STATUS REPORT

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE: UTILIZATION OF TECHNOLOGY OF PROCESS 5 AND PROCESS 6

AUTHORS: R. C. Wise
J. C. Alexander
W. H. Nelson

J. F. Erdmann,
Group Leader

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SUMMARY Experimental polymerizations have been conducted in both production and pilot scale equipment to adapt Process 5 and Process 6 to the Suspension Vinyl Resins Unit at Location 514. Under the guidance of Dr. Hans Bauer of Burghausen, Germany, conditions were established for full-scale operation of these two processes, although only Process 5 is presently capable of producing salable resin.

Process 5, which utilizes Methocel 10 as the suspending agent and sodium bicarbonate as a buffer, produced resin of excellent initial color, as measured by the ICMT mill test. Physical properties of QXSM-5 and QXSJ-5 resins were near enough to the target properties to permit recommendation of immediate commercialization of both, and single experiments with QXSA-5 and QXSC-5 resins demonstrated that the capabilities of Process 5 extend to those resins as well. Process 6 uses a poly(vinyl alcohol), Polyviol VZ 200, as the suspending agent, Mersolat sodium alkyl sulfonate as a surfactant, calcium chloride as a surfactant modifier, and calcium carbonate as a buffer. Experimental Process 6 resins possessed relatively poor initial color, and although the color can be improved by addition of lauric acid to the polymerization charge, these resins cannot be recommended at this time for commercial production.

Process 5 could not be operated successfully in the autoclaves equipped with the Mixco turbine agitation systems, and a complete changeover to Pfaudler-type agitation is therefore necessary for commercialization. Process 6, on the other hand, can be operated with either the Mixco or the Pfaudler system. However, a considerable part of the technology of Processes 5 and 6 is concerned, not with agitation or recipe,

DEVELOPMENT DEPARTMENT
UNION CARBIDE CHEMICALS COMPANY
SOUTH CHARLESTON, WEST VIRGINIA

but with the need for precise, reliable methods of charging and operating the autoclaves if resins of consistently high quality are to be produced. Recommendations in this area include the use of simpler methods of control, arrangement for prompt analyses and evaluations, establishment of fixed responsibility for the various steps in the polymerization procedure, and general modernization and simplification of the production equipment.

These two processes establish new areas for investigation, and extensive developmental effort is planned to adapt the processes to the various types of resin currently being produced. In addition, experimental determination of the full significance of the several process variables must be undertaken to obtain greater insight into the technology behind the new processes.

INTRODUCTION For some time the poly(vinyl chloride) resins produced in the Suspension Vinyl Resins Unit at Location 514 have possessed significant shortcomings, particularly with regard to initial color of plasticized formulations. In addition the manufacture of poly(vinyl chloride) by suspension polymerization has not undergone sufficient technological advance to permit a worthwhile profit under current market conditions. Therefore when samples of poly(vinyl chloride) resin manufactured in Germany by Wacker-Chemie exhibited excellent initial color, equivalent or superior to that of most domestic competitive resins, arrangements were made whereby information regarding the German production would be made available to interested persons in South Charleston. Accordingly, a project team representing the Development, Production, and Engineering Departments visited the Wacker-Chemie plant at Burghausen to determine whether Wacker technology could be applied toward solution of the local problems concerning resin quality and operating efficiency.

The information obtained in Germany (1) indicated that without any exotic methods, materials, or equipment, Wacker-Chemie was able to produce resins of consistently high quality. In particular, two types of resin were considered more readily adaptable than others for domestic manufacture. One of these resins, type HH 100/f, is produced with methyl cellulose as the suspending agent and with sodium bicarbonate as a buffer salt. It has excellent initial color, low fisheye content, good plasticizer absorption, and excellent electrical

properties. The other resin, type H 100, is produced with poly(vinyl alcohol) as the suspending agent, and with an anionic detergent and several salts added. This resin features excellent hot-processing characteristics and extremely low fisheye content, but is inferior to type HH 100/f in color and electrical properties.

The decision was made to operate these two Wacker processes in the suspension polymerization equipment at Location 514, and efforts along this line were begun on production and pilot scale as soon as the tour party returned to this country. However, to assist in expeditious adaptation of the Wacker technology to local facilities, Dr. Hans Bauer of the Burghausen plant was invited to South Charleston. Under Dr. Bauer's guidance, adjustments in recipes and operating conditions were made as required to achieve satisfactory properties in the resulting resin. At the conclusion of his visit Dr. Bauer recommended areas for continuation of the program (2). Those aspects of his recommendations pertinent to production equipment were explored immediately upon his departure.

This report is intended to provide an account of the adaptation of Wacker technology to local equipment and procedural capabilities. To ensure complete understanding, the resins produced under the Wacker influence will be differentiated from regular production runs by addition of "-5" or "-6" to the customary four-letter code designation (3). Specifically, the process for production of HH 100/f resins is Process 5, and that for H 100 resins is Process 6. Reference to any or all procedures and recipes which arise from information from Wacker-Chemie will be accomplished in this and all future reports by use of these new terms. An equally important reason for this designation is to eliminate the possibility of associating future commercial resins with their German origin through accidental references.

DISCUSSION For investigation of the applicability of Process 5 and Process 6 technology to the equipment and organization at Location 514, three production autoclaves were made available for experimentation. Careful control of the production runs was exercised by assignment of technical personnel as monitors for each shift. The first series of experimental polymerizations began as soon as necessary equipment modifications were completed, and continued after

Dr. Bauer's arrival, while he familiarized himself with the Mixco agitation system and with the Suspension Vinyl Resins Unit in general. Operating conditions for this group of experiments, which are presented in Table I, were based on production of Process 5 resin for two reasons. First, that process is inherently the simpler of the two under consid r-ation; and second, all necessary raw materials were available by the time the production autoclaves were ready to use. The remaining production-scale experiments included in this report were conducted under the guidance of Dr. Bauer. While Dr. Bauer was here, pilot-scale operations were made according to his suggestions; other experiments were conducted before and after his visit.

I. Process 5 Resins

The first attempts at production of Process 5 resin (Table I) were performed without alteration of the production equipment except for that associated with resin recovery. Agitation was accomplished by use of three 31.8-inch, 7-bladed Mixco impellers, those in use for normal production of suspension poly(vinyl chloride). Conditions for production of QYSM-type resin were chosen to permit ready comparison of the Process 5 resins with standard production resins and at the same time to eliminate the need for use of a molecular-weight degrader which might have influenced the results to an indeterminate extent. The nominal recipe for Process 5 includes Methocel 10 as the suspending agent and sodium bicarbonate as a buffer salt. The operating conditions utilized as the process was developed locally are listed in Table II.

The particle size of the resins produced with a nominal Process 5 recipe was considerably larger than expected, and the fisheye content was excessive (Experiments 1W through 8W, Table III). By variation of agitator speeds and suspending-agent concentrations the particle size was reduced to desirable levels, but the fisheye content remained intolerably high in all instances. Dr. Bauer explained the deviation from desired performance in terms of the quality of the agitation. In order to prevent the formation of hard resin particles it is necessary to provide high-speed, low-shear flow in the auto-clave. The Mixco agitation system installed in the production autoclaves provides maximum shear with a relatively low amount of flow.

The validity of this explanation was tested by removing three of the four side-mounted baffles from one of the autoclaves and replacing the Mixco turbine impellers with a single 51-inch impeller with three straight, glass-coated blades. The advantageous effect of this alteration was clearly apparent (Experiments 5W and 11W, Table III). The median particle size of the resin was reduced from 280 to 105 microns, and the fisheye rating decreased from 200 to 20 per square foot on 17.5-mil film. The operating conditions were essentially identical for these two runs, with the exception of the type and speed of agitation. With the Mixco agitation system the maximum speed attainable within the power limitations of the drive motor was 110 rpm, whereas the Pfaudler-type impeller could be run at 125 rpm, the maximum drive speed, with only some two-thirds of the available power being consumed. Thus Process 5 was adapted to local equipment with only this relatively inexpensive alteration in the agitation. In all likelihood the final arrangement for full-scale manufacture of this resin will present further benefits, if care is taken to design the optimum impeller-baffle combination for the existing autoclaves.

II. Process 6 Resins

Operation of Process 6 was expected to be more nearly like standard local production than Process 5 had been. Process 6 recipes utilize a combination of poly(vinyl alcohol) suspending agent and an anionic surfactant, and in this respect are not unlike the recipes often used in local production of certain resins. In the nominal Process 6 recipes, Polyviol VZ 200 was the specified poly(vinyl alcohol), and Mersolat sodium alkyl sulfonate was the surfactant. To these basic ingredients calcium chloride was added as a surfactant modifier, and calcium carbonate, as a buffer. The operating conditions of the experiments performed with the Process 6 recipe are listed in Table IV, and the evaluations of the resulting resins are presented in Table V.

In production autoclaves agitated with Mixco turbine impellers, the Process 6 recipe, unlike the Process 5 recipe, produced resin of a fine particle size and a low fisheye rating. The particle sizes, in fact, were somewhat smaller than desired, in that about 30 per cent of the resin passed through 270-mesh screen. Such a resin would probably be unsuitable for several types of processing, notably dry-blending operations. However,

Dr. Bauer had no doubt that the particle size could be increased by suitable adjustment of agitator speed, concentrations of recipe ingredients, and saponification number of the poly(vinyl alcohol) selected as the suspending agent.

The principal deficiency in the Process 6 resins as produced in this series was in initial color (Table V). Dr. Bauer had never observed in this type of resin the yellow cast which overcame the plasticized resin on the mill. He suspected that impurities in the calcium chloride or the calcium carbonate might have been responsible for the poor color. Accordingly, the reagent-grade materials were subjected to semiquantitative analysis by the flame spectrometer, along with samples of the corresponding materials used by Dr. Bauer. The results of this analysis, which are presented as Appendix A, provide no corroboration of Dr. Bauer's suspicions.

Whatever its source, the poor color can be improved markedly by addition of lauric acid to the autoclave charge. The effect of such an addition was demonstrated in the 10-gallon E8 autoclave (Experiments 718 and 720, Table VIII). The only difference between the operating conditions of these two runs was the presence of lauric acid in Experiment 720. Initial color as measured by the ICMT test was improved from a reflectance of 78 to 112. Although experiments in E8 and other autoclaves of similar size have often produced erratic results in the past, the combined evidence of these runs and Dr. Bauer's statements establishes the use of lauric acid in the Process 6 recipe as a practical method of achieving the desired initial color in the Process 6 resin.

III. Process 5 and Process 6 on Pilot Scale

To provide a guide for initial operations in production equipment, several polymerizations were conducted in pilot-scale autoclaves. The conditions employed in these polymerizations are listed in Table VI and Table VII, and the evaluation data for the resulting resin are located in Table VIII. With the Process 5 recipe (Table III), QXSM-5 resin was produced in the 10-gallon, stainless steel E8 autoclave, equipped with 7-inch Mixco impellers. Several different charging and operating procedures were used, but in nearly every instance the particle size of the resulting resin was very large, and the fisheye content was excessive (Table VIII). However, the

initial color of the Process 5 resins produced in E8 autoclave was considerably improved over that encountered in ordinary runs. The average value of the ICMT tests on these resins was approximately 110, whereas a typical resin from previous work would be expected to yield a value of approximately 65.

As in production equipment, the Process 6 recipe proved more appropriate for the Mixco agitation system than had the Process 5 recipe. The Process 6 resin was finer (average median particle size about 235 microns), and the fisheye content was about 30 per square foot on 17.5-mil film. The initial color was not satisfactory for the nominal Process 6 recipe, but upon addition of lauric acid, as mentioned previously, the performance of the resin in that respect was improved significantly.

Polymerizations by Process 5 were performed also in the 500-gallon, glass-lined E16 autoclave equipped with Pfaudler-type agitation. These runs are included in Table III, and the evaluations of the resulting resin are in Table VIII. However, numerous operating difficulties were encountered in E16 autoclave which rendered the results virtually useless. In any event, neither corroborative nor contradictory evid nc was obtained in the short period of E16 operation.

IV. Resin Recovery

From each experiment of this series two resin samples were evaluated. One was the flash-dried resin, and the other was resin drained from the autoclave and dried in the Proctor and Schwartz tray dryer. In practically every run, the resin from the tray dryer possessed significantly superior initial color, compared with the resin from the flash dryer. This relationship has been observed invariably over a long period of time, but has always been challenged on the ground that either the wet-stripping tank or the flash dryer could have produced the ill effect. The elimination of the wet-stripping tank from these runs, accomplished for just this reason, leaves no reasonable doubt that the flash-drying system of the Suspension Vinyl Resins Unit does considerable harm to the resin.

V. Status of the Program with Regard to Specific Resins

The program for utilization of Process 5 and Process 6 technology embraces two distinct areas. One is the development of techniques and recipes to permit optimum operation of production equipment in these processes and to develop the best resins of which the processes are capable. The other area is the application of these techniques and recipes to specific types of resin already being produced in the Suspension Vinyl Resins Unit. To demonstrate the applicability of Process 5 and Process 6 to current resin production, Tables IX, X, XI, and XII present a comparison of several important properties of each with designated target properties (4). The examples in these tables are specific runs whose results are considered representative.

A. QXSM

QXSM-5 resin, the Process 5 counterpart of QYSM resin, exhibited significant improvements over standard production resin (Table IX), particularly in initial color of plasticized formulations and in fisheye content. In comparison with the target, which represents the properties a resin must have to be generally competitive in the current market, QXSM-5 resin is somewhat low both in specific viscosity and in apparent density. The low specific viscosity should not be considered an actual deficiency, since it can be altered readily by simple adjustment of the concentration of molecular-weight degrader in the charge or by small changes in the polymerization temperature. Some development work will be required to increase the apparent density without incurring the generally attendant reduction in absorption. However, the magnitude of this adjustment, from 28 to 32 pounds per cubic foot, is small enough that no great difficulty is foreseen, and in the meantime, the QXSM-5 resin now available should satisfy all but a few of the potential users.

QXSM-6 resin, it can be seen, deviates considerably from the target, principally in possessing too fine a particle size and in exhibiting poor initial color. The high incidence of hard resin particles should not be a significant problem. Process 6 is designed to produce practically no fisheyes, and the occurrence of fisheyes in the experimental resins is considered a result of experimental inadequacies. However, the other discrepancies will probably require considerable effort to attain acceptability.

B. QXSJ

QXSJ-5 resin, as produced in the period covered by this report, falls very little short of complete attainm nt of the target properties (Table X). In fact, insofar as the target properties accurately describe the actual requirements for the resin, QXSJ-5 resin must be considered ready for immediate commercialization.

The properties of QXSJ-6 resin differ significantly from QXSJ-5 resin only in lower absorption and smaller particle size. However, since increasing the particle size often involves a reduction in absorption, the readiness of QXSJ-6 for commercial application cannot be assumed.

C. QXSA and QXSC

The need for processes for manufacture of QYSA and QYSC resins was not so urgent as that for QYSM and QYSJ resin, and therefore experimentation with each was limited to on run at the end of the series. However, in both instances the resin produced under Process 5 conditions possessed properties quite close to those designated as the target (Tables XI and XII). It is apparent that the capabilities of Process 5 extend to both QYSA and QYSC resin, although the exact status in th se areas cannot be estimated accurately.

V. Operating Conditions

The nominal recipes for Process 5 and Process 6 were presented in an earlier report (1), and the recommended range of permissible variation in the concentration of each of the ingredients was listed also. No need for modification of the previously reported limits has become apparent in the studies to date. However, as further efforts are made to provide a complete line of resins with the desirable characteristics of the new processes, departure from these nominal conditions is inevitable. The following discussion of the significance of the several process variables should provide a basis for orderly progress into those areas which future needs will prescribe.

A. Recipe

According to Dr. Bauer, the two recipes which constitute Process 5 and Process 6 may be used interchangeably for production of a given type of resin. That is, a satisfactory resin may be made with either process. However, Process 5 produces a resin of superior color and electrical properties, whereas Process 6 yields a resin of relatively easy processing behavior and with virtually no fisheyes. When these slight differences cover properties of great importance to a particular customer, then the preference for one process over the other becomes significant. In other circumstances the only factor available for making a choice is the relative ease of operation and adaptability to existing equipment. Process 5, by virtue of its fewer components, is inherently easier to operate. Process 6, on the other hand, has proved more readily adaptable to equipment and procedures in use for standard production.

B. Suspending Agents

Of great significance in the technology of Process 5 and 6 is the strong emphasis which is placed on the suspending agent. The exact relationships between physical properti s of the suspending agent and characteristics of the resulting resin are not available, and will not be until considerable, small-scale, experimental effort has been devoted to that problem. However, regardless of the type of suspending agent, several generalizations will apply.

First, the suspending agent must be completely and uniformly dispersed in the autoclave. The only way to ensure this condition is to prepare the charge solution of suspending agent in water in such a way that guarantees the complete absence of gels and undissolved particles. Any of several methods may be employed to achieve this objective, but whatever the means, efficient production of quality resin demands a virtually perfect solution of suspending agent.

Another consideration is the accuracy of charging the suspending agent solution to the autoclave. Although the relatively low precision of this experimental series, which resulted from the many changes in operating conditions, did not permit definitive demonstration of this effect, Dr. Bauer

was quite specific in stating that very small errors in charging the suspending agent can cause significant variations in the characteristics of the resin produced. To satisfy this requirement for accurate charging will necessitate the provision of accurate weighing or metering devices and the implementation of a firm schedule for determining the solids content of the solution.

C. Additives

The recipe for Process 5 includes a buffer salt, sodium bicarbonate. Although there is nothing to indicate that sodium bicarbonate exclusively is required, Process 5 technology teaches that some buffer is necessary for consistent polymerization results.

In operations of Process 6 several polymerization additives are required. Mersolat, a sodium alkyl sulfonate detergent made in Germany, is included for its effect on particle characteristics. In this recipe calcium chloride is added as a modifier of the surfactant, and calcium carbonate is added as a buffer, again to improve reproducibility of results. The basis for the choice of these particular ingredients has not yet been determined, but no doubt exists that these or equivalent additives are necessary for desirable results. Worthy of particular notice in the case of Process 6 is the fact that previous local recipes, which utilized a different grade of poly(vinyl alcohol) and another surfactant, were incapable of producing resin with the required initial color and low fisheye content. Thus, until more specific information can be obtained, these original Process 6 ingredients must be considered an essential part of the process, even though their eventual replacement by domestic materials is quite desirable.

D. Other Polymerization Variables

Process 5 and Process 6 involve no new considerations with regard to the remaining polymerization variables. The molecular weight of the produced resin is determined by the polymerization temperature or may be modified by use of a degrader, such as trichloroethylene. Dilauroyl peroxide, regularly used locally as the polymerization initiator, is utilized in Process 5 and Process 6 as well. High standards of purity are demanded for monomeric vinyl chloride in both the new and the old processes, and the need for water free of dissolved salts and inert gases likewise is recognized in both.

E. Operating Techniques

Recipes and polymerization conditions constitute only a part of the technology on which Process 5 and Process 6 are based. Of perhaps equal importance is the concept of more careful operation throughout all phases of production. At every stage in the investigations into these new processes, from the initial inspection of Process 5 and 6 production facilities to the adaptation of the processes to local equipment, certain considerations were continually demonstrated as vital to successful operation.

Cleanliness over the entire commercial operation is a prime requisite for achievement of good results. Resin remaining in the autoclave from previous runs leads almost invariably to the formation of fisheyes by providing a hard particle on which excessive agglomeration can occur. In addition, particles exposed to several polymerization cycles are likely to exhibit greatly inferior color by virtue of repeated exposure to unnecessary heat. Any particles of dirt allowed to enter the autoclave in the water or monomer will produce similarly undesirable results; and, if the dirt should contain iron salts, for example, the adverse effect on initial color and thermal stability would be sufficient to overcome any advantage gained by improvements in the recipe or the process.

Accuracy in operation likewise has been shown essential to successful production. Previously successful commercialization of the principles of Process 5 and Process 6 has involved careful metering of all components of the autoclave charge, whereas local procedures have been accurate only when conditions were ideal. For example, in normal production the charge of water to the autoclave containing all the other charge ingredients has been measured by a probe in the sight glass of the autoclave. If there is any appreciable amount of foaming or if the sight glass is in any way occluded, two conditions which occur often and without available remedy, the amount of water actually charged can quite conceivably vary considerably from run to run. The quality of resin will vary with the charge, and hence reproducibility will be poor. Reversal of the order of charging so that the monomer is charged last can lead to even greater

chance of error. Accuracy of charging other ingredients of the recipe, while not so obviously inadequate, is in many instances suspect because of an absence of specific controls and checks, which in turn is due in large measure to a lack of fixed responsibility for the numerous steps in preparing and charging the polymerization ingredients.

In short, the secondary aspect of the new technology is care, some of which can be built into the operating procedures by provision of accurate, convenient, and simple measuring devices, and some of which can arise only from increased attention by operating personnel to specifically assigned, carefully detailed procedures.

When such reliability has been achieved, still another need remains. The often-fickle nature of polymerizations and the impossibility of perfect performance by operating personnel and equipment will always result in some poor runs. It is necessary that one poor run not be permitted to destroy the value of other, more satisfactory runs. The only way to guard against such an occurrence is to evaluate the resin for each run before it is included in a blend. The use of a common blow-down tank for the resin from several runs, as normal operating procedures require, makes timely evaluation impossible and may cause one poor run to spoil three or four times its own amount of resin. Tests must be devised and implemented to permit shift-by-shift, run-by-run evaluation of the resin. In instances where Process 5 and Process 6 have achieved commercial success, this has been done.

EQUIPMENT For the full-scale polymerizations which are discussed in this report, three production autoclaves (C15, C16, and C17 at Building 176, Location 514) were used. Each of these stainless steel autoclaves has a capacity of 4600 gallons. At the start of this series each autoclave was equipped with stainless steel Mixco impellers mounted on a vertical shaft, which was supported at the top of the autoclave by a mechanical seal and thrust bearing and at the bottom by a steady bearing. Agitation changes, which have already been discussed, involved only the replacement of the turbine impellers by a single, glass-coated, straight-vane impeller located near the bottom of the autoclave. Each autoclave was equipped originally with four side-mounted baffles; with the altered agitation three of these baffles were removed.

Alteration of the existing recovery system was required because, unlike in normal production, resin from each run had to be processed separately and because the experimental processes called for washing the resin before it was dried. The blowdown tank was therefore bypassed, and a "skin trap" was installed to remove any large aggregates of resin. The wet-stripping tank was used as a wash tank, but no steam was fed to it; the Bird centrifuge and the flash dryer were used without physical alteration. The path of the resin thus was from the autoclave through the skin trap into the wet-stripping (wash) tank, then to the Bird centrifuge and on into the flash dryer.

OPERATING PROCEDURE In many respects the operating procedures used in this series were the same as those used for regular production. However, some of the procedures were sufficiently different to warrant special mention.

I. Process 5

Water at 50°C was first charged to the autoclave to a level measured by insertion of a rod of a specified l ngth. The suspending agent, Methocel 10, was dissolved in water in a 50-gallon stainless steel drum (it was first slurried in 80°C water, then agitated in cold water until completely dissolved) and then pumped through a Ful-Flo filter into the autoclave. Sodium bicarbonate powder was then poured into the autoclave, followed by the dilauroyl peroxide catalyst. The autoclave vapor space was then evacuated for a half hour at a temperature of 50°C and a pressure of 2.2 psia, with the agitator turning slowly to aid diffusion of dissolved gases from the wat r. At the end of the evacuation a small amount of vinyl chloride was added to break the vacuum, and the autoclave was cooled to 30°C. The vinyl chloride charge was added at the lower temperature so that the autoclave pressure would not exceed that of the vinyl chloride supply line. Any required trichloroethylene was also added at that time. With the charge completed the agitator speed was increased to the specified level, and the autoclave was heated to operating temperature.

Polymerization was conducted until the specified termination point was reached, at which time the unreacted monomer was stripped from the autoclave while operating temperature was maintained. Stripping in the autoclave was made practical by the high degree of conversion (greater than 90 per cent) to which these polymerizations could be carried while preserving the desirable resin properties. The stripped charge was next cooled to 30°C and fed to the recovery system by application of nitrogen pressure to the autoclave. The slurry, which contained about 30 per cent total solids, passed first through the skin trap for removal of any large aggregates of resin, and then into the wash tank, where water was added until the total solids content was about 10 per cent. A liquid-level instrument on the wash tank actuated a motor valve which regulated the flow of slurry into the tank. From the wash tank the slurry passed into the Bird centrifuge, where as much wash water was added as was possible without incurring resin losses in the effluent liquid. The resin then entered the flash dryer, and when dry it was directed into a clean storage bin.

II. Process 6

Procedures for Process 6 were essentially the same as for Process 5, with the obvious exception of the charge. The suspending agent, Polyviol VZ 200, was first slurried in cool water and then dissolved in hot water prior to being pumped through a filter into the autoclave. Next the wetting agent, Mersolat, was dissolved in water and pumped into the autoclave. Calcium chloride was also predissolved before being pumped into the autoclave; calcium carbonate was added next, followed by the dilauroyl peroxide initiator. From this point on, the remaining procedures of Process 5 were followed exactly.

RESIN EVALUATION During all steps of operation, samples were taken to permit evaluation of all phases of the experimental series. A list of the samples and the tests performed on each sample is presented as Appendix B.

One of the most critical tests to which the resins were subjected is the "ICMT"* test, which was developed for evaluation of resins scheduled for use by Elm Coated Fabrics, Inc. The details of this test are available (5), and for the purposes of this report complete description is unnecessary. For the ICMT test the resin is first compounded and then worked on a

*ICMT = initial color mill test

two-roll mill for a total of ten minutes. A numerical value for initial color is obtained by measuring the reflectance of specimens taken from the mill at 5 minutes and 10 minutes milling time. The sum of the reflectances of the two specimens is the number which represents initial color, with a value greater than 100 denoting satisfactory performance.

The test listed frequently in Appendix B for aqueous surface tension refers, in addition to the standard determination by the Instron Tester, to a simplified method of determination which was received as a part of Process 5 and 6 control procedure. It consists merely of filling a specially designed pipet with the solution being tested and then observing the number of drops formed as the solution flows out of the pipet. Since both the concentration and the quality of the polymerization ingredients have an effect on the results of this test, it provides a simple check on the accuracy of charging the water-soluble components.

The wet-milling evaluation was an innovation utiliz d in this series. The test produced no new information, but it had the advantage of permitting evaluation of the resin immediately upon termination of the polymerization, before the resin was dried. Although the test itself is of little consequence, such a method would be of great value in decreasing the likelihood of one bad batch of resin being permitted to enter and contaminate an otherwise satisfactory blend. The Development Department now has work underway to modify existing test procedures to permit such wet-milling evaluations. New procedures will probably be available for use by the time the production facilities have been modified to provide temporary storage space for rejected resin.

The remaining evaluation procedures were standard tests, which have been performed and reported many times in the past. The results of all the tests have been evaluated, in order to obtain insight into the relationships between all measurable variables and the properties of the resultant resin. However, only those tests which were demonstrably significant are included in Tables III, V, and VIII.

CONCLUSIONS The experiments included in this report constitute a significant forward step in the development of high-quality, salable poly(vinyl chloride) resins. It is clear that future work in this area will refer back to this series as a plateau of understanding on which much, or perhaps all, additional effort will be based. This establishment of a base for future work is the most important conclusion which can be drawn from this series of runs. However, there are also several features of the study which permit more specific conclusions.

I. By use of Process 5 recipes and techniques, resins with exceptional initial color and with very low fisheye content can be produced immediately. The recipes are simple to employ,and the techniques are not difficult. But Process 5 cannot be operated with the Mixco turbine impellers. Changing to a Pfaudler agitation system, or its equivalent, is an absolute requisite for utilization of Process 5 on production scale.

II. Process 6 can be employed in present autoclaves with current agitation systems, but the resin cannot be expected to have the same excellent initial color as was achieved in Process 5. It is likely that the color of Process 6 resins can be improved to an acceptable level by suitable changes in recipe and in polymerization conditions; and if this can be done, the virtual freedom from fisheyes of the Process 6 resins will be of great value.

III. The adaptability of Process 5 resins to immediate production of current grades of resin has been demonstrated adequately. To the extent that local tests and specifications are true representations of resin requirements, the conclusion can be drawn that QXSJ-5 and QXSM-5 are suitable replacements for their counterparts in present production. However, it must be recognized that the ultimate test of any resin is satisfactory performance for the customer, and therefore final judgment of Process 5 resins must await completion of customer evaluations, such as those already in progress at Elm Coated Fabrics, Inc.

IV. The acquisition of Process 5 and Process 6 places the Company in a position to compete for practically all the major resin accounts in the suspension poly(vinyl chloride) field. The capability is now in hand for production of resin with initial color at the very least equal to competitive resins such as Presto, Marvinol, and Escambia 2185. The only level not yet attained is that filled by Escambia 3185 resin in the premium-quality, "Crystallite" area. This is not to say that Process 5 resins will now overwhelm the market. The particle properties which determine preblend characteristics, ease of extrusion, and the like are not yet wholly satisfactory, and still other deficiencies may emerge when customer demands are heard. But with initial color and fisheye content under control, accomplishing the relatively slight improvements still required in particle characteristics should not present any great difficulties.

V. The optimistic tone of these conclusions is based on reliable results; however, one major condition must be attached to any such predictions. Process 5 is capable of producing these excellent results only if it is properly employed. The Suspension Vinyl Resins Unit at Location 514 is not fit for production of Process 5 resins in its present form. The unit must be altered in accordance with forthcoming recommendations if the full benefit of Process 5 is to be enjoyed.

RECOMMENDATIONS Because of the abrupt shift in thought effected by the emergence of Process 5 technology, specific directions of future effort should be laid out at this point in order to control any tendency toward over-zealous utilization of the new information. The following recommendations are therefore presented, along with a strong urging that they be analyzed most carefully before any alternatives are adopted.

I. Production Department

A. Pfaudler-type agitation should be installed in all production autoclaves as soon as possible. No Process 5 runs should be attempted with the Mixco agitation system.

B. Operational changes in Process 5 conditions should be confined within the range of conditions which have already been explored. Specified limits in recipes and in operating conditions, based in large measure on Dr. Bauer's suggestions, are listed in a previous report (2).

C. A long-range program should be begun immediately for the orderly renovation of the Suspension Vinyl Resins Unit. First, the charging system should be simplified as much as possible to reduce the number of possible sources of contamination and to provide maximum ease of accurate charging and operating. Second, the drying system must be overhauled and modified to ensure that resin hold-up or recycle is eliminated. If reasonable efforts cannot effect this improvement, then the drying system should be replaced completely. Even the very finest process control cannot overcome the damaging effects of an inadequate recovery system.

D. Steps must be taken to impress upon operating personnel the absolute necessity for faithful, unvarying adherence to the newly established operating procedures. The sensitivity of Process 5, and Process 6 as well, to relatively small changes in process conditions demands the very greatest care of operation, without which the major advantages of the new technology will be lost.

II. Development Department

A. Pilot-scale experiments will be undertaken for the development of a firmer understanding of the principles behind Process 5 and Process 6. Modifications of the nominal recipes, including experimentation with alternative materials for use as suspending agents, surfactants, and other additives, will be investigated. The 10-gallon and 15-gallon autoclaves are satisfactory generally for primary experiments with novel operating conditions.

B. For reliable, reproducible engineering data, larger pilot-scale autoclaves must be made available. The ideal size probably lies between 30 and 50 gallons. Autoclaves larger than this would produce good results, but the cost of operation would be unnecessarily high. It is therefore our intention that an autoclave in the size range of 30 to 50 gallons be obtained and that this autoclave be used as the site for basic work on the variations in particle characteristics which will be necessary as the area of application of Process 5 and Process 6 resins becomes broader.

C. Intermediate-scale polymerizations will be undertaken in the available 500- and 600-gallon autoclaves to pursue the leads offered by the smaller equipment. In addition, attempts will be made to establish scale-up factors to p rmit accurate correlation between development and production operation.

ACKNOWLEDGMENTS In the operations described in this report the cooperation and assistance of the following persons are gratefully acknowledged.

I. The Suspension Resins Department under J. C. McIntosh of the Production Department provided the equipment and personnel for the polymerizations.

II. Personnel of R. G. Massey's group of the Development Department were responsible for operating pilot-scale autoclaves and for tray-drying the numerous slurry samples from the production runs.

III. P. T. McCoy, of J. J. Brezinski's group of the Development Department, developed and conducted the novel testing procedures introduced in this series.

IV. Personnel of the Quality Control Laboratory, under the direction of J. W. Chatfield and C. J. Haines, exp nded considerable effort to provide the evaluation results as rapidly as possible, thereby reducing delays between runs in the series.

V. Dr. Hans Bauer, besides providing the polymerization recipes and the technology for the most profitable adaptation to local equipment, contributed greatly to the present measure of success by his never-ending insistence on attention to minute details. In addition, much of the comment of this report is based on his explanations, stated or implied, of the relative importance of the several aspects of the study.

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LABORATORY ACCOUNT: 2612

PERIOD: December 1, 1959 to February 15, 1960

Attachments:
12 Tables
2 Appendixes

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TABLE I

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Operating Conditions for Initial Experimentation with Process 5 in Production Autoclav s

Autoclaves: 4600-gallon, stainless steel with falling-film brine jacket
Agitation: Three 31.8-inch, 7-bladed Mixco impellers and 4 side-mounted baffles
Constant operating conditions: Autoclave charge level, 90 per cent; polymerization temperature, 57 °C; concentration of dilauroyl peroxide catalyst, 0.088 per cent of VCl
Type of resin produced: QXSM-5

Expt No.	Autoclave No.	Monomer-to-Water Ratio	Concentrations, per cent of VCl		Agitator Speed, rpm	Operating Pressure, psig	Reaction Time, hr	Termination Point
			Methocel 10	Sodium Bicarbonate				
1W	C15	37/63	0.09	0.11	84	124	13.0	Pressure drop
2W	C15	30/70	0.09	0.11	84	127	12.2	Pressure drop
3W	C15	30/70	0.068	0.083	84/110	126	13.25	Pressure drop
5W	C15	37/63	0.09	0.11	110	124	11.5	Pressure drop
6W	C15	37/63	0.13	0.127	110	127	12.25	Pressure drop
7W	C15	37/63	0.18	0.127	110	130	10.75	Pressure drop
8W	C16	37/63	0.15	0.153	110	132	10.5	Time

TABLE II

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Operating Conditions for Experimentation with Process 5 in Production Autoclaves

Autoclaves: 4600-gallon, stainless steel with falling-film brine jacket
Agitation: One 51-inch, straight-bladed, glass-lined Pfaudler-type agitator and 1 side-mounted baffle
Constant operating conditions: Autoclave charge level, 75 per cent; monomer-to-water ratio, 37/63

Expt No.	Autoclave No.	Concentrations, per cent of VCl				Polymerization Temperature, °C	Agitator Speed, rpm	Operating Pressure, psig	Reaction Time, hr	Termination Point	Type Resin Produced
		Methocel 10	Sodium Bicarbonate	Dilauroyl Peroxide	Trichloro-ethylene						
9W	C15	0.10	0.13	0.088	-	57	127	129	10	Time	QXSM-5
11W	C15	0.09	0.12	0.088	-	57	123	127	10	Time	QXSM-5
16W	C15	0.113	0.15	0.12	-	57	125	125	10.2	5 psi ΔP	QXSM-5
18W	C16	0.13	0.15	0.12	-	57	125	124	10.5	5 psi ΔP	QXSM-5
19W	C15	0.15	0.15	0.12	-	57	125	124	11	25 psi ΔP	QXSM-5
13W	C15	0.10	0.13	0.088	1.0	57	128	130	13	ΔP	QXSJ-5
15W	C16	0.12	0.15	0.165	1.0	57	125	125	9.7	5 psi ΔP	QXSJ-5
17W	C17	0.13	0.15	0.165	1.0	57	125	127	9.5	5 psi ΔP	QXSJ-5
20W	C17	0.135	0.15	0.165	1.0	57	125	125	9.75	25 psi ΔP	QXSJ-5
21W	C16	0.13	0.15	0.195	2.2	57	125	124	9.5	5 psi ΔP	QXSA-5
22W	C15	0.15	0.15	0.26	-	50	125	102	13.5	5 psi ΔP	QXSC-5

TABLE III

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Evaluation Data for Process 5 Resins Produced Experimentally in Production Autoclaves

(See Tables I and II for Operating Conditions)

Expt No.	Nominal Resin Type	Specific Viscosity	Apparent Density, lb/cu ft	Absorption, per cent	Persorption, per cent	Particle Characteristics				Hard Resin Particles Per Square Foot on Mil Thickness			Color	
						Median, microns	Dispersion	Skew	Through 270-Mesh, per cent	6	10	17.5	ICWT Mill Test	Press "C" Test
1W	QXSM-5	0.175	35	46	172	300	-	-	2	50	200	300	100	74
2W	QXSM-5	0.177	31	45	183	330	-	-	1	200	300	300	109+	80
3W	QXSM-5	0.178	34	46	152	300	-	-	-	70	75	20	107	83
5W	QXSM-5	0.183	33	62	254	280	-	-	8	50	200	200	99+	85
6W	QXSM-5	0.181	29	54	203	235	-	-	2	40	200	200	96+	82+
7W	QXSM-5	0.183	29	49	185	110	-	-	13	70	200	70	103+	71
8W	QXSM-5	0.183	29	55	184	120	-	-	14	50	100	100	109	73
9W	QXSM-5	0.175	30	50	223	160	83	+0.03	6	100	100	20	111	73
11W	QXSM-5	0.173	26	49	218	105	80	+0.40	8	20	30	20	103+	82
16W	QXSM-5	0.175	28	54	-	170	75	-0.07	3	2	2	5	90+	80+
18W	QXSM-5	0.177	28	49	169	115	65	+0.15	10	10	20	5	107+	79+
19W	QXSM-5	0.176	27	47	174	90	35	0	15	2	5	5	105+	84+
13W	QXSJ-5	0.142	29	50	-	180	-	-	6	5	50	50	109+	82
15W	QXSJ-5	0.145	31	50	-	150	83	+0.09	6	0	10	5	116	74+
17W	QXSJ-5	0.143	30	47	-	145	70	-0.15	3	5	10	0	106+	73+
20W	QXSJ-5	0.147	31	42	156	115	45	0	10	0	5	2	112	75
21W	QXSA-5	0.123	32	44	-	165	75	0	7	10	10	5	108	73
22W	QXSC-5	0.217	30	51	-	115	45	0	8	2	2	2	101+	84

NOTE: Color tests to be comparable must be made on standard plaque thicknesses. The + or - associated with some reflectance values indicates a plaque slightly thicker or thinner than prescribed. Thus, a reflectance with a + is an uncorrected reading which is lower than the true value since the plaque is thicker and absorbs more light.

TABLE IV

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Operating Conditions for Experimentation with Process 6 in Production Autoclaves

Autoclaves: 4600-gallon, stainless steel with falling-film brine jacket
Agitation: Three 31.8-inch, 7-bladed Mixco impellers and 4 side-mounted baffles
Constant operating conditions: Autoclave charge level, 90 per cent; monomer-to-water ratio, 33/67; agitator speed, 110 rpm

Expt No.	Autoclave No.	Concentrations, per cent of VCl						Polymerization Temperature, °C	Operating Pressure, psig	Reaction Time, hr	Termination Point	Type Resin Produced
		Polyviol VZ 200	Mersolat	Calcium Chloride	Calcium Carbonate	Dilauroyl Peroxide	Trichloro-ethylene					
10W	C16	0.33	0.098	0.047	0.0094	0.070	0.17	57	129	14.25	85 psig	QXSM-6
12W	C17	0.31	0.095	0.047	0.0094	0.080	1.0	58	132	14	60 psig	QXSJ-6
14W	C16	0.25	0.070	0.045	0.0080	0.080	1.0	58	134	14.25	60 psig	QXSJ-6

TABLE V

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Evaluation Data for Process 6 Resins Produced Experimentally in Production Autoclaves

(See Table IV for Operating Conditions)

Expt No.	Nominal Resin Type	Specific Viscosity	Apparent Density, lb/cu ft	Absorption, per cent	Persorption, per cent	Median, microns	Dispersion	Skew	Through 270-Mesh, per cent	Hard Resin Particles Per Square Foot on Mil Thickness			ICMT Mill Test	Press "C" Test
										6	10	17.5		
10W	QXSM-6	0.172	26	50	198	65	-	-	28	50	70	30	40+	63-
12W	QXSJ-6	0.139	31	35	-	55	-	-	31	5	50	5	97+	73-
14W	QXSJ-6	0.140	30	45	-	65	-	-	30	5	30	10	89	63-

TABLE VI

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Operating Conditions for Experimentation with Process 5 in Development Autoclaves E8 and E16

Autoclave E8: 10-gallon, stainless steel with circulating-water jacket, agitated by two 7-inch, 4-bladed Mixco impellers and fitted with 4 side-mounted baffles
Autoclave E16: 500-gallon, glass-lined with circulating-water jacket, agitated by one 36-inch, 3-bladed, glass-coated, sloped impeller and fitted with 1 suspended baffle

Expt No.	Autoclave No.	Monomer-to-Water Ratio	Concentrations, per cent of VCl			Polymerization Temperature, °C	Agitator Speed, rpm	Operating Pressure, psig	Reaction Time, hr	Termination Point
			Methocel 10	Sodium Bicarbonate	Dilauroyl Peroxide					
696	E8	38/62	0.0875 (1)	-	0.0625	53.5	300	141	12	Time
697	E8	38/62	0.0875 (1)	-	0.0625	53.5	300	147	14.5	Pressure drop
699	E8	38/62	0.0875 (1)	0.1125	0.0625	53.5	300	149	17	Pressure drop
700	E8	38/62	0.0875 (1)	0.1125	0.150	53.5	300	112	12	Time
701	E8	38/62	0.0875	0.1125	0.0625	53.5	300	132	12	Time
702	E8	38/62	0.0656	0.0844	0.0625	53.5	300	127	12	Time
703	E8	38/62	0.0656	0.0844	0.0625	53.5	400	126	12	Time
704	E8	38/62	0.0875	0.1125	0.0625	53.5	400	113	12	Time
705	E8	38/62	0.0875 (1)	0.1125	0.150	53.5	300	113	6	Time
706	E8	35/65	0.0875	0.1125	0.150	58	250	132	8	Pressure drop
707	E8	35/65	0.0875	0.1125	0.150	58	250	128	8.3	Pressure drop
708	E8	35/65	0.0875	0.1125	0.150	58	300	128	8.6	Pressure drop
709	E8	35/65	0.0875	0.1125	0.150	58	350	130	7.8	Pressure drop
710	E8	35/65	0.0875	0.1125	0.150	58	300	130	8.5	Pressure drop
711	E8	35/65	0.0875	0.1125	0.150	58	350	128	8	Pressure drop
715	E8	35/65	0.0875	0.1125	0.150	58	250	128	8	Pressure drop
716	E8	35/65	0.0875	0.1125	0.150	58	300	129	10	Pressure drop
717	E8	35/65	0.0875	0.1125	0.150	58	350	128	13	Pressure drop
29	E16	37/63	0.10	0.15	0.10	57	144	125	12.5	Pressure drop
30	E16	33/67	0.13	0.15	0.10	57	142	125	13	Pressure drop

(1) The suspending agent was Methocel 15, rather than Methocel 10.

TABLE VII

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Operating Conditions for Experimentation with Process 6 in Development Autoclave E8

Autoclave: 10-gallon, stainless steel with circulating-water jacket
Agitation: Two 7-inch, 4-bladed Mixco impellers and 4 side-mounted baffles
Constant operating conditions: Autoclave charge level, 90 per cent; monomer-to-water ratio, 33/67; concentration of dilauroyl peroxide catalyst, 0.08 per cent of VCl

	Concentrations, per cent of VCl										
Expt No.	Polyviol VZ 200	Mersolat	Anhydrous Calcium Chloride	Calcium Carbonate	Trichloro-ethylene	Lauric Acid (1)	Polymerization Temperature, °C	Agitator Speed, rpm	Operating Pressure, psig	Reaction Time, hr	Termination Point
718	0.33	0.0975	0.0472	0.0095	1.5	-	57	250	125	17.5	60 psig
719	0.33	0.0975	0.0472	0.0095	1.5	-	57	350	128	17	60 psig
720	0.33	0.0975	0.0472	0.0095	1.5	0.3	58	250	128	18	46 psig
721	0.25	0.07	0.045	0.008	1.0	0.3	58	250	130	16	60 psig
722	0.33	0.0975	0.0472	0.0095	-	-	57	250	126	15	60 psig

(1) Lauric acid in these polymerizations was Wecoline ABL 70 from E. F. Drew Company.

TABLE VIII

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Evaluation Data for Process 5 and Process 6 Resins Produced in Pilot-Scale Autoclaves

(See Tables VI and VII for Operating Conditions)

Expt No.	Process	Specific Viscosity	Apparent Density, lb/cu ft	Absorption, per cent	Persorption, per cent	Particle Characteristics				Hard Resin Particles Per Square Foot on Mil Thickness			ICMT Mill Test	Press "C" Test
						Median, microns	Dispersion	Skew	Through 270-Mesh, per cent	6	10	17.5		
696	Process 5	0.164	17	208	-	>440	-	-	-	-	-	-	99	-
697	Process 5	0.167	31	46	-	>440	-	-	-	-	-	-	129	-
699	Process 5	0.165	25	64	-	>440	-	-	-	-	-	-	110	-
700	Process 5	0.190	24	66	-	110	-	-	-	-	-	-	85	-
701	Process 5	0.180	17	197	-	>440	-	-	-	-	-	-	61+	-
702	Process 5	0.166	14	227	-	410	-	-	-	-	-	-	91	-
703	Process 5	0.182	12	-	-	315	-	-	-	-	-	-	-	-
704	Process 5	0.189	19	-	-	>440	-	-	-	-	-	-	118	-
705	Process 5	0.181	8	-	-	60	-	-	-	-	-	-	-	-
706	Process 5	0.165	30	49	-	70	-	-	22	-	-	-	93	-
707	Process 5	0.171	27	70	-	>440	-	-	-	-	-	-	126	-
708	Process 5	0.167	27	64	-	>440	-	-	-	-	-	-	103+	-
709	Process 5	0.170	27	60	-	>440	-	-	-	-	-	-	111	-
710	Process 5	0.175	28	60	-	>440	-	-	-	-	-	-	121+	-
711	Process 5	0.171	28	54	-	>440	-	-	-	-	-	-	115	-
715	Process 5	0.172	27	62	-	>440	-	-	-	-	-	-	106	-
716	Process 5	0.167	26	70	-	>440	-	-	-	-	-	-	98+	-
717	Process 5	0.167	21	102	-	>440	-	-	-	-	-	-	-	-
718	Process 6	0.131	28	48	210	170	78	+0.03	5	10	30	10	78	59-
719	Process 6	0.131	33	38	194	315	107	+0.009	19	75	200	200	79	61-
720	Process 6	0.132	32	41	171	275	120	-0.45	10	10	70	40	112+	61-
721	Process 6	0.138	31	39	-	240	-	-	16	10	20	30	120+	-
722	Process 6	0.176	26	60	-	180	65	0	2	30	30	30	29+	-
E16-29	Process 5	0.174	28	52	182	155	68	+0.037	6	-	-	-	-	-
E16-30	Process 5	← Not evaluated →												

TABLE IX

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Status of Development of Process 5 and Process 6 Resins

	Target	QYSM (Average)	QXSM-5 (C16-18W)	QXSM-6 (C16-10W)
	QXSM			
Specific viscosity	0.170	0.170	0.177	0.172
Apparent density, lb/cu ft	32	30	28	26
Absorption, per cent	50	31	49	50
Median particle size, microns	150	100	115	65
ICMT mill test	>100	45	107+	40+
Press "C" test	-	-	79+	63
Hard resin particles, per square foot on 17.5-mil film	<10	10	5	30

NOTE: (1) The average values for QYSM resin are derived from Blend 134 through Blend 148. Some of the properties were not measured for all the blends, but the average includes all available information.

(2) The examples used for QXSM-5 and QXSM-6 resin are taken from specific runs which are considered representative of the over-all series.

TABLE X

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Status of Development of Process 5 and Process 6 Resins

QXSJ

	Target	QYSJ (Average)	QXSJ-5 (C17-17W)	QXSJ-6 (C17-12W)
Specific viscosity	0.145	0.144	0.143	0.139
Apparent density, lb/cu ft	32	37	30	31
Absorption, per cent	50	28	47	35
Median particle size, microns	150	-	145	55
ICMT mill test	>100	59	106+	97+
Press "C" test	-	-	73+	73
Hard resin particles, per square foot on 17.5-mil film	<10	9	0	5

NOTE: (1) The average values for QYSJ resin are derived from Blend 224 through Blend 230. Some of the properties were not measured for all the blends, but the average includes all available information.

(2) The examples used for QXSJ-5 and QXSJ-6 resin are taken from specific runs which are considered representative of the over-all series.

TABLE XI

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Status of Development of Process 5 Resins

QXSA

	Target	QYSA (Blend 2905)	QXSA-5 (C16-21W)
Specific viscosity	0.115	0.116	0.123
Apparent density, lb/cu ft	32	38	32
Absorption, per cent	-	36	44
Median particle size, microns	<200	-	165
ICMT mill test	-	79	108
Press "C" test	-	-	73
Hard resin particles, per square foot on 17.5-mil film	<20	5	5

NOTE: The example of current production of QYSA represents a single blend, the only one for which data are now available.

TABLE XII

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Status of Development of Process 5 Resins

QXSC

	Target	QXSC-5 (C15-22W)
Specific viscosity	0.210	0.217
Apparent density, lb/cu ft	32	30
Absorption, per cent	50	51
Median particle size, microns	<200	115
ICMT mill test	>100	101+
Press "C" test	-	84
Hard resin particles, per square foot on 17.5-mil film	<10	2

NOTE: Since QYSC resin has not been produced locally, regular production results cannot be presented.

APPENDIX A

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Comparison of the Calcium Carbonate and Calcium Chloride Used in Local Process 6 Polymerizations with the Materials Used in Germany

	$CaCO_3$		$CaCl_2$	
Impurity	Local, per cent	German, per cent	Local, per cent	German, per cent
Mg	0.01	0.2	0.01	0.001
Si	0.01	0.05	0.01	0.01
Fe		0.1		
Al		0.1		
Cu		0.001		
Total	0.02	0.451	0.02	0.011

NOTE: These data were obtained by semiquantitative analysis by the flame spectrometer and are considered reproducibl within 30 per cent.

APPENDIX B

SUSPENSION POLYMERIZATION OF VINYL CHLORIDE

Sampling and Testing Schedule during Production-Scale Experimentation with Process 5 and Process 6

Sample	Tests
Autoclave Ingredients	
A. Ion-exchanged water used for the charge	pH, chloride ion, iron, surface tension, conductivity
B. Suspending agent solution	pH, total solids, gel analysis
C. Autoclave water charge before catalyst addition	pH, chloride iron, iron, surface tension, conductivity
Polymerization Slurries	
A. Hourly samples	pH, microscopic inspection
B. Final slurry after stripping (filtrate)	pH, chloride ion, iron, surface tension, conductivity
C. Final slurry after stripping (resin)	
1. Decanted wet resin 2. Tray-dried resin	wet-milling evaluation (optional), specific viscosity, heating loss, ICMT mill test
Flash-Dried Resin	specific viscosity, heating loss, apparent density, sieve analysis, absorption, hard resin particles at 6, 10, and 17.5 mils, ICMT mill test, 7905 preblend evaluation, photomicrographs at 75X

B:20:220-S2

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