Date: Aug 13, 2007

To: **DIERS UG Emulsion Round-Robin Working Group**

From: Joseph Leung, DIERS UG Design/Testing Committee Chair

## Subject: Proposed Emulsion System for Round-Robin Testing

Hi all –

It has been a long hot summer. Harold reminded me again that with the meeting coming up in just two months, we need to get the ball rolling again on the Round-Robin testing. So here is a suggestion for your consideration.

I am proposing an emulsion recipe using Vinyl Acetate (VAc) as the monomer. This system was actually used extensively in a PhD thesis ( Dr. M.F. Kemmere, ChE dept, Eindhoven Univ, Netherland, YR1999 ) which was supplied to me by Harold Fisher.

I am only going to attached the subsequently published paper in the J App Polymer Sci in this email. If you like the PhD thesis also, just let me know and I can forward that separately. This is a batch recipe which uses the following common emulsifier, initiator and pH buffer :

1. Sodium lauryl persulfate (MW=288.4) 0.010 kmol/m3 water basis (also known as sodium dodecyl persulfate)

2. Sodium persulfate (MW=238.1) 0.010 kmol/m3 water basis

3. Sodium carbonate (MW=106.0) 0.009 kmol/m3 water basis

All these ingredients are easily available from Sigma-Aldrich. Note that Item #1 is the emulsifier, Item #2 is the initiator, and Item #3 is the pH buffer. Their molar concentrations are nearly the same (0.01 kmol/m3 water basis).

The attached paper presented reaction rates at 50C (in RC1 under isothermal condition) and at various agitation speeds. It also presented visual experiments in determining when emulsion was obtained. I would suggest that we do the same visual experiment first – just use the water and emulsifier in an open glass test cell with similar baffle and stirrer, add the VAc on top, increase the stirring speed until good emulsion is observed. We might also want to see how stable the emulsion is by turning off the stirrer and water for any phase separation. During the adiabatic experiment, the stirring speed has to exceed the minimum stirring speed for “good emulsion”. According to this paper, VAc being much more soluble in water (2.5%wt) than styrene, an emulsion was formed at a lower rpm than with styrene.

Proposed recipe is as follows:

| | |
|---|---|
| DI water | 100 units (mass) |
| VAc | 66.7 units |
| Emulsifier | 0.288 units |
| Initiator | 0.238 units |
| Buffer | 0.095 units |

Above emulsifier, initiator and buffer concentrations should correspond to 0.01kmol/m3, 0.01kmol/m3 and 0.009 kmol/m3, respectively.

This should produce a 40% wt solid for 100% conversion. The expected adiabatic temperature rise is about 130 C.
Starting temperature is 50C, yielding a peak temperature of 180C and about 200 psi (based on VAc vapor pressure).

Note that I have increased the VAc content from 25% wt in the attached paper to 40% wt in this current recipe in order to get a higher heat kick. Oxygen does affect the reaction, so it is important to purge the oxygen and perhaps replace with nitrogen as a pad.

### RC-1 users

Conduct the isothermal experiment at 50C at a good stirring speed. Use the data in the paper for guidance, it might also depend on the type of agitators used. Purpose is to see how well RC-1 users can reproduce the heat generation curve. Run time is about a day.
Determine particle size and particle number if capability exists in your lab.

### VSP / APTAC / ARSST / ARC users

Conduct the adiabatic experiment starting at 50C with a pre-determined good stirring speed. Note that some stirrer type may provide better stirring than others. Visual test would indicate the minimum stirring rpm for a particular stirrer. Also advisable to have baffles in the sample cell.

Data submission –

Please plan ahead and do these trial runs, submit your data before the DIERS meeting in Manchester so that we can evaluate this system and make a decision on whether to invite everyone to participate. Also any suggestions you have or will discover in the course of the testing would be most welcome.

Thanks in advance for your participation!