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JUL 19 1968

R. N. Wheeler

INFORMAL REPORT

PROJECT NO.: 333A10

DATE: July 16, 1968

AUTHOR: A. A. Peterson

SUSPENSION VINYL RESINS: PROCESSES

FOR PRODUCING SPHERICAL COPOLYMER

FOR POWDERBLEND RECORD MARKET

Process-14 (Elvanol 50-42 suspending system) has now been studied thoroughly as a method for the production of a superior flow, high bulk density copolymer for the powderblend record market. Results are incomplete but the following problems are evident: 1) recipe instability in the acid medium produced by partially hydrolyzed polyvinyl alcohol even with unusually high buffer content (0.3% NaHCO<sub>3</sub> compared to less than 0.1% for most pH sensitive recipes), 2) inability to consistently produce particles in the desired 130-160 micron range, 3) high degree of foaming in the autoclave, creating washing problems and some autoclave fouling (antifoam agents tested have agglomerated the particles) and 4) slightly poorer color and heat stability than desired.

As a back-up approach, Process-4 (Methocel 90HG100) has been studied. It has none of the disadvantages of Process-14 while producing a copolymer with a whiteness index higher (greater than 90) than any copolymer in the powderblend market and with the desired high bulk density and good flowing spherical particles. It now appears that Process-4 is the best method (with no apparent disadvantages) for the production of such a resin. If necessary, more pilot plant work could be done to delineate various trends in the process, but it is felt by the author that we have a recipe now ready for initial scale-up to produce enough resin for customer sampling. Bound Brook personnel have requested 200 pounds of pilot-plant material for sampling at Capitol Records. The evaluation of pilot-plant runs at Bound Brook has shown the resin to have the desired properties.

*Adolph A. Peterson*  
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MONTHLY REPORT  
(DIVISION-SUPPORTED PROGRAMS)

TO: Dr. F. E. Bailey, Jr.

R. N. Wheeler

FROM: Dean E. Richardson

DATE: February 9, 1968

*ms KED*

SUSPENSION VINYL RESINS

Project No.

347 B 10 General Notes on Group Program

The installation of the 900-gallon solvent wash tank is complete, and the first charge of solvent is being used to wash the 600-gal. autoclave between polymerizations. The stainless steel replacement vessels for the present blowdown and reslurry tanks are being fabricated in the Texas City Plant shops. Delivery of the agitators is expected March 15. Several ideas are being considered to revise material flows around the pilot plant fluid-bed dryer, the aim being to reduce back pressure inside the dryer itself and eliminate the Vibra-Screw feeder now required to convey dewatered resin from the Bird centrifugal to the dryer.

A letter was issued to interested personnel outlining revised and expanded areas of primary responsibility for the technical personnel in the group. In cooperative endeavor with operating supervisors, periodic meetings of personnel from the two groups for communications have been inaugurated, the aim being to improve cooperation between Production and R and D in areas of mutual interest.

347 B 10 Blotter-type PVC

The first computer results were received from the statistically-designed series to explore the effects of process variables on the Methocel 90HG100/Aerosol TR recipe. Predictability factors were not as great as hoped for, and several sets of slightly modified data will be processed through the computer in an effort to more completely pin down the effects of the variables.

Additional work is also planned in the 10-gal. stainless steel autoclave with the Elvanol 50-42/Silicone L-5310 recipe. The agreed timetable is to scale up into production a blotter-type PVC, primarily for F and F, during the second quarter.  
(Dean E. Richardson, R. J. Greer)

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### 347 B 10 Mixed Catalyst Studies

The mixed catalyst studies using isopropyl peroxydicarbonate (IPP) with dilauroyl peroxide (DLP) in the 600-gal. autoclave are currently scheduled to begin February 19. Two ratios of IPP/DLP will be checked at QSAN-7 conditions against a control using 100% DLP. The aim is to provide operating personnel with data which will allow them to increase QSAN-7 productivity by 20%.  
(Dean E. Richardson, A. A. Peterson)

### 347 B 15 Graft Polymer Studies

Ethylene-vinyl acetate copolymers have been received from Bound Brook with VAc contents as high as 45%, and studies of the solubility of these materials in VCl monomer are in progress. Meanwhile, various Applications R and D groups have indicated some degree of interest in usage of a PVC/EVA graft polymer, and their comments are being used as a guide to planned work.

One of the problems anticipated is in finding an analytical method for the graft polymer, particularly assuming a grafting efficiency substantially less than 100%. Technical Center personnel are assisting in this area, with the hope of finding a method which will at least indicate grafting efficiency, if not a complete composition analysis.

Small samples of  $\alpha$ -methyl styrene co- and terpolymers have also been received from Bound Brook for solubility studies in VCl. The hope for a graft polymer with VCl in this case would be increased heat distortion temperatures without substantial adverse effect on other properties.  
(Dean E. Richardson, K. L. Meisner)

### 347 B 15 Slurry Compounding Studies

With the transfer of J. W. Fields to Texas City, the responsibility for slurry compounding studies was also moved to this location. The status of the work was reviewed with Messrs. Fields, Hockersmith, Greer, Jack Field and me. Plans for the immediate future are to make 100 lbs. of a wire and cable compound, using equipment available at Bldg. 116 (Vinyl Catalyst Unit). This is scheduled to begin February 12, and will be supervised by Messrs. Fields and Greer. The compound will be evaluated at Bound Brook.

Meanwhile, at the request of the Operations Team, Mr. W. S. Lanier has begun engineering estimates of capital costs involved in a slurry compounding facility and probable profit potential. In this effort, he will seek to coordinate information from Bound Brook R and D, Marketing and Texas City. The studies will admittedly be order of magnitude because the process itself has not been well defined, but the data should provide some basis for guidance of further work if such effort is considered warranted.  
(Dean E. Richardson, R. J. Greer, J. W. Fields)

### 347 B 15 Studies to Support Sale of Technology

At the request of legal personnel involved in the negotiations to sell VCl-ethylene copolymer technology, three runs were made in the 600-gal. autoclave to check examples in two foreign patents which might be construed as bearing on UCC applications now on file. In one case, it was possible to reproduce conditions cited in the example in a single run. In the other case, it was not possible to completely reproduce claimed conditions in two runs, partially because of unfamiliarity with the suspending system. Results were transmitted to legal personnel at 270 Park Avenue for consideration.

(Dean E. Richardson, K. L. Meisner, R. C. Robinson)

### 211 B 10 Intensive Mixer Studies of Plasticized Formulations

The 5-lb. Henschel mixer at Texas City is being used to carry out studies of QYTQ-7 performance in plasticized formulations. The studies were prompted by two reports from customers of problems encountered with QYTQ-7 in Henschel mixers. It was not clear from the field reports whether the problem lay with the resin, the mixer, or a combination of the two. Initial studies were therefore designed to use a formulation similar to the customer's, and were aimed at trying to define the problem. The Henschel cycle was followed by cooling in a slow speed Hobart mixer to simulate the ribbon blender which is used as a cooler in the customer's plant. As an additional measure, Geon 102 and 103 were used as controls, since these resins have historically been excellent resins for extrusion.

Initial work showed little difference in preblend density between QYTQ-7 Bl. B-330 (ethylene modified) and Geon 102, so the range of experiments was expanded to define the effect of Henschel drop temperature on preblend density, ASTM dryflow time and particle size distribution. Data are incomplete, but several preliminary conclusions have been reached:

1. Preblend density increases slowly as the preblend cools in the Hobart.
2. Preblend density can be increased sharply by adding to the Hobart small concentrations of very fine particle materials such as Magnesol, Cabosil, carbon black or dispersion resin QYNV. The increase is from ~23 lb./cf to ~31 lb./cf. This effect cannot be obtained by adding the same materials to the Henschel.
3. The mechanism of the above phenomenon seems to be a simple adsorption effect, using the tremendous surface area of the small particles to attract any available plasticizer. Dry flow properties are similarly improved.
4. The mechanism of the improvement in preblend density mentioned above is not static electricity. This is indicated by photomicrographs and other studies.
5. Some variation in preblend properties is possible by manipulation of the Henschel cycle.

The studies are continuing on a high priority basis, utilizing suggestions from Bound Brook and Tarrytown personnel.  
(Dean E. Richardson, K. L. Meisner)

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### 333 A 10 Improved Dryflow Properties in VCl/VAc Copolymers

A VKF sound record copolymer previously made in the 600-gal. autoclave using Elvanol 50-42 as suspending agent was evaluated favorably at Bound Brook as having excellent dryflow properties and spherical, glassy particles. Attempts to carry out a short, statistically-designed series to define the effect of four variables resulted in the discovery that a fifth variable, charge water temperature, was exercising a controlling effect, so the series has been expanded to sixteen runs to include the fifth variable. This series is now in progress, with completion expected February 16.

(Dean E. Richardson, A. A. Peterson, R. C. Robinson)

### 333 A 10 Antistatic VYCR-10 Copolymer

RCA has favorably evaluated VYCR-10 as a replacement for VYHR, and insists that an antistatic version of this resin will be needed even after a shift to powderblend feed. Accordingly, an upcoming VYCR-10 production run will be aimed at supplying resin for RCA's usage, and antistatic samples of the production resin will be prepared for Bound Brook testing, using Magnesol to obtain satisfactory dryflow properties. (Dean E. Richardson, A. A. Peterson)

### 343 A 10 Scaleup of QXPM-4 Plastisol Additive Resin

Bin and blend analyses of the QXPM-4 production run made in mid-January show the resin to be on target for quality control parameters. Production totalled 76,000 lb., which was bagged out as Blend B-10. Particle size was slightly smaller than expected (51 $\mu$ ), with ASTM sorption just above the 60 phr target at 62 phr. Evaluations are in progress at Bound Brook and the Technical Center, with completion expected in late February. If plastisol evaluations confirm the quality control analyses, marketing personnel plan to release Blend B-10 for field sampling in March. (Dean E. Richardson, R. J. Greer, Operating Supervisors)

  
Dean E. Richardson

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