

B.F.GOODRICH CHEMICAL COMPANY  
DEVELOPMENT CENTER

Project 5038-3208  
TOP #38

REMOVAL OF VOLATILE MONOMERS FROM  
GEON 450X3 LATEX IN THE  
RODNEY HUNT TURBA-FILM EVAPORATOR

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### MAJOR OBJECTIVE

Residual volatile monomers present in Geon latex products after polymerization represent a potential safety problem. Our present method for reducing the volatile monomer level in latices in the Production plants consists of blowing the hot latex from the polymerizer into an evacuated stripping vessel. The method is neither economical or adequate. Problems associated with latex foaminess, vacuum control, and high production schedules have resulted in a situation where Production stores and ships latex products containing 0.2 to 0.5 percent residual volatile monomer. This is well above what is believed to be a safe limit.

In an effort to reduce or completely eliminate the possibility of these monomers, vinyl chloride and vinylidene chloride, forming a flammable or explosive mixture in the vapor space above the latex, a number of investigations have been started to evaluate improved stripping methods.

### LIMITED OBJECTIVE

Evaluate the Rodney Hunt Turba-Film Evaporator as a method of removing vinyl chloride and vinylidene chloride from Geon 450X3, a soft, film-forming copolymer (46 vinyl chloride - 17 vinylidene chloride - 37 ethyl hexyl acrylate) latex.

### CONCLUSIONS

1. The level of volatile monomers in Geon 450X3 latex can be successfully reduced by the Rodney Hunt Turba-Film Evaporator without adversely affecting latex properties.
2. A variety of conditions are suitable for stripping Geon 450X3 in the Development Center Rodney Hunt (Model #1, with 4 sq.ft. of heat transfer surface).
3. The best results, considering latex properties and monomer removal, were achieved in a single pass under the following conditions:

|                       |   |             |
|-----------------------|---|-------------|
| Feed Rate             | - | 4.0#/Minute |
| Vacuum                | - | 53 MM Hg    |
| Temperature on Jacket | - | 46°C.       |

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4. Other operating conditions produced the following changes in the volatile monomer level:
  - a) Increasing the feed temperature above 50°C improved stripping efficiency slightly. It produced two undesirable side effects - decreased latex pH and increased the outsize particle level.
  - b) Reducing the vacuum toward 100 MM Hg decreased stripping efficiency, but increasing it below 50 MM Hg had little influence.
  - c) Two passes were not necessary to obtain a satisfactory reduction in the residual monomer level.
5. Residual ethyl hexyl acrylate monomer could not be detected.
6. The length of the run was limited by the size of the product receiver rather than the operation of the Rodney Hunt or the stability of the latex.
7. The feed rate was limited by the physical size of the pilot plant Rodney Hunt and its auxiliary condenser and vacuum system. Nothing was observed that would indicate a satisfactory run could not be made in a larger unit properly designed.

#### FUTURE COURSE OF ACTION

Additional work to remove volatile monomers from Geon 450X3 in the Rodney Hunt is not planned by Development. We would strongly recommend, however, that Avon Lake General Chemical Plant arrange for an extended production run on one of Louisville's larger Rodney Hunt units. The initial trial should be made under the following conditions:

|                  |   |                                              |
|------------------|---|----------------------------------------------|
| Feed Temperature | - | 50°C.                                        |
| Vacuum           | - | 50 MM Hg                                     |
| Feed Rate        | - | Maximum possible under the conditions above. |

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## DISCUSSION

Studies at Louisville by G. D. Schaaf and L. D. Pringle have shown that the Rodney Hunt Turba-Film Evaporator can efficiently remove vinyl chloride monomer from a hard, resinous copolymer latex such as Geon 351. While hard, resinous latices represent the major portion of our latex production, a substantial quantity of softer, internally plasticized latices are also produced, principally at the Avon Lake General Chemical Plant.

Removal of monomers from softer, film forming Hycar nitrile latices in the Rodney Hunt is usually accompanied by a greater increase in the outsize particle level than that observed with the harder Geon resin latices. Consequently, information pertaining to the behavior of the softer Geon latices in the Rodney Hunt was of considerable importance.

Stripping studies were conducted in the Rodney Hunt unit located in Building 422 at the Development Center. This particular evaporator (Model #1) has four square feet of heat transfer surface. The unit and its auxiliary equipment was designed primarily to strip small batches of experimental latices made in the pilot plant. It does not lend itself well to continuous operation. This type of operation is possible, however, with close technical supervision. For our purposes, it was felt that the unit should be used to obtain preliminary operational information. Two objectives were established:

1. Determine if Geon 450X3 latex could be handled in a Rodney Hunt.
2. Select a favorable set of operating conditions as a starting point for larger scale operations at a later date in a production size unit. Consequently, a series of short runs was made. Feed rate, vacuum, and temperature were varied.

Two lots of fresh Geon 450X3 latex were supplied by Avon Lake General Chemical Plant - one from the blend tank (20035), the other from a reactor (Charge 72). A 55-gallon drum of latex was placed on a scale, tared and sampled. The Rodney Hunt and product receiver were evacuated and checked for leaks. The vacuum was adjusted to the desired pressure and the feed was sucked from the drum into the unit through a polyethylene hose.

Initially, the jacket of the Rodney Hunt was cold and the rotor was revolving. An initial set of readings was taken and after approximately 30 seconds, the jacket was adjusted to the desired temperature with hot water. A rotometer was used to control the flow rate.

Several preliminary trials were made to establish approximate operating conditions and familiarize ourselves with the equipment. None of this data is reported. It was immediately obvious that the feed rate was limited by two factors:

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1. The general foaminess of the latex.
2. An inadequate foam knock-down bottle between the Rodney Hunt and the vacuum jets.

Above 9 pounds per minute the foam would appear as a white liquid in the barometric condenser between the Rodney Hunt and the two stage non-condensing jets. Since these same jets are used in many other pilot plant operations, we were reluctant to risk fouling them. Installation of a larger, better designed foam trap would probably overcome most of our early operating problems and permit a higher feed rate, if desired.

After the preliminary trials were complete, two runs were made at 9 pounds of feed per minute. Foaming was a problem in both runs and all subsequent runs were made at a much lower rate. After 18 minutes operation (Runs 1 and 2), the rotor on the Rodney Hunt stalled. Examination of the Process log revealed that a Hycar nitrile latex had been stripped in the Rodney Hunt between the preliminary trials and Run 1. Some of this rubbery material remained firmly attached to the rotor blades despite flushing and stalled the unit. After a thorough cleaning, the unit performed satisfactorily for the remainder of the trial.

In most runs, the latex was collected in the product receiver until the trial was completed and then pumped out into 5 gallon pails. For a number of runs, however, the product was sampled and then passed through the Rodney Hunt for a second stripping operation.

The percentage of residual volatile monomers in the feed and product samples was measured at Avon Lake General Chemical Plant by the standard steam distillation method. Latex properties were measured at the Development Center. All data are summarized in the attached table.

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Properties of Geon 450X3 Latex Stripped in the Rodney Hunt Turba-Film Evaporator

| Run Number              | 450X3 Feed Latex | Rodney Hunt Conditions |            |             |                  | Total Solids % | Residual Monomer |     | pH  | Surf. Tens. Dynes/cm | Brook. Visc. cps. | Latex Spec. Grav. | Part. Size Beck. DU - A | Outsize Millipore Filter |
|-------------------------|------------------|------------------------|------------|-------------|------------------|----------------|------------------|-----|-----|----------------------|-------------------|-------------------|-------------------------|--------------------------|
|                         |                  | Jacket Temp. °C.       | Vac. mm Hg | Ave. #/min. | Rotor Amps       | Oper. Min.     | VCl              | RHA |     |                      |                   |                   |                         |                          |
| 4/27 Feed               | Lot 20035 Dr-59  | -                      | 24         | -           | 4.8              | -              | 0.16             | n11 | 7.7 | 41.6                 | 58                | 1.122             | 1401                    | 0.23                     |
| 4/27 Run 1              | Lot 20035 Dr-59  | 32                     | 44         | 9.0         | 6.8              | 9              | 0.00             | n11 | 7.6 | 40.0                 | 53                | 1.125             | 1408                    | 0.41                     |
| 4/27 Run 2 <sup>b</sup> | Lot 20035 Dr-59  | 34                     | 37         | 9.0         | 6.8 <sup>c</sup> | 9              | 0.17             | n11 | 8.1 | 42.3                 | 59                | 1.124             | 1404                    | 0.31                     |
| 4/30 Feed               | Lot 20035 Dr-58  | -                      | 100        | -           | 4.0              | -              | 0.06             | -   | 8.0 | 41.3                 | 43                | 1.121             | 1411                    | -                        |
| 4/30 Run 1              | Lot 20035 Dr-58  | 29                     | 102        | 4.0         | 4.1              | 25             | 0.00             | n11 | 8.0 | 40.1                 | 53                | 1.123             | 1440                    | 0.45                     |
| 4/30 Run 2              | Run 1 4/30       | 29                     | 53         | 4.3         | 4.2              | 20             | 0.04             | -   | 7.9 | 39.5                 | 50                | 1.123             | 1413                    | 0.38                     |
| 4/30 Run 3              | Lot 20035 Dr-58  | 29                     | 54         | 4.5         | 7.5              | 16             | 0.17             | n11 | 8.1 | 40.6                 | 62                | 1.124             | 1410                    | 0.38                     |
| 5/5 Feed                | Lot 20035 Dr-57  | -                      | 49         | -           | 4.1              | -              | 0.01             | -   | 8.1 | 39.4                 | 82                | 1.127             | 1444                    | 0.46                     |
| 5/5 Run 1               | Lot 20035 Dr-57  | 46                     | 53         | 4.0         | 4.2              | 28             | 0.00             | -   | 8.0 | 39.9                 | 73                | 1.128             | 1374                    | 0.82                     |
| 5/5 Run 2               | Run 1 5/5        | 46                     | 53         | 4.0         | 4.1              | 20             | 0.03             | n11 | 7.7 | 38.7                 | 84                | 1.126             | 1425                    | 0.60                     |
| 5/5 Run 3               | Lot 20035 Dr-57  | 58                     | 100        | 4.0         | 4.2              | 30             | 0.25             | n11 | 8.1 | 38.8                 | 17                | 1.113             | 2028                    | 0.21                     |
| 5/7 Feed                | Chg. 72 - Poly 1 | -                      | 49         | -           | 4.0              | -              | 0.04             | n11 | 7.8 | 38.9                 | 16                | 1.113             | 1999                    | -                        |
| 5/7 Run 1               | Chg. 72 - Poly 1 | 30                     | 52         | 5.5         | 5.5              | 15             | 0.00             | -   | 7.7 | 38.6                 | 24                | -                 | 2012                    | 0.24                     |
| 5/7 Run 2               | Run 1 5/7        | 44                     | 50         | 7.5         | 5.7              | 8              | 0.00             | -   | 6.7 | 38.1                 | 27                | 1.113             | 1986                    | 0.57                     |
| 5/7 Run 3               | Chg. 72 - Poly 1 | 59                     | 58         | 4.3         | 4.5              | 21             | 0.04             | -   | 6.9 | 38.5                 | 26                | -                 | 1991                    | 0.49                     |
| 5/7 Run 4               | Chg. 72 - Poly 1 | 59                     | 100        | 4.0         | 4.5              | 30             | 0.00             | -   | 6.7 | 39.0                 | 24                | 1.127             | 1938                    | 0.30                     |
| 5/7 Run 5               | Chg. 72 - Poly 1 | 46                     | 37         | 3.0         | 5.0              | 32             | 0.00             | n11 | 6.0 | 38.0                 | 185               | 1.142             | 1991                    | 0.80                     |
| 5/7 Run 6               | Run 5 5/7        | 60                     | 43         | 3.5         | 5.4              | 24             | 0.00             | n11 | 6.0 | 38.0                 | 185               | 1.142             | 1991                    | 0.80                     |

- a) Cumulative percent greater than 0.45 microns.  
b) Run 1 and Run 2 4/27 blended together before testing.  
c) Rotor stalled after 9 minutes operation.

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