

MONSANTO CHEMICAL COMPANY
SPRINGFIELD, MASSACHUSETTS

May 26, 1961

TO: R. A. Coffey (2)

AT: PVC-PTS

CONFIDENTIAL

SUBJECT: FINAL REPORT: EVALUATION OF
6.5% DILATOMETER POLYRATE VCM
ON OPALON 330 - P88-60

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INTRODUCTION

Monsanto at Texas City has undergone equipment and process revisions which gives them a process capable of producing vinyl chloride monomer at about a 6.5% dilatometer polyrate level compared to the early 1961 average of 5.2 - 5.5%. Before placing the entire polymerization system on this higher polyrate VCM, this P-Number was authorized to try to predict any effects it might have on homopolymer (300 series) polymerization and resin quality. Of specific interest were the effects of high polyrate VCM on resin particle size.

SUMMARY

Four batches of Opalon 330 were made in late February, 1961 using unscrubbed 6.5% dilatometer polyrate VCM produced by Texas City for Springfield evaluation. For comparison three routine batches of Opalon 330 were picked for control purposes. The SDP for Opalon 330 was used throughout the study. Slurry samples of these seven batches were taken for analysis by the control lab. for screen, bulk density, kitchen mixer dry time and extrudability. Virgin and recovered monomer samples were taken from selected batches and tested by dilatometer polyrate and gas chromatography.

CONCLUSIONS

1. Based on the results of this study, 6.5% dilatometer polyrate VCM should give the following polymerization and resin properties for Opalon 330:

- A. Screen - significantly coarser resin should be produced. Vacuum adjustment will probably be needed.
- B. Bulk Density - slightly lower

- C. Extrudability - normal grade, slightly lower rate.
- D. Dry Time - no significant change
- E. Cycle Time - no significant change at the same vacuum

2. There is poor correlation between Springfield and Texas City on dilatometer polyrate testing.

3. Subsequent plant experience with VCM up to 6.2% polyrate confirmed the shift to coarser particle size, but vacuum adjustment was inadequate to bring particle size in specification.

DISCUSSION

Number five washed monomer tank was segregated for this study. The tank cars of 6.5% dilatometer polyrate VCM were unloaded directly to this tank, bypassing the tank farm tanks and the caustic scrubber. Lines common to standard Texas City monomer had to be used for this operation and in kettle charging. For this reason all kettle charges using 6.5% VCM were contaminated with approximately 5 - 10% standard VCM.

Particle size for Opalon 330 in 88 Bldg. was fluctuating during the latter half of February, tending to run on the coarse side. This was attributed mainly to a higher VCM dilatometer level (above 5.7% average compared with a past normal of 5.1 - 5.5% average). During the experimental study the particle size of the individual batches became variable (reason unknown) giving very flat blend distributions as fine and coarse batches were mixed.

A product change in 88 Bldg. necessitated curtailing the experimental plan and using additional kettles for experimental and control batches. The three standard batches picked for sampling for controls were all out of specification (too fine) on resin particle size. It is felt that this must have been a coincidence as the product blends do not reflect a series of fine batches during this time.

The four batches produced with 6.5% VCM had an average SAF of 99. Using the control batches slurry SAF average of 181 or the routine production blend average SAF of 136 it is obvious that the experimental batches were significantly coarser. Although two of the experimental batches were out of specification (coarse) on slurry samples, all experimental blends were within specifications (+40 - mesh resin scalped off).

Extrusion grades were normal, however the rates were slightly lower than other samples extruded at the same time (196 vs. 225). No direct comparison can be made between the resins made with the two types of VCM used because the control lab. could not blend the control samples for extrusion (particle size too fine).

Kitchen mixer dry times for the xperimental batch s were normal. The dry times for the control batch s were out of specification due to the fineness of the re in particle size.

The Springfield dilatometer results compared to Texas City dilatometer results were further evidences of the lack of correlation between the two control labs. Sampling was determined to be a possible cause of part of the discrepancy. Samples taken from the unloading line during the unloading of the tank cars tested higher and closer to Texas City's results than those taken from the diptube. Sampling procedures have been changed and all routine samples are presently being taken during the unloading operation. The results of the unloading sample of GATX 37873 proved that there was also difficulty in the test itself. This sample of a car which originally tested 6.48% in Texas City gave a value of 5.63% in Springfield. The sample was then sent to Texas City where an average value of 6.31% was obtained. Cause of the discrepancy is presently being pursued by the two laboratories.

One limitation of this experiment was that the effect of vacuum adjustment was not tested. During the two weeks after this experiment the dilatometer blend level was steadily increasing to a high of 6.2% (based on Texas City tests) as Texas City had difficulty maintaining a lower polyrate level. The 300 series resins produced during this period became too coarse in particle size to meet specifications and they could not be brought back into specification by the normal method of lowering the vacuum. This problem was finally circumvented by converting the building to the 600 series resins.

R. R. Saxton
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APPENDIX I

Ba No.	Kettle	Cycle Time Hr.	Kitchen Mixer Dry Time Min.	Extrusion Grade	Rate	Resin Bulk Density	SCREEN						Thru	
							40	60	80	100	140	200	Pan	100 SAF
2150	1	8:45	5:00	W2B-5	204	.459	1	16	46	25	10	2	2	14 95
2160	1	9:45	4:30	W2C-6	174	.454	3	6	30	34	23	4	2	29 108
2167	1	8:45	4:30	V2C-5	204	.436	T	11	51	23	12	2	1	15 94
2168	2	9:00	4:45	V2C-5	203	.411	T	5	48	28	15	3	1	19 99
Ave.		9:04	4:41		196	.440								99
2153	4	8:45	20:00+	--	--	.478	T	1	1	8	31	29	31	91 188
2155	1	8:55	12:00	--	--	.415	T	1	1	7	35	36	20	91 179
2165	3	9:00	15:00	--	--	.451	T	1	1	6	40	34	18	92 176
Ave.		8:53	--	--	--	.446								181

All batches 23 1/2" vacuum
Lot 357 PDR-1

Other batches produced under same conditions

2146 1 8:55
2148 4 8:45
2154 3 9:00
2159 3 8:55

Blends produced during experimental study:
Routine:

61055	.467	T	12	21	24	21	12	10	43	126
61056	.487	T	6	28	19	18	14	15	47	136
61058	.470	T	1	11	13	29	25	18	72	158
61059	.464	T	4	29	26	21	10	9	40	124
Ave.	.472									136

Experimental:

61057	(Batch 2150)	.459	T	13	49	22	12	3	2	17	97
61060	(Batch 2160)	.454	T	4	27	33	25	7	4	36	116
61061	(Batches 2167 & 2168)	.439	T	5	43	28	16	5	4	25	110
Ave.		.451									108

APPENDIX II - VCM DATA

Impurity	6.48 GATX 37873 (222A)	6.47 GATX 76245 (2)	Batch 6.48 Virgin 223A	2160 6.48 Recovered 223B	Batch STD (5.77) Virgin 223C	2165 STD (5.77) Recovered 224B	Batch 6.47 Virgin 224A	2167 6.47 Recovered 224C
C-2								
Propane	6.0	1.4	3.2	14.8	1.4	7.2	3.8	16.4
Propene		2.1						
Isobutane	132.	88.88	86.6	297	100	304	100	323
n-Butane	257	228	176	525	212	522	200	509
Butene-1, Isobutene	1.8	1.6	1.7	5.2	10.0	17.6	1.6	6.8
t-Butene-2	2.2	2.2	1.8	4.3	4.2	7.4	1.8	4.4
c-Butene-2	0.6	0.4	0.3	0.7	0.7	1.0	0.4	0.6
Isopentane			0.2					
1,3-Butadiene	9.7	9.0	7.5		17.0		7.6	0.4
Methyl Acetylene	4.0	2.5	2.8	9.0	2.4	6.4	3.0	7.4
Methyl Chloride	21.4	13.3	14.4	42.4	17.6	40.8	17.1	41.0
VCM	∞	∞	∞	∞	∞	∞	∞	∞
Ethyl Chloride								
2-Chloropropene								
U-6							0.7	
U-3			0.6					
U-4							2.4	4.9
Vinylidene Chloride								
Dilatometer Polyrate (average of 2 Springfield Lab)	5.64%	5.82%	5.35%	4.90%	5.36%	5.73%	5.07%	5.74%

Impurities are recorded in mm. peak height - gas chromatograph data (G.L.P.C.)

Dilatometer data (Springfield individual values %)

GATX 37873 6.48 Texas City Test

Diptube sample 5.20, 5.24, 5.30 5.25 Ave.

Unloading sample 5.48, 5.78 5.63 Ave.

GATX 76245 6.47 Texas City Test

Diptube sample 5.22, 5.29, 5.33, 5.34 5.30 Ave.

Unloading sample 5.78, 5.87 5.82 Ave.