I-184 (50M)

INTER-OFFICE MEMO **TENNECO CHEMICALS, INC.**

APR 10 1969

To M. W. Williams At Piscataway

From P. F. Albanese At Flemington

Subject MONTHLY REPORT - MARCH, 1969
PROCESS DEVELOPMENT

Date April 7, 1969

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RAW MATERIALS

MVC

A 42,000 gallon tank car of Allied MVC and 9 tank cars of PPG monomer were processed through the Burlington site as part of a monomer evaluation program. All of the data gathered is still under review and at this point, the PPG monomer is comparable to good Tenneco monomer. The major significant effect noted was the quality of the Allied recovered monomer which gave a noticeable improvement in reaction kinetics over the Tenneco monomer. A report will be issued shortly with a detailed list of savings that could be expected with a better quality monomer.

In-process monomer sampling, to more closely relate to process performance, is being delayed because of the heavy load of monomer samples resulting from the Allied and PPG evaluations.

A memo was issued detailing the cost savings that could be ascribed to a decrease in propadiene level which was noted during the early part of February. The cost savings reported was based on the improvement in reaction times noted for 1730 and calculated to represent an addition to annual profits of approximately $75,000.

P.E. has been monitoring the storage life of Lupersol-11 and catalyst storage facilities in Burlington. After one month storage, the Lupersol-11 was in specification for activity and the storage temperature was held below 28°F. It is planned to continue with this study.

It was reported in the last report period of the number of unusual occurrences in PVC reaction behavior at the Burlington site; poor reactions and long dry blend times. Observations to date have noted some improvement and more consistent homopolymer quality; coarser particle size, low SHDBT and higher bulk densities which also coincides with the dispersion resin plant using their own recovered monomer.

DHW-80

No change in status.

MC-15 Makeup

Centrifuge studies were completed and an interim report (LOP 729) was issued. The data shows an overall reduction in gel level and a reduction in the number of large "fibers" but not appreciably affecting the total number of "fibers." Data is still being collected to determine if the improved filtration of water has also had an effect on the gel level.

Solution cooling of MC-15 is still being considered for a trial run at Burlington. Engineering has been requested to estimate the cost of providing the Methocel make-up tank with "brine" water.

PRODUCTIVITY IMPROVEMENTS

Suspension

2-EHP - Two single-batch runs were made this period; the first into a cleaned reactor followed by a second into the same reactor uncleaned. Both batches were sluggish and were dumped early with the prime suspect being the activity of the 2-EHP. Q.C.'s evaluation of the catalyst showed a 25% reduction in activity. Although the data may be meaningless, the most critical property, gels, was in spec, 2 and 8, respectively. Large-scale runs will be made using 7-batch lots as soon as a new shipment of 2-EHP is received.

IPP - A program was started in the Flemington plant on evaluating the use of IPP as a possible raw material savings in catalyst cost. It is planned to conduct the IPP program in Flemington whereas the 2-EHP will be studied in Burlington. Information and data on the Flemington runs will be reported during the next report period.

Aposet 435 - No change in status. Apogee was notified of the Lucidol patent on Lupersol-11. We have not heard Apogee's comments on the patent, and at this point, if the patent is valid, there is not sufficient incentive to replace Lucidol with Apogee's material.

EHP - Reflux - No change in status. It is not planned to conduct any development work in this area pending the return of Tenneco Plastics personnel from Tenneco Oil.

Increased Reactor Loading - Burlington and Flemington Production are continuing with their programs via LOP to increase reactor loading. The monomer charge for the 315 formulation was increased, however, due to excessive vibration in the copolymer centrifuge, the charge was lowered again. At this point, we have no reason to believe higher monomer loading is the cause.

250-2 - No lumpy batches were encountered this period.

Dispersion Resin

Sodium Bisulfite - At 0.03 phm of sodium bisulfite in our 1740 formulation, induction times were eliminated and cycle times correspondingly reduced. A memo was issued reporting a savings of $63,000 per year based upon the current 1740 production rate. The evaluation is still in progress on an every-other-batch basis for the 1740 formulation. The program has not been started for the 1730 formulation or on the addition of sodium bisulfite to the emulsifier.

Recovered Monomer - LOP 746 was approved to evaluate the effect of recovered monomer on dispersion reaction kinetics and product properties; a maximum usage of 2,000 lbs. per batch is authorized. Samples of the recovered monomer will be submitted for G.C. analysis and bottle polymerizations.

Two vs. Three Stage Homogenization with 1740 - LOP 725 was issued to evaluate two vs. three stage homogenization and the effect on resin properties, especially Brookfield viscosity. Although all data has not been evaluated, three stage appears to produce resin with one day Brookfield viscosities approximately 5 poise higher than that produced with two stage.

Lots 939011 thru lot 939014 have been evaluated. None of the eight pallets produced via three stage had one day Brookfield viscosities lower than 45 poises, while five of seven pallets produced via two stage had one day Brookfield viscosities lower than 45 poises. As soon as time permits, a report on this LOP will be written.

Antifoam DF-190 - No action this month.

DRY BLEND RESIN PROCESS

A status report is being prepared and will issue the first week of April. Results to date and recommendations for future work will be included. It is now apparent that large particles alone do not produce a dry blend resin. Agglomeration of fines caused by the presence of a surfactant is required to produce the required dry blend and dry flow properties.

The program was expanded to also include a 200 type resin primarily to obtain a less expensive resin for Elm Coated Fabrics. There is insufficient data available at this time to base any conclusions.

VP-631

The second batch charged during the present plant VP-631 program did not initiate and had to be recovered. Complete coverage of the batch was provided by R & D and the reason for not initiating is not known. A follow-up batch, the third in the series, was charged after taking certain precautions of eliminating only traces of recovered monomer entering the charge. This batch failed to initiate during the prescribed time period but did initiate after raising the temperature ten degrees. The resulting latex gave acceptable blending resin properties after being processed in the modified 10MM line, however, after 3 days, it had to be scrapped because of poor latex stability. A more detailed report will follow during the next report period.

BLENDING RESINS

Copolymer Blending Resin

Nine batches of copolymer blending resin were run under NPI Extension 733-1. This run was our first attempt to dry and screen this product through the large copolymer line. Two batches of lot 799004 started processing using a 100 mesh screen, but screen rejects ran high at approximately 30%. The balance of this lot was dried into a bin and held until later when it was screened through production's standard 50 mesh screen.

The seven batch lot (799005) was processed through 80 mesh to reduce tailings and provide Wheaton with a truckload of resin. We had 16% tailings and an average drying rate of 2,700 lbs. per hour. An Interim Report (NPI Extension 733-1 Copolymer Blending Resin dated March 25, 1969) was issued. Slurry samples from each batch have been forwarded to the Pilot Plant for drying and subsequent evaluation by Surface Coatings Group.

Based on the low production rate and the screening problem on the 40MM line, it is recommended future development work be done on the 10MM line.

Additional runs are planned pending Wheaton's evaluation of this latest resin and their subsequent orders. As time permits, classifying and screening studies will be conducted on the resin produced during this latest run.

The addition of silicone to a copolymer formulation has shown a tendency towards a finer particle with less agglomeration taking place. This was also evaluated in the Pilot Plant using a copolymer blending resin formulation with the expectation this would improve the fineness and decrease the amount of agglomeration. Samples were submitted to Product Development for evaluation.

Medium Particle Size, PD-03333

No change in status.

Coarse Particle Size, PD-03334

It was also planned to evaluate processing on the 40MM lb. line with PD-03334. However, based on our recent experience, development work for this resin should be done on the 10MM lb. line. NPI Extension 677-3 is circulating for approval to run four batches. This will be done following the copolymer runs.

Fine Particle Size

No change in status. No additional work is planned in the Pilot Plant pending the return of A. C. Shah from Tenneco Oil or until the modified 380 resin program progresses to a satisfactory end point.

AMP's

The rough draft covering the formulations and process conditions for the Flemington plant is completed. This draft will be reviewed with Production within the next report period.

The 1716 AMP was modified so that unless a special request is made, lauryl alcohol will be used. For a specific non-FDA use, Production will be specifically requested to use tridecanol. Each of the variations are on separate AMP's with different formulation numbers.

NEW PRODUCTS

Dispersion

No work was done in this area at the Plant or Pilot Plant level.

1738 - Polymerization work is pending Product Development's evaluation of a 1738-type resin by means of an intermediate reaction temperature and TAIC.

0565 - Samples were submitted to Product Development representing 0565 that was polymerized at a lower temperature. This approach was taken to improve the rheological properties since previous work on 1738 had shown an increase in gel time with a decrease in polymerization temperature.

Suspension

Modified 380 - NPI 737 was issued to produce a faster fluxing resin than the previous attempt, NPI 695. A stringent clean-out, a reduction in Methocel level and the elimination of a forced heat kick were incorporated as an attempt to lower the fusion time. Product Development found the resulting resin to be an improvement over the previous run as far as cross-contamination was concerned, however, the fusion time was not significantly improved to the point where it would be acceptable. The Pilot Plant is presently studying the effect of agitation and colloid level on particle size, bulk density and fusion time to determine if this will lead to further improvements in fusion time.

An additional program was initiated to develop an absorptive α-olefin modified 380 by way of a surfactant/colloid system. Runs made to date have given comparative fusion times to Escambia 6180Y and at times has surpassed the Escambia resin. Work is needed to optimize the formulation in terms of reproducibility, reactor cleanliness and process controllability. Agglomeration at certain formulation conditions is a problem. Also, for some unknown reason, kinetics appear to be poorer with the surfactant system.

Film Grade Resin - 17F - TAMI's evaluation of the 6,000 lb. sample of 17F (589008) showed it to be within tolerable limits for their "Rolla-Glass" operation. They requested a 10,000 lb. sample of another lot of 17F in order to make a final decision based on our ability to reproduce. A second attempt to make this resin resulted in over 150,000 lbs. of resin (lots 589013 and 589014) comparable in properties to lot 589008 based on Q.C. properties. Product Development is presently evaluating this lot along with in-process samples.

Twenty-four thousand lbs. of lot 589008 was shipped to Edison Plastics after this resin was released by CSL and P.D.

Polymix - No change in status.

Tenneco 10R - No change in status.

Solution Grade Resin (VYHH type) - No additional work will be carried out until the FDA status of the Ganex V-816 is clarified. A 10 lb. sample of batch 10-3120-C has been sent to C. W. Johnston for transmittal to GAF for extraction studies.

Fast Flowing 150 Resin - A scaleup to a 200 gallon reactor was made to provide sufficient quantities of a fast flowing low relative PVC resin for customer evaluation. Although the relative viscosity was the same as a previously-accepted batch, the resin had twice the melt flow expected. Additional runs are needed at a revised level of TCE to provide the desired melt flow.

16RS - Reduction of Fines - No change in status.

Graft Copolymers - A graft copolymer with 10% Hypalon 20 was attempted in the Pilot Plant. It is planned to go to a 5% graft since a considerable amount of undissolved and unreacted Hypalon 20 was present in the final product.

PVC 300 - No change in status. No further work is planned until receipt of the expected orders for this resin.

180/11R - NPI 742 was issued to produce a resin with properties somewhere between 10R and 12R plus improved heat stability and a slightly coarser resin. This resin is expected to meet the requirements for FDA bottle formulations. All target properties were met with the exception of heat stability, 98% vs. 100%. Resin is presently being evaluated by CSL and Product Development.

PRODUCT REFINEMENTS

Tenneco 389 - Two SPA extensions were issued, 735-1 and 735-2. The primary objectives were to better define the particle size requirement at Capitol and to also lower the relative viscosity so as to lessen the dependence of extrusion time on particle size. The resin objectives were met and we are presently waiting for an evaluation at Capitol.

Modified 12R (SPA 728) - We have not received any feedback on the special resin prepared for a compound shipment to Sloane.

Modified 315-2 (SPA 743) - Marketing requested a finer particle size version of Tenneco 315-2 so as to sample to GAF. Approval at GAF would mean an additional 5MM lbs./year of business at GAF. The SPA was issued and approved, however, because of the tight scheduling in the copolymer plant, this resin has not been made.

PVC 200 (SPA 724) - SPA (724-1) was extended so as to produce a lower cost resin by reduced venting. The extension covered the period from 2/25/69 to 3/25/69 during which 25 lots were produced in the No. 3 Plant, exclusively. Since there have been no customer complaints on this resin, it is expected that the longer dry blend times will remain in effect.

NEW DISPERSION RESIN PLANT

Process Engineering has advised Production on the excessive surge resin capacity in their dispersion resin bagger hoppers. The capacity of the new hopper is 800 lbs. vs. 200 lbs. in the old bagger. As a result, the additive system can only be checked within ±40%. The recent erratic resin rheology could be the result of our inability to check the additive levels. An attempt will be made to lower the high level alarm to the halfway mark. However, even at this level, we could only expect to be able to control the additive system within ±20%. If control continues to be a problem, a more expensive modification may be necessary, e.g., install a hopper scale.

P. F. Albanese

PFA:em

The above next burdens could become additional ~~...~~ if ~~...~~ right ~~...~~ distillation columns were installed in the MVR area. To explain further, ~~...~~ addition of the Allene Cut to the nitrogen monomer from 100 to 5 would translate to a ~~reduction~~ in the recovered monomer from 15% to 75%. In essence, the recovered monomer would be equivalent to the Pennco monomer we are presently using. ~~In the long run, it will be to the complication~~

An alternate solution for effectively lowering impurity levels in Recovered Mon

A n alternate solution would be distilling recovered monom at Tinker Plas- however ~~...~~ complex product - mix would make this cumbersome.

Discussion

[illegible] increased incentives? If these rention time differences are [illegible] and I feel that they are conservative, we [illegible] would save about 2¢ minutes less per [illegible] light end free increased and design over most. This would directly translate to a 1M pound per year increase or reduction of the extra time used to make 225/16K resin. This would amount to a $5000 per year profit increase and a $23,000 per year savings in direct and labor costs because of greater reactor efficiency. It would interesting to note that the additional 1M pounds of resin that we cannot produce because of the limited monomer we must use, would require ~~an incremental capital~~ an investment of $100,000 (based on 11¢/pound investment) to increase the plants capacity.(4) (about the same cost of installing a light ends column in the PVC unit at Houston)

Insert (2)

The Tenneco reaction times increase as more recovered monomer is charged per batch because "light end, reaction inhibiting" impurities are concentrated 7 to 50 times in the recovery. It is interesting to find that, in general, saturated impurities, such as propane and butane, concentrate about 30 to 50 times. Unsaturated impurities such as Vinyl acetylene, concentrate about 9 to 15 times, indicating that some are being used in the reaction. 1,3 butadiene is completely used in the reaction and does not concentrate. When Phillips ~~monomer was used, the reaction times did not increase as drastically with increased recovered monomer charge as did Tenneco.~~

For the same evaluation, average reaction times of 225 NOK (6-tek) were plotted as a function of the average amount of recovered monomer charged per batch (See Fig 5). Batch points were determined by averaging the reaction times of all batches, segregated by the amount of recovered monomer per batch, since the first of the year. The Allied points were determined by averaging the reaction times during the evaluation. [The reaction time increased as more recovered monomer was charged per batch, ~~reaction inhibiting impurities~~ were concentrated] to 50 [INSERT] The Allied reaction times do not increase as ~~drastically as~~ the recovered monomer charge [this trend is verified by bottle polymerization tests using Allied

~~For~~ the above chart ~~was~~ constructed to show that the ~~above~~ chart was truly representative. (Fig 4 ~~above~~) All of the E grade resin ~~copolymers~~ and copolymers were plotted chronologically over the years starting E grade. Upon superimposed statistically, were the same results as above. In general these charts show the same trend as above. For example, PP recycle recovered monomer appeared to have no detrimental effect on any of the copolymer types. PP monomer appeared to have a problem effect on 315 PTU's (5 out of 6 E grade), but this will be discussed later. All other E grade lots ~~on~~ whether polymer and copolymer appear to be for some reason other than monomer quality (~~or~~ contamination, ~~odorless viscosity volatiles~~). No - To determine, etc.

The easiest method to determine what is being sacrificed by the ~~high~~ level of light and impurities in ~~tonnage~~ monomer is to run a ~~copolymer~~ ~~monomer~~ that is known to be free of light and impurities and compare resin quality ~~and kinetics~~. Then ~~what is done~~ use the Allied and Monomix evaluations.

It was felt necessary that no similar trend could be seen from the 315° capacity results due to the use of potential 'skewed' readings. However, sufficient testing existed to find any trends during the period.

However, this could be directly related to virgin monomer quality.

In spite of this we need to note that during the period

of the experimental increased viscosity was being used in

the experience, there was a definite lowering of gloss and

dry blend time, lack of specification occurred, the

NOT

resin. At the same time a favorable increase in impact

strength also occurred. Since resin quality on homopolymer

drastically improved during this period, it appears

that the concentrating of light end impurities in the

plastisol process has a tremendous effect on the

resin properties when used in homopolymers. The fact that

Discussion

During the months of January and March, 1969, the monomer and resin qualities were monitored in an effort to determine the sales dollar losses due to unreacted and excessive impurity levels in Tennessee virgin monomer. Both the Allied and PPG monomer evaluations took place consequently a good basis for comparison existed (during this period).

Monomer Quality

Three unsaturated impurities, Allene, methyl acetylene and 1, 3, Butadiene, were monitored. Their concentrations were determined by graphing Houston's GC analysis of the monomer tank car, based on the day it was unloaded into the monomer storage tanks (see Fig 2). I feel that this is a reliable technique since good correlations has been shown between Houston and Burlington G.C analyses.(6)

During the two months period, the concentration of Allene peaked around the first of March at a level of 140 to 150 ppm. At the beginning of the period the level remained fairly constant at 50 60 ppm and at the end of the period the level remained at about 75-100 ppm. The 1, 3 Butadiene (concentration) on

the other hand, peaked at 7-11 ppm about the middle of the manway in the end of the second, the other remained low, at a level of 3-5 ppm Methyl Acetylene did not change much at all it ranged between 25-40 ppm.

The third monomer evaluation started on February 25th, and ended on February 27th. One 42,000 gal tank car was involved in the ~~evaluation~~. The GC analysis indicated that the monomer was "clean" of known type light end impurities. The monomer did, however, contain 150 ppm of methyl chloride[2] (~~the high level is~~ peculiar to the oxy-chlorination process). Since methyl chloride is saturated, (~~it is not as reactive and~~ ~~it is not~~ ~~does~~ should not affect the polymerization as does alkene or 1, 3, butadiene.

The PPG monomer evaluation took place between the 25th and 21th of March. Nine tankcars were involved in this evaluation. G.C analysis of every other car indicated it was also clean of light end impurities. The methyl chloride concentration ranged between 35 and 50 ppm.[2] The monomer, however, appeared to be heavily contaminated with unidentified heavy end impurities. Based on the

performance of PPG monomer in the plant, it appears that these impurities are affecting the reactions of light and impurities

RESIN QUALITY

During the period, several aspects of resin quality were monitored in an effort to locate trends which would correlate to the above plotted monomer quality. For example, figures 2 and 3 show 225/16R and 315 resin qualities, respectively, plotted chronologically. Also, dry blend time and +100 mesh sieve analysis were monitored ~~for the Dispersion~~ because these ~~qualities~~ were most often out of specification. PTU and +100 sieve analyses were plotted for the 315 resin for the same reason. These two resins were chosen to be monitored because they are most frequently made at Burlington and it was felt that their quality would be most indicative of plant wide quality. ~~Superimposed~~ Chronologically were other events that may affect resin quality, such as the period that the copolymer plant used plastisol recovered monomer, the Allied and PPG monomer evaluations and the date after which plastisol recovered monomer was used by the plastisol plant.

Definite trends were found in the 225/16R plot.

Possible [illegible] project and reduced [illegible]	$24,000	$110,000	[illegible]
	$41,000	$270,000	[illegible]

PPE and Allied monomer were very clean, light and comparable as compared to Tenneco. However, based on bottle polymerization tests and GC analyses, the PPE monomer was considerably more contaminated with heavy end impurities so much so that it appeared to have as detrimental an effect on reaction kinetics and resin properties as Tenneco impurities.

RECOMMENDATIONS

Digest

A rigorous study of monomer quality and its effects on reaction kinetics and resin properties was initiated during February and March, 1969. It was concluded that about $1,200,000 (annualized rate) was lost due to varying and excessive impurity levels in Tennessee monomer. Based on this study it is recommended that a light ends distillation column be installed in the NVC unit at Houston to insure a stable and low level of impurity. No direct correlation between virgin monomer purity and product quality could be found. However, longer reaction times were experienced in homopolymer due to highly contaminated recovered monomer and in plastisol due to high and variable impurity levels in the virgin monomer. This translated to an annual loss in production of 1.6 MM pounds. Competitive monomers were evaluated during the study. In general, Allied monomer (low in light and impurities) gave better kinetics and resin properties than Tennessee, especially when using recovered monomer. PPG, on the other hand, (low in light and impurities, but substantial heavy end impurities) gave kinetics and resin properties very similar to Tennessee with increasing amount of effort ~~[illegible]~~ spent on stripping

[illegible] quality and its effect on [illegible] and item [illegible]. It is anticipated that $69,000 would be spent this year.

Conclusions

Based on a two month study of monomer quality [illegible] quality, a total of $ 121,000 [illegible] has been attributed to poor monomer quality [illegible] and recorded. By using Tennessee monomer 1.7¢/pound [illegible] chemical production is being saved [illegible] production due to extended reaction times. This translates to an incremental capital expenditure of $270,000 to bring the plant up to the capacity. It is also interesting to note, that based on 200 M pounds a year monomer usage, the above losses represent a [illegible] .06¢/pound of monomer used. This indicates that perhaps we are paying [illegible] premium for Tennesco monomer.

(Covering [illegible] pounds)

	Savings and extra profits	equivalent incremental Capital invest.	Savings and extra profits / Monomer used
1. Possible yearly savings by not using [illegible] and in Copolymer resins	$3800		2×10^{-3} ¢/L
2. Possible extra profit due to shorter reaction times in plant as based on monomer study	$82,000	$1,60,000	4.1×10^{-2} ¢/L
3. Possible extra profit due to Tenn E-grade 225/16C due to better monomer quality	$ 7800		3.65×10^{-3} ¢/L

POLYMER (PVC) CONVERSION TEST

WITH DIFFERENT VCM SAMPLES

Reagents: To 75 cc. of 0.5% Sodium Lauryl Sulfate add 0.25 grams of $K_2S_2O_8$ in 10 cc. of H_2O and 25 grams of VCM.

Test Conditions: Polymerize for 5 hours at 40°C (104°F) and determine the percent conversion.

Monomer Source	% Conversion	Date
Conoco (CONX 9054 - Burlington)	80.0	8/1/68
Ethyl (UTLX 83804 - Burlington)	84.8	7/19/68
Pantasote (SACX 9015 - Burlington)	55.6	8/8/68
B. F. Goodrich (GATX 21968 - Burlington)	62.0	8/9/68
Stauffer (GATX 98227 - Burlington)	81.6	July, 1968
B. F. Goodrich (UTLX 82521 - Flemington)	56.0	
B. F. Goodrich (Recovered - Flemington)	32.8	
Tenneco (NATX 38053 - Burlington)	61.6	5/17/68
Tenneco (NATX 38037 - Burlington)	18.4	6/10/68
Tenneco (Car 38042 - Flemington)	66.4	Sept., 1968
Tenneco (Recovered - Flemington)	18.8	Sept., 1968

SUSPENSION POLYMERIZATION KINETICS

Reagents: 150 grams of H_2O + 0.40 grams MC-15 + 0.125 grams L_2O_2 + 80 grams VCM.

Test Conditions: Polymerize for 10 hours at 132°F (55.5°C) and determine the percent conversion.

Monomer Source	% Conversion
100% B. F. Goodrich (UTLX 82521 - Flemington)	84
80% B. F. Goodrich (UTLX 82521 + 20% Recovered Goodrich VCM)	84
50% B. F. Goodrich (UTLX 82521 + 50% Recovered Goodrich VCM)	80.5
20% B. F. Goodrich (UTLX 82521 + 80% Recovered Goodrich VCM)	76
100% Tenneco (Car 38042 - Flemington)	87.5
80% Tenneco (Car 38042 + 20% Recovered Tenneco VCM)	75 (?)
50% Tenneco (Car 38042 + 50% Recovered Tenneco VCM)	77.5
20% Tenneco (Car 38042 + 80% Recovered Tenneco VCM)	72.5
100% Tenneco (Cars 38042, 38016, 38038, 38039)	86
50% Tenneco (Cars 38042, 38016, 38038, 38039) + 50% Recovered Tenneco VCM	80