Seeing Is Believing - Learning Fundamentals of Chemical Engineering

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Chemical Engineering has become a very pivotal discipline which enjoys commonality with all cutting edge sciences. Chemical engineers are the most versatile species who are adept at converting themselves into chemists, physicists, material scientists, biologists, biotechnologists, mathematicians, environmentalists and all sorts of engineers. They can certainly disguise themselves as one such species and still retain their identity. This has been possible due to the sound training chemical engineers receive in basic sciences, mathematics and systems approach. The importance of experiments in any scientific field can be hardly overemphasized and more the number of experiments invoking different basic principles, the better it is. Apart from the regular experiments, it is important to introduce many concepts in chemical engineering through visualization.

In 1978, M.M. Sharma, the legendary Professor and Head of Chemical Engineering at this Institute, returned from his trip to the Cambridge University UK and discussed with me a simple demonstration experiment performed by J.F. Davidson on the similarity between bubble flow in liquids and fine solids. Davidson apparently held two glass tubes in his hand side by side, each filled with water and fine particles to the same level with a little air on the top, closing the tops with his palm. He just inverted the tubes and asked Sharma to observe the bubble flow. What Sharma could not believe was that the bubble travel in gas-solid and gas-liquid systems was strikingly similar. He asked me make a simple set up to show this experiments to the UG students. As a young Associate Lecturer, a sort of demonstrator and also a Ph. D. student, I was heavily involved in Chemical Engineering Laboratory and enjoyed setting up experiments for the UG students. I enjoyed doing experiments and did all sorts of experiments before doing theory.

Immediately I made the first set-up with two glass tubes of 1 cm diameter and 1 m in height, with silica powder (~300 µm) and water-glycerol as the ‘fluids’ leaving 2 cm air space at the top. I fixed these tubes side-by-side on a wooden plank (10 cm diam x 1.20 m length) made a rotary device with a handle at the back and put it on a frame (Fig. 1). With a few trials, I could replicate Davidson’s experiment. Only after the experiment was done, I looked at Davidson’s work and tried to understand the theory! A bed of solid particles fluidized by gas behaves like a fluid. There is a minimum gas flow for the fluidization at which the pressure drop is just enough to support the weight of the bed. When the gas flows at a velocity greater than that required for minimum fluidization, each particle is suspended and the whole bed becomes mobile and resembles a liquid. The bubble does not break and maintains its fidelity and much of the gas travels as a bubble.

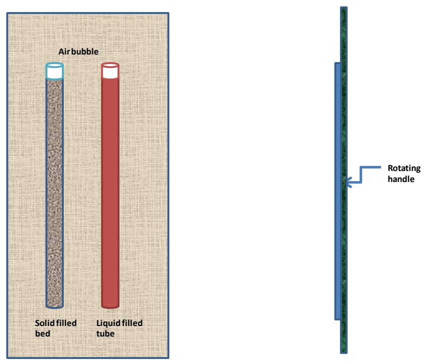

Fig. 1 : Similarity of bubble flow in gas-solid and gas-liquid systems

Professor Sharma was too excited to settle at one experiment and made a continuous effort to generate ideas as well as gave us the freedom to add experiments of such kind. Sharma wanted me and some of my other junior colleagues teaching Chemical Engineering Lab to set up a Demonstration Laboratory where all fundamentals of Chemical Engineering could be taught through simple experiments to reinforce the concepts. Being a celebrity and active consultant, Sharma thought this unique laboratory should be developed and made a regular 'habit' for the disbelieving students. He was very enthusiastic about Marangoni effect, three phase fluidization, mixing, etc. Ours was a very poorly funded lab which did not have the material and money to buy costly equipment/apparatus and we had to live on ideas and some easy to fabricate set-ups. He used to throw challenges at us and asked all research students to come with any demonstration related to their work which could become part of this lab. We have developed a special lab called 'Demonstration Experiments' which was introduced. Several concepts when visualized become very clear to the doubting students and they appreciate them greatly.

How to bring Marangoni effect to the lab and in a country, where even looking at an effervescent beer is a taboo, was a big problem ! (at least in 1970s!!). Marangoni's famous story is related to mass transfer when a bottle of champagne is opened up and the interfacial turbulence. A surface tension lowering solute on transfer from one phase to another causes imbalance in the interfacial turbulence. So we chose acetone as the surface tension lowering solute being transferred from toluene to water. A 4% v/v solution of acetone in toluene makes the continuous phase whereas water droplets are added from a burette. When the drop moves in the organic phase, it disturbs it resulting in non-uniform eddies. This brings an acetone rich element to the interface, which lowers the interfacial tension and changes the pressure of the drop and the drop 'kicks'. In the case of gas-liquid this can be demonstrated by putting a few drops of diethyl ether over water in a watch glass and sprinkling talcum powder.

Thus the next experiment on our radar was the internal circulation in droplets, which was done with carbon tetrachloride and aluminum particles to demonstrate how fluid droplets behave in different manner vis-à-vis solid particles while travelling in a liquid medium. There is onset of internal circulation because of the mobile interface. This simple experiment was done in a glass cylinder containing water and releasing organic phase droplet containing fine aluminum powder, with a light source at the background. The internal circulation leads to higher mass transfer rate in liquid-liquid systems. Now this experiment has graduated to an elaborate set-up.

Another concept was based on the direction mass transfer, coalescence and identification of continuous and dispersed phases. It is commonly believed that the phase with lesser volume is a dispersed phase. However, with a phase volume upto 70%, it can still be a dispersed phase. So a simple demonstration is to take two immiscible liquids, one of them with a dye having no distribution in the other phase and agitate it. As the agitation is stopped, the interface is distinguished after some time. The dispersed phase droplets, still coalescing, crowding and merging with its bulk phase will be seen next to the interface in that phase. The other side of the interface is free of any droplets. No great funds are required to show this behavior!! The volumes can be varied to see which would be the dispersed phase in 30: 70, 50:50 and 70:70 phase ratios. The idea can be extended to make one of the phases as the continuous or dispersed phase by locating the impeller above or below the interface. If the heavier phase is desired to be the continuous phase, then the impeller is placed in it and the lighter phase becomes dispersed. On the contrary if the impeller is placed in the lighter phase, the heavier phase is dispersed in it. However, this may be an unnatural position for the impeller. Hence if the lighter phase is required to be continuous, then placing the impeller near the bottom of the vessel, adding the lighter phase firstly, agitating it and then adding the heavier phase leads to the desired effect.

A related experiment in this regard is the phase inversion in liquid-liquid contactors, such as mechanically agitated contactors and rotating disc contactors, which is governed by several parameters such as interfacial tension, phase ratio, density, viscosity, etc. Phase inversion occurs when the rate of coalescence exceeds that of redispersion. It is difficult to disperse a high surface tension liquid at high hold up in a low surface tension liquid. An organic phase such as toluene and colored aqueous can be selected to observe phase inversion. Dispersed phase flow rate is increased till the creation of a dense layer of droplets at the bottom of the column. This inversion normally occurs at the bottom compartment and passes in upward direction from compartment to compartment.

I had initiated research on phase transfer catalysis (PTC) in mid-1970s for my doctoral thesis and this new technique was hardly known to chemical engineers. Liquid-liquid reaction between reagents in two different phases is extremely sluggish and takes days to complete. The enhancement in rates of reaction is seen due to transfer of the nucleophile from aqueous phase to organic phase, where the substrate resides, by using a phase transfer agent (Fig. 2).

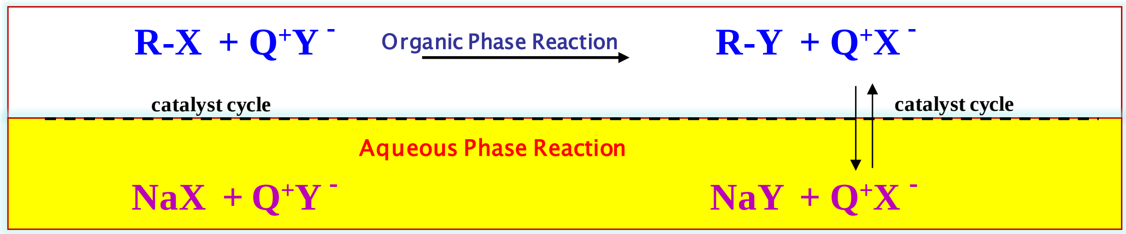

Fig. 2 : Liquid-liquid phase transfer catalysis

To demonstrate PTC, a simple experiment would be the esterification of nitrophenol with acetic anhydride in a stirred vessel. The sodium salt of nitrohenol is taken in the aqueous phase and has a yellow color. Acetic anhydride is taken in mixed xylene as a solvent and is colorless. Even after intense mixing, no reaction takes place and two layers are separated. Addition of a small quantity of tetrabutylammonium bromide as a catalyst and agitation shows that the reaction has taken place in the organic phase and there is a discoloration of the aqueous phase and coloration of the organic phase due to transfer of quaternary nitrophenoxide to the organic phase. This experiment can be done in a beaker without any agitation while observing gradual discoloration and coloration of the phase or the rates could be enhanced by agitation.

Weissenberg effect is well known to polymer chemists and engineers. The role of mixer is quite adequately demonstrated in non-Newtonian fluids where the ordinary M.A.C. will not be a good idea; for instance, viscoelastic materials which possess properties of viscous materials as well as elastic solids. Materials possessing elasticity exhibit normal stress difference. In a rotational flow, the fluid elements experience centrifugal force and elastic force. At lower rotational speeds where the elastic force exceeds the central force, the material moves towards the centre and to keep continuity climbs on the shaft. A 4% w/w aqueous polyvinyl alcohol with 3% v/v glycerol can be used as a viscoelastic material to demonstrate this ‘climbing’, known as Weissenberg effect. This demonstration can be made dramatic by taking two identical beakers without any baffles, one containing water and another the viscolelastic liquid and asking the students to predict the outcome of the agitation. One can literally trick the students and then explain the phenomenon.

Three phase catalytic reactors are so common in industry and the reactor performance is marred by poor mixing. A complete suspension of solids is very important and the operation has to be beyond the critical speed. Contrary to the common belief, when gas is introduced in an agitated solid liquid system, the particles will settle down if the speed is in the critical region. This is detrimental to many catalytic reactions such as dead end hydrogenations. This experiment is done with a fine sand particles in water in a stirred tank and the speed is increased gradually to keep the solids suspended. Then air is introduced into the tank to find that the particles settle down. The speed has to be increased to a higher value to make suspend the particles.

Table 1: Demonstration Experiments

1 Laminar flow in pipes
2 Internal circulation
3 Direction of mass transfer & coalescence
4 Interfacial turbulence
5 Agitation in liquid-liquid systems: Identification of continuous and dispersed phases
6 Phase Inversion in agitated contactors
7 Wetting characteristics
8 Induced coalescence
9 Liquid maldistribution in packed towers
10 Wire mesh type of entrainment separator
11 Gas-inducing type of impeller
12 Co-current gas-liquid down flow in packed columns
13 Flooding in gas-liquid and liq-liq contactors
14 Bubble rise in gas-liquid and gas-solid system: A similarity
15 Three phase fluidization: Contraction of liquid fluidized bed on introduction of gas.
16 Three-phase reactor
17 Weissenberg effect
18 Cyclone separator
19 Instantaneous gas-liquid reaction: Existence and movement of reaction plane
20 Selectivity in two-phase liquid-liquid systems
21 Phase transfer catalysis
22 Interfacial polycondensation
23 Foam Fractionation
24 Hydrotropy
25 Small scale experiment with surfactants
26 Detecting reverse micelles in non-polar media
27 Microemulsions
28 Incompatibility of polymers (Aqueous two phase extraction)
29 Segregation due to flow (coning)
30 Relaminarization
31 Enhanced sedimentation in settling tanks (Boycott effect)
32 Air lift reactor
33 Experiment with granular particles
34 Circulation cells in bubble column
35 Origin of viscoelasticity in fluids
36 Oscillating reactions
37 Langmuir monolayer of surfactant
38 Ultrafiltration
39 Laser Doppler Anemometry
40 Cavitation
41 Hydrogenation reactions in high pressure vessel
42 Jet mixer

A large number of these demonstration are a result of our own research in various areas and some published work of other as listed in the references. The following is a partial list of such experiments which we have developed over last 30 years is given in Table 1 .

A totally renovated lab is now set up to house all these experiments, video demonstrations, posters and portraits of great scientists and engineers to inspire the young minds. With advent of new equipment and tools, we have been striving hard to include nanotechnology, green chemistry and engineering and biotechnology experiments. Several international visitors have appreciated our efforts and now we are according the University Grants Commission’s Networking Centre in Chemical Engineering to educate teachers, students and practitioners from industry on a sustainable basis. When M.M. Sharma emphasized the need of such demonstration lab during 1970s, hardly anybody would have realized its importance in chemical engineering curricula.

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