure 1. The focus of this course: how to integrate building blocks into integrated process design.

know-how and tools are covered in the course.

There are three main themes:

1. The *philosophy* of integrated design
2. The *tools* for an integrated design methodology
3. The *application* to an actual industrial situation

The underlying *philosophy* taught in this course is what has been called the "Science of the Big Picture," or SOTBP, as a simple heuristics set for design that entails the following steps:^[1]

Consider the "big picture first" by looking at the global process as an integrated system

Use fundamentals at each step to establish a priori targets for design efficiency and the consumption of materials and energy.

Worry about details (later) to finalize the design and support the global view.

The term "fundamentals" here denotes thermodynamics laws and basic mass and energy balances. Virtually all established integrated design *methodologies*, such as thermal pinch analysis^[4,5] and mass exchange networks,^[6,7] follow this general heuristics set. Following this approach, process design evolves through a series of decisions that provides increasing levels of detail. Each decision will be sound from the overall viewpoint and will be consistent with fundamentals. Details are worked out later, only after the overall structure of the process has been established. Thus, a global understanding of the process is obtained first. Next, methods of structured thinking, including how to decompose the design problem and to apply rules-of-thumb and experience, are used. And finally, simulations and/or experimental analyses are used in support of fundamental understanding and to complete design details.

The *tools* reviewed in the course are methodologies for integrated design. The hierarchical review methods of Douglas and others^[2,8-10] are covered in detail and contrasted to traditional approaches that break down a process into its unit

operation components instead of levels of analysis. The point is made that while breaking the system down into units has been a traditional way of dealing with complex processes, it provides little information about the interconnection of the elements and sheds no light on how these elements should come together to form an efficient process. Techniques for heat and mass integration are also reviewed with an emphasis on visualization techniques as a key to understand the problem, generating solutions, and communicating. Legal and regulatory issues are also covered and are viewed as an integral part of the “big picture” of the pollution prevention problem.

Finally, the *application* component of the course is an actual industrial project that is developed as an assignment for the students in collaboration with an industrial sponsor. The students are given free rein to solve the problem at hand using any of the tools available and including experimentation. The project has to be conducted within the framework imposed by the industrial sponsor, however.

COURSE ORGANIZATION AND CONTENT

The course was team-taught and involved the participation of several experts. Approximately half of the class time was in a lecture format, while the remaining time was spent working on the course project (see Table 1). In the first offering, eleven doctoral students took the class. They were divided into two design teams, each with an appointed team leader. Regular meetings were scheduled with the course instructors.

The course began with an overview of the driving forces at the root of pollution prevention in industry (environmental regulation, product quality and consumer demand, and efficiency and cost reduction), of the industrial obstacles to its implementation (unfavorable balance between long-term environmental benefit and short-term profit, capital requirements, inertia), and of regulatory impediments to pollution prevention (complexity and inconsistency of environmental regulation, regulatory focus on individual media, narrow definitions of pollution prevention).

Next was a detailed coverage of the legal principles of environmental regulation and of regulatory aspects of management strategies for achieving pollution prevention. Four lecture hours given by two practicing environmental lawyers covered the history of modern environmental law, the relationship between federal and state government in the environmental arena, and the common elements of statutory programs such as ambient and performance standards, permit programs, self-reporting requirements, and enforcement tools. The structure of Virginia’s environmental programs (water and air) was also covered as an example.^[11] These lectures included an overview of the Pollution Prevention act of 1990 and of ISO 14000 and 14001 standards.^[12]

The next set of lectures introduced a general framework for pollution prevention through integrated design.^[13] We began by defining design as a multifaceted problem that depends on both “technical” and “nontechnical” factors, including the overall business environment the company operates in, how the design work is organized, and the importance the company places on process design. The key point illustrated here was that pollution prevention is not just a “technical” problem. And, it is not just a matter of management. It is both—which makes it especially challenging. Considerable emphasis was then placed on correctly structuring “the problem.” The instructors’ experience is that engineers, even experienced ones, must be helped to see the big picture first and to fill in the details later. It is a process that must begin with a clear definition and setting of goals: the sponsor’s goals, the goals of the individual participants, and the time-frame within which these goals are to be attained.

The students were challenged to define their own personal goals in this class, pointing out that the ultimate measure of

TABLE 1
Course Syllabus

<i>Week</i>	<i>Topic</i>
1	Introduction; course outline, driving forces, and impediments to pollution prevention
2	Legal principles of environmental regulation; management strategies for pollution prevention
3	Process integration for pollution prevention—fundamentals
4	Introduction of course project
4-5	Hierarchical review of chemical processes for pollution prevention
5-7	Heat and mass integration—HEN’s and MEN’s
8	Waste assessment and pollution prevention measures
9-14	Course project, team meetings, interaction with sponsor, and presentations

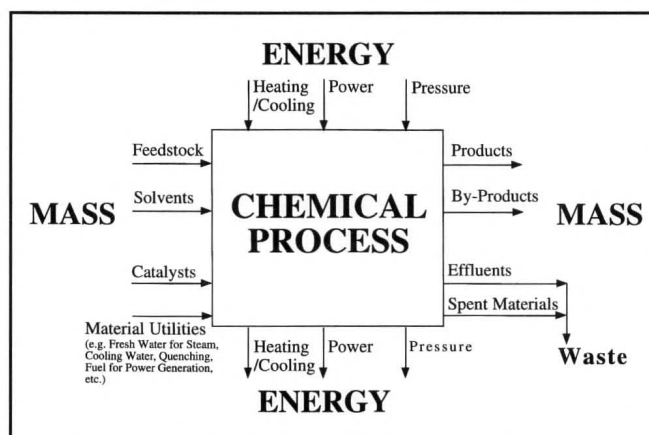


Figure 2. Global view of a chemical process as a closed system with integrated mass and energy flows.^[1]

success might be the actual implementation of the solution proposed for the course project. We made the point that just arriving at a solution is not sufficient—getting the sponsor to appreciate its value and to implement it is part of the job. Thus, we emphasized the need for effective contact with the sponsor as a key to credible, implementable solutions. The next set of two lectures was devoted to a presentation of the course project (described later). Two process engineers from the sponsoring company presented the situation defining the problem, the technical company constraints, and the expectations.

Following this introductory material, a set of lectures was devoted to a review of process integration tools. Students were challenged to view chemical processes as integrated systems such as the one depicted in Figure 2. Our job is to find the most favorable allocation of mass and energy flows

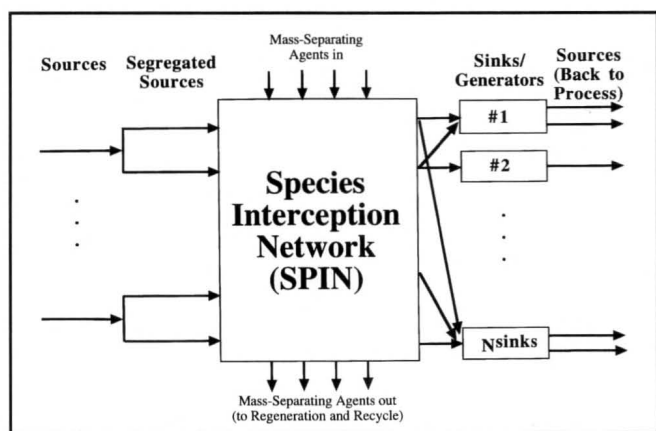


Figure 3. Mass allocation network with segregation, interception, and recycling strategies.^[1]

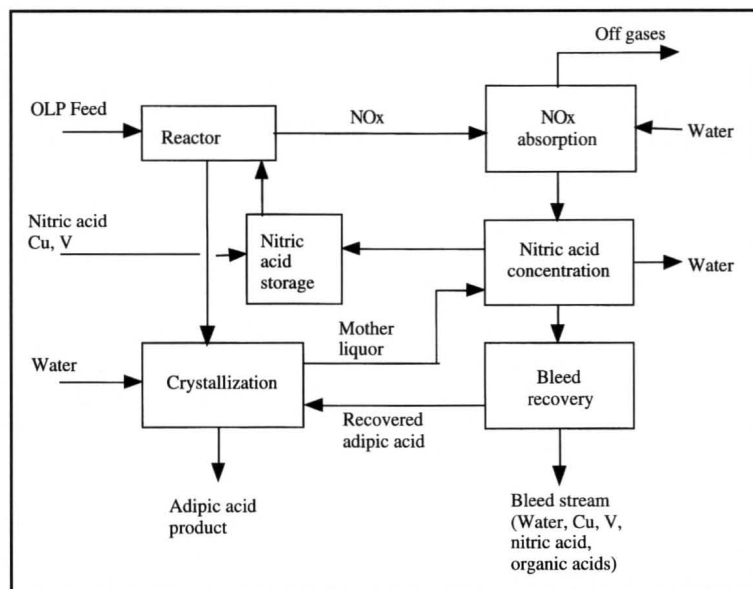


Figure 4. Simplified process flow diagram for adipic acid manufacturing process used as course project.

that would minimize waste. The first step in the analysis is a hierarchical review of design decisions at different levels of detail. Following Douglas' work,^[8,9] we made the point that this procedure, originally developed for process synthesis, can also be applied for waste minimization in an existing facility, as discussed, for example, by Rossiter, et al.^[10] In fact, by understanding how waste is generated from decisions made at different structural levels, one can systematically develop retrofit alternatives.

Beyond the hierarchical review approach, two important dimensions of the problem were considered in detail: the creation and routing of chemical species (mass integration or mass exchange networks—MEN's) and the application of energy (energy integration, or HEN's). In heat integration we examined tools such as pinch analysis (or pinch technology),^[4,5] mixed-integer nonlinear programming, and simulated annealing^[14] that shift the focus away from the heating and cooling of individual streams to the global allocation of energy in an integrated system. Similarly, in mass integration, we examined recently developed techniques^[6,7] that shift the focus away from merely linking unit operations (reactors and separation units) to a global allocation of chemical species by considering the efficient creation of desirable species, the minimization of undesirable components, and the routing of species to their most desirable destinations. A recent monograph by El-Halwagi^[15] provided reference material and case studies in mass integration. Figure 3 shows the generic approach for species allocation presented in this work.

When the overall process is considered, waste minimization targets can be established *a priori* based on fundamental application of overall balances. This is true for both energy and mass integration. A *species allocation diagram* similar to that in Figure 3 is drawn for each chemical species (e.g., water in a waste-water minimization problem). The first step is then to consider segregating each "source" or stream containing that species. Often, major waste reduction can be obtained simply by avoiding mixing of process streams and reusing the segregated sources with a direct recycle to the process.

The next step is to consider implementation of a "species interception network" (SPIN) where mass-separating agents are used to upgrade segregated sources to a quality adequate for in-process recycle to appropriate "sinks"—ordinarily unit operations that can use the upgraded sources. Shortcut design methods are used for screening alternatives at this level on the basis of cost estimates and economic potential.

Finally, the last step is to consider manipulation of "sinks" and "generators" so as to reduce the creation of undesirable species and improve their efficiency.

Clearly, a hierarchical level exists in this analysis. Usually, acceptability (and, in many cases, impact) is greatest for simple segregation and direct recycle modifications of an existing process while acceptability, and sometimes impact, is normally smallest for more costly sink/generator manipulations.

In a final set of lectures, we covered quantitative aspects of pollution monitoring and emission inventory^[16] and examples of pollution prevention measures based on the work of Nelson.^[17] The rest of the semester was spent working on the course project in close collaboration with the industrial sponsor.

COURSE PROJECT

The project for this course was suggested by AlliedSignal personnel. It deals with an existing problem concerning copper emissions from an adipic acid manufacturing plant. At a facility in Virginia, AlliedSignal produces about 30 Mlb/yr of adipic acid via the oxidation of a waste stream composed of cyclohexanol and cyclohexanone, which is generated in another process. Figure 4 shows a simplified process flow diagram. The process produces 3,000 to 5,000 lb/h of crystalline adipic acid as well as a bleed stream at a rate of about 2,000 to 3,000 lb/hr that contains water, nitric acid, organic acids by-products (principally glutaric and succinic), as well as copper and vanadium, which are catalysts for the oxidation process. Currently, the bleed stream is neutralized and sent to a regional waste-water treatment plant. The company anticipates that changing waste-water regulations will require the virtual elimination of copper discharges. The following objectives were thus set by the sponsor

1. Recover copper and vanadium from the bleed stream
2. Recover nitric acid from the bleed stream and recycle to the process
3. Recover the organic acids from the bleed stream

The sponsor specified that the first objective *must* be accomplished. Objectives 2 and 3 were to be pursued if economically feasible. The sponsor required that the solution should be implementable in an 18-month time frame and that a 35% simple rate of return should be used for economic evaluation. The short time frame precluded most solutions involving changes in the process chemistry that would entail major modifications of the existing adipic acid facility.

The eleven PhD students enrolled in the class were divided into two independent teams that engaged in a friendly competition. Each team conducted its own literature search, visited the plant on several occasions, obtained samples of the bleed stream and other data from the plant, conducted their own experimental investigation, and carried out preliminary design calculations and economic estimates. An initial proposal was to install end-of-pipe treatment devices

(ion-exchange columns, membrane systems, electrochemical methods, etc.) that would remove copper from the bleed stream. When the process was analyzed from a global viewpoint, however, these alternatives were quickly discarded since it was recognized that it would be sufficient to recover the organic acids in order to produce an aqueous stream containing copper and vanadium catalysts and nitric acid that could be recycled directly to the process. This required the definition of a waste interception network capable of removing the approximately 400 to 800 lb/hr of glutaric and succinic acid that is produced as a by-product in the oxidation process.

The teams recommended different solutions. One team followed a more traditional approach of esterifying the acids with an excess of methanol and extracting the diesters with an organic solvent. The students initially followed a US patent^[18] describing a reactive-extraction process, but then decided to perform their own experimental investigation finding that no-catalyst was required for esterification in the bleed stream and that toluene could be used as an effective extraction solvent. The other team followed a more innovative approach, discovering in the laboratory that, for the conditions of the bleed stream, the organic acids could be extracted into an existing process feedstock. Although the partition coefficient is not as favorable as in the case of extraction of the dimethylesters, the proposed approach has the advantage that the extraction solvent is a reactant in the process so that no contamination of the existing adipic acid facility with extraneous species would occur. The raffinate, devoid of a large portion of the organic acid by-products, could be recycled directly to the adipic acid process.

Based on preliminary design and economic estimates, each alternative met the sponsor's objectives. In each case, in addition to virtually eliminating copper emissions, potential sales of the recovered organic acids would provide a substantial economic return. At the conclusion of their work, each team presented their recommendations to the sponsor. This task was made rather challenging by the fact that the presentations took place at the plant and were attended by a large group that included senior plant management, several process engineers, plant operators, environmental officers, and chemists and engineers from the technical support group of the company. The students were asked to meet this challenge with a balanced presentation addressing the diverse audience. The company is now evaluating implementation of one of the two proposals made by the students and is acquiring equipment for pilot-scale testing.

Finally, it should be noted that although the project scope was limited by the sponsor to water emissions, a broader-scope project could be developed by considering air emissions as well.

CONCLUDING REMARKS

This course was an invaluable experience for both the students and the faculty. It provided the students with an opportunity to do something that is rare in graduate education, but common in industrial practice—the opportunity to work in teams on the solution of a real problem from the basics to virtually its implementation. The students who took this course are involved in doctoral research in extremely diverse areas, from catalysis to biochemical engineering to separations. Thus, each student brought particular skills to the completion of the project. Through the team's efforts, each student learned to treat real problems from a global perspective within defined constraints. One of the main lessons was that optimum solutions to environmental problems often come from an intelligent reengineering of the process itself, rather than through the adoption of end-of-pipe approaches.

The course was geared for doctoral candidates and most of the students were at an advanced level. Although a different version of such a course could be developed for all graduate students, the time commitment required for a successful interaction with industry could be difficult to meet by beginning graduate students who are engaged in other coursework.

A key to the success of this course was the commitment of the sponsor to the effort. The company sponsor shared valuable data and experience with the students and provided guidance at a level that the faculty alone could not have provided. Likewise, external experts who participated both in the conception and in the offering of this course provided a different and unique perspective that added tremendous value to the educational experience.

Finally, there was a "real" value in return to the company sponsor. Aside from the direct technical contributions, company personnel were exposed to a way of thinking about chemical processes that is different from traditional, unit-operations-based approaches. Essential to this success was the sense of trust and respect that developed among the participants through frequent interactions at different levels.

This course provided several opportunities for the development of instructional modules for use in our undergraduate design class. One of the modules is focused on legal and regulatory aspects, with emphasis on pollution prevention. The other is a design project based on the data obtained by the graduate students and their proposals for the solution of the copper emission problem. The latter was offered as a senior undergraduate design project. The undergraduates who took the project enjoyed working on a real problem using data developed "in house" as the basis for their design calculations and learned a great deal by working within the constraints of an actual industrial situation. A version of this project including technical specifications and economic data is available upon request.

ACKNOWLEDGMENTS

Development of this course was supported by a grant from the Academic Enhancement Program of the University of Virginia. We are indebted to Stan Sitnik of AlliedSignal, Inc., and to Wayne Halbleib of Mays & Valentine for their participation in the course. Support from the National Science Foundation Graduate Research Traineeship program is also gratefully acknowledged.

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