REVIEW: Developments in Distillation and Separation Technology

Kenneth E. Porter
Speedwell House, Birmingham B5 7PR, U. K.

With the focus on the power of theoretical concepts to enable the creation of new technologies, separation technology is discussed in terms of "intellectual tools" derived from ideas in diffusion accompanied by reaction and from "pinch point" techniques. Ideas are the intellectual tools shaping the changing world (we live not in the information age, but in the intellectual age). Developments in distillation equipment during the last 20 years reveal the importance of business drivers for stimulating change, and why the 1980s was the decade of structured packing and the 1990s of high throughput trays. Because of out inadequate understanding of phase equilibrium (no molecular theory of liquids) and of two-phase flow (except for when one phase is very dilute), process and equipment design is still essentially empirical. In conclusion it is noted that more might be done to encourage the development of young intellectual tool-makers. Research in this field might help speed up technical innovation.

Introduction

In recognition of the changes brought about by computers and information technology, it is now commonplace to refer to the present time as the *information age.* However, information is only useful if it is organized in some way and at best formed into patterns by theoretical concepts. The technological world we live in today has been defined and made in the minds of engineers. It has been constructed by ideas which are the intellectual tools by which we shape the world. We should perhaps describe the present time as the *intellectual age,* rather that the *information age.* The most useful ideas are concepts which not only explain previous observations, but which cause us to make predictions which are then found to be true. Some of these predictions, expressed in terms of new technology, may lead to technical breakthroughs. These new developments generate yet more information some of which cannot be explained even by the novel concept which generated it. Thus, information builds up yet more quickly and even newer concepts are needed to organize it and produce the next technical breakthrough. The process is illustrated in Figure 1 where the rectangular blocks represent information arranged in patterns by concepts and the dots represent things which are known but not yet understood.

In this article I will discuss separation technology, and in particular distillation, in terms of information and ideas or concepts. I will suggest that we have today an abundance of information, half structured by empiricism and empirical relationships, and are more than ready for new concepts to provide the new intellectual tools for new technical breakthroughs. In conclusion I will suggest that the creation of new concepts is the unique function of the universities, that those

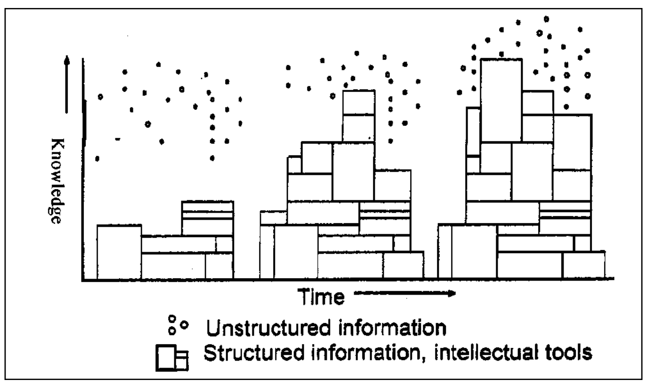

Figure 1. Development of a subject over time.

Correspondence concerning this article should be addressed to K. E. Porter.

few people who have the peculiar ability to do this original work may be identified, and that universities should be managed to support them.

Business Drivers and Intellectual Tool Box

In the context of this article, *business driver* is a term used to describe how the wider needs and desires of society are translated into financial targets for engineers. Intellectual tools may provide the means for solving technical problems, but the market place determines which problems are to be solved at a particular time. The business drivers which have been important in recent years have been a result of the energy crisis of the early 1980s, the increasing importance of "green" issues such as ecology and pollution, and at most times, the cost of investment.

It then follows that, sometimes, valuable ideas are presented at the wrong time and must wait in the intellectual toolbox until they are needed. Examples mentioned below are the work of Huber (Huber and Hiltbrunner, 1966) on scaling up packed distillation columns and to a lesser extent that of Papendick (Papendick and Runkles, 1965) on microbial processes in soil and that of Hatta (1932) and Danckwerts (1970) on the diffusion reaction theories of mass transfer.

Recent Developments in Distillation Technology

At the time of the energy crisis of the early 1980s, in response to the need to design distillation columns "leaner and harder" (Rush, 1979), it was desired to install more theoretical trays into columns so as to work at lower reflux ratios. Most columns were trayed columns, and it was clear there would be great difficulties welding in new support rings to support more trays. It was known that, in principle, more theoretical trays might be obtained by removing the trays and replacing them with packing. However, there had been too many scale-up failures of packed distillation columns for packing to be a credible solution. There followed intensive research programs by Fractionation Research Inc. and by the equipment suppliers, and, by approximately 1983, the scale-up problem was solved. The solution is based on a concept for scale-up which may be applied in many situations. This is that channeling and maldistribution reduce performance, cross mixing acts to correct this, but cross mixing only works over a limited distance, so large diameter equipment may fail when small diameter equipment succeeds. See, for example, Porter et al. (1972). Thus, the solution to the problems of safe scale-up is to ensure that channeling and maldistribution cannot occur by putting better distributors into large equipment. This applies to packed distillation columns where the key to safe scale-up is to provide excellent distributors for the liquid, and sometimes for the vapor. See for example, Kunesh et al. (1987).

It is interesting to note that the theory of scale-up of packed columns had been published some fifteen years before by Huber and Hiltbrunner (1966), who worked with the Sulzer company. Sulzer had demonstrated the validity of these ideas and had built several large diameter packed columns for vacuum distillation. The use of packing in energy-saving revamps at higher pressures and, therefore, higher liquid irrigation rates permitted the use of the structured sheet metal packing which became the dominating equipment supply of the 1980s. The structured packing which had been used previously by Sulzer for low-liquid rate vacuum distillation was made of self-wetting wire gauze.

The massive energy-saving driven column revamping was completed by the 1990s, and it was to be expected that the industry would revert to the previous pattern of packing for vacuum distillation and the cheaper trays for all else. However, the increased volumetric efficiency of structured packing was such that it was claimed that a packed column might

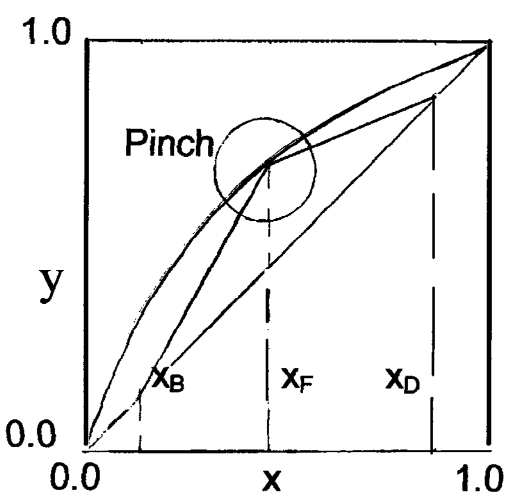

Figure 2. Original pinch point diagram of McCabe and Thiele (1925) for distillation column design.

x_D, x_F, x_B = concentration of most volatile component in top product, feed and bottom product respectively, mvc = most volatile component, x, y = concentration of mvc in liquid, vapor respectively.

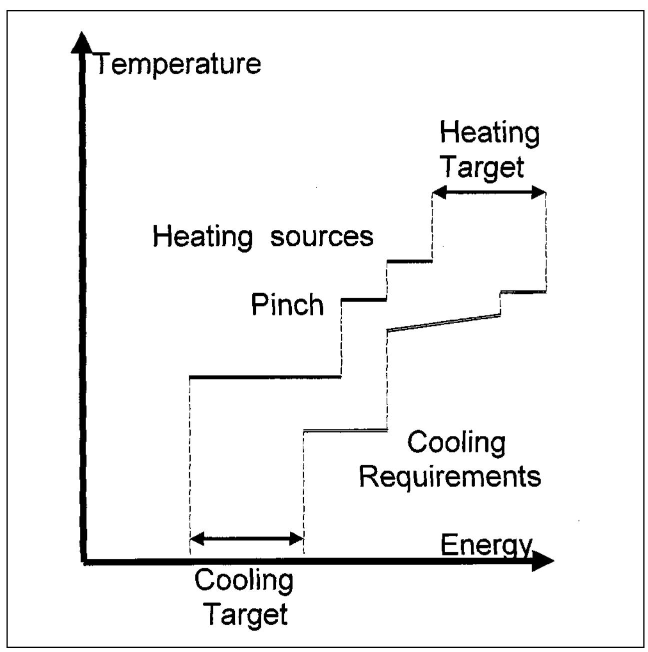

Figure 3. Pinch point diagram for process energy integration.

now be smaller (and, therefore, cheaper) than a trayed column. This combined with financial pressures provided the opportunity for the high throughput trays, which were developed in the 1990s.

Most of the high throughput trays are based on various ways of suspending the downcomers clear of the tray below, so as to release more area for the vapor, for example, as will be seen in Figure 9. They have demonstrated that the traditional empirical equations used for downcomer design, pressure drop, and so on work in these slightly modified situations. That is, more experience based information is now available. As far as I know, no high throughput tray has yet been designed by predicting from first principles how to increase the vapor and liquid rates for a given froth height. Is this because, at present, we lack a theory of two-phase-flow which will adequately describe the tray dispersions? It may be that before too long such a theory will become available and this may result in significant changes in tray technology.

Ideas as Intellectual Tools

To best illustrate the power of ideas, I have chosen two examples of concepts which have each been applied to several quite different practical situations. This emphasizes the importance of the concept rather that that of the techniques developed for using it for a particular purpose. The ideas are those of identifying a minimum flow rate by pinch point theories and those concerned with diffusion accompanied by a reaction near an interface.

Perhaps, the most widely known example is the use of "pinch" technology for finding the minimum energy required by a process after integrating the energy flows arising from different units. This is described in *A User Guide on Process Integration for the Efficient Use of Energy* (1982) and was introduced and championed by Linnhof (1979). More recently, a similar "pinch" idea has been used by Wang and Smith (1994) to reduce process water requirements by identifying where polluted water from one part of a process may be best used in another part.

The original pinch point analysis was described many years ago by McCabe and Thiele (1925) for finding the minimum reflux ratio in a distillation column. It is still one of the first examples of chemical engineering thinking which is taught to undergraduates. These three examples of pinch technology, minimum reflux, minimum process energy, and minimal process water are illustrated in Figures 2, 3, and 4.

All the pinch concepts are concerned with identifying a minimum: a minimum flow rate of pure water or of heating or cooling supply. They are based on simple principles, that of heat or mass balance, and that a temperature or concentration difference is needed to transfer heat or material from one stream to another. When this difference approaches zero, (the pinch), the cost of the equipment becomes infinite or the process impossible. The power of these intellectual tools is that they simplify complexity. They permit the engineer to think about the design problem in terms of some limiting minimum, a minimum reflux ratio, minimum energy requirement, or minimum water supply in a way that no numerical computer driven information processing calculation ever could. The minimum provides a means of assessing his final design decisions, which will be based on technical and eco-

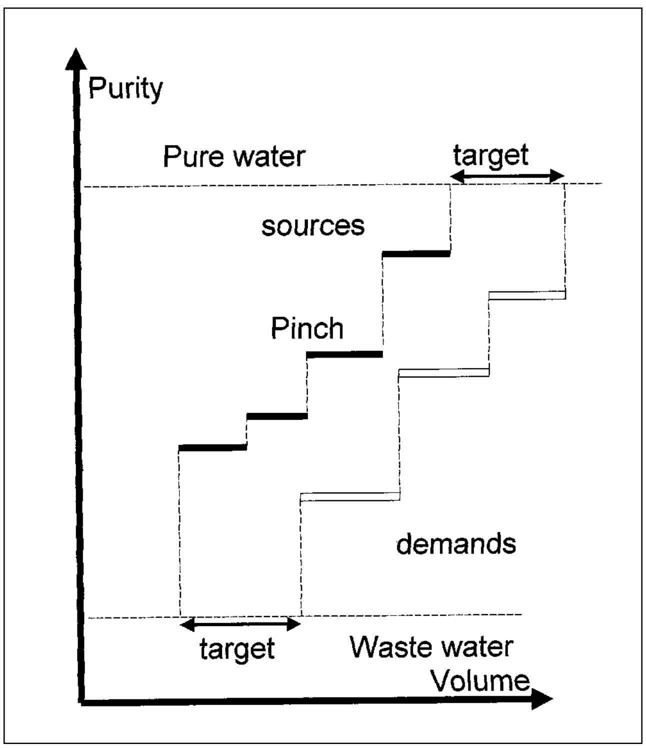

Figure 4. Pinch point diagram for economizing for process water use.

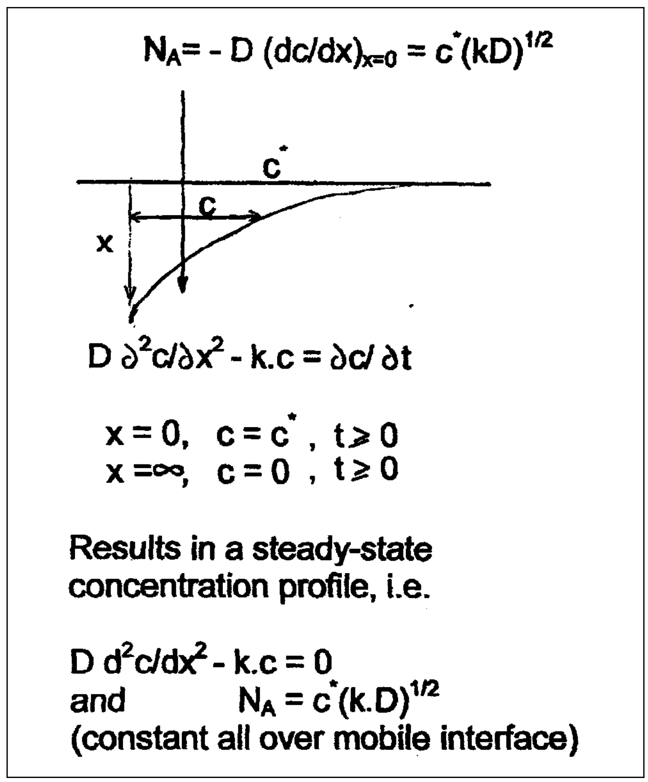

Figure 5. Diffusion of a species from an interface into a region where it is consumed by a reaction (in this case a first-order reaction).

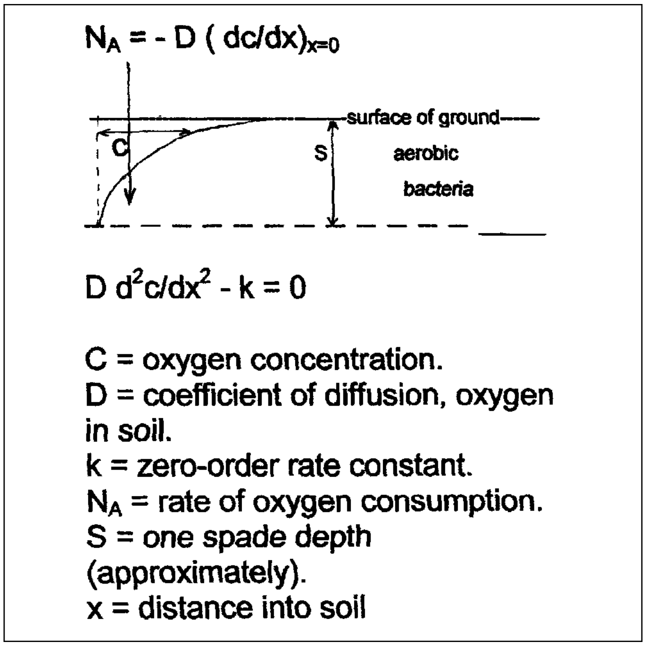

Figure 6. Diffusion of oxygen from air into soil where it is consumed by a zero-order aerobic microbial reaction, but only over a limited distance.

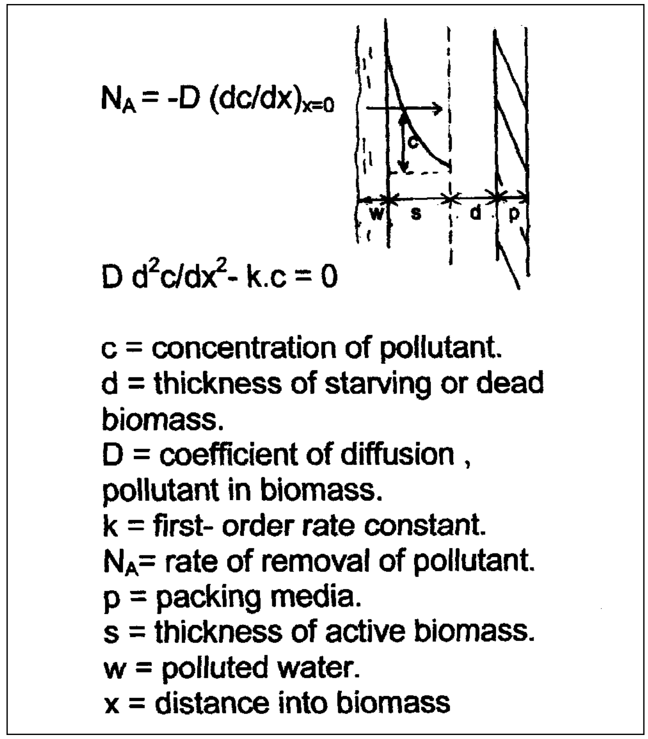

Figure 7. Diffusion accompanied by a microbial reaction controlling the thickness of the active layer of biomass in a packed bed used for removing water pollution.

nomic calculations and on the need to avoid overcomplex processes or unnecessary technical risk. The contributions of pinch technology to process design has been recognized by several prestigious awards.

The power of the diffusion with reaction concept is probably less well known. The mathematical basis for it, shown in Figure 5, was first introduced to Chemical Engineers some 50 years ago by Hatta (1932) as part of his analysis of gas absorption accompanied by a chemical reaction, and by Thiele in his well known description of the activity of catalyst particles. The properties of the solution shown in Figure 5 that I wish to emphasize here is that of the *steady-state* concentration profile which occurs when the rate of diffusion of a species through the interface becomes exactly the same as the rate at which the species is removed by the reaction in the medium adjacent to the interface.

In gas absorption, this provides a situation where the rate of absorption per unit area of interface is constant and predictable from the physicochemical properties of the system. It is the basis for the chemical method of estimating gas-liquid interfacial area, as shown by Danckwerts and Sharma (1966) and Porter et al. (1966a). In catalyst design it illustrates that, in some circumstances, only that part of the catalyst which is near the surface of the pellet will be contributing to the reaction.

The same concept has been used to explain several other quite different situations. For example, as shown in Figure 6 by Papendick and Runkles (1965) to estimate the limited depth of the aerobic active layer near the surface of the ground (one spade depth?), or as shown in Figure 7 by Porter (1982) to identify the mechanisms of biomass growth and death by starvation in an irrigated packed bed used for removing organic pollutants from water, and as shown in Figure 8, perhaps the most unexpected application, the reason for scale-up failures of sieve plat distillation columns (Porter et al., 1972).

The chemical method for determining interfacial area was developed some 30 years ago as part of investigations into the science of mass transfer with chemical reaction by Danckwerts and others. The use of this science in design depends both on being able to calculate several different physicochemical properties, often in salt solutions, and on a suitable calculation procedure based on mass-transfer coefficients. Only recently have such procedures become widely available (the rate based methods of Krishna and Taylor). At present, these methods do not usually include the ‘‘chemical’’ area, but it seems likely that this will be needed, particularly for situations where more that one species is reacting. Recent work and a convenient technique is described by Wermer and Schaber (1997).

The calculation of the active depth of soil will have been of interest to horticultural scientists since it was published. More recently, it might be used to help those concerned with cleaning up oil spillages by answering the question of whether we can expect microorganisms in the soil to consume the spilled oil? The answer is yes, but only near the surface of the ground.

Several valuable insights are obtained from the application of the diffusion with reaction theories to biomass growth in packed beds. The overall mechanism of removing dissolved organic pollutants from water by biological means is to transform a dissolved pollutant (at a very low concentration) into a solid which can be settled out. The solids grow as ‘‘biomass’’

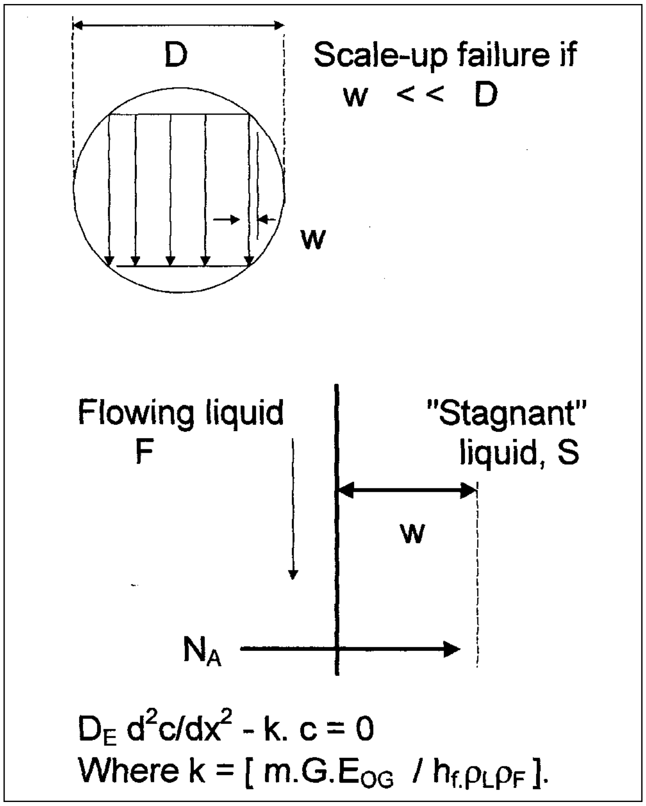

Figure 8. Mixing and mass transfer on a distillation tray, analogous to diffusion and reaction, provides an explanation for scale-up failures.

c = concentration in the liquid of mvc component, D_E = froth mixing coefficient, E_{OG} = point efficiency, G = vapor mass velocity, h_f = froth height, m = slope of equilibrium line, x = distance into stagnant region ρ_L = liquid density, ρ_F = froth liquid fraction.

on the surfaces of the packing, and it might be expected that they would eventually fill all the spaces in the bed and block it up. Fortunately, the solids break away from the packing and fall through the bed. The reason for this is explained by the "active layer−starving layer" of Figure 7, that is, the diffusion with a biological reaction process limits activity to a limited distance and when the biomass film grows to more than this, those parts of the film beyond the active layer are starved, die, and break away. The theory not only explained the basis for the first-order and zero-order (in pollutant concentration) rate equations which were then used to design the packed beds, but also predicted the half-order rate situation. This had been found for a few cases, for example, by La Motta (1976). This is all described in the textbook by Winkler (1981).

The last example, that of predicting scale-up failures on sieve plates, provides an excellent example of the superiority of a simplifying conceptual analysis over that of a numerical solution of the more exact equations. The initial objective of the work was to explain unexpected liquid concentration profiles, which had been observed on a distillation plate of 1.2 m diameter. Lines of constant concentration were U-shaped. A part of the work produced numerical solutions. On their own, these numerical calculations would have predicted the U-shaped concentration profiles on the experimental plate but they would not have predicted scale-up problems when the size of a "stagnant" area is bigger that the "width of a mixing zone". (It was subsequently shown that scale-up problems had occurred and had been corrected by design changes which may have been suggested by the theory.)

Need for New Ideas

In at least three important fields we have an insufficient understanding of a very great deal of knowledge accumulated from past experience. Most new designs are based on what has gone before. We advance by trial and error rather than by radical new changes predicted by some new conceptual analysis. See, for example, Figure 9. [This is discussed further in Porter and Jenkins (1979) and Porter (1995).]

The challenge is to provide answers to difficult questions that have remained unanswered for decades. New intellectual tools are needed based on:

(1) A theory of two-phase flow which is able to predict froth height from first principles. This will probably include a circulation in the vertical plane which changes position in a chaotic way.

(2) A theory of liquids able to predict vapor-liquid and liquid-liquid equilibrium *ab initio*, which takes into account polarity, hydrogen bonding, and allows for a restricted rotation of large molecules. As an introduction see Homer et al. (1992).

(3) A theory of separation processes which not only identifies the best combination of separation methods but explains why it is the best.

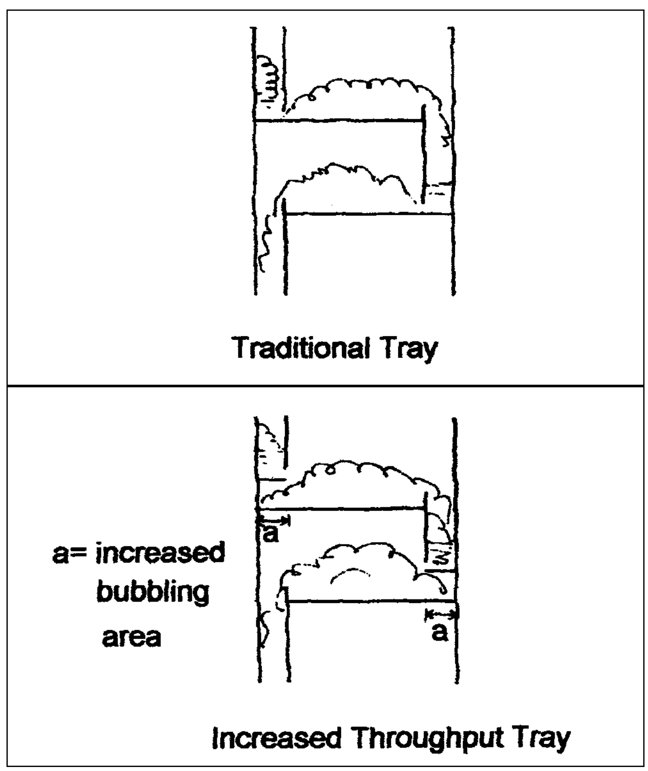

Figure 9. Typical example of changing tray design to increase throughput.

Encouraging Creative People

I have tried in this article to illustrate the power of ideas and concepts as intellectual tools for creating new technology. In all of the examples described above, the creative step was the realization that a particular concept (pinch point or diffusion-reaction confined to a limited distance) could be usefully applied to a new situation. Other examples might have been chosen where the concept used is unique to one application. Indeed, a comprehensive review of useful concepts in chemical engineering might form the subject of a new work. If the value of ideas is accepted, we might reasonably ask if anything can be done to cause more ideas to be produced sooner rather than later. Where should ideas be produced? Although two of the ideas I have used to illustrate this discussion (packed column scale-up and pinch technology) came to people when they were working in industry in Sulzer and in ICI, it is not the main objective of industrial research to identify ideas, but rather to make a contribution of commercial benefit to the company which supports the research. Ideas for general use are essentially a byproduct of industrial research. Intellectual tool making (often supported by public funds) is the unique function of a university where commercial research results, if they occur at all, may be the byproduct. Can anything be done to help universities to encourage the development of new ideas? The great Dr. A. J. V. Underwood once remarked, ''that every idea starts in one head.'' If this is so, we should seek to identify those individuals who have the capacity for creating useful ideas. Some years ago, I discovered a technique for doing this based on the citation indices. This is illustrated in Figure 10 which shows a group of full professors placed in rank order of citations to their work in reputable journals. Perhaps, the most important discovery is that some few people are cited very much more often that others. Some are rarely cited at all. This large difference in impact is probably sufficient to disarm or neutralize any criticism of the accuracy of the technique. It illustrates the reasonable hypothesis that creative ability in chemical engineering is as rare as it is in other creative activities such as writing, painting, or music. Another property of Figure 10 is that it provides a way of ''calibrating'' a particular subject in a particular country. Thus, an ambitious young academic wishing to find out if he has what it takes, after say five years of choosing his own research topics, may evaluate himself against the correlation of professorial impact of Figure 10. He may then decide whether to go on doing what he is doing or to seek success in some other field in this exciting world. (His situation is similar to that of someone anxious to become a concert pianist who may evaluate his likelihood of success by how many people turn up to his concerts.) His research must of course be supported during this period of evaluation.

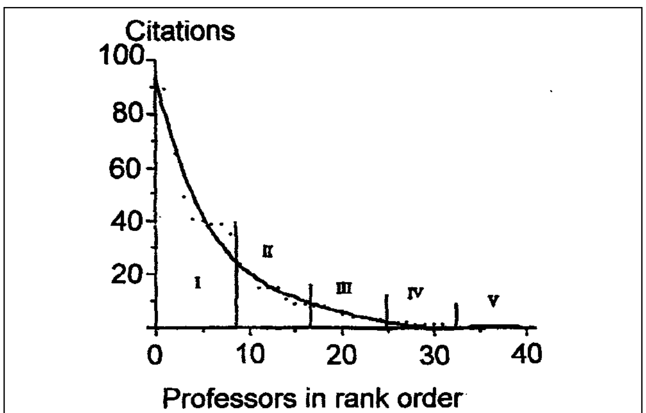

Figure 10. Comparison of professors of chemical engineering in the U. K. by citations to their work in reputable journals (*AIChE J., Chem. Eng. Sci., Ind. Eng. Chem., Trans IChemE, Chem. Eng. J.*), for the period 1983 to 1986.

A university should strive to achieve a situation where at least one intellectual toolmaker is teaching in each department. These people may credibly give one or two lectures in which they present ideas which they themselves have produced. This is important so that students may understand that new ideas are made in human heads by human people and not discovered carved on tablets of stone or published in books. The more ambitious student may reach the healthy conclusion that if an old fool can have new ideas, then so can I, and then be prepared to spend a few years finding out if this is so.

Those who have the responsibility for the wise distribution of money to support research should judge their own performance by the degree to which the amounts distributed matched the measure of ability shown in Figure 10. Figure 10 was correlated as an intellectual tool to help develop intellectual toolmakers.

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