NEBRASKA'S INTEGRATED PROCESS DEVELOPMENT/DESIGN LABORATORY

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SEEMINGLY MOST RESISTANT to change in the wave of innovation that has been so well documented in this journal is the traditional unit operations laboratory. Typically rather set experiments are conducted by two or three-man teams on fairly large pieces of equipment, followed by the submission of formal reports. There are obviously some advantages inherent in this format. Turning valves and watching apparatus approach steady state are good experiences for any chemical engineering undergraduate, and exposure to technical report writing has value. On the other hand, this type of exercise has limited flexibility. The equipment can be operated in only so many ways, and a file of previous years' laboriously written reports effectively squelches much new thinking by even the best of students.

Until 1969 Nebraska had a three-credit senior laboratory course in much this mold, with wetted wall and packed column mass transfer, concentric pipe heat transfer, and vapor-liquid equilibrium experiments. (Distillation and evaporator exercises had previously been abandoned.) In addition there were junior transport and senior process control laboratories, each for one credit. With the exception of the process control course, which featured nearly all modular equipment, we were encountering the same lack of enthusiasm and originality seen elsewhere.

This was probably sufficient to dictate a change, but we had yet other reasons. Our department has a strong tradition in thermodynamics and vapor-liquid equilibrium and therefore offers 1½ terms of them and only about one-half term of kinetics. The increased emphasis on kinetics in the last few years encouraged us to offer more, but we did not want to de-emphasize a strength at the same time.

These factors suggested to us that a laboratory sequence stronger in kinetics that required more originality from our students would distinctly strengthen our curriculum. Properly formulated, this sequence could serve as a wrap-up to our whole program in that previous material could be re-emphasized, and could in addition give our students a taste of industrial problem-solving. The last point was especially important to us, because Nebraska is a primarily agricultural state and few students have any contact with either industry or engineers.

NEW SEQUENCE

THE BEST METHOD to achieve these goals seemed to be replace the unit operations and transport laboratories and a three-credit design and economics workshop course offered to second semester seniors with a two-semester, seven-credit, integrated laboratory and design sequence. This sequence was conceived to allow seniors to employ their own experimental data, along with those they could estimate or find in the literature, to achieve a preliminary design of a plant producing some simple but commercially unavailable chemical. Laboratories and design sessions were to be fairly free-form, specified laboratory times being necessary only because some equipment had to be used by more than one group. Equipment was to be simple, essentially glassware with some mixers and small metering pumps. Faculty were to serve primarily as consultants and were not to specify either experiments or treatment of data.

> We fully believe in the advisability of asking students to provide their own direction, even though wasted time and poorer experiments and designs result from it.

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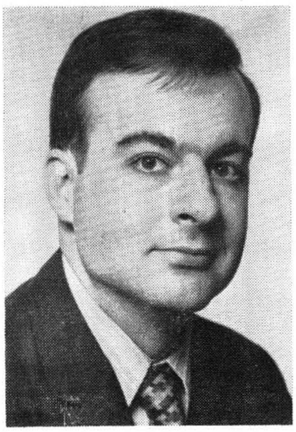

James M. Eakman received both his B.S. and Ph.D. from the University of Minnesota. He worked in the area of process research for Archer-Daniels-Midland and Ashland Chemical Co. before coming to Nebraska in 1968. His teaching and research interests include process engineering, computer aided design, optimization, and statistical computations. (Above)

The only totally fixed feature was a weekly one-hour economics and design lecture during both semesters.

After piloting this concept with a group of six volunteers during the academic year 1969-1970, we have offered this laboratory sequence to the last four senior classes. In each year the specified product has been an ester, hexyl caprylate for two years; ethylene glycol dipropionate for one year; and 2-ethylhexyl oleate for two years (Table I). While the whole class studies the same reaction, groups are assigned different catalysts and operational modes to investigate so that little duplication in experimental systems occurs.

These systems were picked with several factors in mind. First, they were safe in that they were liquid at ordinary temperatures and were not flammable or explosive. Second, the kinetic mechanisms were known but were fairly complex. Third, there were problems in separating products from reactants—heterogneneous azeotropes in the first and third systems and a homogeneous one in the second. In addition, while the raw materials were fairly inexpensive, the products were not made commercially in large amounts. In fact, it is quite possible that we were the largest producers of one or the other. This prevented our students from discovering the process in the literature.

Table I. ESTERIFICATION SYSTEMS

ALCOHOL	ACID	PRODUCT
n-Hexanol	Octanoic acid	Hexyl octanoate
Ethylene glycol	Propionic acid	Ethylene glycol Dipropionate
2-Ethylhexanol	Oleic acid	2-Ethylhexyl oleate

CATALYSTS
None
Dibutylin oxide
Sulfuric acid
p-Toluenesulfonic acid
Strong acid ion exchange resin (amberlyst 15)

OPERATIONAL MODES
Continuous
Batch

Groups are expected to obtain kinetic data in approximately ten experiments to fit a model that is applicable at different temperatures, catalyst concentrations, and reactant ratios. This is, of course, a tall order for such a few runs, and any model obtained is admittedly only very roughly accurate; but we have found that there are diminishing benefits to the students by prolonging this line of attack any further. This rather draconian limit puts a great premium on a proper choice of experimental conditions for each run. We have become exceedingly hard-nosed in expecting a plan to be formulated before any experimentation begins, though we realize that changes often are necessary after some data have been gathered.

This kinetics investigation occupies virtually the whole first semester. The second semester is devoted to vapor-liquid or liquid-liquid equilibria, originally an experimental project where data from each group were pooled, but more recently where the behavior of each system was estimated; physical properties, now also estimated or found in the literature; and the plant design itself. To add a bit of realism to the design, each group is required to choose a specific plant location within Nebraska. Our more adventurous students occasionally interview chambers of commerce and take pictures of available buildings and land. We do find, however, that often plants locate near the students' home towns rather than at the most advantagious location. The State Department of Economic Development has been extremely cooperative in this venture, providing literature on sites and lectures on factors that are important in the choice of a location.

Seemingly most resistant to change in the wave of innovation, is the traditional unit operations laboratory.

STUDENT PERFORMANCE

SINCE THE MAIN GOAL of any curricular change is to increase knowledge learned and retained by students, this sequence must ultimately be judged on these grounds. How have our students done since the curriculum was changed?

Performance is appreciably better than in the old arrangement. Participants now are challenged to use kinetics, distillation and extraction design, vapor-liquid equilibrium and thermodynamics. Often they must go beyond classroom knowledge, for instance when confronting multicomponent distillations that behave in a highly nonideal manner. They have found that sophisticated computer methods are a great aid in design and optimization. While often they can obtain computer programs already written, on many occasions they have had to do their own programming.

Students are confronted with the concept of using their experimental data further. We would like to believe that this causes them to be more careful and more complete, but we notice that in many cases data are not yet fully trustworthy. This is partly due to the use of equipment for specific purposes for which it was not necessarily designed, inadequate analytical methods, and a general lack of time.

Undoubtedly more work is expended. Most of it is gainful, but some is wasted through improper choice of experiments, uncertain data, and lack of proper data-collecting. We expected this when we refrained from specifying experiments. To some extent the wasted motion is beneficial, in that it emphatically teaches the lesson that proper preparation ultimately saves time.

We do not claim that the students are fully happy at all times over the change. They realize that they are working harder, and they have several specific complaints in addition. Most deal with the intentional formlessness of the sequence. They would like more faculty direction and equipment more specifically designed for the experimental program. They are quite upset when work does not immediately lead to useful results.

We fully believe in the advisability of asking students to provide their own direction, even though wasted time and poorer experiments and designs result from it. This sequence is quite a change from their other laboratories and lecture courses, which are highly structured, and we expect them to be quite discomfitted.

The new sequence provides the opportunity for procastination, especially during the beginning and middle of the second term when the initial enthusiasm has disappeared and the task appears endless. We partially combat this by requiring periodic reports (Table II) and by informal questioning of results. This is a thin line to tread, since we want the students to provide their own direction.

Ironically, many groups are loath to seek faculty help or advice even when bogged down. Occasionally faculty initiative is required to open communciations.

Students now are challenged to think independently, since very little information is being fed to them. If they are successful it is a strong prod to their self-confidence. Talks with graduates of this sequence leads us to believe that their prac-

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tice in independent thinking and their added self-confidence have been helpful to them in industry.

Table II. REPORT SUBJECTS

First Semester

- Analogous commercial processes—Esterification mechanisms
- Possible rate equations—Proposed kinetics experimental design
- Interim kinetics data
- Final kinetics data and model evaluation

Second Semester

- Batch, tubular, and CSTR sizing
- Reactor costing
- Estimated boiling points
- Estimated vapor pressure curves
- Estimated vapor-liquid or liquid-liquid equilibria
- Final report—Plant design, product cost, and location

The more perceptive students quickly recognize the link between theory and experiments, and they use theory successfully to design experiments. The lesson is later solidified, since experimental data must be extrapolated with theory to obtain proper design.

Eventually the usefulness of proper experimental design becomes apparent to nearly everyone. Unfortunately for many of the students, this awareness often does not come early or easily. If we were primarily interested in good results during this laboratory, we would take a more direct hand in helping students set up their experiments. Since, however, we view the laboratory mainly as a learning tool, we allow quite a bit of slipping and sliding before stepping in.

One of the unexpected results was that three-man groups were over any extended period more unstable than groups of either two or four members. Usually one or occasionally two of the three did not carry a fair share of the load. This of course led to hard feelings, even though we pointed out to the aggrieved parties that they probably learned more that way. The problem was solved by going to two-man groups. Only a very exceptional person has the temerity to leave the whole load on his partner. Conversely, the dominant partner, if one emerges, realizes that civility enlists more cooperation than alternate modes of behavior.

POSTSCRIPT

TO SOME EXTENT this approach has some flavor of reinventing the wheel, since we learned long after undertaking it that the late Professor Vilbrant at VPI had trod a similar path a number of years ago. However, the development of this sequence has educated us during the past five years perhaps more than any of our students. Our appreciation goes to those students who have volunteered advice, some of it perhaps not so well received at the time, that indicated which of our many changes were successful and which were not. No course of this type can ever stand still. We, therefore, hope that our students in the future will be equally free with their help. □

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