

ESCAMBIA CHEMICAL CORPORATION
INTEROFFICE MEMORANDUM

To: Mr. R. W. Beech

Date: October 2, 1970

From: D. V. Lowe

Copies:

Re: PVC Process Improvements
Progress Report - September, 1970

CEA 7009 - "Addition of One 5,000 - Gallon Polymerizer
with Electrically Driven Agitation to the
Existing PVC Plant"

1. Revised refrigerated water piping requirements around the new surge tank supply pumps. Concrete foundation for the surge tank is in progress.
2. Wrote maintenance work request to extend existing chemical cleaning supply and return pipes to service the new reactor after it is put in service.
3. Upon further study of the agitator drive mounting flange, a question arose concerning the exact design of the flange and proper mounting procedures. To clarify our questions, a trip was made to Mixing Equipment Company by Mr. E. Battles.
4. Escambia was notified of the impending liquidation of Glascote Products, Haveg Industries, the vendor of our new 6" glass coated cooling baffles. A visit to Pfaudler Company by Mr. Battles confirmed the feasibility of substituting the three-fingered baffle delivered with our vessel from Union Carbide until the cooling baffles are available from Glascote.
5. The shutdown date has been moved back to October 19, 1970 to allow better coordination of maintenance effort in PVC and the other plants. Mr. Kerler has a list of shutdown maintenance items. All necessary preparations for construction work during the shutdown appear in order. All critical equipment deliveries have been properly coordinated except the cooling baffles and the new Yarway drop valve. The latter will be covered with a spare. Detailed shutdown and startup sequence planning will be accomplished during the week of October 5, 1970.

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6. It must be noted again that there still is no definite provision for emergency power to the agitator and cooling water circulating pump for the new reactor. It would be unsafe to start the reactor without such emergency power available. Mr. Speed has made considerable progress concerning these requirements, and has presented our case to Escambia management.

CEA-7037 - "Expansion and Modernization of the PVC Plant
at Pace, Florida"

1. The wet cake drying tests at Louisville Rotary Dryer Company were cancelled during September. The purpose was to have been a projection of increased rates available with our equipment if the use of Bird screen-bowl type centrifuges could greatly reduce volatiles in the wet cake. It appears that Bird Machine Company's test work indicates that only certain types of Escambia PVC resins are benefited by the screen-bowl centrifuge design. Therefore, the rotary drying tests were cancelled and we must install two new flash drying systems for series operation with our rotaries as originally planned.
2. PVC Research and Development conducted bench scale tests to determine the effect upon residual volatiles of percolating ambient temperature dry air through a small sample of 6180. It was found that volatiles were reduced from 1.0% to 0.4% after a period of three days. Costs have been accumulated for installation of fluidizing air pads in the bottom of one of the new silos. Total cost would be only an additional \$5,000. However, the project was abandoned since it was calculated that the fluidizing air requirements for a tank of the size planned would be tremendous and unreasonable.
3. Contact was made with representatives of Fluid Energy Systems Company for further information on their new design in flash drying equipment. A preliminary quote has been obtained along with some specific information as to operation of their equipment. Mr. Speed has requested that Fluid Energy revise their preliminary estimate to permit easy comparison to the firm quote on hand from Bowen Engineering. The writer has requested Fluid Energy requote based on slightly smaller sizes compatible with our actual needs. Samples of 1225 and 6180 wet cake have been collected to allow test work at Fluid Energy pilot facilities if so warranted.
4. Specifications and requirements for complete bulk storage and transfer systems were given to three more vendors for quote. All three were given personal tours of our existing bulk handling facilities to fix locations, distances involved, and interconnecting procedures. Although much time remains available for firming this portion of the project, quotes should be in by November.

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PVC REACTION SECTION

Hot Water Charging to Reactors

Charging of hot process water was again tried as a means of decreasing heatup time. Again, there is no indication why this procedure cannot be instituted as normal plant routine if only (a) methocel is replaced by PVP as a suspending ingredient (to eliminate cold water solutions), and (b) a new rinse water header is installed to replace the existing line. Hot rinse water is not practical since it must be manually handled. This line cannot be relocated to another source at this time since it must be at least 100 psig for good reactor cleaning (process water supply pumps exert 100 psig). After inception of chemical cleaning, the line may be relocated to a source of low pressure water.

Inspection of Catalyst Systems

Past experience has shown that certain combinations of lauroyl peroxide and IPP are advantageous in reducing cycle times under certain conditions, especially if the polymerization takes place above 140°F. It was determined that at least 9 of our resin types are candidates for application of this catalyst system. Until such time as new initiator systems are proven and recommended by PVC R&D personnel, it is planned to slowly begin investigation of the effects of the combination system upon yields and resin quality.

CEA 7012 - "Automation of PVC Reactor Drop Valves"

The Ingersoll-Rand air driven angle-wrench was finally received in the plant. The automated Yarway valve which has been on hand for several months was finally put in service. Instructions were issued for its proper operation. The automatic valve has been successfully operating in PVC service for three days with no problems. It is planned to leave this valve in service for one month as a practical test of its feasibility. After that period of time, the remaining money in CEA 7012 will be spent to order parts for complete conversion of all the other reactor drop valves.

External Heat Exchanger on PVC Reactor

As a simple experiment, a single tube stainless steel heat exchanger was connected to one of the nozzles atop No. 4 reactor. It was piped with refrigerated cooling water to observe the rate of heat removal during polymerization. Condensing vinyl chloride vapors may be a feasible means of external temperature control providing chemical cleaning of the condenser were available. It was likewise planned to study fouling characteristics in the stainless steel heat exchanger. Results of the four-day long test indicated no appreciable heat exchange with this setup, however. It was concluded that

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External Heat Exchanger..... (cont'd)

external heat exchange may be insignificant compared to liquid to liquid exchange through the vessel walls. To prove such a conclusion would require larger scale tests.

New PVC 6180 Recipes

To alleviate the need for constant temperature variation in the 6180 recipe for changes in relative viscosity, we are studying one 6180 recipe which will be polymerized at a fixed 146°F. Future variations in relative viscosity requested by our customers will be accomplished through variations in TCE level only. This may tend to more firmly fix the range of bound vinyl acetate in our 6180 copolymer, regardless of relative viscosity variations.

Reactor Rupture Discs

Seven of the newer style Fike rupture discs have experienced rupture since the changeover to that type disc. Consultation with the manufacturer and his representative reveals that there is most probably nothing amiss with the discs themselves. We are apparently operating our reactors, since PVC 2150 manufacture has begun, above the recommended lower safety zone for long life. Long life is supposedly impossible if alternate vacuum-pressure cycles are imposed on the discs and if the pressure is over 80% of the disc rupture pressure rating. To rectify this, we are investigating a disc from Fike which will be rated at 210 psig (with a tolerance range) versus the presently allowed range of 180-200 psig. ASME codes will be consulted and complied with.

FINISHING SECTION

Auxiliary Duct for South Exhaust Fan

A cost estimate was obtained from Barrios and Acosta Company for installation of a stainless steel duct to allow the Alpine exhaust fan to service the south line product collector in parallel with the south exhaust fan. The increased air flow through the south rotary dryer would allow additional resin throughput as long as residence times were not found to be adversely affected by the increased air velocities. This project has been set aside for further study at a later date when it might be fitted into the maintenance effort.

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North Product Collector Air Grids

The 304 s.s. air supply header grids in the north product collector were found badly corroded and contributing to product contamination. The problem appears to be wet and/or salty air from our inoperative process air dryer. (There is a new air dryer included in CEA 7037). All of the corroded air grids are to be replaced during the impending plant outage. The s.s. bag cap nipples have also corroded badly for the same reasons. The solution here will be to buy one spare lot of bag caps, allowing alternate lots of the existing caps to be removed from service for nipple replacement.

Ionized Air for South Rotex Screener

A quote for ionized air equipment adaptable to the rotex screener was received and the equipment is being put on order for plant trials. The use of a moving ionized air stream should reduce static charges on resin particles and reduce tailings. In addition, the need for humidifying steam would be eliminated, allowing proper use of rotex inspection port covers. If the ionizing air equipment is proven not suitable for our service, it may be returned to the vendor for 90% refunding.

Magnet Boxes for Contaminant Reduction

Drawer-type magnet trays are on order for installation under each of the product rotary valves in finishing. The magnets, constructed of "Alnico" and encased in 304 stainless steel, will serve to trap metal and rust contaminants in the process stream and thus reduce the level of finished product contamination. Cabinets are being fabricated by our shop to contain the magnet trays and are likewise to be constructed of 304 s.s. Installation of these items is expected during October but can be accomplished without plant outage.

GENERAL

A report entitled "A Treatise of PVC Off-Grade and Procedures" was issued to Mr. A. K. McMillan to clarify the major reasons for and method of reporting off-grade PVC production. We hope that definition of the terms involved in reporting unusual production will promote better agreement among all interested parties.

David V. Lowe
David V. Lowe

AP00046765

ESCAMBLA CHEMICAL CORPORATION
INTEROFFICE MEMORANDUM

To: Dr. W. M. Smith Date: October 2, 1970

From: D. J. Mann Copies:

Re: Monthly Report
PVC Research and Development
September, 1970

OPERATIONS

I. Fusion Resins

Objective: To develop a process for manufacturing a PEARL^R fusion resin by direct polymerization having a median particle size ~60-90 microns and a bulk density of 20-25 lbs./cu.ft.

A. Properties Laboratory

Field evaluation results to-date on the MP-2A MICROPEARL^R indicate that it behaves differently than 4185-2S, even though initial fusion work at Pensacola demonstrated the equivalency of both products. For example, fusion samples from GS Plastics showed a smoother, glossier surface for 4185-2S than for MP-2A. It is readily apparent that such variables as level of pigment, type of stabilizer, etc., all affect the properties of the resultant fused surface.

We intend to study these factors with the objective of qualifying MP-2A in applications now being satisfied by 4185-2S and certain competitive resins.

II. Saran

Objective: To prepare sufficient quantities of both extrusion and solution grades of saran to permit selective field sampling and laboratory studies.

A. Properties Laboratory

Crystallization rate studies were made this month on both pilot-plant and plant-produced resins for the following purposes.

1. Develop a quality control tool
2. Measure the performance of different saran types
3. Compare plant versus pilot-plant resin

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OPERATIONS (Cont'd)

Data is currently being accumulated on all saran runs, and this will be continued until confidence in reproducibility and performance of all resins is established.

The test procedure is as follows:

The resins are blended with 4.0 parts Epoxol 9-S and pressed into films 5-9 mils thick. The hot films are then quenched in an ice bath and cut into strips 3/4" X 3". These strips are then conditioned in a water bath at the desired temperature for a varying period of time. The crystallinity behavior is then determined by measuring tensile properties.

The following crystallinity data (minutes to obtain maximum tensile strength) was obtained on the two recently produced plant saran resins.

	<u>30°C</u>	<u>40°C</u>	<u>50°C</u>	<u>60°C</u>	<u>70°C</u>
SV1710E	>70'	>70'	37.5'	18'	----
SV1706E	----	>70'	28'	11'	7.5'

As expected, the plant SV1706E crystallizes somewhat faster than the SV1710E. We are presently attempting to correlate this data with similar pilot-plant resins. Also, we wish to learn the effect of inhibited versus uninhibited vinylidene chloride on crystallization rate. These data are being obtained and should be available next month.

III. Bottle-Grade Resin (PVC 2185)

Objective: To support the efforts to establish a joint working agreement with Fosfatbolaget/Lonza by developing a bottle-grade resin which will match the performance of Lucovyl RB8010 (Pechiney-St. Gobain) in blow-molded applications.

Efforts were continued during September to prepare a resin matching the performance of Lucovyl RB8010. Towards this end, the following three objectives (discussed previously in the August report) were pursued.

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OPERATIONS (Cont'd)

1. Increase the Geon latex in the standard manufacturing recipe for PVC 2185.
2. Use PVP K-90 as the sole suspending agent.
3. Add a supplementary amount of protective colloid to the PEARL^R recipe, viz, PVP K-90 which is highly compatible with PVC.

A. Plant Trials

Last month it was reported that attempts to make a small particle size PEARL^R resin (0.4 part Geon latex) in the plant failed, due presumably to the use of water-insoluble ethylene dichloride solvent for reactor cleaning. It was believed that the walls were coated with this solvent and actually enhanced wall fouling and agglomeration. This trial was repeated in the plant using reactors in their normal state, and better results were obtained as follows:

	PVC 2185 #1504	PVC 2185 #1516
M.P.S. (°C)	155	155
Bulk Density	28.4	29.8
Fisheyes at 4.5'	-----	4
6.0'	-----	0.8

Although plant trials were successful in obtaining the desired physical properties, the resins were not superior to the PEARL^R resins previously sampled to Lonza as determined in film extrusion trials.

Another plant trial was run (#1518) based on a non-PEARL^R recipe using .50 part PVP K-90. This trial also gave the desired physical properties but failed the extrusion test.

It is believed that the condition of the plant reactors (wall fouling, etc.) contributed to the failure; and, on this basis, it was decided to prepare all resins for shipment to Lonza in the pilot plant.

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OPERATIONS (Cont'd)B. Pilot Plant

As discussed above, difficulty in preparing gel-free resins in the plant made it necessary to return to the pilot plant. Although efforts were made to prepare both a PEARL^R resin containing increased Geon and a non-PEARL^R resin (PVP K-90), each with a median particle size approaching 110 microns, we were successful only in preparing the latter resin. In order to use the minimum amount of PVP K-90, a filled reactor with K-water was employed. The following recipe gave, in eight batches, median particles from 90-110 microns and bulk densities from 35-39 lbs./cu.ft.

	<u>Parts by Weight</u>
Water	265
VCl	100
LPO	0.09
PVP K-90 (20% solution)	0.5
Temperature, °F	155

Three hundred pounds of this resin was prepared and sent to Lonza. The eight pilot-plant runs were scalped through a 100-mesh screen and then blended. The composite, designated 3-1236-43, had the following properties:

	<u>3-1236-43</u>	<u>Lucovyl RB8010</u>
Screens, % on 100	T	T
120	9.0	8.0
140	20.0	57.0
170	20.0	26.0
200	29.0	4.0
PAN	22.0	5.0
Median Particle Size (μ)	90.0	108.0
Bulk Density (lbs./cu.ft.)	38.0	39.1
Relative Viscosity	1.80	1.76
Oil Absorption (ml. DOP/gm.)	0.65	0.60
Fisheyes at 4.5 min.	5.0	0.0
6.0 min.	0.6	0.0

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OPERATIONS (Cont'd)

3. At the higher emulsifier and K-90 levels, the bulk density is lower, with the most stable recipes having bulk densities in the range of 20-23 lbs./cu.ft.

This bottle polymerization study is being continued.

IV. Heat-Stable PVC 6180

Objective: To develop a PVC 6180 with improved thermal stability such that bulk-rail problems are eliminated and storage problems are minimized.

A. Properties Laboratory1. Melt Flow Testing

All plant-produced PVC 6180 lots are now to be tested for melt-flow behavior in anticipation of using this as a quality control test in the future.

The data obtained thus far indicate that the melt flow is more related to relative viscosity than acetate content. The melt flow range is 10.7-13.0 grams/10 min. for R.V.'s in the range of 1.78-1.81. If the R.V. drops below 1.78, the melt flow increases proportionately.

Efforts are being made to correlate this with customer melt-flow data (Nixon-Baldwin) and with customer acceptance/rejection on the basis of flow properties.

2. Volatiles Reduction

Efforts to reduce the volatiles by passing dry air through a bed of PVC 6180 resin were studied with the following results obtained:

<u>Time (hrs.)</u>	<u>% Volatiles</u>
0	1.1
19	1.0
27	0.9
50	0.5
74	0.4
102	0.6

This technique is being considered by the plant as a method for reducing PVC 6180 volatiles (which consist of ~ 85% unreacted vinyl acetate monomer) during storage in silos.

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OPERATIONS (Cont'd)B. Analytical Laboratory

Difficulties have been encountered in developing a reproducible procedure for measuring bound vinyl acetate; and this, in turn, has delayed our efforts to determine the limits of the present manufacturing process, in terms of maximum/minimum bound acetate levels.

The analytical laboratory believes a reproducible method has been developed, and the Linwood analytical laboratory is in the process of confirming this.

V. General Process/Product Development

Objective: To evaluate new processes, ingredients, equipment, techniques, etc., necessary to remain abreast of advanced polymerization technology.

A. Initiators

In an attempt to find an alternate catalyst system for IPP, two systems were investigated.

1. Lupersol 223M

Lupersol 223M is the di-2-ethyl hexyl peroxydicarbonate (40% in mineral spirits) from Lucidol. It was believed that this catalyst did not fall within the Uniroyal patent and therein lay the incentive for this study. Economically, it does represent an attractive alternate, and polymerization/polymer properties appear also to be equal to those of IPP. However, Mr. Nikaido, in a recent visit to Escambia, did not agree with our interpretation of this patent and felt that it did fall within the Uniroyal patent.

Until this matter is fully resolved, no further work will be conducted with this system.

2. Fosfatbolaget "In Situ" System

The teachings of Swedish Patent No. 315,404 (Fosfatbolaget) for the "in situ" formation of IPP were studied in the 10-gallon reactor. The cycle times were found to closely match those of IPP. A higher amount of coarse resin was formed during the polymerization than with IPP, but recipe modification may be able to solve this.

In view of Mr. Nikaido's comments on the Lucidol initiator, this catalyst system will be studied in more depth.

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OPERATIONS (Cont'd)B. Pipe-Grade Resin (PVC 1230-P)

It was reported last month that considerable work had been performed in the pilot plant directed towards a higher bulk density for PVC 1230-P, and that the following trends for increasing the bulk density had been observed.

1. Increase ΔP from 40 psi to 90 psi.
2. Reduce or eliminate Tween.
3. Reduce the level of protective colloid (PVA).

Using these guidelines, a plant trial was conducted during September which indicated that the most feasible approach to increasing the bulk density was the removal of Tween plus a decrease in the water/monomer ratio. Increasing the ΔP was effective but uneconomical; reducing the PVA was ineffective.

The recipe modifications should permit the manufacture of PVC 1230-P with an average bulk density of ~ 33 lbs./cu.ft. but with a slightly coarser particle size distribution.

D. J. Mann
D. J. Mann

/pad

AP00046772

ESCAMBLA CHEMICAL CORPORATION
INTEROFFICE MEMORANDUM

To: W. M. Smith

Date: October 2, 1970

Copies:

From: W. K. Clark

Re: Monthly Summary
PVC Technical Service
September, 1970

I. Customer Service

A. American Vinyl Company (TSR 851)

The 1000-hour weatherometer results on samples submitted by this customer have been reported. (Reference 1)

B. Polymers, Incorporated

An internal evaluation report was issued to determine a suitable Escambia resin to replace Geon 106 at customer's plant. PVC 2175 was recommended for sampling. (Reference 2)

C. International Shoe Company

Samples of Escambia's resins were compared for physical properties against Conoco's resin now being used by customer.

One thousand pounds of PVC 2200 has been selected for a future trial at customer's plant.

An early visit and trial at their plant by W. K. Clark is urged as a prerequisite to recommendations of a new screw design for increased output. (Reference 3)

D. Cerro Wire & Cable

A sample of competitive resin (fast drying) from this customer was evaluated in comparison with our PVC 1230-P and PVC 1225. (Reference 4)

E. Intervinyls, Inc.

This customer requested aid in the development of a dry blend formulation, incorporating Escambia's resin, for the

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I. Customer Service (Cont'd)

- E. extrusion of crystal-clear rugrunners. A letter and sample molding to this end was forwarded to Mr. Sparks with the recommendation of 2185. In view of the low output rates now being achieved by customer, a visit is being planned to ascertain that we can help them in this direction. (Reference 5)

F. Ethyl Resin Evaluation

Two samples of Ethyl's resin used for making re-wetable battery separators were evaluated. The initial evaluation gives some indication that this resin may be an emulsion type. (Reference 6)

G. Sekisui PVC Evaluation

Two samples of resin used by Sekisui for pipe extrusion and injection molding were evaluated. The viscosity of 2.14 confirms that their pipe is primarily unmodified type I. (Reference 7)

H. Surface Research and Development Company

General information on post-chlorinated PVC was written in a memorandum to P. F. Baker for transmittal to the customer. (Reference 8)

I. General Tire and Rubber - Akron, Ohio

Certain quality control results on three shipments of PVC 1250 were submitted at customer's request.

J. Tenneco Chemicals, Inc. - Nixon, New Jersey

At customer's request, we submitted certain quality control results on five shipments of PVC 6180.

K. Kerr Glass Manufacturing Company

This customer requested certain quality control results on sampled PVC 2185. (Reference 9)

L. Evaluations

An internal report was issued listing the physical properties of five competitive resin samples we recently evaluated. (Reference 10)

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October 2, 1970

I. Customer Service (Cont'd)

M. Vycell PVC Foam

A memorandum requesting further information on the subject product has been sent out. Implications that this may enter into the shoe sale market will be investigated.
(Reference 11)

II. Technical Service to APCI

Patent - Parallel Plate Extruder

Through the efforts of our patent attorney, Mr. Barry Moyerman, a patent application has been filed for a new extruder type of potential value in several new applications by W. K. Clark.
(Reference 12)

III. Samples

A total of 69 resin samples (734 pounds) and one dry blend (45 pounds) was shipped to 22 companies including APCI.

W. K. Clark

W. K. Clark

/pad

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Reference 1	Letter dated 9-1-70 from P. R. Browne to Mr. Reetz, American Vinyl Company
Reference 2	Report dated 9-1-70 from P. R. Browne
Reference 3	Memo dated 9-10-70 from P. R. Browne to W. K. Clark
Reference 4	Memo dated 9-14-70 from P. R. Browne to D. J. Mann
Reference 5	Letter dated 9-28-70 from R. E. Kunc to J. H. Sparks, Intervinyls, Inc.
Reference 6	Memo dated 9-10-70 from P. R. Browne to Dr. R. K. S. Chan
Reference 7	Memo dated 9-30-70 from P. R. Browne to Dr. W. M. Smith
Reference 8	Memo dated 9-30-70 from W. K. Clark to P. F. Baker
Reference 9	Letter dated 9-18-70 from P. R. Browne to Mr. Clarence Rohrer, Kerr Glass Mfg. Company
Reference 10	Report dated 9-17-70 from P. R. Browne
Reference 11	Memo dated 9-24-70 from W. K. Clark to A. R. Adams
Reference 12	Disclosure of 8-20-70 from W. K. Clark to W. M. Smith

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ESCAMBIA CHEMICAL CORPORATION

PLASTICS AND RESINS

MONTHLY REPORT

October, 1970

AP00046777

ESCAMBIA CHEMICAL CORPORATION
INTEROFFICE MEMORANDUM

To: Mr. T. L. Carey

Date: November 11, 1970

Copies:

From: A. Ross Adams/J. M. Davidson

Re: Plastics Division - Monthly Report
October 1970

COMPANY CONFIDENTIAL

In October, the Plastics Division lost \$58,000 after taxes against a budgeted loss of \$33,000. The October results showed the following variances from budget:

	October M\$	Year to Date M\$
Sales Revenues	(225)	(225)
Production	3	3
Inventory Adjustment	(2)	(2)
Total Cost of Sales (74%)	79%	79%
Selling	4	4
Distribution	24	24
G&A & Corp. Services	(19)	(19)
R & D	0	0
Interest	(6)	(6)
Other Income/Expense		

(Unfavorable)

Sales for October were \$390,000 vs. \$615,000 with the variance due primarily to reduced volume. The loss in volume was attributed to the general economic slow down as well as the automotive strike. In reviewing the "key" accounts we show a positive variance with all indications that this will continue. Average prices improved in October caused by a reduction in sales volume and a consequent per cent increase in bulk 6180 resin sales.

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	Oct. '70 ¢/lb.	Sept. '70 ¢/lb.	Aug. '70 ¢/lb.	YTD Act. ¢/lb.	Budg. Std. ¢/lb.
Bulk Prime	12.36	11.15	11.34	12.36	11.42
Bag Prime	11.72	12.30	12.17	11.72	12.22
Total Prime	11.87	11.71	11.84	11.87	11.97
Off Grade	6.22	6.66	7.47	6.22	7.25
Total Plastics	11.41	11.17	11.50	11.41	11.51

PRICING:

During the month pressure on pricing continued. Prices at Plastic Calendering were reduced marginally and we have been able to regain the business in spite of still holding prices higher than Conoco.

Allied and Goodyear announced price increases for their general purpose resins. These increases put general purpose bags at 12.5¢, bulk rail at 11.5¢, and bulk truck at 11.75¢. The increased prices are attributed to higher costs of production and are certainly overdue.

SALES ANALYSIS AND OUTLOOK:

Key account analysis shows a positive (185,000 pounds) variance although we were off in meeting budget goals. The automotive strike, business generally being off, and shipments to Suave considerably below budget accounted for the deficiency. Suave Shoe, who is under contract, was 400,000 pounds below budget for the month. Shipments to Suave Shoe should be on target by November.

The strike at Elm was settled during the last week of the month which should improve sales in November. Increases in business during November as anticipated at Plastic Calendering, Universal Pipe, and Industrial Vinyl. We expect to regain business at Tenneco which was lost due to a quality problem. Kerr presents a special problem where losses are anticipated to continue until we can establish a proper specification which will assure acceptance by the account. Generally, it appears that business will improve in November.

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ACCOUNT ANALYSIS

<u>ACCOUNT</u>	<u>Oct. 1970</u>	<u>6-Mo. Avg.</u>	<u>Oct. 1969</u>	<u>Variance</u>
Goodyear	692	502	204	+190
Plastic Calendering	280	213	400	+ 67
USM Chemical	233	398	190	-165
Suave Shoe	216	132	0	+ 84
Tenneco (N. J.)	200	191	412	+ 9
Harte & Co.	161	134	121	+ 27
Industrial Vinyl	160	65	80	+ 95
Armstrong Cork	144	102	72	+ 42
Goodrich	144	104	240	+100
Glamorgan Pipe	117	157	114	- 40
Elm Coated Fabrics	108	341	737	-233
Universal Pipe	108	61	0	+ 47
Custom Plastics	80	113	40	- 33
Tenneco (Ill.)	80	47	120	+ 33
Zapata	80	131	120	- 51
Kerr Glass	73	106	72	- 33
Industrial Solvents	72	47	30	+ 25
Jarrow	60	86	40	- 26
Plicoflex	55	0	0	+ 55
East Coast Chemicals	45	39	250	+ 6
American Vinyl	40	66	40	- 26
NAFCO	40	0	0	+ 40
New England Plastics	39	34	32	+ 5
Lynn Vinyl	35	40	0	- 5
Texon	31	65	37	- 34

PRODUCTION:

Significant production statistics for October are given in the table below:

	<u>Actual</u>	<u>Budget</u>
Prime Production, M lbs.	3,552	3,640
Off Grade, Tails & Sweeps, M lbs.	336 (8.6%)	232
Total Product, lbs/gal/year	1,250	
Reactor Availability, % *	86.4%	
VCM Efficiency, %	95.4%	95.0%
Production Runs/Grade	17/13	None

*Exclusive of plant shut down

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Prime production was lower than budget due to production problems on 2185. Prime production would have equalled budget except for the excessive off grade.

A six day shut down which accounted for 1378 hours was required for the 5000 gallon reactor tie-in. During this time, general plant maintenance was performed that would normally be due in the near future. The 5000 gallon reactor is reported to be producing resin of prime quality on a continuous basis although there are numerous small jobs required to complete the project. During October 402, 000 pounds of production was regraded prime for certain Escambia customers.

MARKET DEVELOPMENT:

An initial technical session was held at Delco-Remy which will commence in pilot plant trials during November. This work will provide us with knowledge of how our product will work in the application or what modification will be necessary.

The program on 6180 has led to improved product and expected reduced rejections at Goodrich as well as sales to Tenneco (Chicago) which had been lost. Although the plant problem is not completely solved further upsets should be minimal.

A brochure on Robbins has been prepared and several preliminary meetings have been held with interested parties.

The re-evaluation of the Norton EAR was completed based on lower cost input. However, this did not materially affect our over-all appraisal.

A meeting was held with FMC concerning solution PVDC which unquestionably confirmed that we have not and still do not have a suitable material. The deficiency is primarily in the area of MVTR and work has started toward improving this area. It is felt that when we develop a competitive product business will be available.

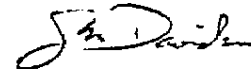
AP00046781

RESEARCH & DEVELOPMENT:

A study of the PEARL^R piggyback system has indicated that, while the new additive provides a synergistic effect on particle size reduction, it also reduces the bulk density below the minimum believed desirable for bottle grade resin.

Initial pilot plant studies on 2225 indicate that a significant increase in bulk density by wetting agent adjustment is not feasible. It is currently estimated that the maximum bulk density achievable will be 35 - 36 pounds/cu. ft.

Processing characteristics of Escambia 2000 series determined that these materials fuse from a powder into a melt faster than bulk polymerized PVC in both rigid and plasticized formulations. Lubricated rigid compounds are more tolerant to excess lubricant than bulk PVC typified by a proportional extrusion rate to screw speed. The 2185 is the only exception being more like the general purpose suspension resin except for a faster fusion time from powder to melt.



A. Ross Adams/J. M. Davidson

/acw

AP00046782

PVC INVENTORY

	Pensacola Warehouses 10/1/70 <u>M Pounds</u>	Off-Site Warehouses 10/1/70 <u>M Pounds</u>	Total <u>M Pounds</u>
Prime	4,872	2,459	7,331
Off-Grade	1,431	32	1,463*
Tailings & Sweeps	<u>163</u>	<u>0</u>	<u>163</u>
	6,466	2,491	8,957

*301,000 pounds sold and invoiced to H. Muehlstein
but not yet shipped.

	Pensacola Warehouses 11-1-70 <u>M Pounds</u>	Off-Site Warehouses 11-1-70 <u>M Pounds</u>	Total <u>Pounds</u>
Prime	4,326	2,912	7,238
Off-Grade	1,563	68	1,631*
Tailings & Sweeps	<u>186</u>	<u>0</u>	<u>186</u>
	6,075	2,980	9,055

*300,000 pounds sold and invoiced to H. Muehlstein
but not yet shipped.

ESCAMBIA CHEMICAL CORPORATION
INTEROFFICE MEMORANDUM

To: Mr. T. L. Carey

Date: November 9, 1970

From: A. K. McMillan

Copies:

Re: PVC Monthly Report
October, 1970

Prime production for the month was 3,552M pounds as compared to a budget of 3,640M pounds. Off-grade continues at a higher level than budget, 8.5% vs. 6.0%, and was responsible for not meeting prime budget. The high off-grade resulted from quality problems with 2185 production which accounted for 198M pounds of 277M pounds of off-grade exclusive of tails and sweeps. This results to some extent from a recipe change to make a finer particle size for the Kerr bottle application and a catalyst change to improve reactor cycle time.

The six-day shutdown for the 5M gallon reactor tie-ins was completed as scheduled. Several minor jobs remain to be done before the project will be complete. These will be done over the next two six-weeks. A number of maintenance items requiring a shutdown were done during the outage. Significant among these were repairs to the north product collector, dryer air seals and north stripper. Operation of the 5M gallon reactor has been excellent on the first few batches processed at the beginning of November.

The 5M gallon reactor will be used to increase production and at the same time obtain useful information for production of our resins in a larger capacity system and under improved reaction conditions. Special procedures have been established for collection of this important information.

Exclusive of the shutdown, no major outages were required for the month. However, a cutback was necessitated by a steam outage at the boiler to replace a main header steam valve resulting in 62 hours of lost reactor time.

Shipments for the month were 4,316M pounds and plant inventory decreased by 381M pounds to give a closing inventory of 6,085M pounds.

Other major changes have been made which will have a significant favorable effect on the plant operations. The new refrigerated water storage tank was placed in service near month end and has improved the operation of the Trane absorption machines and provided a more constant source of low temperature cooling water. The new automatic monomer and water metering

AP00046784

Mr. T. L. Carey
PVC

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November 9, 1970

systems are working well and should help resin reproducibility. The new rotex and modified heating coils were installed on the north line and are working well.

Cost variance for the month was \$21M unfavorable versus \$30,000 unfavorable forecasted. This unfavorable variance is due primarily to plant downtime to install the 5M gallon reactor. Variance for November is forecasted at \$1.5M favorable.



A. K. McMillan

AP00046785

ESCAMBIA CHEMICAL CORPORATION
INTEROFFICE MEMORANDUM

To: Mr. A. K. McMillan

Date: November 4, 1970

From: R. W. Beech

Copies:

Re: PVC Production Report
October, 1970

SUMMARY

Production of prime resin for the month of October was 3,552M pounds versus a budget of 3,640M pounds. Off-grade, tails, sweeps totaled 336M pounds or 8.6% of total production. Prime production would have equaled budget except for the excess off-grade.

Shipments for the month were 4,316M pounds with local inventory decreasing by 381M pounds to 6,085M pounds.

Vinyl chloride efficiency was 95.4% versus a budget of 95%.

Seven types were produced in nine runs on the south line and eight types were produced in eight runs on the north line.

Routine reactor downtime exclusive of the shutdown was 13.6%. Approximately 1,378 hours were lost during the shutdown about 18.5% of the total hours for the month.

Off-grade, tails and sweeps amounted to 336M pounds. This is 8.5% of the total PVC produced for the month. Fisheyes were the major source of off-grade for the month accounting for 41.4% of the total exclusive of tails and sweeps.

A six-day shutdown for the 5M gallon reactor project was completed as scheduled, however, numerous small jobs remain to be done to finish the project.

Production and variance for October are shown below along with the forecast for November:

	<u>October</u>	<u>November</u>
Production, Prime, M lbs.	3,552	4,820
Variance (favorable), \$	20,947	-0-

AP00046786

Mr. A. K. McMillan
PVC

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November 4, 1970

STATISTICS

	<u>October</u>		<u>Year to Date</u>	
	<u>Actual</u>	<u>Budget</u>	<u>Actual</u>	<u>Budget</u>
Prime Production, Homopolymer	2,811	3,089	2,811	3,089
Copolymer	741	551	741	551
Total Prime	3,552	3,640	3,552	3,640
Off-Grade, Homopolymer	236		236	
Copolymer	41		41	
Tails, Homopolymer	54		54	
Copolymer	5		5	
Floorsweeps	0		0	
Total Off-Grade	336	232	336	232
Total Production	3,888	3,872	3,888	3,872
Off-Grade Yield, %	8.6	6.0	8.6	6.0
Bulk Production, % Prime	41.1		41.1	
VCI Efficiency	95.4	95.0	95.4	95.0
Reactor Availability, % of Total Hours	86.4	-	86.4	-
	<u>Prime Only</u>		<u>Prime + Off-Grade</u>	
Production, Lbs./Gal, Yr.	1,250		1,368	
<u>Downtime Analysis*</u>				
	<u>Hours</u>	<u>% of Downtime</u>	<u>% Total Reactor Hrs.</u>	
Clean & Wash Reactors	439	52.0	7.1	
Plugged Drop Valve	21	2.5	0.3	
Changing Types	30	3.6	0.5	
Mixing Solutions	20	2.4	0.3	
Behind on Recovery	49	5.9	0.8	
Hydraulic Motor Maint.	10	1.2	0.2	
Scheduling Charges	85	10.3	1.4	
Miscellaneous	181	21.9	3.0	
Total	825		13.6	

* Calculated exclusive of shutdown.

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Mr. A. K. McMillan
PVC

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November 3, 1970

	<u>Hours</u>	<u>% of Downtime</u>	<u>Total Reactor Hrs.</u>
Downtime for 5M Gal. Reactor Shutdown	1, 378		18.5

	<u>South Line</u>	<u>Batches</u>	<u>North Line</u>	<u>Batches</u>
Production Sequence, M lbs.	2185	30	2200	5
Total	1250	62	2225	60
	1230	15	1225	84
	2160	4	1200	20
	6180	94	2250	45
	3185	10	1250	20
	2185	24	1230	10
	6240	5	2150	1
	1230	6		

Production Summary

<u>Type</u>	<u>Budget</u>	<u>Prime</u>	<u>Off-Grade</u>	<u>Tails</u>	<u>Total</u>	<u>%</u>
1200	76	162	0	0	162	4.2
1225	889	594	38	9	641	16.5
1230	355	247	0	5	252	6.5
1250	226	669	0	6	675	17.4
2150	76	6	0	0	6	0.2
2160	101	29	0	0	29	0.7
2185	277	221	198	8	427	11.0
2200	172	42	0	0	42	1.1
2225	341	453	0	6	459	11.8
2250	242	339	0	10	349	9.0
3185	90	59	0	10	69	1.8
6180	545	715	41	4	760	19.5
6240	16	26	0	1	27	0.7
All others	<u>234</u>					
Total	3640	3552	277	59	3888	

Floorsweeps

- 0 -

Total

3888

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Mr. A. K. McMillan
PVC

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November 3, 1970

Off-Grade Analysis

<u>Type</u>	<u>Lot</u>	<u>Pounds</u>	<u>Reason</u>
2185	6548	40,000	Contamination
	6549	40,400	Relative viscosity
	6555	41,000	Bulk density
	6636	40,400	Fisheyes
	6637	36,400	Fisheyes
1225	6576	38,000	Fisheyes
6180	6594	7,300	Sediment
	6596	34,000	Sediment
Total		277,500	
Tails		59,000	
Floorsweeps		<u>-0-</u>	
Total Off-Grade		336,500	

MAINTENANCE

The significant maintenance jobs for October are listed below:

1. Repaired valves on the north recovery compressor.
2. Repair work on the Pennsylvania compressor.
3. Hydraulic motor repairs on No. 10 reactor.
4. Replaced worn step bearing on No. 10 reactor.
5. Replaced leaking relief valves on north and south recovery compressor.
6. Installed rebuilt rotary valve on north bulk transfer system.
7. Repaired north dryer automatic feed valve.
8. Installed new pressure element on No. 1 reactor.

The following maintenance jobs were performed during the 5M gallon reactor shutdown:

1. Installed new air grids in north product collector.

AP00046789

2. Installed new damper boxes on north product collector.
3. Repaired patches in north stripper.
4. Cleaned out blend tank vent lines.
5. Cleaned tube bundles in refrigeration machines.
6. Cleaned out monomer and water charging systems.
7. Replaced dryer air seals.
8. Repaired cracks in north dryer wind tunnel.
9. Repaired all miscellaneous steam leaks.

DISCUSSION

A six-day shutdown was required in October to complete the 5M gallon reactor project. The project was completed as scheduled, in fact, lost reactor time was 1,378 hours versus a planned 1,440. This vessel is expected to be in full operation for November. Numerous jobs remain undone but will be completed by construction over the next few weeks.

Numerous quality problems were encountered in the production of 2185. This partially resulted from a recipe change to finer particles to try and improve acceptance at Kerr and a catalyst change to improve reactor cycle times. Total off-grade 2185 for the month was 198M pounds. In addition, we have encountered a number of rejections at Kerr. This problem will be pursued at Escambia and Kerr to try and resolve the difficulty.

A serious packing leak developed on the low pressure stage of the Pennsylvania compressor. Inspection revealed the wear rings were worn out and the piston was riding on the cylinder walls. The machine is currently undergoing repairs.

Considerable improvement was observed this month in the amount of resin not meeting manufacturing specifications. This material amounted to 679M pounds or 17.5% of the total production. Of this material, 402M pounds was regraded as prime and 277M pounds was off-grade. Off-grade yield remained high, however, due to the quality problems with 2185.

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Mr. A. K. McMillan
PVC

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November 3, 1970

ANALYSIS OF VARIANCE FOR OCTOBER

The variance for PVC bulk production was \$20,947 unfavorable. This is primarily a result of nonproductive reactor time required for the 5M gallon reactor outage. The major factors are listed below:

Raw Materials	\$(6,358)
Process Costs	1,937
Fixed Costs	(2,633)
Volume	29,375

R. W. Beech
R. W. Beech

AP00046791

ESCAMBIA CHEMICAL CORPORATION
INTEROFFICE MEMORANDUM

To: Mr. R. W. Beech

Date: November 6, 1970

From: D. V. Lowe

Copies:

Re: PVC Process Improvements
Progress Report - October, 1970

CEA 7009 - "Addition of One 5,000 Gallon Polymerizer with
Electrically Driven Agitation to the Existing PVC
Plant"

1. Issued a report (Reference 1) describing shutdown and startup plans, production plans, and objectives. The intent was to record general plans for operation and use as a learning tool.
2. Prepared firm PVC plant shutdown schedules and sequences, and startup procedures (References 2 and 3).
3. Provided shift coverage during shutdown and liason with engineering and construction to assure proper equipment arrangement, installation, and interconnection.
4. Established a list of uncompleted construction items during shutdown week of October 26 and communicated with Engineering to get the jobs done. Several items such as building siding and piping insulation are presently being completed since startup. Two items which will remain uncompleted for another month or so are installation of the automated Yarway drop valve and the two cooling type baffles.
5. Participated in checkout and final preparation of the reactor instrument systems and later prepared operating instructions (Reference 4).
6. The refrigerated water cooling system was removed from the emergency electrical generator and the electric drive for No. 11 reactor was installed on the system instead. In lieu of refrigerated water during power outages, we can use cooling tower water and a reaction shortstop if necessary.

AP00046792

7. Initiated new log and charge sheets for collection of all data on 5,000 gallon reactor operation. Established a central log book, in addition, where results of each run will be entered individually for the next year or so. Wrote recipes for the products to be produced in the new reactor after startup.
8. Tied in the piping for the new refrigerated water storage tank outside the building. There is now a much more constant temperature source of refrigerated water for cooling reactors and operation of the Trane absorption machines is improved.
9. The VCL and H₂O metering systems are working very well and these materials are now being automatically and reproducibly metered into all reactors. The vinyl acetate and trichloroethylene metering systems have not been in demand as yet.
10. The new north rotex is in operation and doing well by all indications. No study of increased efficiency has been made as yet. The north line air heating coils were modified to include three 600 psig steam coils, identical to south line operation. Dryer temperatures at air inlet have increased 50 - 80°F and steam flow control at the higher pressure has been good.
11. The first batch of PVC was charged in the evening of November 1, 1970. No problems of any lasting significance were encountered and the production was prime. (First batch was 4300 gallons of PVC 8217). Full 5,000 gallon batches are now being made with full size catalyst charges. Significant data is already being collected and conclusions about resin quality being formed.
12. Objective during November will be to continue to initiate prime resin production as we encounter each new resin type. Next, as production of types is repeated through subsequent runs, modifications will be made in attempts to improve quality. (As an example, full size charges of PVC 1250 are being made with reduced methocel level and prime resin resulting).

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REACTION SECTION

CEA 7012 - "Automation of PVC Reactor Drop Valves"

Progress resumed when the angle wrench driver was received in the plant. One converted Yarway valve was put in service and works well in the automated mode. Conversion parts for all remaining Yarway drop valves were put on order. A second angle wrench was likewise put on order.

Additional Revisions in Reactor Rupture Discs

Flat-seated scored rupture discs at a higher burst pressure rating of 206 psig were received and put in service. This allowed us to produce PVC 2150 successfully, a high-temperature, low relative viscosity resin. We will continue to specify only this high pressure rating, having received approval from our insurance carrier to do so.

Production of PVC 1200

Reaction cycle time of PVC 1200 has been lowered by 45 minutes via usage of lauroyl peroxide catalyst in conjunction with IPP. There were no process control problems and quality was good.

Steam Desuperheater Tie-ins

During the plant shutdown, tie-ins to the low pressure steam supply to our Trane absorption refrigeration machines were made to allow future installation of a steam desuperheater. There is indication that past crystallization problems may have stemmed from overconcentration of lithium chloride solutions by this highly superheated steam. A quote for such an equipment item is now in hand. We will wait to determine whether or not operation of the new refrigerated water surge tank is going to greatly reduce past problems of this nature. If not, justification for an RFE for capital expenditure will be prepared and a desuperheater installed when approved.

FINISHING SECTION

Trials with Magnet Trays Over Rotex

One new tray of magnets was installed under the south product rotary valve and over the rotex screener to catch iron contamination. A fair test could not be conducted since steam sparge which is used for static charge dissipation caused PVC to cake up in the magnet box and plug. When the

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Trials with Magnet Trays Over Rotex (continued)

humidifying steam is replaced with ionized air for static dissipation, the magnet trays will be permanently reinstalled.

Ionized Air Supply for Rotex Screeners

The ionizing devices for blowing ionized process air into the rotex screens for static removal were received but not installed. After they are installed, steam usage can stop and product resin will be slightly drier. Ionized air could result in lowered tailings yield and increased prime if steam is eliminated and resin remains dry.

North Product Collector Air Grids and Air Supply

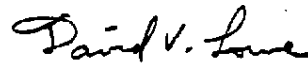
During the plant outage, stainless steel air supply grids were installed in the north product collector. Old ones had badly corroded and contaminated product. The main external air supply header was replaced with a stainless steel line for the same reason. Stainless steel air damper valves were installed in two locations on the same product collector for that reason.

New Product Collector Bag Caps and Nipples

One complete lot of stainless steel bag caps and nipples for the north Dustex product collector were put on order. The nipples on all existing caps in service are cadmium-coated carbon steel and are badly corroded. When these caps and nipples arrive, they can be put in service and the lot which is taken out of service can then be reconditioned with stainless steel nipples. Possession of this extra lot of caps and nipples will permit us to maintain one lot of these out of service at all times. It is planned to repair one lot each month on a round-robin basis until all four lots are new. This also will reduce contamination of resin.

Reinstallation of Automatic Product Samplers

The reconditioned automatic product samplers were installed under the Rotex screeners during shutdown. They now have teflon seals and are designed to extract resin samples without adding to contamination. Not enough time has elapsed to allow us to draw conclusions.


David V. Lowe

AP00046795

Mr. R. W. Beech
PVC

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November 6, 1970

REFERENCES

1. Memorandum to Mr. T. L. Carey, "Initiation of Polyvinylchloride Production in 5,000 Gallon Polymerizer, CEA 7009", from D. V. Lowe, October 1, 1970.
2. Memorandum to Mr. W. J. Kerler, "Tentative PVC Shutdown Schedule", from D. V. Lowe, October 7, 1970.
3. Memorandum to Mr. A. K. McMillan, "Initial Startup Plans - 5,000 Gallon Reactor", from D. V. Lowe, October 29, 1970.
4. Memorandum to PVC Shift Supervisors and Operators, "Operation of Instrumentation for New 5,000 Gallon Reactor and Control Procedures", from D. V. Lowe, November 2, 1970.

AP00046796

ESCAMBLA CHEMICAL CORPORATION
INTEROFFICE MEMORANDUM

To: Dr. W. M. Smith

From: D. J. Mann

Re: Monthly Report
PVC Research & Development
October, 1970

Date: November 2, 1970

Copies:

OPERATIONS

I. Powder Coating Resins

Objective A: To develop a process for manufacturing a MICRO-PEARL[®] fusion resin by direct polymerization having a median particle size of ~60-90 microns and a bulk density of 20-25 lbs./cu.ft.

A. Properties Lab

Efforts were continued during this reporting period to resolve fusion differences reported in the field with those obtained in the laboratory. As reported previously, potential customers have observed the fusion properties of MP-2A to be inferior to those of 4185-2S in certain formulations. Upon further checking, it was found that these differences were most readily apparent in barium-cadmium stabilized systems, since the fusion qualities of both 4185-2S and MP-2A were shown to be excellent with tin stabilizers and definitely superior to those with barium-cadmium. When compared in the latter system, 4185-2S appeared to fuse better. It had a slight gloss, good color, and a few large pinholes, whereas the MP-2A was dull and grainy with many small pinholes and some discoloration.

Presently, studies are underway to determine if these differences are definite manifestations of product quality and, if so, whether they can be corrected by,

1. type of Ba-Cd stabilizer (Ba-Cd ratio)
2. stabilizer level
3. order of ingredient addition in preparing dry blends
4. addition of surfactants (for more efficient wetting)
5. formulation change (i.e., lower pigment level)

AP00046797

November 2, 1970

OPERATIONS (Cont'd)

In the course of pursuing this problem, additional quantities of bagged MP-2A, obtained from the plant, were found to contain much coarser resin than originally represented by the composite sample and so reported by Quality Control. Further investigation revealed that the coarse fraction had an R.V. = 1.50 and that the MICRO-PEARL^R plant trial was conducted immediately after a PVC 2150 run, thereby establishing the fact, with reasonable certainty, that the coarse fraction was a PVC 2150 cross-contaminant.

The following screen data reveal the particle size differences between "pure" and "contaminated" MP-2A.

	Lot #6227 #1	Lot #6227 #2
Screens, % on 60	----	6.0
80	----	18.0
100	----	9.0
120	----	5.0
140	----	3.0
200	4.0	5.0
230	26.0	14.0
270	26.0	14.0
325	24.0	18.0
400	12.0	8.0
PAN	8.0	----

The coarse MP-2A does not fuse satisfactorily in either tin or barium-cadmium systems. It is not known, at this time, whether any coarse MP-2A resin was field sampled and therefore whether this material contributed to the poor field results. This matter will be pursued with Market Development.

Objective B: To develop an improved suspension system which will permit the manufacture of PVC 4185-SP and 3185, respectively, to targeted product specifications at a high level of reproducibility.

A. Plant Trials

In an attempt to prepare PVC 3185 by a more reproducible process, two experimental recipes were evaluated in batch trials, using the following recipes.

AP00046798

OPERATIONS (Cont'd)

	<u>T₂₁</u>	<u>XT-1321</u>
Water	200	200
VC1	100	100
Alperox F	0.16	0.16
PVA/VA I-535 (50% sol'n.)	-----	0.5
Sulframin 85	0.75	-----
MBCP	0.20	-----
Geon 450X3	0.25	-----
PVP K-90	0.20	-----
TCE	0.80	0.80
Temperature, °F	140	140

The first recipe shown (T₂₁-1312) was an outgrowth of the PVC 2185 bottle-grade resin studies (and will be discussed in that section of this report), whereas the second (XT-1321) was a modified MP-2A recipe employing a lower level of I-535. The results obtained are shown below:

	<u>T₂₁-1320</u>	<u>XT-1321</u>	<u>PVC 3185</u> <u>Objective</u>
Screens, % on 30	14.0	----	-----
40	18.8	----	-----
50	12.8	0.2	-----
60	8.6	0.4	-----
80	8.4	2.0	-----
100	8.0	2.0	-----
120	10.4	2.4	(
140	10.0	2.8	(75-90
200	8.0	17.2	(
PAN	1.0	73.0	10-25
Bulk Density, lbs./cu.ft.	29.5	25.3	21-25

The T₂₁-1312 recipe failed completely, yielding not even the median particle size (115 μ) obtained in the pilot plant. The XT-1321 recipe gave resin too fine for PVC 3185 but ideal for MICRO-PEARL^R applications. This resin will be evaluated in the fusion studies reported above versus MP-2A-6227.

II. Saran

Objective: To prepare sufficient quantities of both extrusion and solution grades of saran to permit selective field sampling and laboratory studies.

AP00046799

OPERATIONS (Cont'd)A. Properties Laboratory

Crystallization studies of pilot-plant resins were continued, using the test procedure described in the R&D Monthly Report for September, 1970.

The results obtained to-date are summarized in Table I, with the time defined as "minutes to develop a tensile strength of 1000 psi or greater" at that particular temperature.

The conclusions reached, thus far, are as follows:

1. Plant-produced SV1710E is at least equivalent to pilot-plant SV1710E in crystallization rate and perhaps slightly faster.
2. Plant-produced SV1706E is faster than SV1710E, as expected, but not as fast as one previous pilot-plant resin (3-1171).
3. There is fairly good reproducibility in the crystallization test from batch to batch. The discrepancies observed could probably be explained through chemical analysis.
4. There is, presently, insufficient data to reach a definitive conclusion regarding the effect of MEHQ stabilizer on crystallization rate.
5. A higher polymerization temperature gives a faster crystallization rate.

B. Pilot Plant

Fifteen pounds of an 85/15 copolymer saran was prepared and shipped to Linwood for extrusion studies. Although crystallization studies have not yet been made, a very fast rate is anticipated.

III. Bottle-Grade Resin (PVC 2185)

Objective: To support the efforts to establish a joint working agreement with Fosfatbelaget/Lonza by developing a bottle-grade resin which will match the performance of Lucevyl RE8010 (Pechiney-St. Gobain) in blow-molded applications.

AP00046800

OPERATIONS (Cont'd)A. Polymerization Laboratory

Considerable effort was devoted this month in the bottle laboratory to the study of PEARL^R/FVP K-90 combinations, in the hope that such a system could be developed offering the desired bottle-grade properties and, in addition, a less fouling recipe more suitable for the Fosfatbelaget stainless steel reactors.

Bottle polymerizations were carried out at 140°F with 0.8 part of TCE to give an R.V. = 1.85. The variables studies were,

1. Sulframin 85 and Ultrawet 60L surfactants at 0-0.1 part
2. FVP K-90 (20% solution) levels from 0-0.45 part
3. Geon 450X3 latex levels from 0-0.5 part

The following conclusions were reached from the above bottle polymerization data:

1. All recipes showing a propensity for reduced particle size (M.P.S. $\leq$ 200) gave bulk densities lower than 30 lbs./cu.ft.
2. Sulframin 85 is a more efficient surfactant for particle size reduction than Ultrawet 60L, with or without Geon latex; but it also gives lower bulk densities.
3. In Geon latex recipes, the optimum level of FVP K-90 solution appears to be in the range of 0.2-0.3 part for reduced particle size. As the K-90 level increases, the bulk density decreases.

Considerable bottle polymerization work was done to study the effect of addition order on the PEARL^R/K-90 combinations. With the water added first, all other possible addition orders were evaluated with no significant differences noted. The resin particle size was fairly reproducible regardless of how the emulsifier, salt, latex, and K-90 were added.

B. Pilot Plant/Plant Trials

Although no recipes from the above studies looked encouraging for PVC 2185, several did look attractive as candidates for PVC 3185 or 4185-SP.

AP00046801

OPERATIONS (Cont'd)

Thus, the following recipe was run in the pilot plant three times with the results as shown.

Water/VCl/LPO/ S-85/MBCP/Geon/K-90 sel'n./TCE
 200 100 0.2 0.075 0.2 0.25 0.2 0.8 (T°F = 140)

	<u>3-1273</u>	<u>3-1275</u>	<u>3-1276</u>
Screens, % on 80	2.0	3.0	3.0
100	16.0	16.0	16.0
120	27.0	28.0	23.0
140	26.0	27.0	20.0
170	14.0	14.0	16.0
200	10.0	7.0	12.0
PAN	5.0	5.0	10.0
Bulk Density	25.6	24.8	25.6
M.P.S. (K)	115	115	115

Based on the pilot-plant results, this recipe appeared ideal for PVC 4185-SP. However, when scaled-up during two plant batch trials, this recipe did not translate as expected and yielded instead coarse resin in the region of 250 microns. The reason for this is unknown at the present time.

IV. General Process/Product Development

Objective: To evaluate new processes, ingredients, equipment, techniques, etc., necessary to remain abreast of advanced polymerization technology.

A. PVP K-90

There has been a growing interest on the part of Production in replacing Methocel with PVP K-90, since it offers certain process advantages as well as producing resins having, generally, a lower gel content. During this reporting period, two plant batches of PVC 1225 were prepared as follows,

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OPERATIONS (Cont'd)

Recipe: Water/VCl/ IPP /K-90 sol'n.
200 100 0.0175 0.34

and yielded the following properties:

Screens, % on	80	2.0
	100	21.0
	120	38.0
	140	26.0
	170	8.0
	200	3.0
	PAN	2.0

Bulk Density 30.2

This resin is currently being evaluated versus standard PVC 1225 in the following tests: Brabender flow, Killion extrusion, color and clarity, hot dry blend time, and oven heat stability.

B. PVC 6180 - Incremental Addition Technique

Efforts were initiated to prepare a PVC 6180 copolymer by a gradual addition technique, since it has been postulated that the resultant, more uniformly distributed copolymer should have improved heat stability.

Thus far, copolymers have been prepared with little difficulty using a VCl/VAc charge of 89/11 in which 50 parts of VCl are charged initially and the remainder added incrementally at a constant equilibrium pressure. The resins prepared to date have not shown good heat stability, but only because they contain 13-15% bound VAc rather than the 8.5% typical of PVC 6180. Efforts are currently underway to prepare, through recipe adjustment, a uniformly distributed PVC 6180. In addition, a correlation is being established between % bound acetate and equilibrium pressure for the VCl-VAc system in the pilot-plant reactors.

Similarly, a PVC 6145 (85/15) copolymer was prepared by this technique which hopefully would have better solution properties than the standard batch prepared resin. The first attempts followed a procedure recommended by M. Langsam and yielded a copolymer containing 18% bound acetate. This resin exhibited poor thermal stability on drying as manifested by discoloration. Higher equilibrium pressures are being studied to reduce the VAc content to 15%.

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OPERATIONS (Cont'd)C. PVC 6180 - Melt Flow

Plant PVC 6180 composite samples are continuing to be monitored for melt flow properties on the Tinius-Olsen Melt Indexer. The results appear to indicate that R.V. has a much greater affect on melt flow than the relatively small variances that occur in vinyl acetate content. It is intended that melt flow become a product specification for PVC 6180 after an adequate correlation has been established with either customer melt flow data and/or customer acceptance/rejection history.

D. PVC 2225 - High Bulk Density

In an attempt to prepare a PVC 2225 resin with a bulk density > 35 lbs./cu.ft. for pipe applications, several recipes were studied in the 10-gallon reactor. Initial efforts were directed towards reducing the Ultrawet 60L level in the standard plant recipe to raise the bulk density. Severe wall fouling occurred in all of these recipes with little or no change in bulk density. The bulk density obtained using the plant recipe was 31.6 as compared with a plant range of 32-35 lbs./cu.ft.

By polymerizing PVC 2225 at a pH of 3.5, considerably less wall fouling was noted, indicating a more stable suspension. However, the bulk density also dropped to 22.7. By reducing the surfactant in systems of this type (lower pH), it was demonstrated that the bulk density could be increased; but this system has not yet been optimized.

D. J. Mann
D. J. Mann

/pad

AP00046804

Table ISaran Crystallization Data

<u>Resin</u>	<u>30°C</u>	<u>40°C</u>	<u>50°C</u>	<u>60°C</u>	<u>70°C</u>	<u>Comments</u>
SV1710E X-20	>100	100	32	16	10-11	Standard SV1710E made in plant
" " 3-1117	----	----	----	20	10	Distilled VdCl
" " 3-1118	----	----	----	34	20	" " " " "
" " 3-1119	----	----	----	30	20	" " " " "
" " 5-438	----	----	----	> 40	> 40	MEHQ-inhibited VdCl, .4 IPP (C-D 35 hours)
" " 5-439	----	----	----	14	3	MEHQ-inhibited VdCl, .45 IPP (C-D 21 hours)
" " 3-944	----	----	----	> 36	> 36	Washed VdCl
" " 3-1141	----	----	----	> 38	30	MEHQ-inhibited VdCl
V1710E 5-465	----	----	----	20	7	Distilled VdCl
" " 5-467	----	----	----	8	4	" " " " ", 140°F poly. temp.
SV1706E X-21	>100	80	20	6	4	Standard SV1706E made in plant
" " 3-1171	2	< 1	----	----	----	MEHQ-inhibited VdCl
SV1704E 5-484	45	6	----	1	----	" " " " " " "
SV1804E 5-485	45	10	----	1.5	----	" " " " " " "
Dow 864	8	2-3	----	----	----	" " " " " " "

ESCAMBIA CHEMICAL CORPORATION
INTEROFFICE MEMORANDUM

To: Dr. W. M. Smith

From: W. K. Clark

Re: Monthly Summary
PVC Technical Service
October, 1970

Date: November 4, 1970

Copies:

I. Customer Service

A. B. F. Goodrich

On October 9, 1970, P. R. Browne visited this customer's plant to ascertain the validity of their rejection of approximately 16,000 pounds of PVC 6180. A number of samples were tested via the Goodrich gel solution test and found to have a very high gel content. Further work is being done between the two laboratories to eliminate future rejections. (Reference 1)

B. Lynn Vinyl Plastics Corporation

P. R. Browne made a courtesy call at this customer's plant on October 9, 1970 to discuss the developments of our new low R.V. 2150 resin. As a result of their laboratory tests, we have shipped one lot of low R.V. 2150 for plant trials and subsequent customer acceptance. (Reference 2)

C. International Shoe Corporation (TSR 849)

As a result of a technical service visit to customer's plant in Bryan, Texas, the following progress was made.

1. Poor extrusion rates were found to be due to the excellent dry blend flow of PVC 2200 blends. Flow was excellent but "grab" was poor.
2. Mechanical and condition changes were made which resulted in 10%-15% increase in output.
3. An improved screw design was recommended along with additional changes for an estimated increase of 30% in production rate and improved quality.

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I. Customer Service (Cont'd)

4. Validity of a number of the recommendations above will depend on accuracy of present amperage and pressure measurements on their equipment. Present measurements indicate very low head pressure and low amperage loads. (Reference 3)

D. Evaluations

A preliminary report was issued on the dry blending characteristics of our PEARL^R resins versus the Lucevyl bulk-polymerized resins using the Brabender ASTM powder mix test and Hebart hot dry blending test. Preliminary results showed our PEARL^R resins to be comparable overall with the Lucevyl bulk resins in dry blending characteristics. (Reference 4)

E. Owens-Corning

Per V. J. Grande's request, we made one fusion blend for this customer incorporating ECC resin. (Reference 5)

F. Anger-Clew

Pipe resin evaluation and development evaluations to date indicate that BK-75, the Goodyear bulk polymer (now the preferred resin for Anger), has an extremely fast-fluxing rate in Brabender tests. This in spite of Anger claims that a fast-fluxing resin is not desirable. Present indications are that relatively minor changes in resin protective colloids will necessitate appreciable changes in compound formulations with respect to lubrication.

A trial date for the resin(s) chosen will be arranged with Anger. Further Brabender trials will be made prior to Anger trials.

G. Southwire Company

Two samples of unidentified competitive resins from this customer were evaluated and reported. (Reference 6)

H. Abbett Laboratories

This customer requested and was supplied with general information regarding formulations for medical tubing. (Reference 7)

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November 4, 1970

I. Customer Service (Cont'd)I. General Tire and Rubber

Certain quality control results on five shipments of PVC 1250 were submitted at customer's request.

J. Tenneco Chemicals, Inc.

At customer's request, we submitted certain quality control results on six shipments of PVC 6180.

K. Kerr Glass Manufacturing Company

This customer requested certain quality control results on sampled PVC 2185. (Reference 8)

L. Ohio Sealer and Chemical Corporation

Above customer requested certain quality control results on PVC 3185 and PVC 4185-28. (Reference 9)

M. Killien Blow Molder

We have received notice that our Killien blow molder has been shipped as of the last of October. It is hoped that the machine will become operational by the end of November.

II. Technical Service to APCIA. Battery Separators

A proposal for preliminary laboratory work prior to special resin development has been made to Market Development. (Reference 10)

B. Experimental Plate Extruder Design

1. Several design parameters have been worked on with respect to ease of manufacture.
2. Verbal expressions of interest in building initial prototype have been made by Killien Extruders and Hartig Extruders.

III. Samples

A total of 60 resin samples (274 pounds) and one dry blend (8 pounds) was shipped to 23 companies including APCI.

WKC/pad

W. K. Clark
W. K. Clark**AP00046808**

- Reference 1 Report dated 10-20-70 from P. R. Browne to W. K. Clark
- Reference 2 Report dated 10-27-70 from P. R. Browne to W. K. Clark
- Reference 3 Report dated 10-30-70 from W. K. Clark to W. M. Smith
Letter dated 10-30-70 from W. K. Clark to Mr. John W. Juett, International Shoe Corporation
- Reference 4 Report dated 10-20-70 from R. E. Kunc to W. M. Smith/W. K. Clark
- Reference 5 Memo dated 10-6-70 from P. R. Browne to V. J. Grande
- Reference 6 Memo dated 10-14-70 from P. R. Browne to J. L. Harvey
- Reference 7 Memo dated 10-2-70 from W. K. Clark to P. F. Baker
- Reference 8 Letter dated 10-22-70 from P. R. Browne to Mr. Clarence Rehner, Kerr Glass Manufacturing Company
- Reference 9 Two letters dated 10-23-70 from P. R. Browne to Mr. Harvey Hepner, Ohio Sealer
- Reference 10 Memo dated 10-15-70 from W. K. Clark to A. R. Adams/V. J. Grande

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