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PLANT PROCESS DEVELOPMENT DEPARTMENT

RESINS GROUP

TEXAS CITY PLANT

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G. J. Hanks, Jr.

BUSINESS AREA 200 THERMOPLASTICS GENERAL

Project No.

320 L 10

Suspension Vinyl Resins: Azo Catalyst for High Temperature Use

In cooperative work with Operating Supervisors, a series of runs was made in D17 autoclave during the recent production run on VXJD-10. The work was confined to D17 because it is the only south line autoclave presently equipped with a catalyst charging pot. Twelve polymerizations were made with azo (azobisisobutyronitrile) at the same conditions being used for DLP (dilauroyl peroxide). The operating recipe for use of DLP included 1.6% trichlorethylene in order to hold the autoclave temperature to 80°C. The use of TCE with azo is not anticipated or desired, but was unavoidable in this case because the TCE was premixed with the vinyl acetate in Tank Area 4 and charged as a single ingredient. Nevertheless, useful information was gained relative to 1) the tendency toward secondary agglomeration with azo; 2) the relative efficiency (lbs. resin per lb. catalyst) of azo and DLP, and 3) vinyl acetate conversion. The production run on VXJD-10 was terminated before all questions could be resolved, but the available data support several tentative conclusions:

1. The unreacted VAC in the resin was generally lower in the azo runs.
2. The relative efficiency in lbs. of resin per lb. of catalyst, at 80°C with 1.6% TCE, is approximately 3.2/1 in favor of azo. In other words, 3.2 lbs. of DLP are equivalent to 1 lb. of azo.
3. VAC conversion, as indicated by terminal autoclave pressure and PVC content of the resin, was generally higher with azo.
4. Secondary agglomeration in the azo runs was minimized by increasing the PVP (polyvinyl pyrrolidone) concentration from 0.14% to 0.15%. The production run was terminated before this problem could be completely resolved.

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Further test work using azo is planned during the VYCA/VYCR run beginning about October 15. The target is to hold unreacted VAc in all of the copolymer resins below 1.0% as quickly as possible, in order to minimize drying problems.

(D. E. Richardson, Vinyl Operating Supervisors)

320 L 10 Effect of Suspending Agent Quality

As part of the continuing effort to define the effect of raw material variations on resin quality, a meeting was held with personnel from Suspension Production, Development, and Resins Laboratory to discuss Cellosize products with Technical Center R and D personnel knowledgeable in this area. Emphasis was on Cellosize HEC WP-300, which is used locally as a suspending agent. Results were gratifying, and South Charleston will endeavor to supply WP-300 of constant quality, minimum variation in molecular weight spread, and minimum glyoxal content. Some cooperative effort to define the effect of molecular weight spread will be undertaken later.

Also, in consideration of the fact that Institute production specifications permit a wide spread in Carbowax 20M (Process 12) molecular weights, five runs were made in the pilot plant to produce QXAN-12, using five C20M samples covering the entire spread of C20M molecular weights allowable under the specification. Under the conditions of the test, no substantial differences were observed in resin quality. This conclusion should be qualified because the effect of oxygen on C20M recipes is not yet clearly defined.

(D. E. Richardson, G. G. Harkreader, R. C. Robinson, E. W. Dennard, J. J. Keil, J. H. Williams)

Suspension Vinyl Resins: Drying System Design for New Production Line

In the course of cooperative effort with Works Engineering personnel to specify and design drying equipment for the next increment of suspension vinyl resin capacity, a visit was made to South Charleston to observe fluid-bed dryers and other drying equipment, and to review operating experience with drying equipment. The information gained was combined with Texas City experience and test data, and the suspension resin drying equipment was sized and specified. Design of the fluid bed dryer is in progress, cooperatively with Works Engineering personnel.

(G. G. Harkreader)

320 L 10 Wacker-Chemie Suspension Vinyl PVC Samples

After reviewing the regular production grades of PVC marketed by Wacker-Chemie (Burghausen, W. Germany), samples were requested of four of the resins, representing a range of recipes and molecular weights. The

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samples arrived October 5, and are currently being analyzed by the local Resins Laboratory. They are as follows:

Vinnol H100/65 v, a plastisol additive type resin based on Methocel 90 HG.

Vinnol H100/70 d, a Process-7 resin of about 1.0 inherent viscosity.

Vinnol H100/75 f, a Process-5 resin between QYSC and QYSL in molecular weight.

Vinnol EB59823, a Process-7 resin in the QYSA-QYSJ range.

Evaluation results will be reported as soon as analyses are completed.

(D. E. Richardson, Texas City Resins Laboratory Personnel)

320 L 10 Suspension Vinyl Resins: General Notes on Pilot Plant

In view of the current situation wherein the local suspension resin plant capacity is essentially sold out, some realignment of development project priorities has been made. The number of new resins to be scaled up will be minimized, and it will undoubtedly be advisable to sample some of the anticipated new resin types from the pilot plant, rather than from limited scaleup runs, to selected users for evaluation. The present situation should allow more time in the pilot plant for study of recipe variables than previously.

BUSINESS AREA 210 WIRE AND CABLE RESINS

320 G 10 Suspension Vinyl Resins: QXAN-12 for Wire and Cable Dryblend

In view of the favorable results with QXAN-12, QEX-1215, Bound Brook Wire and Cable Development personnel also evaluated QXAN-12, QEX-1216, which was slightly different in particle size. Results were again good in all respects, and W and C personnel indicate that a resin like QEX-1215 or -1216 would be an excellent candidate for marketing as a wire and cable dryblend resin. Further activity awaits an extended scaleup run on QXAN-12 in production.

BUSINESS AREA 230 MOLDING AND EXTRUSION RESINS

Suspension Vinyl PVC Resins by Process-10 for Blown Vinyl Bottles

Bound Brook R and D personnel have evaluated QYAC-10 and QXAJ-10 for vinyl blowmolded bottles. The QYAC-10 was excessively soft, indicating too low a molecular weight. The performance of QXAJ-10, Bl.1, however, was good in all respects save fisheyes, and it was known in advance that Blend 1 was slightly contaminated.

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Prior to the above evaluation, and at the request of Bound Brook R and D personnel, 1000 lb. of QXAF-10 was made in the pilot plant and shipped to Bound Brook to be used in trials of the new Le Seuer blowmolding machine being purchased for that location.

Based on the favorable results reported above for QXAJ-10, Marketing personnel plan to make powder-mixed vinyl bottle compound in a Henschel mixer at Bound Brook, and sample it to three potential customers in the blown vinyl bottle field. The compound will be based on QXAJ-10. (D. E. Richardson, G. G. Harkreader, R. C. Robinson, E. W. Dennard, J. J. Keil, J. H. Williams, Bound Brook R and D Personnel)

BUSINESS AREA 240 CALENDERING, RECORD AND FLOORING RESINS

324 A 10 Suspension Vinyl Resins: Effect of Monomer Quality

In view of data collected by the Suspension Vinyl Technology Team showing that VC1 quality at the Suspension Vinyl Plant varies widely, two steps have been taken in the pilot plant: 1) a chromatographic analysis is being obtained for each tank of VC1 received, and 2) recent data are being reviewed to see what effect might be expected from substantial concentrations of allene and other contaminants. Pilot plant VC1 analyses recently have been in the 320-360 ppm allene range. Efforts to correlate VC1 quality with resin quality will continue.

(D. E. Richardson, G. G. Harkreader, R. C. Robinson, E. W. Dennard, J. J. Keil, J. H. Williams)

324 A 10 Suspension Vinyl Resins: Single Resin System for Sound Records

In response to demands for a "Single Resin System" copolymer for sound records, Brabender melt viscosity data were examined for several resin blends known to be preferred by record manufacturers. Based on this data, and after consultation with Bound Brook R and D personnel, it was agreed that the target resin with which to sample Columbia Records is a Process-10 copolymer of 88% PVC and 0.530 inherent viscosity. In order to bracket the target, three resins will be prepared in the pilot plant, all at 88% PVC, and having inherent viscosities of 0.520, 0.530 and 0.540. These resins will be shipped to Bound Brook R and D for making up the compounds and delivering to Columbia. Preparation of the resins is scheduled to begin October 18.

(D. E. Richardson, Resins Lab personnel, Bound Brook R and D personnel)

324 A 10 High Molecular Weight PVC Resin for Columbus Coated Fabrics

One run was made in the pilot plant to provide an ultra-high molecular weight calendering PVC for a special application at Columbus Coated Fabrics. The resin was designated QXAZ-12, QEX-1219, and 100 lb. was shipped to the Cincinnati sales office for delivery to CCF. The inherent viscosity

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of QEX-1219 was 1.57, and the particles were typical of dyblend resins, with good fisheye characteristics and good dryblendability in the CCF plasticizer system. Evaluation by CCF is awaited.

(D. E. Richardson, G. G. Harkreader, R. C. Robinson, E. W. Dennard,
J. J. Keil, J. H. Williams)

324 A 10 Suspension Vinyl Resins: QXAN-12 for Critical Calendering Use

Bound Brook evaluation of QXAN-12, QEX-1216, in Gloss -06 sheeting showed good clarity and fisheye levels but a graying effect in roll stock, normally associated with inferior heat stability. A review of the data on QEX-1216 indicated that this was probably due to the substandard ICMT rating of 93. The low ICMT is not inherent in the recipe, since other batches of QXAN-12 have exhibited ICMT levels well over 100. No continuing difficulty in this area is therefore expected.

Inasmuch as pilot plant data on QXAN-12 indicated the possibility of oxygen sensitivity in the recipe, six additional runs were made in the pilot plant to check the effect of oxygen variations in the autoclave, as well as variations in C2OM concentration. Unfortunately, oxygen in the pilot plant autoclave can only be checked by the change in chloride ion concentration over the run, and efforts to intentionally raise oxygen levels turned out, with one exception, to be unsuccessful. Suspending agent concentration effects were approximately as expected. One or two additional runs to resolve the oxygen question are planned.

Efforts are being made to schedule an extended scaleup run on QXAN-12 in production, and it is hoped that such a run will be possible late in October. Production of 100,000 lbs. of resin is anticipated. Such a run would permit assessment of such intangibles as autoclave fouling, reproducibility, and general debugging.

(D. E. Richardson, G. G. Harkreader, R. C. Robinson, E. W. Dennard,
J. J. Keil, J. H. Williams)

324 A 10 Suspension Vinyl Resins: Process-11 Terpolymers for Flooring

As agreed with Dept. 240 management, two Process-11 terpolymer resins were prepared in the pilot plant for limited customer sampling. The two were designed to duplicate earlier resins evaluated as QEX-1174 and -1175, and analyses were similar except that equilibrium melt viscosities (Brabender) were almost exactly the same despite differences in comonomer content. These results were reviewed by Dept. 240 personnel, and it was agreed that we should plan limited sampling of two resins, having 85-86% PVC, 1.0% comonomer and inherent viscosities of 0.530 and 0.550, in order to maximize melt viscosity difference. One of the earlier resins (QEX-1221) corresponds to the 0.530 level, and the 0.550 resin will be made in the pilot plant this week. Marketing has designated four flooring manufacturers for sampling.

(D. E. Richardson, G. G. Harkreader, R. C. Robinson, E. W. Dennard,
J. J. Keil, J. H. Williams)

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BUSINESS AREA 360 COATINGS AND ADHESIVES RESINS

336 J 41 Suspension Vinyl Resins: Plastisol Blending Resin
Studies in the Pilot Plant

The plastisol blending resin samples previously forwarded to Technical Center R and D have been evaluated with favorable results, both for the PVC resins and the Process-11 versions. A sample of QEX-1218, the Process-11 resin with 1.0% comonomer, was also sent to Bound Brook for evaluation. Based on the Tech Center data, it was agreed that QEX-1218 would be the target for a single autoclave scaleup run, tentatively scheduled for the week of October 18. The scaleup will be attempted in D17 autoclave, and dried through the pilot plant. Target comonomer content is 1.0 - 1.2%, at an inherent viscosity of 0.95 to 1.0. A report was issued covering the plastisol additive work in the pilot plant.

(D. E. Richardson, G. G. Harkreader, Tech Center R and D personnel)

REPORTS

1. Status Report, "Suspension Vinyl Resins: Plastisol Additive Resins (QXPA) - Based on Methocel 90 HG 100," by D. E. Richardson and G. G. Harkreader, October 6, 1965; Project 336 J 41.
2. Status Report, "Suspension Vinyl Resins: Factors Affecting Quality of Production PVC Resins," by Suspension Vinyl Technology Team (Fisher, Schlichter, Humphreys), September 30, 1965.


Dean E. Richardson

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