Multi Phase Reactions in Flow

Multi-phase reactions can be anything where immiscible reagents are combined.

There are numerous liquid / gas scenarios, some including dedicated reactor designs that have been developed with a great deal of research over recent years (much work is still continuing in this field), and some simply combining a liquid and gaseous flow in an otherwise fairly conventional flow reactor.

And there are simpler liquid/liquid scenarios, generally involving an aqueous and an organic phase. These biphasic liquid reactions are the subject of this article.

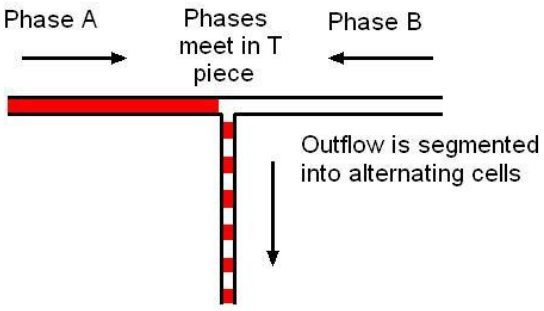
Biphasic reactions in Batch

The attraction of a biphasic reaction is that it may be possible to carry out a process which results in a two phase end result where one phase contains almost pure end product and the other phase contains waste products and/or unused reagents. Final separation is trivial and relatively expensive solvent removal is avoided. A final aqueous workup would come under this heading.

Typically in batch this might be achieved by energetically agitating two immiscible phases to create an emulsion in which the interphase boundaries act as the reaction sites. The extent of the agitation is then in effect a reaction variable.

Biphasic reactions in Flow

In flow, however, things are quite a bit simpler. When two immiscible fluids are brought together in a simple T mixer, in broadly similar proportions, the outcome tends to be what is known as "Taylor Flow" (see right).

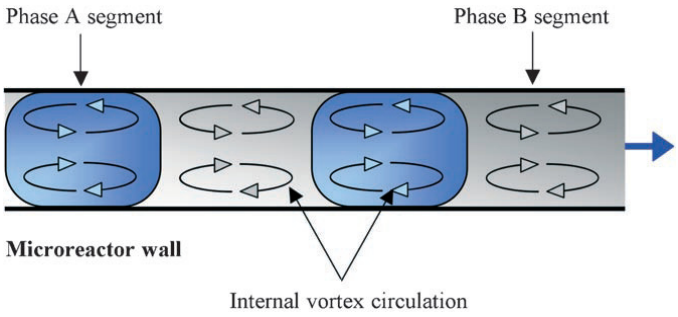


Taylor flow after a T mixer

The sizes of the alternating cells depends on viscosity, surface tension and mixer geometry but tends to be very stable with flow rate and temperature over the typical working range.

These cells then persist all through the tube reactor and only recombine when the flow is collected in a vessel with a free surface at the end of the process.

The actual cells have been shown to "roll" along the tube, mixing at the interface between the alternating cells. The mixing is very thorough, and reactions take place rapidly.



Minimal Dispersion

Dispersion is generally seen in a flow system at the leading and trailing ends of a plug as it moves through the system, whereby mixing occurs between the plug and whatever comes before or after it. The upshot of this is that the leading and trailing edges of a product stream emerging from a flow system need to be discarded as they do not represent a steady state. The Vapourtec Flow Commander™ software has an algorithm to model this dispersion and automatically predict when the steady state output will be achieved.

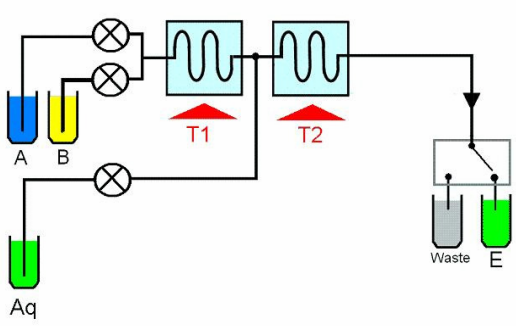
With Taylor flow as illustrated above, however, the dispersion is almost non existent so there is more efficient use of reagents (especially when very small reaction volumes are used.

Examples

For more details of a biphasic reaction in action, look at www.vapourtec.co.uk/applications

For a recent publication showing a segmented flow Heck reaction, look at www.tinyurl.com/segmentedheck

Another example of biphasic flow is the use of a 3rd pump in the Vapourtec system to achieve aqueous workup after the main reaction. (see illustration, right)



The thorough mixing referred to above along with the controllable time and temperature ensures that the workup is both effective and totally reproducible.

Things to watch out for.

Usually the flow system user will run solvent through the system while the system achieves the chosen temperature conditions, then switch to actual reagents for the reaction proper. With fully miscible reaction components, this solvent would often be the same for both reagents channels.

However, in a biphasic flow experiment, it may be necessary to use different solvents for the different phases, for the following reason When the flow into the pump switches from the reagent to the solvent flow or viceversa, the re-wetting of the pump may not be complete if the reagent and solvent are not fully miscible and compatible.

(Of course, should a pump fail to perform correctly because of this, the Vapourtec pump monitoring system will immediately alert the user, but this would still represent lost time and is therefore best to avoid).