BUSINESS CONFIDENTIAL

MONTHLY REPORT
BUSINESS-SUPPORTED PROGRAMS)

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

FROM: Dean E. Richardson

DATE: March 17, 1967

SOLVENT VINYL RESINS

Project No.

347 K 12 Dewatering and Washing Solvent Vinyl Resins

Test work using the Bird-Young rotary vacuum filter was completed at Bldg. 121 and the filter was returned to suspension vinyl use on March 15, 1967. During the course of the test work, the pilot filter was operated to dewater and wash VMCH slurry (both first stage and second stage) and VYNS first and second stage slurry. Several observations are pertinent:

1. In VMCH service, the filter could not be used satisfactorily for dewatering hardening tank slurry (first stage) because the coarse, spongy nature of the particles did not permit the cake to be held on the drum long enough. With wash tank (second stage) slurry, the filter performed well, and water washing on the filter drum reduced the free VAc from 1.0% to 0.5%.

2. In VYNS service, the filter worked well with both hardening tank and wash tank slurry. In the case of the hardening tank slurry, water washing the cake on the drum reduced the acetone to less than half its normal level. Isopropanol was reduced only slightly, but vinyl acetate was decreased to about half the concentration in the unwashed cake. In dewatering wash tank slurry, cake washing reduced acetone by about 25%, isopropanol by 15% and vinyl acetate by 15-20%. Total solids of the filter cake varied from 45-60%.

Although the test work was admittedly not exhaustive, two firm conclusions can be drawn:

1. Vacuum filtration as a process appears to be both practical and promising for dewatering solvent resins, and savings in solvent losses might well justify the cost of the equipment.

2. The Bird-Young rotary vacuum filter does not appear to be the optimum machine for use with solvent resins. Vacuum belt filters such as the Eimco model recently tested in suspension vinyl service appear to be much better suited.

A small pilot model vacuum belt filter is being sought for limited further test work.
(Dean E. Richardson, R. C. Robinson)

RESEARCH AND DEVELOPMENT DEPARTMENT
CHEMICALS DIVISION
UNION CARBIDE CORPORATION

SUSPENSION VINYL RESINS

347 B 10 General Notes on Group Program

A meeting was held on March 7 and 8 in Bound Brook with Technology Managers and other interested parties to review resin requirements, communications channels and applications developments. Good progress was made in establishing closer liaison with various marketing technology personnel, and it was possible in most cases to define rather closely the resin characteristics needed by each group. There was unanimous agreement as to the value of the discussions, and it was tentatively agreed that a similar session should be held early in the summer to update the technology as new market areas come more clearly into focus.

Maintenance is finally in motion again on reinstalling the two 10-gallon autoclaves, and although the progress is painfully slow, there is hope that the installation can be completed in May. A large backlog of projects awaits the completion.
(Dean E. Richardson, R. C. Robinson)

347 B 15 Evaluation of Gantrez AN as a Suspending Agent

Glass bomb polymerizations were made in preliminary evaluation of the copolymer of methyl vinyl ether and maleic anhydride (General Aniline's Gantrez AN) as a suspending agent. Polymerizations were made at 60°C, and concentrations of 0.1%, 0.2% and 0.3% Gantrez AN-169 were checked in both homopolymer and copolymer recipes. Also, in view of the manufacturer's claim that the effectiveness of the Gantrez copolymer is enhanced by the use of a small concentration (1% of the Gantrez AN-169) of particular types of surfactants, the same recipes were repeated adding the recommended concentration of Tergitol NPX.

In the case of the homopolymer recipes, all bombs contained excellent well-formed particles, but the ones containing the surfactant yielded slightly smaller and more regular particles at all concentrations. In the copolymer bombs, all polymerizations yielded excellent particles, and there was no apparent difference between those with and without surfactant.

Photomicrographs of the resin revealed that all of the resin had one property in common: the particles displayed a startling roundness, being in the form of almost perfect spheres. This property would be of particular interest in copolymer resins and plastisol additive types. Scaleup runs in the pilot plant are scheduled for April.
(Dean E. Richardson)

347 B 15 Designed Experiments to Study Effect of Agitation

The statistically designed series of pilot plant runs to study the effect of agitation variables (18 runs) was completed. Analyses are complete, and the data is being prepared for the computer. The data will also be compared with other runs on QXAH-12 from earlier studies.
(Dean E. Richardson, A. A. Peterson, T. R. Smith, R. C. Robinson)

223 B 10 QXAM-12 for Wire and Cable Dryblend

The plan of action for QXAM-12 mentioned in last month's report was partially deferred to allow experimental work on a phenomenon which became apparent subsequent to the February report. Storage stability tests by Jim Hockersmith's group showed that a significant reduction in ICMT occurred during three week's storage, and that the diminished heat stability was most evident at extended times (15 and 20 minutes on the 2 roll mill). Bound Brook testing using the high speed mill confirmed the observations. As an order of magnitude, ICMT values decreased from 107 to 98 in three weeks.

Conferences with Technical Center personnel indicated that the phenomenon was probably due to degradation of the residual Carbowax 20M on the resin, and that Ionol should be effective if added just before the end of the polymerization. This information coincided with an overseas recommendation for the use of Ionol for the same purpose with another suspending agent. Accordingly, runs were made in the pilot plant to compare QXAM-12 with and without Ionol addition. Stability tests on this resin are now underway. If the tests indicate that the use of Ionol is effective, the scaleup timetable previously agreed on will be implemented immediately.

In other work, dryblendability tests by Bound Brook W. and C. personnel demonstrated that:

1. QXAM-12 (two blends) from the earlier extended production run was fully competitive with a leading W. and C. dryblend resin from another manufacturer. This was true in both the Flexol 1010 and 1313 formulations. In addition, it showed good dryblend flow characteristics and good fisheye ratings.

2. Of four blends of QXOM-7 tested, all were superior in the Flexol 1010 formulation, but only one would dry at all in the Flexol 1313 mix. This indicates that O resins are probably not suitable as an across-the-board replacement for PVC in W. and C. dryblend usage.

3. While QYTQ-7 was satisfactory with Flexol 1010, it would not dry with Flexol 1313.

From the above, it is apparent that QXAM-12 is the only candidate we currently have which meets the dryblend requirements for a fully competitive W. and C. dryblend resin. This conclusion only adds urgency to the current work aimed at stabilizing ICMT values and sharpening particle control.
(Dean E. Richardson, T. R. Smith, R. C. Robinson, J. L. Hockersmith, Bound Brook W. and C. Personnel)

223 B 10 Resistivity of Double-Washed QSAN-7

Although single-washed QSAN-7 from an earlier production run showed excellent electrical resistivity, two samples from the current run showed poor resistivity when tested at Bound Brook. Inasmuch as the resin is intended for electrical compound at Bound Brook, it became urgent to correct the deficiency, so the pilot model Bird-Young rotary vacuum filter was hastily moved from Bldg. 121 to the spare slurry blending tank and installed to permit double-washing of QSAN-7. R. and D. technicians

worked overtime to train operating personnel in the use of the filter, and production of double-washed QSAN-7 is in progress. Samples have been forwarded to Bound Brook for test. Meanwhile, engineering work on the permanent double-washing installation is nearing completion.
(Dean E. Richardson, R. C. Robinson, Production Supervisory Personnel)

211 B 10 QXAF-5 for Extrusion Usage

Bound Brook personnel requested preparation in the pilot plant of a Process-5 PVC with inherent viscosity between QYSA and QYSJ for use of a customer in Massachusetts who extrudes needles for synthetic Christmas trees. Such a resin was prepared without difficulty, and 200 lb. was shipped to the customer for test, along with a smaller quantity to Bound Brook for evaluation. The resin is designated as QXAF-5, and the shipment was identified as QEX-1280.
(Dean E. Richardson, T. R. Smith, R. C. Robinson)

211 B 10 QXAC-5 for Injection Molding

Bound Brook personnel requested preparation of a Process-5 resin at the "C" inherent viscosity level for an injection molding application at General Electric involving a glass-filled vinyl compound for injection molding. Two runs in the pilot plant were made to produce the resin, one run with trichlorethylene and one without. Samples were shipped to Bound Brook for evaluation prior to trials at GE.
(Dean E. Richardson, A. A. Peterson, T. R. Smith, R. C. Robinson)

211 B 10 QXOS-7 for Food Products Division

R. and D. personnel of the Food Products Division's Chicago labs are interested in QXOS-7 for plasticized extruded film. After discussion, they were furnished with 200 lb. each of two production blends for evaluation.

In addition, a letter was written to Food Products Division personnel, pointing out that quality levels for QYTQ-7 have improved substantially, to the point where most of the current production should be suitable for extruded meat wrap film.
(Dean E. Richardson)

333 A 10 VYCR-10 Particle Control in Production Autoclaves

The recent VYCR-10 production run demonstrated that current control of particle properties is inadequate. Of the first 4 blends produced, two met the tentative particle requirements requested by Steve Krumm (140-150 microns median, 40 lbs/cf bulk density, <15% fines) while the other two were near misses. Subsequently, however, particle size slid abruptly downward, eventually levelling out at 110-110 microns median, for no apparent reason. Since Bound Brook personnel plan to sample various customers with Blend 179 (one of the first four) as a powderblend resin for records, it is imperative that our particle control in production be sharpened substantially.

Data, recipes and procedures were reviewed with Production supervisors on March 16. Jack Erdmann was able to document that the vinyl chloride monomer quality went from good to poor at approximately the same time that the shift in particle size occurred. No other change was apparent. Several other observations were made:

1. Both the pilot plant data and production experience indicate that the VYCR-10 recipe in production is relatively insensitive to most of the prime variables normally controlled.

2. Variations in production VYCR-10 particle size have normally been accomplished by varying degrees of secondary agglomeration rather than by changes in primary particle size.

3. Pilot plant data indicate that primary particle size can best be controlled by varying agitation, i.e., lower agitation, larger primary particles.

Timewise, no further VYCR-10 is scheduled for production in the near future. However, experimental runs of VXKF-10 are scheduled about March 27, and particle control measures developed for VXKF-10 should also be applicable to VYCR-10. Therefore, the strategy for the next VYCR-10 run will be largely determined by the VXKF-10 studies proposed below. (Dean E. Richardson, A. A. Peterson, T. R. Smith, Production Personnel)

333 A 10 Development of VXKF-10 "Single" Resin for Records

After monomer quality returned to normal, VXKF-10 production experimental work was resumed on a one-autoclave-at-a-time basis. Since the particles were too small (∿110 microns median), several successivle changes were made, using pilot plant parameters, to adjust the particle size upward. The effect was slight, increasing the median from 110 microns to 120 microns. One run was then made to produce a 150 micron median particle, but again it was noted that the increase was due mainly to secondary agglomeration. A repeat run yielded only 119 micron particles.

In view of these results and the observations mentioned above for VYCR-10, it was agreed that further efforts to produce powderblend quality VXKF-10 in production will be as follows:

1. One of the South-2 autoclaves, probably D-23, will be equipped with the 104 rpm agitator gear set. This reduced speed should increase the primary particle size. Resin will be recovered through the spare blowdown and slurry blending tanks.

2. 0.1% PVP will be used at 30/70 mon./H_2O ratio. Vinyl chloride monomer quality will be checked frequently and correlated with other data.

3. It is expected that production equipment for this work will be available about March 27. No further work is anticipated before that time. (Dean E. Richardson, A. A. Peterson, T. R. Smith, R. C. Robinson, Production personnel)

343 A 10 QXPM-4 Plastisol Additive Resin

Discussions with Bound Brook personnel indicated that, by their evaluations, QXPM-4 as recently made in production, is sufficiently close to target to be competitive. They further point out that QXPM-4 can be used to a greater extent in plastisols (63 phr plasticizer) without settling than PVC additive resins, and that the rigidsol viscosity tests currently used represent performance in only a very small segment of the business. Viscosity stability characteristics are much less critical in the highly liquid systems commonly used. It was agreed that Bound Brook personnel would review with marketing personnel the previous marketing decision to offer a low-fusing plastisol additive resin rather than a straight PVC.

Meanwhile, prior to an extended scaleup run on QXPM-4, pilot plant runs have just been completed to check out the effect of lower molecular weight homologs of the currently used Methocel 90 HG 100. These suspending agents were recommended by Dow Chemical as being likely to produce a smaller particle than the Methocel 90 HG 100. Resin from the pilot plant runs is currently being analyzed.
(Dean E. Richardson, T. R. Smith, R. C. Robinson)

Dean E. Richardson

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