Organic Process Research & Development

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Highlights from the Literature

Some Items of Interest to Process R&D Chemists and Engineers, Selected by the Editor

It is always interesting to see how different companies cope with environmental demands. In a recent article in *Chem. Technol. Eur.* (**1996**, May/June, 19−25) Dr. Claus Christ from Hoechst AG (Germany) shows how his company has reduced environmental impact by redesigning processes, rather than looking for end-of-pipe solutions. He describes several processes where an integrated environmental approach has led to economic advantages as well as reduced pollution. These include the following:

(a) Changing from iron reduction to catalytic hydrogenation in the process for production of aromatic amines from nitro compounds. As a result, process waste was reduced from 237 to 1.5 kg per 100 kg of product, with discharge to the atmosphere reduced from 0.176 to 0.000 05 kg.

(b) Changing from a chemical process to an enzymatic process for the production of 7-aminocephalosporanic acid (7-ACA), which resulted in the following changes in the amounts of waste (per tonne of 7-ACA): waste requiring incineration was reduced from 31 to 0.3 t; waste gas emissions were reduced from 7.5 to 1.0 kg; residual zinc, requiring recovery as $Zn(NH_4)PO_4$, was eliminated; waste water COD, however, increased by a factor of 17. As a result, the environmental costs associated with 7-ACA production were reduced by 90%.

(c) Carrying out reactions without solvents. By designing a special double-helix agitator for a vessel, a pharmaceutical intermediate could be produced in a solvent-free process, eliminating the use of methylene chloride.

In a process for production of 2-acetamidonaphthalene-8-sulphonic acid, a flat-bottomed vessel with special agitator was used to carry out a dry acetylation process (previously carried out under Schotten−Baumann aqueous conditions). This new process eliminated waste water production and allowed recycling of acetic acid byproduct.

In contrast, chromium wastes produced during oxidations are separated and recycled by electrolysis.

At Hoechst, new processes or improvements in existing processes have prevented the formation of 300 000 t of residues, and a further 2.37×10^6 were recycled.

Of course, not all environmental issues relate to kilograms or tonnes of pollutants: a nuisance to local inhabitants can result from odours in very small quantities. A recent article (Siegell, J. H. *Chem. Eng. Prog.* **1996**, January, 35−41) indicates how to “Solve Plant Odour Problems”. A useful list of odour thresholds and exposure limits for common odorous compounds is included, as is a table of suggested sources of odour reduction.

One way to reduce odour is by activated carbon absorption, but regeneration of carbon may be a problem. C. C. Leng and N. G. Pinto (*Ind. Eng. Chem.* **1996**, *35*, 2024−2031) discuss chemical regeneration techniques as an alternative to thermal regeneration.

Having mentioned electrochemistry above, I am reminded that at least two articles in the past couple of years have been devoted to industrial electrochemical synthesis. In the first, Frank Walsh (University of Portsmouth, UK) and David Robinson (ICI, Runcorn, UK) (*Chem. Technol. Eur.* **1995**, May, 16−23) outline the principles of cell design and discuss scale-up issues in relation to commercially available reactors. Specific reactions are discussed, particularly the electrosynthesis of N_2O_5: this is a nitrating reagent which exists primarily as the nitronium ion, ensuring rapid nitrations at low temperature (in contrast to nitric sulphuric acid mixtures, which may need temperatures up to 120 °C). Pilot plant processes are now used in the UK for the synthesis of “energetic” compounds such as nitratomethyloxetane and glycidyl nitrate (See: *Nitrations: Recent Laboratory and Industrial Developments*; Albright, L. F., *et al*., Eds.; ACS Symposium Series 623; American Chemical Society: Washington, DC, 1996).

Electrochemical processes are now being used for pollution control; generation of Ag(II) by anodic oxidation of Ag(I) solutions allows oxidative destruction of organics in waste streams. The process is particularly useful for cyanides, phenolics, chlorinated phenols, organosulphur and organophosphorus and hydrocarbon residues in aqueous systems. Electrochemical processes have also been developed for the conversion of aqueous sodium sulphate wastes to more valuable sulphuric acid and caustic soda.

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Electrosynthesis is also touted as being useful in the synthesis of pharmaceuticals in a short review (Genders, J. D.; Pletcher, D. *Chem. Ind.* (*London*) **1996**, 682). The authors emphasise that electrolyses scale with the electrode area, not reactor volume, and that conversion is limited by the rate at which reactant reaches the electrode. So scale-up requires an understanding of cell design, electrode materials and membranes. Since there are reliable flow cells around and lots of experience to guide the newcomer, there is no reason why rapid scale-up cannot be achieved. Scale-up of electrochemical processes is dealt with in detail in an earlier book by the same authors.

The scale-up of processes involving gas/liquid and gas/liquid/solid reactions seems a perennial problem in stirred tank reactors. The Rushton turbine agitator has been the system of choice for many companies, but it still has its weaknesses. A recent review (*Trans. I. Chem. E.* **1996**, *74A*, 417) from an expert in this field, Professor A. W. Nienow (University of Birmingham, UK), compares more modern commercial developments, which eliminate some, but not all, of these weaknesses. The advantages and disadvantages of a variety of impellers are discussed. A new impeller, designed by a joint project team working at the University of Birmingham with APV (UK) Plc under one of the UK's LINK schemes (designed to encourage university–industry co-operation), promises to eliminate the disadvantages of earlier designs, and a patent is pending.

In the same journal (*Trans. I. Chem. E.* **1996**, *74A*, 595) Dr. S. S. Kattu of Dow Chemicals (Midland, USA) gives an industrial perspective on gas/liquid/solid systems, concentrating on a detailed understanding of mass transfer in these chemical processes to improve selectivity and reduce byproduct formation.

Finally, in an article (*Chem. Eng. Prog.* **1996**, February, 110) intriguingly entitled "Improve Your Chances for Successful Process Development", the author, D. Mukesh, Alchemie Research Centre (ICI India), indicates 15 points which, I guess, most chemists and engineers would have already discovered. For example, he emphasises the need for a product champion, for a multidisciplinary team, knowing the limitations of the pilot/manufacturing plant and designing the process to fit it, and suggests that short cuts in data collection only lead to scale-up problems. Perhaps the most important piece of advice is—remember Murphy's law—if something can go wrong, it will. That's what makes process R&D so interesting and challenging.

Trevor Laird
Editor

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