UNION CARBIDE INTERNAL CORRESPONDENCE

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To (Name) Dr. D. R. Montgomery
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Location Texas City

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Dr. K. L. Hoy, 511
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Dr. C. E. Moyer, 515
✓Mr. R. N. Wheeler, 514

Subject Removal of Residual VCl via Redox Initiator Systems

Dear Don:

In a recent conversation with Dr. Moyer, I reviewed some of our current efforts in the removal of residual VCl from our latices via redox polymerization.

With latices containing both vinyl chloride and vinyl acetate, we have no difficulty in going below 10 ppm on the resin basis. With VCl/VCl_2 latices in which the residual vinylidine chloride is quickly used, we can readily get below 100 ppm and probably to the 10 ppm level. The technique is the use of a redox system over a period of time up to 8 hours.

Our redox system is comprised of t-butyl hydroperoxide and erythorbic acid activated with ppm quantities of EDTA-complexed iron. If your system is acidic, you should not need the EDTA. The recipe:

Oxidant Solution:

350 parts water, 14.4 parts TBHP (Trigonox A-W70)
1.5 parts of 0.844 $FeSO_4.7H_2O$/1.156 EDTA/200 H_2O at pH 9.1

Reductant Solution:

300 parts water, 21 parts erythorbic acid neutralized to operating pH.

Feed:

0.03 parts oxidant, 0.023 parts reductant per minute per phr.

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Several points come to mind:

1. Studies should be done either with or without blowdown, but with care to exclude oxygen. Contamination with air appears to interfere with the redox polymerization more than would be expected.

2. High-speed agitation should be maintained such that VCl will be removed from the vapor phase. Since the rapid removal of the VCl will also lead to a vacuum in perhaps a half-hour, some nitrogen should be added to hold the pressure above atmospheric, again to prevent the possibility of introducing air into the reactor.

3. We have successfully operated this redox system from 40 to 85°C, but these temperatures are above the glass transition temperature of our products. In your case you should test at conditions both above and below the transition temperature because of differences in diffusion of VCl from the particle.

Since your particles are much larger than ours, the success of the redox system for you is in question both in the removal of VCl and possible damage to resin porosity. Nevertheless, the advantages of completely avoiding your vacuum stripping system via this technique could be highly significant.

Sincerely yours,

R. J. Hanna

RJH/d