DIERS Users Group Round-Robin Testing - Vinyl Acetate Emulsion System

As part of the ongoing DIERS UG Design/Testing Committee activity, the next round-robin testing system involves a vinyl acetate emulsion. An earlier effort by our working group (and THANKS TO ALL THEIR HARD WORK) has demonstrated success in achieving the emulsion and yielding good agreement among different users.

This recipe uses the following common emulsifier, initiator and pH buffer:

(1) Sodium lauryl sulfate (MW = 288.4, CAS#151-21-3) 0.010 kmol/m^3 water basis (also known as sodium dodecyl sulfate) [Emulsifier].

(2) Sodium persulfate (MW = 238.1) 0.010 kmol/m^3 water basis [Initiator].

(3) Sodium carbonate (MW = 106.0) 0.009 kmol/m^3 water basis [Buffer].

All these ingredients can be obtained from Sigma-Aldrich. Their molar concentrations are nearly the same (0.01 kmol/m^3 water basis).

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 0.01 kmol/m^3, 0.01 kmol/m^3 and 0.009 kmol/m^3, respectively. This should produce a 40% wt solid for 100% conversion – you are urged to determine the conversion by solid content after the test. The

expected adiabatic temperature rise is about 100ºC. With a starting temperature of 50ºC, that should yielding a peak temperature of about 150ºC (and a peak pressure < 200 psi based on VAc vapor pressure).

APTAC/ARSST/ARC/DEWAR/VSP Users

Conduct the adiabatic experiment starting at 50ºC with a pre-determined good stirring speed. For ARC users, stirring is necessary. 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. Conduct the test at a stirring speed above the minimum needed to achieve good emulsion. After the test, determine the solid content and hence conversion by evaporating off the water and any unreacted VAc in the sample.

Data Submission

Send via E-mail to Joseph Leung at leunginc@cox.net and copy Amy Theis at theis@fauske.com

NO LATER THAN OCT 1, 2008

Submitted Data Should Include

- Summary sheet filled in (see attached sheet).
- Tabulated data with columns (label and units clearly identified).
- Any relevant plots.

Note that your data will be archived for future use by the DIERS UG; thus all necessary data are urged to be submitted.

Background Information

This system was actually used extensively in a doctoral thesis (Dr. M. F. Kemmere, ChE Dept., Eindhoven Univ., Netherland, YR1999) and a summary paper was subsequently published in the Journal of Applied Polymer Science in YR2001 (paper attached as a PDF file).

Kemmere paper presented reaction rates at 50ºC (in RC1 under isothermal condition) and at various agitation speed. It also presented visual experiments in determining when an 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, emulsion was formed at lower rpm than with styrene.

Note that I have increased the VAc content from 25% wt in the Kemmere's work 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.

Round-Robin Test Data Sheet (to be submitted with each test)

Contact Person:_____ Tel. No.:_____

Company:_____

E-mail:_____

Test ID:_____

Equipment:_____

Test Cell Description (including internals and stirrer):_____

_____

_____

Sample Weight: _____(gram)

	Actual	Target
DI Water	_____	100 units (mass)
VAc	_____	66.7 units
Emulsifier	_____	0.288 units
Initiator	_____	0.238 units
Buffer	_____	0.095 units

$$\text{Phi} - \text{factor} = 1 + \frac{(mC_p)_{\text{test cell}}}{(mC_p)_{\text{sample}}} =$$

(show calculation, use C_p = 0.8 cal/gºC for emulsion sample)

Agitation comments - (stirrer speed during test, indicate minimum stirring speed for achieving emulsion in visual experiment, and note duration for phase separation after stirring is turned off)_____

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Other Remarks - _____

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