BUSINESS CONFIDENTIAL

MONTHLY REPORT

TO: Dr. J. B. Saunby
Mr. W. E. Whitehurst

FROM: R. J. Hanna

DATE: May 31, 1974

DISPERSION, SOLVENT, AND SPECIALTY VINYL RESINS

HIGHLIGHTS

VCl Monomer in Dispersion Resin Latices - In one of the techniques under study to reduce the residual monomer content of dispersion resin latices, a series of batch stripping operations was conducted in the laboratory at temperatures of 50, 60, 70, and 80°C with a constant foam level and vacuum increasing with time. Under these conditions the latex, which contained 1.5% monomer initially, yielded values with increasing temperature of 0.25% (50°C), 0.03% (60°C), 100 ppm (70°C), and 10 ppm (80°C) with 2.5 hours of stripping.

Vinyl Chloride-based Latexes for Can Interiors - The reproducibility of product and process for the UCC water-borne vinyl terpolymer for can interiors has been demonstrated by consecutive, triplicate polymerizations in a 100-gallon autoclave. The data from these runs are required for the FDA petition. The latex will be used for future customer trials.

RESEARCH AND DEVELOPMENT DEPARTMENT
CHEMICALS AND PLASTICS
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216H DISPERSION RESINS, OPERATION TEAM

Vinyl Chloride Removal from Latices - One method being studied to reduce the residual vinyl chloride in PVC latexes is to strip the latex under vacuum in a batch process. Preliminary studies using a one-liter charge in a jacketed, 2-liter flask yielded the following results.

Temperature	VCl Content After 2.5 Hrs.
50°C	0.25%
60°C	0.03%
70°C	100 ppm
80°C	10 ppm

In each case the vacuum was controlled to maintain a constant foam level of approximately 10 cms. above the liquid surface. The initial residual VCl content of the latex was about 1.5%. In all of the runs, the stripped latex was found to be acceptable. A 2-4 percent increase in total solids was observed. However, if a greater degree of foaming can be tolerated, or a more effective anti-foam agent can be found, it may be possible to concentrate the latex by operation at a pressure considerably below the vapor pressure of water at the stripping temperature. In addition to continuing the above study, a continuous falling film stripping operation is being examined.

(P. F. Lobo, R. J. Hanna)

Use of a Rototherm Thin-Film Evaporator Under Vacuum to Remove VCl From QYNV Latex Containing 500 ppm Nalco-22-73 Antifoam Agent - The employment of the one quarter-square foot Rototherm evaporator at atmospheric pressure for the removal of VCl from QYOH latices with and without the use of antifoam ag nts has been previously reported. At temperatures to 85°C, 2/3 of the monomer was removed. The results of more recent runs conducted under vacuum conditions are presented below.

Run No.	Latex Feed Rate, lbs./hr.	Latex Temp., °C	Absolute Pressure, mm Hg	Latex Solids, %	Increase in Latex Solids, %	VCl in Latex, %	VCl Reduction in Latex, %
Control A	--	Room Temp.	Atmospheric	34.3	--	0.83	--
1	19.6	64-71	350	35.1	2	0.24	71
2	11.7	74-76	340	35.4	3	0.10	88
Control B	--	Room Temp.	Atmospheric	37.0	--	1.28	--
3	23.1	70-72	300	38.5	4	0.26	80
4	7.5	72-78	300	44.3	20	0.06	95
Control C	--	Room Temp.	Atmospheric	37.5	--	1.40	--
5	10.8	73	300	36.7	--	0.12	91
6	10.7	68	215	37.4	--	0.07	95
7	10.7	64	165	38.1	2	0.05	96
8	10.8	62-64	125	39.2	5	0.12	91

Under these conditions, removal of 95 percent of th VCl was approached. Latex samples taken from the B control and Runs 3 and 4 were spray dried, ground and checked for rheology. No significant difference was noted.

The results obtained above used hot oil in the jacket of the Rototherm. When steam was used, the heat flux became much higher and difficulty was experienced in avoiding broken latices. With high latex flow rates (30 #/hr.), operation was satisfactory but excessive foaming resulted. At low flow rates (10 #/hr.) latex stability could not be retained. These problems tend to indicate that, like the breakage experienced upon blowing latex from an autoclave, a rapid release of VCl from a latex particle tends to disrupt the protective layer around each latex particle.

The best operation, therefore, results from a low heat flux and long residence time. This, however, would require a large thin-film evaporator. The studies will be continued by Dr. Lobo.

(L. G. Reed, R. J. Hanna)

320D05 POWDER COATINGS

Drying Studies - Sufficient data are now available to make an initial comparison of the various methods of reducing the volatile content of spray dried resin. The favorable economics associated with bagged drying suggest that this is probably the best method. However, as the volatiles lost are not recoverable, the feasibility of the method would have to be reevaluated in the light of anticipated solvent emission laws.

The three auxiliary drying processes studied yielded the following results:

Drying System	Approximate Drying Time Required
Microwave augmented drying in an air slide	5-10 minutes
Hot air drying in an air slide	2-5 hours
Hot air over resin bed	5-10 hours

For a given feed rate, the size of a dryer required is invers ly proportional to the residence time or drying time and hence the first method appears most favorable. The recovery of volatiles is comparatively easy because of the small amount of air required in this method. The drying studies are being continued using

spray dried resin samples of various compositions and volatile contents.

(P. F. Lobo, R. J. Hanna)

Closed Cycle Spray Drying Tests - An additional week of tests on the Niro closed-cycle test equipment in Copenhagen has been set for the first week in July. During these tests we intend to evaluate the spray-dry characteristics of GD-7105A, the current "best choice" for a thermoplastic clear vinyl powder for interior can coatings. We also intend to seek further information regarding the necessity of impregnating the feed: substantial process simplification would result if satisfactory performance can be achieved without impregnation. The final item for further examination during the series is the effect of including vinyl acetate monomer in the feed at a level corresponding to what would be found if a "normal" stripped autoclave varnish were diluted to use as feed to the spray drier.

(D. R. Montgomery, R. J. Hanna)

320C17 VINYL CHLORIDE COPOLYMER LATEX FOR CAN INTERIORS

Polymerization Rate

Recent experiments in the one-gallon autoclave have demonstrated that the magnitude and reproducibility of the polymerization rate for the production of terpolymer latex (QEX-2033) can be improved by the addition of small quantities of soluble iron to the premix charge. Results of duplicate experiments, illustrating the improvement in conversion for a constant polymerization time (23-24 hours), are summarized in the following table (Table I):

TABLE I

ENHANCEMENT OF POLYMERIZATION RATE BY THE ADDITION OF SOLUBLE IRON TO THE PREMIX

Soluble Iron Added (ppm of Premix Charge)	Latex Total Solids (Percent in 23-24 hrs.)
0.	21.5
0	22.0
0.19	27.1
0.19	27.5
0.37	37.4
0.37	35.9

A latex made with 0.75 ppm of soluble iron added to the premix charge, i.e. twice the quantity of iron required for good polymerization rate, was evaluated by Mr. W. I. Wertz. Its performance was comparable to standard QEX-2033.

(T. L. Dawson)

Scale-up - The FDA petition on the water-borne vinyl terpolymer beer can interiors must contain data to demonstrate reproducible production of the latex in large-scale facilities. The only problems encountered in the initial 60/100 gallon scale-up were slow and variable polymerization rates.

The use of low concentrations of soluble iron in the premix charge, as described in the preceeding paragraph, has now been employed in a series of polymerizations in the 60/100 gallon autoclaves. Consecutive, triplicate polymerizations (22-23 hours) in these larger autoclaves gave latices with total solids on target and near perfect reproducibility of polymerization rate. The reproducibility obtained with identical charges (2033 composition) is illustrated in the following table (Table II):

TABLE II

REPRODUCIBILITY OF QEX-2033 PROCESS

IN 60/100 GALLON AUTOCLAVES

Run No. (7981-)	Soluble Iron Added (ppm of Premix Charge)	Latex Total Solids (Percent in 22-23 hrs.)
34	0.37	36.6
35	0.37	35.2
36	0.37	36.0

The latex compositions and coatings properties were also reproduced closely and were on target. The three drums of latex prepared during these studies will be employed for customer sampling as needed.

(T. L. Dawson, R. T. Wood)

FDA Petition - The action plan for the vinyl terpolymer latex petition requires that all data be in Dr. W. B. Ackart's hands by July 1. The petition is scheduled to be written and filed by August 1, and the regulation hopefully would issue by February 1, 1975. The current status of the various component parts is as follows:

1. Total non-volatile extractions at Bound Brook.

   Three solvents have been completed. The fourth is in progress and a completed report is planned by June 1.

2. Monomer migration data at South Charleston.

   Test development work is in progress. The completed report is due July 1.

3. Characterization of extracts.

   The revised report has been completed. No additional laboratory work is planned.

4. Seven-day rat feeding at Mellon Institute.

   Polyester sample and low molecular weight terpolym r sample have been shipped to Mellon. Feeding studies are scheduled to begin June 7. The completed report is due on month after receipt of the samples to be fed.

There are some additional routine pieces of information that must be provided by July 1 if we are to maintain the tim - table. These are as follows.

1. Identity of the product.

   Common name, chemical name, trade name, empirical formula, structural formula, molecular weights, and methods used to determine.

2. Manufacturing process.

   Raw materials, RM specifications, and test methods. Polymerization process, recipe, and equations for the re- action.

3. Specifications for the copolymer and test methods.

4. Reproducibility of the products.

   Data for 3 or 4 "commercial" batches according to Item 3 above. The data from the consecutive, triplicat 60/100 gallon runs described in a previous section of this report will be used.

We will also need to file an environmental impact statement with the petition which means that Dr. Ackart will need these data by July. There is some uncertainty about how

complete the EIS need be because FDA has proposed some new, l ss stringent requirements. However, at the very least we will need data identifying the pollutants emitted during manufacture, the applicable Federal, State, and local emission requirements, and a statement that we are in compliance. In the absence of actual production facilities for the vinyl terpolymer latex (QEX-2033), perhaps we can use environmental data from one of our plants that currently produces other vinyl chloride latices. At the worst we will need a full EIS covering manufacture, use, and disposal of the polymer. This implies that we must know in which state the product will be produced.

(W. B. Ackart, J. J. Brezinski, T. L. Dawson, H. Senman, C. S. Weil, W. I. Wertz)

Project Review at Glidden - A general review of the status of the vinyl terpolymer latex (QEX-2033) project was held at Strongsville, Ohio on May 10, 1974. The following people attended the meeting:

Glidden Personnel	UCC Personnel
Andy Engstrom	Watson Ackart
Ed Eubanks	Bill Buttrick
John Hafeli	Lowell Comstock
Bob Moorman	Tom Dawson
Bob Woodruff	Maynard Sherwin
Jim Woebkenberg	Frank Williamson

Special emphasis was given to discussions of the FDA status, th odor emission problem, and overspray.

FDA Discussion - Dr. W. B. Ackart, UCC Manager of FDA and USDA Liaison for Plastics, gave a summary review of the current and potential problems associated with the recent activity related to vinyl chloride monomer. Dr. Ackart pointed out that in addition to FDA we are also concerned about OSHA (manufacture) and EPA (environment) regulations, but that the relevance of these factors cannot be measured accurately until the regulations are finalized. These regulations will impact on the total PVC industry, as well as the water-borne vinyl project. Glidden was advised that this could complicate our ability to supply the latex commercially.

Dr. Ackart briefly reviewed our FDA-related laboratory studies on the terpolymer latex and the results of the pre-petition review at FDA. Dr. Ackart expressed the opinion that there ar no technical barriers to FDA approval of the coatings under curr nt regulations, if we can satisfy OSHA and EPA requirements. Our

current goal for FDA approval is early 1975, but final approval will depend on FDA's reaction to our petition. Mr. Andy Engstrom, Glidden advisor on FDA activities, was optimistic and recommended that we should not retard the program because of the current controversy relating to vinyl chloride monomer. This opinion prevailed and it was decided that we should continue the program as planned.

The next step at Continental Can is a 13-week pack test, so Continental has requested a better definition of the potential for FDA approval. Dr. Ackart will write a letter to Mr. Engstrom outlining our plans, our data, and our expectations regarding the FDA status of the vinyl terpolymer. A draft of this letter is currently being circulated internally for approval before it goes to Glidden. Hopefully, this letter will convince Glidden and Continental that we can obtain FDA clearance for the terpolymer.

Odor Emission During Baking - Bob Moorman emphasized that the emission of the "soapy" odor, which was first detected during the baking of the coatings at Continental Can, is of concern because they are afraid that it might lead to a problem with OSHA. Brief attempts at Glidden to mask the odor with additives hav not yet been successful. The odor is very characteristic of lauric acid, so we have been investigating the use of a less volatil , reactive surfactant (Avirol POL 40).

This FDA-approved surfactant (Avirol POL 40) has be n identified in our laboratory as the ammonium salt of 9,10-dihydroxy stearic acid. A terpolymer latex made with this surfactant gav coatings with blush and adhesion properties similar to those obtained when ammonium laurate is used as the surfactant. Befor submitting this sample (QEX-2067) to Glidden, Mr. W. I. Wertz conducted and reported the following evaluation of its performance properties: Three formulations were prepared (1) QEX-2067 + 1 PHR Carbopol 940, (2) QEX-2067 + 1 PHR Carbopol 940 + 3 PHR ammonium laurate (no Avirol was available in Bound Brook), and (3) QEX-2067 + 1 PHR Carbopol 940 + 3 PHR ammonium laurate + 1 PHR ERL-2774 and 2 PHR Resimene X-735. Systems 1 and 2 were evaluat d on two different samples of Continental Can lubricated budium stock at bakes of 4 minutes at 340°F and 365°F. System #3 was evaluated on E-81 epoxy and lubricated budium using a bake of 4 min. at 320°F. All films were completely blush free after pasteurization and no adhesion failure occurred. An odor test suggested by Glidden was also conducted by Mr. Wertz using unmodified QEX-2033, QEX-2067, and QEX-2067 + 1 PHR Carbopol 940. The conclusion was that QEX-2067 had less and different odor than QEX-2033 and that the addition of the Carbopol seemed to reduce the odor even more.

In view of these encouraging results, a sample of th terpolymer latex made with Avirol POL 40 (QEX-2067) was given to

Bob Woodruff at the meeting on May 10. It was tested in the Glidden laboratory during the visit. The test confirmed less odor emission from the 2067 latex relative to the 2033 latex, although some (different) odor was emitted during the 350°F bake of the 2067 coating. Neither the structure nor the trade-name of the Avirol POL 40 surfactant was given to Glidden personnel. However, Bob Woodruff was told that it contains hydroxyl groups, so that he would investigate its reactivity in an effort to tie up the surfactant in the coating. Bob was impressed with the inherent improvement in the odor emission, and requested a one-gallon sample for further testing and spray studies.

Overspray Problem - Glidden feels that they can solve the overspray problem. It had been suggested earlier that the resin in the overspray be recovered and dissolved in solvent for use as a touch-up lacquer.

(T. L. Dawson)

328L PVA OPERATIONS TEAM

Recovery of Autoclave Varnish - The current production unit uses a plate dryer to remove the acetone and residual vinyl acetate from the PVA autoclave varnish. In preparation for a new plant for the production of low-profile PVA, a possible improvement was the use of a thin-film evaporator in place of the ponderous plate dryer.

Tests in R and D with an Artisan thin-film dryer show d the feasibility of this approach; hence, tests were set up at Artisan and Luwa. These tests, witnessed by Mr. J. V. Carroll of Engineering, did not go well at Artisan because of mechanical problems but did go well at Luwa. The use of a Luwa dryer was, therefore, seriously considered by Engineering for the LP plant design, but finally rejected because of the proven performance of the plate dryer for drying PVA varnish.

(J. V. Carroll, R. J. Hanna)

REPORTS

D. R. Montgomery, Project Report, "Vinyl Solution Polymerizations: Effect of Initiator Type on Lead Content of the Autoclave Varnish," April 10, 1974, File No. 19371.

D. R. Montgomery, Project Report, "Vinyl Acetate Polymerization for Low-Profile Additives: Some Studies on the Existing Process," May 10, 1974, File No. 19453.

D. R. Montgomery, Project Report, "Vinyl Acetate Polymerization for Low-Profile Additives: Process Studies for Expansion of Capacity," May 21, 1974, File No. 19491.

R AND D FINANCIAL SUMMARY

SOUTH CHARLESTON VINYL RESINS (RJH, JJB)

Number	Title	Expenses, $M 1974 April	YTD	Budgeted	Budget
	Coatings				
321H10	Dispersion Resins	2.1	9.8	25	118
321K10	Solution Resins	3.4	19.2	60	96
321K12	FDA Approval	0.1	1.8	15	36
	Total Coatings	5.6	30.8	100	92
	AS and EI				
335H10	Dispersion Resins	4.8	22.8	53	129
	Operations Team (328)				
328K	Solution Resins	8.5	37.9	140	81
328G	PVEE Resins	0.0	0.0	2	0
328L	PVA	3.7	11.2	29	116
	Total Operations Team	12.2	49.1	171	86
	Operations Team (216)				
216H	Dispersion Resins	15.0	60.2	167	108
	Non-Business				
320C17	PVC-Based Latexes	9.0	55.9	107	157
320D05	Powder Coatings	5.8	23.6	70	101
	JJB Miscellaneous	0.4	2.7	17	48
	Total Non-Business	15.2	82.2	194	127
	COMPLETE TOTAL	52.8	245.1	685	107

R AND D FINANCIAL SUMMARY

SOUTH CHARLESTON VINYL RESINS, MISCELLANEOUS

Number	Title	April	YTD	Budgeted	% Budget
216B12	ASTM Activities, Suspension Resins	0.25	2.10	5.0	126
897N42	AS and EI, Sales Travel	0	0.16	6.0	8
897N47	Tech. Service, UCI	0.15	0.49	6.0	25
		0.40	2.65	17.0	46.5

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