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STATUS REPORT

SUSPENSION POLY(VINYL CHLORIDE) RESINS: QXPA PROCESS DEVELOPMENT IN PRODUCTION EQUIPMENT WITH "CELLOSIZE" QP-4400 SUSPENDING AGENT

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SUMMARY Experimental polymerizations have been performed in the 4600-gallon production autoclave C14, in ord r to develop a process for manufacturing QXPA resin. This resin is needed as a plastisol additive for companion sales with QYNV and QXKV-2 resins, for which considerable sales volumes are dependent upon availability of the additive resin.

As a result of this program, a process has been developed for interim manufacture of QXPA resin in quantities sufficient to fill current needs. In this process, agitation is provided by a Pfaudler-type, retreat-curve impeller, th same as is used for standard production of Process 5 resins. The suspending agent is CELLOSIZE QP-4400, which promotes formation of the glassy beads of polymer which are characteristic of a satisfactory plastisol additive resin.

The resin produced by the interim process possess s excellent rheological properties, equal or superior to comp titive resins. The only significant shortcoming is a questionable heat stability, which may necessitate a change to a suspending agent other than CELLOSIZE QP-4400.

The reproducibility of the interim process was demonstrated by successfully conducting six consecutive production runs, in which the essential features of the polymerization were maintained while several minor conditions were varied. However, some undetected variables which have caused difficulties in the past have not yet been eliminated and may emerge at any time to override the benefits of recently imposed improvements.

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During the course of the experimentation, frequ nt inconsistencies in results were observed. These results, upon analysis, provided a basis for a fresh evaluation of the effects of such process variables as agitation, suspending agent concentration, autoclave charge level, and monomer-to-water ratio.

INTRODUCTION The Development Department was called upon to develop a process for the manufacture of QXPA resin in the present facilities of the Suspension Vinyl Resins Unit at Location 514. A considerable urgency was attached to the problem, because a significant volume of sales of QYNV and QXKV-2 resins depends upon the availability of a suitable plastisol additive resin for blending with these dispersion resins by our customers. Obtaining competitive additive resins for resale as a stopgap measure was not desirable, since the known competitive products do not always perform adequately with QYNV and QXKV-2 resins. Thus, the preferred solution of the problem was the rapid development of our ability to produce QXPA resin.

The resin sought in this series was one which would perform well as a plastisol additive in decreasing the viscosity while increasing the resin content of the plasticized formulation. The particle structure known to produce these desirable properties is that of small, spherical, glassy beads of polymer, with low porosity and high apparent density.

A resin possessing these properties was produced in production equipment over a year ago. Agitation was provided by Mixco turbines, and Elvanol 50-42 (E88H) was the suspending agent. However, for the current series, use of the Pfaudler-type, retreat-curve impellers was desired by the Production Department, so that equipment changes would not be necessary when shifting production from one type of resin to another. The use of CELLOSIZE QP-4400 as the suspending agent was undertaken on the basis of several completely successful polymerizations in the 600-gallon autoclave E17 (1).

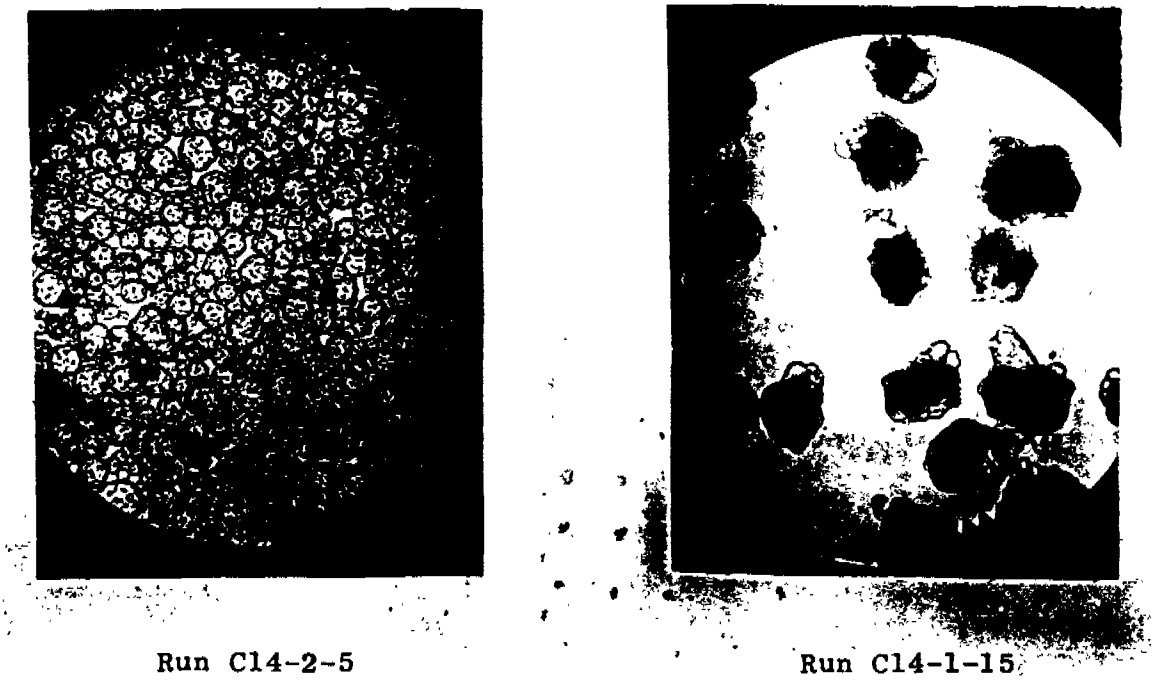
This report deals with the operation of production autoclave C14 on an experimental basis to scale up the succ ssfully applied conditions of operation in E17 autoclave for manufacturing QXPA resin. Topics discussed are the present status of the program, the difficulties involved in reaching the present status, and the effects of variables observed during the experimentation.

DISCUSSION The experimental production of QXPA resin in C14 autoclave was begun under the same conditions which had been employed successfully in the 600-gallon autoclave E17 (1). Early in the program a resin was produced (Table I, Run C14-2-5) which possessed the properties required of a plastisol additive resin. However, the results of that polymerization could not be duplicated in two further attempts at the same conditions (Table I, Runs C14-1-6 and C14-2-16).

The discrepancy which changed this experimental series from a simple scale-up to a considerable task was th onset of agglomeration during the latter stages of the polymerizations. The result of this agglomeration was that, instead of small, glassy beads of polymer (median size 75 to 90 microns), large aggregates (150 to 200 microns) of those beads were obtained. Comparison of the photographs of resin from a successful run (C14-2-5) with an agglomerated run (C14-1-15) in Figure 1 below clearly demonstrates the difference between satisfactory and unsatisfactory resin. The large agglomerat s typified in Run C14-1-15 are wholly unsuited for use as a plastisol additive.

Figure 1

Satisfactory and Unsatisfactory Particle Geometry for Plastisol Additive Resins



Run C14-2-5 Run C14-1-15

A great deal of unforeseen effort was expended before this problem of agglomeration was brought under control. The result of this effort was the development of a process for interim manufacture of QXPA resin in quantities sufficient to fulfill immediate needs while a more reproducible and more productive process is developed. The results of all the runs in this series are summarized in Table I and Table II. Photographs of representative resins are presented in Figures 2, 3, and 4. The extent of deviation from run to run is demonstrated again in Figure 5 where photographs of the resin slurry of two runs ar compared at equivalent stages in each polymerization.

Status of Development Program

The interim process which has emerged from this period of operation has already been published (2), and is presented in Appendix A. It provides for use of the Pfaudler-type, retreat-curve impellers which are used for standard production of Process 5 resins at the Suspension Vinyl Resins Unit. The use of CELLOSIZE QP-4400 as the suspending agent, sodium bicarbonate as the buffer salt, and dilauroyl peroxide as the polymerization initiator involves no novel techniques and provides a simple, straightforward process. The disadvantage which prohibits the process from being specified for full commercial production is low productivity, resulting from the 50 per cent charge level and from the lengthy, 2-hour mix period prior to heat-up.

The resin which is expected from this interim proc ss is equal or superior to competitive resins in the viscosity of blended formulations and in the tensile strength of the resultant plastics. Disadvantages are two: somewhat inferior heat stability, due probably to the QP grade of CELLOSIZE, and slightly large particle size, which results in graininess in thin film applications, but which can very likely be overcome by moderate adjustment of agitator speed.

The reproducibility of the results obtained with this process has been demonstrated (Runs C14-3-23 through C14-2-28, Table II and Figure 4). It is noteworthy that the major characteristics of the plastisol additive resin were retained in all six of these runs, even though some slight changes in recipe and operation were imposed. However, the full importance of the several process variables, some controllable and some not, has not yet been established, and consequently a vestige of doubt hovers over these seemingly well-established conclusions.

Effects of Variables

During the course of this series, many changes were imposed on the process variables in attempting to overcome the problem of agglomeration. Although these changes were insufficient to establish definitely the effects of the variables, several tentative interpretations can be made. These are proposed explanations of observed behavior and are based largely on intuitive reasoning rather than on concrete observations.

Agitation

The agitation system in which QXPA resin is produced seems to be a primary factor in determining whether a resin will be satisfactory or not. Inadequate agitation does not provide enough turnover in the autoclave to maintain the suspension, and huge agglomerates of resin are formed. Likewise, too much agitation results in formation of very small initial particles and greatly increased surface area of the monomer droplets. Th suspending agent often is unable to provide adequate protection over this increased area, particularly in the later stages of the polymerization, and agglomeration of the small, glassy beads occurs.

The degree of agitation is determined by impeller speed, by the type of baffles installed, and by the relative position of the impeller and the baffles. At the start of this series, C14 autoclave was equipped with pipe baffles, as are the other production autoclaves in use for manufacture of Process 5 resins. The pattern of agitation in this system differed greatly, based on visual observation, from the pattern in E17 autoclave (4). Changes were required; namely, installation of 5-inch flat baffles and relocation of the impeller at a higher position in the autoclave - before the initially successful run (C14-2-5, Table II) could be performed. Then, with basic similarity in agitation pattern, the impeller speed had to be decreased from 100 rpm to 75 rpm before the reproducible interim process was found.

Concentration of CELLOSIZE QP-4400

After the basic particle size has been set by the agitation, enough suspending agent must be included to protect these monomer globules from coalescing early in the polymerization and from agglomerating later on. Other than in providing this protection, the concentration of the CELLOSIZE QP-4400 has little effect on the size or the nature of the particles. The CELLOSIZE should be used at the lowest possible level, since excessive amounts reduce the polymer heat stability significantly.

Autoclave Charge Level

The 75 per cent charge level which is in standard use in the Suspension Vinyl Resins Unit was initially used in this series as well. However, in order to decrease the distance from the impeller to the surface of the charge, the charge level was later reduced to 50 per cent of capacity. The principle behind this action was that, as compared with E17, the average particle in C14 autoclave had to travel a greater distance from the impeller. The particle thus would lose more energy than its counterpart in the smaller autoclav , and the lower energy level might lead to agglomeration. Increased agitation would impart more energy to the particle, but could lead to agglomeration, as previously pointed out, as a result of lost protection later in the run. The extent of this equipment size effect has not been definitely established, but it may explain why many competitive resins are produc d in autoclaves considerably smaller than those used in our Suspension Vinyl Resins Unit.

Five of the six successful polymerizations were conducted with a 50 per cent charge level, and this was specified as a requirement for the interim process. However, since th latest run at 75 per cent of capacity (Run C14-3-26, Table II) produced a resin with satisfactory properties, the permissible charge level may reasonably be expected to increase as familiarity with the interim process is gained.

Monomer-to-Water Ratio

At one point in the series (Runs C14-1-9, -2-10, and -3-11, Table I) the monomer-to-water ratio was decreased to 30/70, to determine whether greater dilution would prevent agglomeration. The opposite occurred; agglomeration was even worse than when the ratio was 35/65. This was true even when the CELLOSIZE concentration was increased to the point which would provide the same aqueous-phase viscosity present at the 35/65 ratio. Hence, a logical future step would be to increase the monomer-to-water ratio; perhaps the suspending action would be improved, just as it was reduced in going the other way.

Oxygen Concentration

Before charging the vinyl chloride to these runs in C14, a thorough evacuation procedure was carefully followed. The autoclave vapor space was then tested for oxygen content. Within the reliability of the sampling and measuring techniques employed (Analytical Systems Trace Oxygen Analyzer), the oxyg n level was very low (less than 50 ppm) in all the runs included in this report, with the result that the effects of oxygen have not been considered in this series.

Buffer Salt

The use of sodium bicarbonate as a buffer salt certainly exerts an influence on the polymerization. In bomb polymerizations it increases the tendency to form spherical particles, but also apparently promotes the formation of translucent spheres of polymer, which are of uncertain identity and which contribute significantly to poor heat stability. Isolating these particles, analyzing them, and identifying the role of sodium bicarbonate in their formation are included in prospective plans for future work.

Physical Properties of the Polymerizing Mixture

Inspection of the autoclave slurry samples taken during the early stages of each polymerization uncovered a phenomenon which may prove of benefit in explaining some of the inconsistent results obtained in this series. Within th first three or four hours of polymerization, the consistency of the resin slurry provided a forecast of the eventual outcome of the run. The slurry from runs which turned out successfully had a "creamy" feel when rolled between the thumb and forefinger. On the other hand, the slurry from unsuccessful runs did not feel "creamy", but rather it rolled together in th fingers somewhat like wet tissue paper.

It seems likely that a difference which could be detected so readily by feeling the resin should also appear as differences in measurable physical properties. Therefore, routine laboratory analyses were performed on filtered autoclave slurry samples to determine viscosity, surface tension, pH, and content of gels and insolubles in the water phase of the slurry. The analyses demonstrated differences from run to run, and from sample to sample, but the variations were essentially the same for all runs investigated, whether or not a satisfactory resin was produced. As a further test, samples of the aqueous charge to several of the C14 runs were used as the aqueous charge in laboratory polymerizations in 50-ml glass bombs. There was some variation in resin from bomb to bomb, not unusual in polymerization on this scale, but the differences could not be related to the differences observed in the resin from the production runs.

Mixing Period

In five of the six successful runs, the autoclav charge was agitated at 20°C for two hours before the start of heat-up to the polymerization temperature. This period was

added to the standard charging cycle in order to ensure equilibrium between the phases before the start of polymerization. The report of a Development Department study of agitation (3) had stated that a considerable amount of time was required for protected suspension systems to achieve equilibrium.

The one successful run conducted without the two-hour mixing period (Run C14-1-27, Table II) produced a resin of essentially the same properties as were encountered in the runs with the mixing period. The only difference was a slight decrease in the amount of fines, as if starting the polymerization prevented the monomer droplets from reaching their minimum, equilibrium size. Further study along this line is also required, and the longer mix period should be continued until it can be proven unnecessary.

CONCLUSIONS

1) A process has been defined for interim manufacture of QXPA resin in the 4600-gallon autoclaves of the Suspension Vinyl Resins Unit, Location 514. The process is capable of producing plastisol additive resins which are equal or superior rheologically to competitive products and which at the time of our study were of sufficient quality in other respects to me t competitive materials of similar type.

2) The principal difficulty in scaling up from E17 600-gallon autoclave to C14 4600-gallon autoclave arose from basic differences in agitation, which have been partially accounted for, but which will require additional work in pilot equipment for complete resolution.

3) Developmental effort in C14 autoclave has achieved practically all it can within the present state of knowledge. Further expense for experimental production in th full-scale autoclave is not justifiable; however, the Production Department should be able to gradually improve the process further by employing small, systematic changes (EVOP methods) while producing salable resin.

4) Much of the variability in the results of polymerizations in this series has not been explained, in spite of all the efforts to maintain uniform conditions and to identify variations which might have occurred. In this regard, the quality of the vinyl chloride monomer is considered a possible source of difficulty, although no proof of its harmfulness has been discovered.

RECOMMENDATIONS The most advantageous use of the results of this series can be obtained if the following recommendations are included in future scheduling.

1) The Production Department should use the interim process, as recommended in Appendix A, for producing required amounts of QXPA resin.

2) During interim production of QXPA resin, the Production Department should keep additional records to permit more effective analysis of operating data. A proposed log sheet to use in recording such necessary information is presented as Appendix B.

3) In general, the Production Department should assume responsibility for and control over the semidevelopmental, semicommercial production of QXPA resin in C14 autoclave. If reproducibility on this operating basis is obtained, implementation of a program such as the "random-walk EVOP" would provide a simple, orderly method for process improvement. The Development Department has prepared a specific proposal along this line and will present it separately.

4) If reproducibility should be unattainable at any future point in the production-scale program, the Pfaudler impeller and the present recipe should be abandoned in favor of a return to Mixco agitation and the Elvanol suspending agent system in order to duplicate the conditions under which resin QEX-0125 was produced two years ago.

EQUIPMENT AND PROCEDURE C14 autoclave, in which this work was carried out, is a 4600-gallon, stainless steel autoclave equipped with a brine jacket for falling-film cooling. Agitation was provided by a 56-inch diameter, Pfaudler-type, retreat-curve, stainless steel impeller. The impeller was placed on the shaft 28" (one-half the impeller diameter) above the bottom of the autoclave. Baffling, as previously discussed, was provided initially by two 3-inch pipe baffles which were replaced by four 5-inch flat baffles. In addition to the obvious differences in the two kinds of baffles, the pipe baffles were short and did not extend down to the l vel of the impeller, whereas, the flat baffles extended the entire distance of the straight side of the autoclave.

The operating procedure comprised (1) charging wat r, suspending agent, buffer salt, and catalyst to the autoclave, (2) evacuating the autoclave vapor space at 50°C, (3) charging trichloroethylene molecular weight degrader and vinyl chloride, (4) mixing this charge for two hours at 20°C, and (5) conducting the polymerization at 57°C until autoclave pressure had fall n to 70 psig. Specific procedures are presented in the discussion of the interim process in Appendix A.

FUTURE WORK Although no further experimental work in th production autoclave is intended at this time, considerable experimentation on a smaller scale is planned.

1) Scale-up factors will be investigated by scaling down from the C14 autoclave results in this series to future work in 600-gallon and 10-gallon autoclaves. Such a study, when pursued to a satisfactory conclusion, will provide bases for easing scale-up problems involving many different resins in the future.

2) Additional experiments in glass bombs and in the pilot autoclaves will be undertaken as required to promote less sensitive, more reproducible operation and to improve resin properties found lacking as the resins are evaluated more fully.

ACKNOWLEDGMENTS Assistance in conducting these developmental operations was supplied by the following persons:

I. The Suspension Resins Department under J. E. Deitzler and D. W. Finn of the Production Department provided the equipment and the personnel for the polymerizations.

II. Personnel of the Quality Control Laboratory, under the direction of J. W. Chatfield, performed the routine evaluations included in this report.

III. A. F. Rogers, of J. J. Brezinski's group of the Development Department, conducted those special tests required for evaluation of a plastisol additive resin.

BIBLIOGRAPHY

(1) Alexander, J. C., Suspension Poly(Vinyl Chloride) Resins: QXPA Polymerization in the 600-Gallon Autoclave with CELLOSIZE QP-4400, Development Department, April 1961, (B:20:835-S9).

(2) Erdmann, J. F., et al., QXPA Resin: Recommendation for Interim Production Process, Development Department, March 6, 1961 (B:20:835-M11).

(3) Rodriquez, F., Suspension Process Studies: Effects of Agitation and Suspending Agents in a Model System, Development Department, November 25, 1959 (B:20:240-S2).

(4) Erdmann, J. F., and Nelson, W. H., QXPA Resin: Factors in Agitation Scale-up from 600-Gallon to 4600-Gallon Autoclaves, Development Department, in preparation (B:20:835-S13).

LABORATORY ACCOUNTS: 81753-0-2156
81753-0-3033
81761-0-3248

PERIOD: November 29, 1960, to February 25, 1961

Attachments:
2 Tables
4 Figures
2 Appendixes

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TABLE I

EXPERIMENTAL QXPA POLYMERIZATIONS IN C14 PRODUCTION AUTOCLAVE

Operating Temperature: 57°C
Trichloroethylene concn: 1.0 per cent
Dilauroyl Peroxide concn: 0.09 per cent
Sodium Bicarbonate concn: 0.15 per cent
Autoclave Level: 75 per cent full

C14 Run No.	1-4	2-5	1-6	2-16	2-7	3-8	1-9	2-10	3-11	1-12	2-13	3-14	1-15
CELLOSIZE QP-4400 concn, per cent	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.26	0.26	0.20	0.30	0.30	0.20
Agitator speed, rpm	120	100	100	100	90	90	100	100	120	100	100	115	115
Monomer-to-water ratio	35:65	35:65	35:65	35:65	35:65	35:65	30:70	30:70	30:70	35:65	35:65	35:65	35:65
Specific viscosity	0.141	0.115	0.137	0.138	0.145	0.141	0.144	0.141	0.139	0.141	0.141	0.141	0.137
Apparent density, lb/cu ft	33	43	38	29	39	38	36	38	41	36	39	38	35
Absorption, per cent	29	15	23	34	20	21	24	20	23	30	20	13	32
Heating loss, per cent	0.2	1.7	1.1	1.3	1.6	1.4	1.3	2.0	1.0	1.9	1.5	1.4	1.6
Screen analysis,													
% thru 60 mesh	100	100	100	74	95	99	99	100	98	100	100	100	86
80 "	100	100	99	14	49	75	82	84	83	97	100	100	21
100 "	99	99	92	7	20	39	43	37	39	84	97	36	9
140 "	80	95	78	3	10	24	25	23	25	43	27	27	5
200 "	54	61	53	1	5	10	8	4	23	27	8	17	3
270 "	32	30	32	0	2	6	5	2	22	23	7	16	2
Median particle size, microns	70	65	80	205	185	155	145	145	140	110	115	125	210
ICMT	101	88	88	110	100	106	83	97	85	97	94	77	100
Brookfield viscosity, poises (a)													
24 hr, 2-1/2 rpm	640	135	340	-	-	-	-	-	-	-	-	-	-
20	350	150	300	-	-	-	-	-	-	-	-	-	-

(a) These samples were not washed in the autoclave.

TABLE II

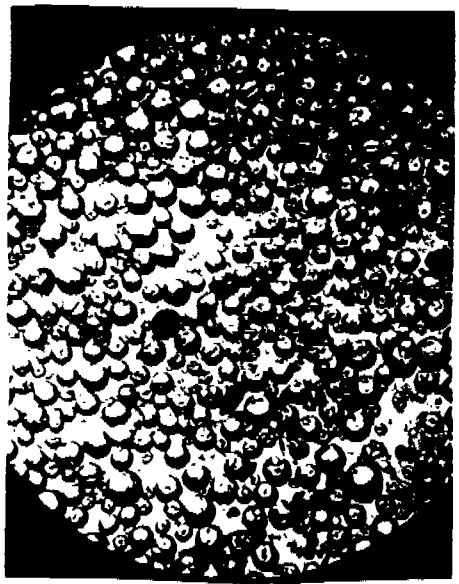
EXPERIMENTAL QXPA POLYMERIZATIONS IN C14 PRODUCTION AUTOCLAVE

Operating Temperature: 57°C
Trichloroethylene concn: 1.0 per cent
Dilauroyl Peroxide concn: 0.09 per cent
Sodium Bicarbonate concn: 0.15 per cent
Monomer-to-water ratio: 35:65

C14 Run No.	3-17 (a)	1-18	2-19	3-20	1-21	2-22	3-23 (a)	1-24 (a)	2-25 (a)	3-26 (a)	1-27 (a)	2-28 (a)	Geon 202
CELLOSIZE QP-4400 concn, per cent	0.20	0.20	0.20	0.20	0.20	0.20	0.30	0.30	0.30	0.30	0.30	0.30	-
Agitator speed, rpm	100	100	100	100	100	75	75	75	60	75	75	75	-
Autoclave level, per cent full	50	50	50	50	50	50	50	50	50	75	50	50	-
Specific viscosity	0.143	0.136	0.143	0.137	0.138	0.142	0.141	0.136	0.131	0.139	0.141	0.135	-
Apparent density, lb/cu ft	43	37	35	37	38	40	45	39	45	43	42	40	-
Absorption, per cent	13	45	46	28	24	25	12	17	13	15	16	18	-
Heating loss, per cent	1.4	1.2	1.6	1.4	1.6	1.3	1.6	1.7	1.7	1.8	1.7	2.1	-
Screen analysis,													
% thru 60 mesh	98	97	94	54	59	13	95	100	93	93	96	98	-
80 "	96	62	46	12	24	4	88	99	80	78	82	89	-
100 "	91	38	28	6	15	2	83	98	65	67	67	76	-
140 "	74	20	15	3	8	1	54	87	33	46	42	52	-
200 "	39	9	9	2	6	-	25	48	14	20	16	23	-
270 "	26	5	6	1	4	-	18	34	10	13	9	15	-
Median particle size, microns	80	160	185	250	230	390	95	65	125	120	120	100	-
ICMT	91	95	93	88	90	95	90	77	82	80	88	87	-
Brookfield viscosity, poises													
24 hr, 2-1/2 rpm	228	-	-	-	-	-	176	272	160	176	160	200	356
20 rpm	182	-	-	-	-	-	143	236	126	150	146	145	257

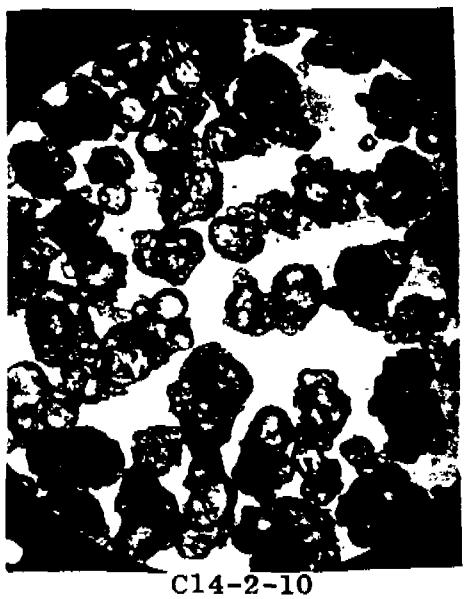
(a) The resin from these runs was washed in the autoclave by addition of TERGITOL NPX after stripping.

PHOTOMICROGRAPHS ILLUSTRATING AN ACCEPTABLE [illegible] PLASTISOL ADDITIVE RESIN AND THE EFFECTS OF THE PROCESS VARIABLES STUDIED AT A 75 PER CENT CHARGE LEVEL

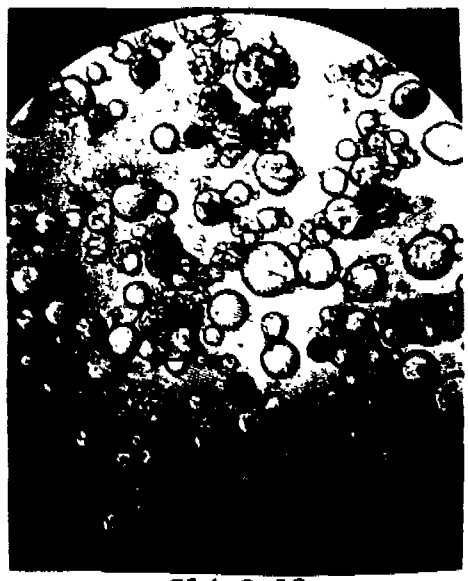


C14-2-5
100 rpm
0.20 Per Cent CELLOSIZE
35:65 Mon. to Water Ratio

C14-2-7
Lower Agitator Speed
(90 rpm)



C14-2-10
Lower Monomer to Water
Ratio
(30:70)

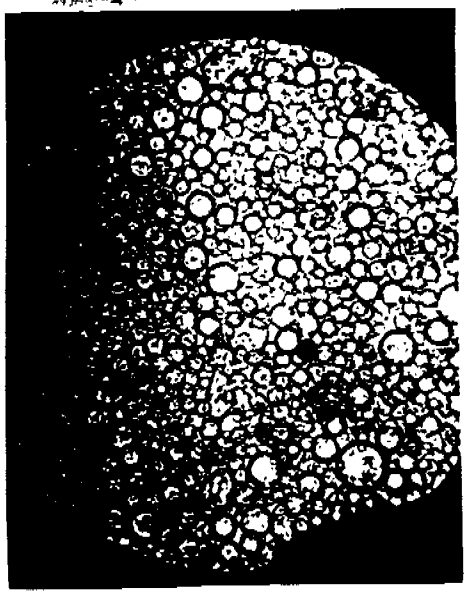


C14-2-13
Higher CELLOSIZE QP-4400
Concentration
(0.30)

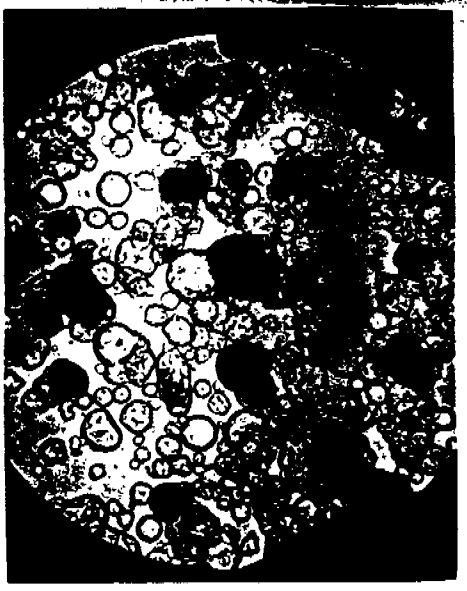
DEVELOPMENT DEPARTMENT

PHOTOMICROGRAPHS ILLUSTRATING [illegible]

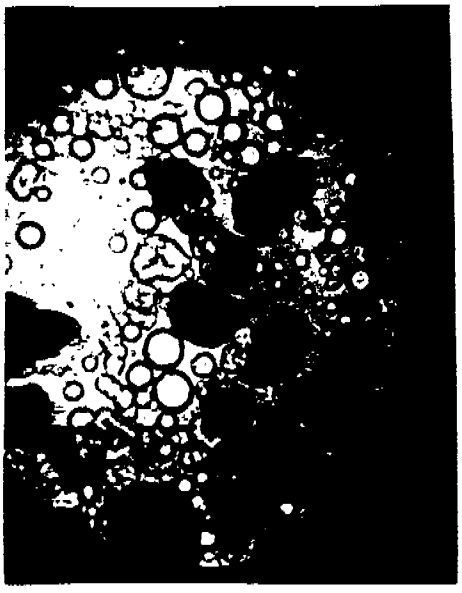
REPRODUCIBILITY AT THE 75 PER CENT CHARGE



C14-3-17



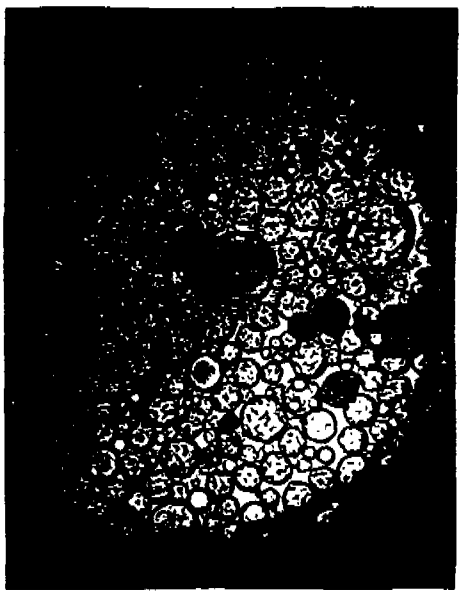
C14-1-18



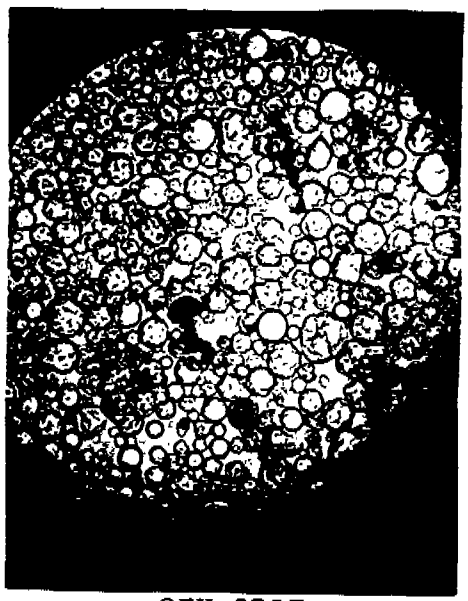
C14-2-19

UCC
039717

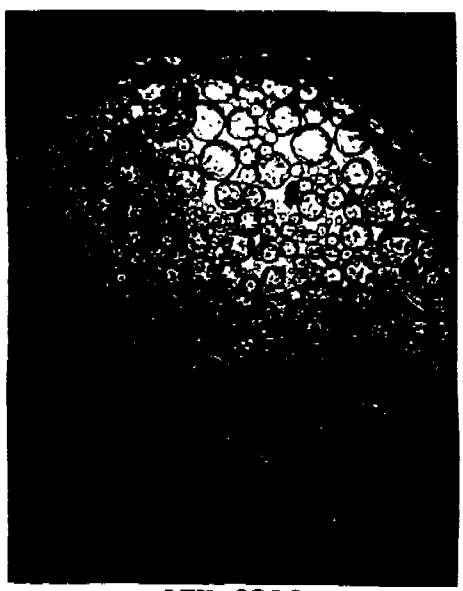
PHOTOMICROGRAPHS ILLUSTRATING REPRODUCIBILITY AT THE 50 PER CENT CHARGE LEVEL



QEX-0325
Blend of C14-2-25
and C14-3-26



QEX-0327
Blend of C14-1-27
and C14-2-28

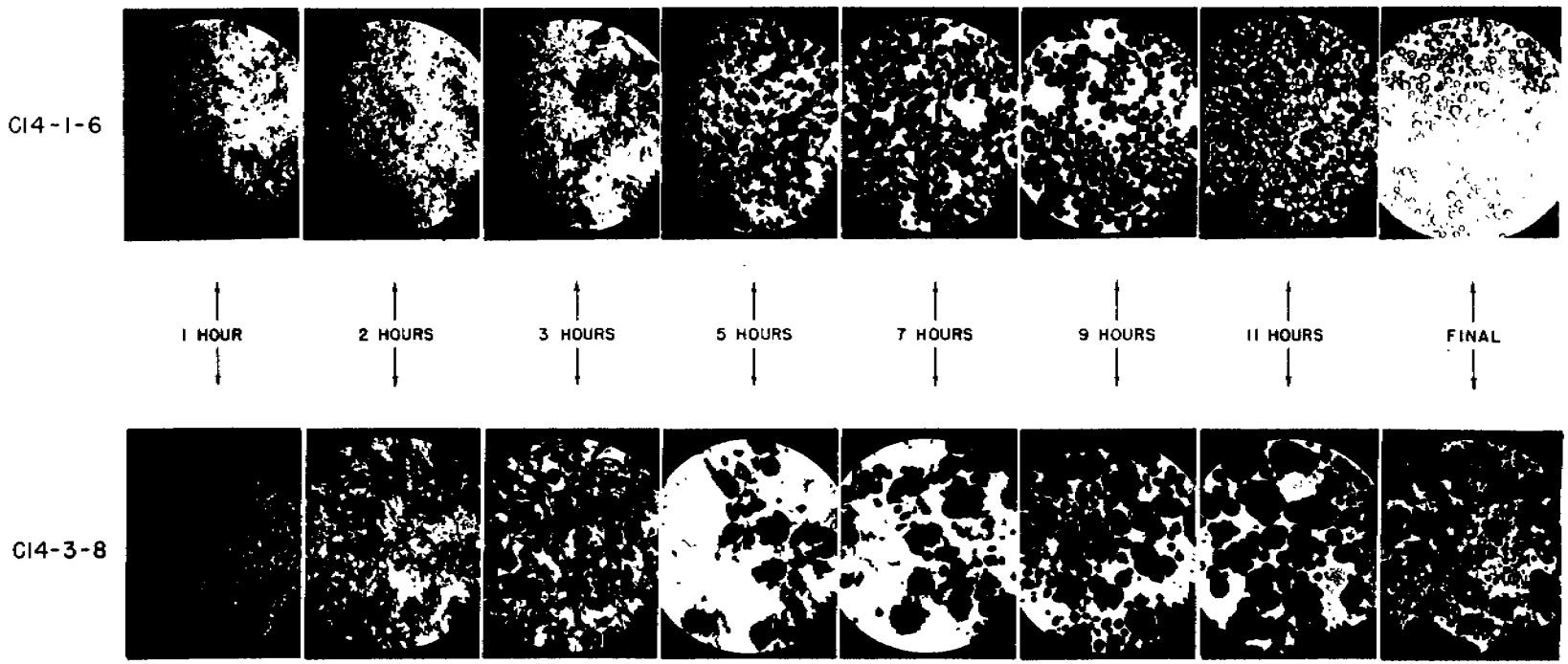


QEX-0310
Blend of C14-3-23
and C14-1-24

DEVELOPMENT DEPARTMENT

B:20:835-S12
FIGURE 5

PHOTOMICROGRAPHS OF RESIN SLURRY SHOWING VARIATIONS IN PROGRESS OF POLYMERIZATION



QXPA - STANDARD INSTRUCTIONS FOR INTERIM PROCESS

I. Autoclave Preparation

1. Clean autoclave after every third run, including sight glasses and nozzles.
2. Before each run wash out autoclave thoroughly to remove all loose resin remaining from previous runs.
3. Pressure-test autoclave carefully and accurately so that later evacuation will be successful in removing oxygen.
4. Put a new chart on each instrument using 24-hour charts so that we will have a continuous record of each run on a single chart.

II. Autoclave Charge - For 50 per cent volume charge in 4600-gallon autoclave

1. Water (50°C) (to 119-inch rod) (1450 gallons, if water meter is used)	12,100 lb.
2. CELLOSIZE QP-4400 (0.30% of VCl)	19 lb. 8 oz.
3. Sodium bicarbonate (USP) (0.15% of VCl)	9 lb. 11 oz.
4. Dilauroyl peroxide (0.09% of VCl)	5 lb. 14 oz.
5. Trichloroethylene (Hooker) (1.0% of VCl) Liquid level increment on metering tank	65 lb. 23.5 inches
6. Vinyl chloride (to provide a ratio of 35/65 vinyl chloride/water, by weight) Counts on Pottermeter	6500 lb. 823

III. Charging Procedures

Charge the autoclave in the order listed, observing the following procedures.

1. Add enough water at 50°C to fill the bottom head of th autoclave before charging CELLOSIZE.

2. CELLOSIZE QP-4400 (dry powder): draw into the autoclave with cold water aspirator. Run the agitator at 30 rpm. Feed carefully to the aspirator to avoid plugging the line. Feed CELLOSIZE rapidly enough to get it all into the autoclave before the water level reaches the end of the rod. Note that cold water should be used in the aspirator since better dispersion of the CELLOSIZE results.

3. Water (50°C): after the CELLOSIZE has been added, fill the autoclave to the designated rod level. Be sure agitator is off when filling to rod level. Because of the foam which often forms from the CELLOSIZE, th use of an accurate water meter is preferred to the rod technique.

4. Sodium bicarbonate - add through funnel.

5. Dilauroyl peroxide - add through funnel.

6. Evacuation:

   a. Blank off rupture disc and stripping header.

   b. Agitate at 30 rpm.

   c. Evacuate to 26" vacuum for 1/2-hour at 50°C.

7. Purge the VCl supply line to the atmosphere for a few minutes and close off vacuum line. Break vacuum with VCl and take to 50 psig autoclave pressure.

8. Remove blanks from rupture disc and stripping header.

9. Cool autoclave contents to 20°C; maintain at least 10 psig on the autoclave with VCl at all times.

10. Make trichloroethylene addition after temperature has fallen to 20°C.

11. Add vinyl chloride by count on Pottermeter at 20°C.

IV. Operating Instructions

1. While charging VCl, purge out lines to oxygen analyzer, and begin measuring the amount of oxygen in the autoclave. Set the analyzer on the 0-500 ppm scale and continue the analysis throughout the run. Label the chart with the run number and time, using a clean chart for each run. Special instructions for use of the analyzer will be issued to all Chief Operators, who will be responsible for the use of this instrument.

2. The oxygen reading is necessary for determining the reproducibility of the evacuation steps, but it is not to be used to stop operation of a given run if a particular level of oxygen is not achieved.

3. When vinyl chloride is added, increase agitator speed to the speed specified for the run. Count by hand to be certain.

4. Hold at 20°C for the specified mix period (2 hours).

5. After the mix period, begin heating as fast as possible to operating temperature.

6. Record on charts and log sheets the times of evacuation, beginning and end of mix period, and beginning of heat-up. (See attached log sheet.)

7. Zero hour is the time when operating temperature is reached.

8. When autoclave pressure has dropped to 70 psig, b gin recovery procedures below.

9. At the 70 psig point, add 1 gallon of TERGITOL NPX by dividing it into four 1-quart portions and diluting each to 1 gallon with HOT WATER before addition. Make sure all the TERGITOL is dissolved in the water (ignore the foam) - this will be greatly helped by using HOT WATER for dilution. Note that the TERGITOL solution must be added against approximately 70 psig autoclave pressure through a double-valve charging system. Continue agitation at operating temperature for 30 minutes; then transfer the autoclave contents into the blowdown tank.

V. Resin Recovery

1. Strip off unreacted monomer in the blowdown tank.

2. Fill the tank with water for dilution.

3. Feed the diluted slurry to the Bird slowly and st adily to ensure complete, uniform drying. (Resin must be at least 99.5% ₩ before bagging.)

4. Recover in No. 10 flash dryer through the east skin trap as usual.

5. Dryer Air Temperatures:

   First stage outlet 60°C
   Second stage outlet 65°C

6. This glassy, bead-like resin particle should be easier to dry than porous resin particles, but probably will require some adjustments of Bird operation and dryer feed because of the smaller particle size. The acceptance of this resin by potential customers depends a great deal upon the dryness of the resin, since more than 0.5% moisture greatly reduces the physical properties of the cured materials.

APPENDIX B

QXPA Log Sheet

Date _____

Please fill in all information desired as the work progresses. If the data is not available or other problems aris , use back of this sheet to explain your troubles. This information is needed for improved efficiency of this process.

Shift	Operator	Chief Operator
7-3	_____	_____
3-11	_____	_____
11-7	_____	_____

		Time Completed
1.	Results of pressure test at _____ psig:	
	Leakage rate _____ psig per hour	_____
2.	Autoclave charge	
	CELLOSIZE QP-4400 _____ lb. ____ oz.	_____
	Water to _____ inch rod	_____
	Sodium bicarbonate _____ lb. ____ oz.	_____
	Dilauroyl peroxide _____ lb. ____ oz.	_____
3.	Autoclave temperature at end of charging ____ °C	_____
4.	Evacuation of autoclave	
	Time started vacuum	_____
	Maximum vacuum on autoclave _____ inches Hg.	_____
	Time stopped vacuum	_____
5.	Breaking vacuum	
	Vinyl chloride used _____ counts on Pottermeter	_____
6.	Temperature after cooling _____ °C	_____
7.	Trichloroethylene _____ inches on metering tank	_____

QXPA Log Sheet (Contd.)

	Time Completed
8. Vinyl chloride _____ counts on Pottermeter NOTE: No. 5 plus No. 8 should equal specified charge.	_____
Source of Vinyl Chloride Tank No. ____ at Building 178. What kind of VCl is it? Synthesis, cracked, mixture of these two, or recovered. (Confirm by telephone call to Unit.)	_____
9. Temperature at start of mix period _____ °C	_____
Agitation speed _____ rpm	_____
Temperature at end of mix period _____ °C	_____
10. Start of heat-up	_____
End of heat-up (zero hour)	_____
11. Recheck of agitation speed _____ rpm at zero hour	_____

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Resin Physical Properties	X
Resin Particle Agglomeration	X
Suspension Polymerization	X
Plastisol Additive Resin	X