Air Products and Chemicals INC.

STRIPPING OF VINYL CHLORIDE MONOMER FROM RESINS IN AQUEOUS SLURRIES AND FROM DRY RESIN POWDERS

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I. INTRODUCTION

The unreacted monomer remaining in polyvinyl chloride can be a source of worker exposure and environmental emissions in polymerization plants, and is, of course, the only source of vinyl chloride released during a fabrication process or present in the final product.

Because of the very strict requirement imposed by OSHA, serious efforts have been expended during the last year by polymer producers to reduce this residual monomer to the lowest possible concentrations. This paper will report on some of the steps which can be utilized for this purpose in production of resin by the suspension process. Our work still is in progress and many of the conclusions presented here are preliminary.

II. DESCRIPTION OF THE PROCESS

Approximately 78% of the polyvinyl chloride produced in the United States is made by the batch suspension process. This amounted to nearly four billion pounds in 1974. A simplified flow diagram for a typical suspension plant is shown in Figure 1. Monomer and initiator are dispersed in water by means of agitation and suspending agents and polymerized at an appropriate temperature to give the desired molecular weight. The reaction cycle is terminated when the rate becomes uneconomical to maintain, or earlier if enhanced porosity is desired. Conversion is normally between 83% and 90% at this point. Most of the unreacted monomer is recovered and recycled to the process, but the last few tenths of a percent historically has been left in the slurry because it is difficult to remove. It is this residual monomer which is now of primary concern.

Monomer is usually recovered by vacuum stripping of the reaction slurry, either in the polymerization reactor or in a special stripping vessel. The monomer so obtained is compressed and condensed for re-use. The parameters of the stripping cycle, time, temperature and pressure are controlled to optimize equipment and monomer utilization.

After stripping, the polymer slurry is transferred to open equipment, and any remaining free monomer eventually enters the environment. The amount and location of this release depends on the amount of residual monomer, the physical nature of the polymer particles and processing conditions to which the resin is exposed.

Blends of several batches of polymer are mixed in slurry tanks and pumped to centrifuges for partial dewatering. Drying is done in either cocurrent rotary kiln driers or fluid bed driers. Dried resin is collected and conveyed pneumatically to storage or packaging. Resin may be transferred several times and stored for a few days to a few weeks before it finally reaches the fabricator and is blended with necessary additives and processed into finished goods. During each of these stages there is further loss of monomer content, dependent upon time, temperature, partial pressure of monomer over the resin, and properties of resin particles.

III. MONOMER LOSSES DURING MANUFACTURE OF RESIN

Monomer concentrations decrease approximately exponentially with time. This exponential decay is seen in every condition to which resins are subjected, but rates may be quite different under varying conditions. For example, the half life of monomer concentration will be on the order of two or three days for general purpose resin stored in paper bags, six to eight hours for well aerated resins at ambient conditions, or a few minutes for resins at very high temperatures. Thus, while it is possible to produce a final product with very low vinyl content by treatment during the last stages of processing, it is more effective in terms of preventing exposure and release to the atmosphere to remove as much monomer as possible during the stripping process.

Figure 2 shows a typical curve relating losses of monomer in resins, water and vapor phases as a function of time during a plant stripping operation. The slurry temperature during this run was 170°F (77°C) and pressure was 8 psia. The data in Figure 2 were obtained from a plant slurry batch of a Type B resin, described later in Table III, taken to about 85% conversion and transferred to an evacuated stripper. During transfer the pressure in the stripper rose to about 40 psig in about two minutes. With the pump capacity and steam input then available, a period of approximately 15 minutes was required to reach the specified temperature and vacuum. Under these conditions, about 50 ppm of monomer remains in this medium porosity resin at the end of one hour total resident time in the stripper. Under comparable conditions a lower porosity general purpose Type A resin (Table II) will have a higher residual monomer content at this point.

Let us follow the monomer content of a Type A non-porous general purpose resin as it moves through the steps downstream of the stripping process. Table I lists these processing steps and approximate average losses of VCM at each step. These concentrations, however, are relatively high compared with our most recent data. The table shows that the greatest absolute loss of monomer downstream of the stripper occurs in the blending step; it shows a reduction of approximately 60%. A further 60% loss is also shown in the drying step; with a more porous resin, this can be as high as 80%. Resins with lower initial residual vinyl chloride show a lower absolute rate of loss. For example, if initial content of VCM were 500 ppm instead of 2,000 ppm, the driving force would be less and the final concentration under the same conditions would be 50 to 75 ppm or 10-15% of the starting concentration rather than the 5% shown in Table I.

IV. STRIPPING PROCESS

The primary variables which have been identified in a plant stripping operation are time, temperature, pressure and resin properties. Stripping should be carried out for as long a time as practical at high temperature and at as low a pressure as practical to achieve maximum reduction in VCM levels. Better results are obtained on highly porous resins of small primary particle diameter.

For convenience in the discussions which follow, we have defined three consecutive stages in the stripping process. Figure 3 and Table II show the changes in monomer content, pressure, and temperature which occur in these stages when a batch of slurry is dropped from a reactor into an evacuated stripper. As noted earlier, there is a very rapid increase in pressure, generally rising to about 40 psig in the first few minutes, followed by a slower fall to the operating pressure. Operating temperature is reached at about the same time. This is defined as Stage I and generally results in a monomer reduction from approximately 18% in the solid to about 2-4% depending upon conditions of the drop. In Stage II residual monomer has dropped to about 500 ppm. The last Stage III is where residual monomer is below 500 ppm and where rates of VCM removal are at their lowest values. Table II summarizes approximate monomer losses during the three stripping stages. About 80-85% of unreacted VCM is lost in Stage I in a few minutes. We have not studied Stage I stripping. We believe, however, that improved rates of monomer removal can be achieved in Stage I by any method that speeds transfer of slurry and monomer vapor, for example, large drop lines, high pumping capacity and use of a gas holder.

In Stage II the point at which our monomer stripping studies generally begin, there is a slower rate of monomer removal. About 15% of unreacted monomer is removed. We have established that higher temperatures, higher porosities, and increased liquid/vapor interface via boiling favor more rapid removal of monomer in Stage II. Adequate pumping capacity and steam input into the stripping system is desirable and necessary to maintain temperature and vacuum.

It may be of interest to comment on some factors which do not appear important in the stripping process. Within limits of agitation ranges normally available, the degree of agitation does not seem to effect stripping rate. We must assume, therefore, that turnover rates in the slurry were adequate to produce optimum stripping in our plant equipment. In addition, we have found no marked discontinuities in stripping rates in temperatures approaching the normal glass transition temperature for PVC homopolymer (70-80°C).

There are process variables that do play an important role in the reactor prior to dropping the slurry batch. Batches which have been polymerized passed the optimum cut-off point result in lowered resin porosity and slower stripping. We have found that batches which have been cooled for scheduling reasons not only require longer time for heating to stripping temperatures but also appear to give slower stripping rates. One generalization discussed later in greater detail is that low molecular weight resins polymerized at higher temperatures are generally less porous and require longer stripping times than higher molecular weight resins manufactured with similar recipes at lower temperature.

V. EXPERIMENTAL

Resins, equipment and experimental procedures used in this work are described in the following sections.

A. Experimental Procedures and Materials

Five resin types used in this study and important property ranges are described in Table III. Resins A and B are general purpose resins made in recipes which contain a conventional colloid stabilizer. Resin Type C, made in laboratory or plant reactors, in the absence of the conventional polymeric colloid stabilizer does not contain a pericellular membrane. Resin C is measurably more porous than A and B and the primary particle structure can be easily identified using a scanning electron microscope. Other resins of Table III are copolymer resins containing either vinyl acetate or propylene.

B. Laboratory Studies

Resins, Type A, B, and C, used in the laboratory stripping experiments were prepared in a 3¼ gallon reactor using commercial recipes. Resin slurries after polymerization were cooled, vented and stored in gallon widemouth glass jars. Aliquot portions were taken at intervals over a period of weeks. No significant losses of monomer occurred in the solid phase during this period.

Laboratory stripping was conducted in a one-liter, three-necked glass flask fitted with a Friedricks condenser. Stirring was supplied by a magnetic stir bar. The slurry mixture from the master batch and added water were mixed, heated rapidly by immersion into a pre-heated five-gallon oil bath. The overall water/resin ratio was approximately 2 to 1. Compressed nitrogen at 5 psi or steam at atmospheric pressure were used in sparging experiments. The water/polymer mixture was sampled prior to stripping to determine VCM content in resin. At completion of each stripping experiment a second sample was removed to determine VCM content of resin and in some cases, resin porosity.

C. Plant Studies

A 10,000 gallon stripper equipped with vacuum and high pressure steam was used in plant stripping. The stripper was fitted with an agitator located just above the steam inlet port near the bottom of the stripper. The procedure for dropping the charge from reactor to stripper was described earlier. Initial samples for analysis and zero time were taken when the pressure rise in the stripper had reached its maximum value before falling.

Analytical samples were removed using a "sampler" of special design located at the bottom of the stripping vessels. This attachment allowed separate collection of slurry and water phases under conditions where no monomer losses occurred. The sampler and analytical procedures will be described in a separate publication.

D. Analytical Procedures

Analysis for VCM in PVC solid was conducted using a solution of the resin in tetrahydrofuran and a gas chromatograph. Impurities in the solvent were removed previously by a passage through a column of basic alumina with or without activated carbon. Gas chromatographs were fitted with flame ionization detectors and used nitrogen or helium as carrier gas. Column packing was either Porapak Q® and Chromasorb W®.

Concentrations of monomer in solid and liquid phases are expressed in parts per million (PPM) by weight. Concentrations in the gas phase are expressed in mole fraction.

VI. INFLUENCE OF VARIABLES ON REMOVAL OF VCM IN THE STRIPPING PROCESS

The stripping operation is the most effective and safest step for removal of unreacted VCM from PVC resin. The following sections report on laboratory and manufacturing plant work designed to establish important process variables and their quantitative effects.

A. Variables of Time, Temperature and Pressure in Stripping

As noted earlier from Figure 2 there is a loss of monomer at a continually decreasing rate with time during a typical plant stripping operation during Stages II and III. Time is an important variable, and under any given set of conditions, the more extended the stripping the lower is residual monomer in resin and aqueous phases. In the following sections, primary emphasis is given to removal of monomer from the solid resin phase.

Figure 4 is a semi-log plot of residual monomer in a Type B resin as a function of time at two temperatures in a plant stripping study. Slurry and vapor temperatures for the two runs appear in Table IV. From these data a 20°F (11°C) increase in slurry temperature produced an increase in rate of removal in Stage II of approximately two times at the 2,000 to 3,000 ppm VCM level and an increase of three times at 200 and 300 ppm in Stage III.

Laboratory scouting experiments were carried out with the medium porosity resin Type B to determine influence of temperature on stripping rate in Stages II and III. The master batch slurry contained about 35,000 ppm in polymer solids. Figure 5 is a semi-log plot of data obtained in the temperature range of 50-77°C (122-170°F). In a 15-minute stripping period an increase of 25°C (45°F) decreases residual VCM in the resin by almost two orders of magnitude. We note that at a temperature of approximately 170°F (77°C) residual monomer content in this medium porous resin was about 200 ppm after 15 minutes of vacuum stripping. Temperature has a profound effect on removal of VCM in Stages II and III. For comparison, plant data for a Type B resin are also plotted in Figure 5. At 77°C rates of VCM removal from the two systems are fairly close considering significant differences in procedures, equipment and times to reach stripping temperature.

Influences of stripping pressure and steam sparging in laboratory stripping studies are shown in Figure 6. Percentage monomer losses from resin is plotted as a function of temperature at a fixed stripping time of 15 minutes. There is a distinct advantage to using vacuum and a further advantage for vacuum plus steam as opposed to operating at atmospheric pressure. Remarkable increases in rate of VCM removal are noted at approximately 203°F (95°C) under atmospheric stripping conditions and at approximately 158°F (70°C) in vacuum stripping with steam. We associate these rate increases with the onset of "pseudo boiling" or nucleation of a high liquid to vapor interface. The importance of this interface to promote rapid stripping is discussed below.

B. Surface Area for Evaporation

Type 8 resin was stripped under atmospheric conditions in laboratory equipment using nitrogen sparging to determine influence of a gaseous phase in slurry stripping. Figure 7 shows that a nitrogen sweep slightly above the surface is without effect. The accelerating effect of nitrogen sparging into the liquid is temperature dependent and is greater as stripping temperature is increased. Apparently the liquid-vapor barrier for VCM removal under these laboratory conditions becomes more important as rate of removal from the solid phase is accelerated.

C. Particle Structure

Particle micro-structure has an important influence on rate of VCM removal during stripping. Figure 8 is a graphical plot of VCM removal from general purpose non-porous resin "A" and general purpose medium porosity resin "B" in plant strippers. Table V summarizes slurry and vapor temperature data for these two runs. Data show that the more porous resin "B" loses monomer at a rate of about 1.5 to 1.8 times faster than resin A at VCM levels between 200 and 2,000 ppm.

Laboratory studies established influence of resin porosity on stripping rates under atmospheric conditions. Figure 9 and Table VI summarize results using master batch slurries containing resins Type A, B, and C stripped at temperatures between 60°C (140°F) and 100°C (212°F) for 15 minutes. We noted again an abrupt increase in rate at a temperature close to but measurably below the boiling point of water. Figure 10 is a plot relating resin porosities of A, B and C to residual VCM content after 15 minutes of stripping at 100°C. For purpose of comparison, data from resins Type A and B plant runs (Figure 8) are plotted also in Figure 10. Response of stripping rate to resin porosity is remarkably similar in these laboratory and plant strippings. An increase of one unit of IPTU of resin porosity in the range measured increases stripping rate by about 15% in Stage III.

An unexpected effect on resin porosity was produced by rapid stripping in Stages II and III in laboratory work. Table VII summarizes stripping conditions and resultant particle porosities as measured by the procedure of Carleton and Mishuck[1]. Rapid removal of monomer from a PVC slurry containing 4.5% residual VCM in Type B resin markedly increased plasticizer absorption from a value of about 26 to values as high as 31. This phenomenon may be related to the process of producing "blotter" resins where liquid vinyl chloride monomer is removed from resin at low conversion forming a highly porous structure and sometimes referred to as the "popcorn" effect. Work is underway to establish the influence of temperature, pressure and unreacted monomer on resin porosity in Stage I stripping.

VII. MODEL FOR STRIPPING VCM FROM A SOLID PVC AQUEOUS SLURRY

A model for removal of VCM from a PVC particle suspended in an aqueous medium in contact with a vapor phase is shown in Figure 11. From this model, equations relating rates of removal from the three phases as a function of operating variables and polymer properties were developed. The model assumes that there are four barriers for removal of monomer from a suspended particle in an aqueous medium. There are two boundary layers above and below the solid/liquid and liquid/vapor interfaces. Among these, the vapor side boundary layer above the liquid/vapor interface is believed to be of no great importance and for practical purposes can be disregarded. Calculations using the correlation in Reference 8 show convincingly that the liquid side boundary resistance at the solid/liquid interface is also negligible. This leaves two boundary layers as important: one on the solid side of the solid/liquid interface and one at the liquid side of the liquid/vapor interface.

PVC particles have a micro-porous structure as discussed in a number of papers[1,3,4,5]. Particle diameters in our model relate to primary particles or aggregates of them shown in Figure 11 rather than the diameter of the particle measured by typical screen analysis.

Concentrations of VCM in solid, liquid and vapor phases are expressed in terms of concentration or VCM partial pressures. Their relationships during a typical stripping operation are shown in Figure 2 for a particular stripping experiment. These are expressed in the model as C_S, X_L, and P_{VCM}, respectively. Mass transfer coefficients of VCM at the barriers for the remaining two important boundary layers are given a K_{SL} and K_{LV}. The model envisions a driving force which is measured as a deviation of VCM concentration from equilibrium in two adjacent phases. It is necessary, therefore, to measure Henry Law constants for solutions of VCM at equilibrium in the liquid, solid and vapor phases.

From a consideration of the model, mass balance equations relating rates of losses in solid, liquid and vapor phases, have been formulated in terms of mass transfer coefficients, pumping capacity, reactor dimensions, the amount of slurry charged and the vapor pressure of water at the temperature of stripping. We are currently in the process of determining the mass transfer coefficients by a computer assisted curve fitting procedure.

In addition, we are determining Henry's Law constants H_{SL} and H_{LV} at a variety of temperatures. Experimental values obtained for a Stage III slurry at 170°F (77°C) are as follows:

For Solid/Liquid Interface: $$H_{SL} = \frac{X_L}{C_S} = .08 \pm .01$$

For Vapor/Liquid Interface: $$H_{LV} = \frac{P_{VCM}}{X_L} = (1.00 \pm .05) \times 10^{11} \frac{\text{dyne-cm}}{\text{g. mole}}$$

$$= 0.024 \pm .001 \text{ psi/ppm}$$

This liquid/vapor Henry's law constant is nearly identical with the value obtained using Berens's VCM water solubility data[5] and a vapor pressure of 10,200 mm Hg at 76°C; H_{LV} = 9.71 x 10^{10} dyne-cm/g.mole. The solid/liquid constant is somewhat lower than the 76°C value calculated from Berens's liquid/vapor and solid/vapor data, using the relation, $H_{SL} = H_{SV}/H_{LV}$; H_{SL} = 0.10.

VIII. STRIPPING OF VCM FROM PVC SOLIDS

When freshly manufactured PVC resins are "stripped" in a small fluid bed type drier, rates of monomer removal in general resemble rates obtained on monomer slurry stripping. Figure 12 is a plot of residual monomer content obtained with resin Type B and resin Type E-2 as a function of time in fluid bed stripping. Percentage losses from both resins were essentially identical over a 24-hour period. Under these conditions, approximately 50% of contained residual monomer was removed in about 2-3 hours at 115°F (46°C).

Very rapid removal of residual monomer from resins is achieved by mixing in an intensive mixer under vacuum at temperatures between 130° and 200°F (55° and 93°C). Figure 13 is a plot of residual monomer content of a propylene copolymer resin Type E-1 at various temperatures for 9 minutes in an intensive mixer. At approximately 170°F (76°C) 50% of the original monomer content was removed and 90% at 200°F. Extensive studies conducted in our laboratories and in others as well show that in typical compounding operations of this type rapid and nearly complete monomer removal can be achieved at high temperatures with no resin discoloration and no loss of thermal stability.

Influence of drying temperature in a commercial rotary drier in manufacture of Type A resin is summarized in Table VIII. As expected, increasing the air inlet temperature in the range of 220 to 280°F (105 to 138°C) had a marked effect on the residual monomer content.

IX. DISCUSSION OF RESULTS

Reduction of monomer content in both aqueous and powder stripping work (Figures 2 and 12) show continuing rate losses with time. The curves are similar to those reported by Berens[6] for PVC powders at 90°C. He interpreted reduced monomer losses with time to arise from a lowering of monomer diffusion rates inside particles as monomer content is reduced and in later stages to the presence of glass particles. Since our glassy particle contents were in the range of 0% to 2%, we do not believe that glassy particles played a dominant role in our work. This aspect will be quantified in later studies.

We note no discontinuity in Figure 13 for VCM loss in the region of the second order transition temperature of PVC, 160-175°F (70-80°C). Hopfenberg and Stannett report a number of examples where no discontinuities in diffusion coefficients were noted in the region of the glass transition temperature for a number of polymers and a number of penetrating gases.[7] The increase in monomer removal rate in atmospheric stripping at 90-95°C in Figures 6 and 9 we associate with the on-set of bubble nucleation.

Rate controlling processes for VCM in Stages I, II and III of aqueous slurry stripping appear to differ. In Stage I, extremely large amounts of monomer are lost in very short periods of time. It is likely that this process occurs by direct formation of bubbles at the solid/liquid interface which move directly to the slurry liquid/vapor interface. Quantitative effects of resin porosity, temperature and pumping capacity remain to be established. The special on-line sampler will make this possible. Stage II rates are influenced markedly by temperature, by boiling or nitrogen sparging and by resin porosity. Present work on our model will establish quantitatively the relative importance of the two important barriers at the solid/liquid and liquid/vapor interfaces. Our work suggests that both are playing a role. It is clear, however, that a boiling or rapid degassing condition in Stage II is necessary to promote optimum rate of VCM removal. Stage III stripping rates appear to be more dependent upon the solid/liquid interface and on diffusion rates inside resin particles.

The very rapid losses of VCM in Stage I and rapid increases in rates at about 90°C in atmospheric stripping suggests that pseudo boiling or degassing occurs below the normal boiling point of water. How bubbles form and how they are influenced by primary particle diameter, pore size and pore distribution remain to be established.

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ACKNOWLEDGEMENTS

We wish to express our appreciation to many persons in the Analytical Department of Air Products and Chemicals, Inc. and to many members of the industry who have shared so generously their knowledge toward the solution of a problem affecting the health and safety of our employees and theirs. It is in this same spirit that we have elected to share our preliminary findings with others in this progress report. We are continuing our work and will be reporting additional findings in the future.

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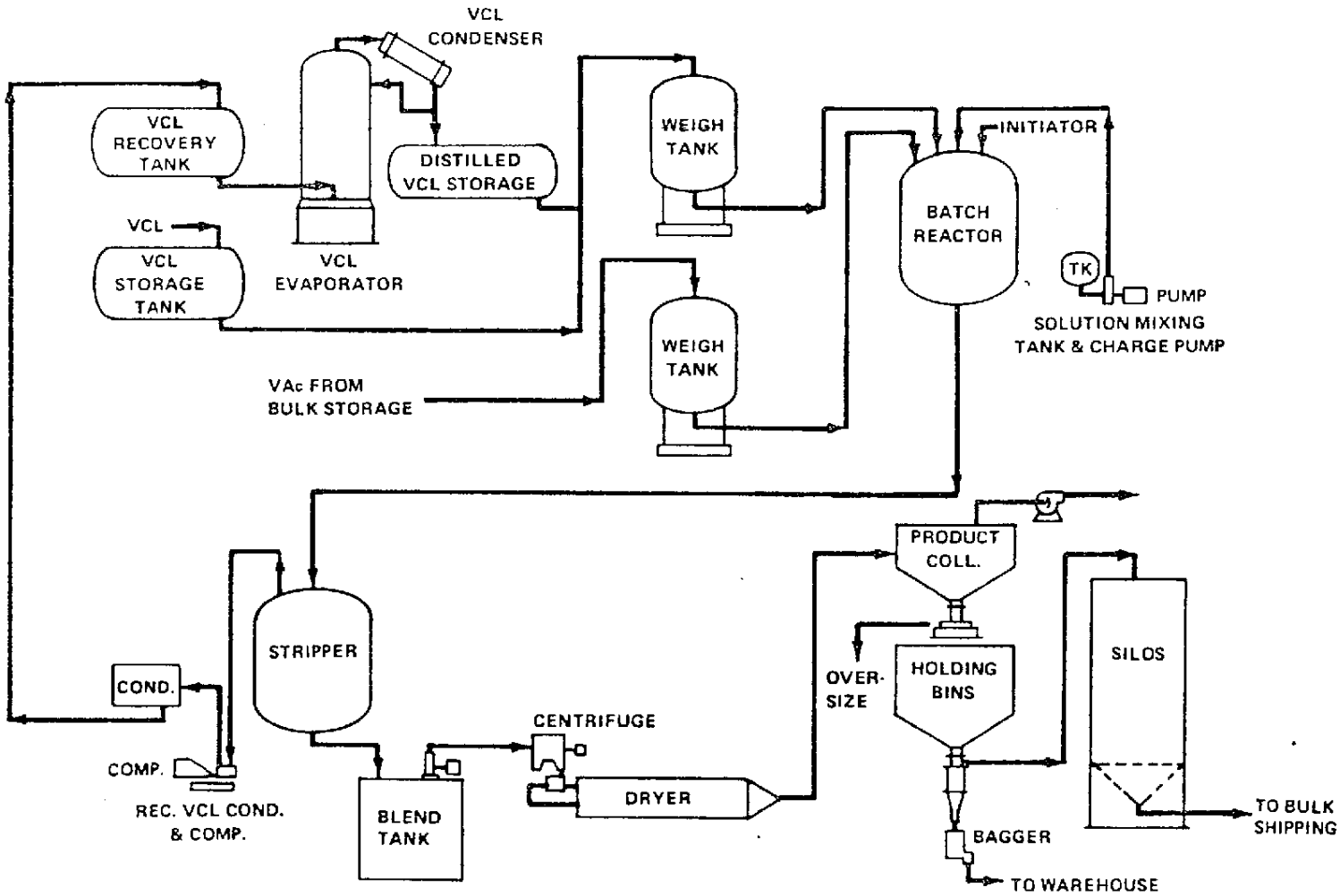

FIG. 1. TYPICAL SUSPENSION PROCESS POLYVINYL CHLORIDE PLANT FLOW DIAGRAM

VVC 00002045

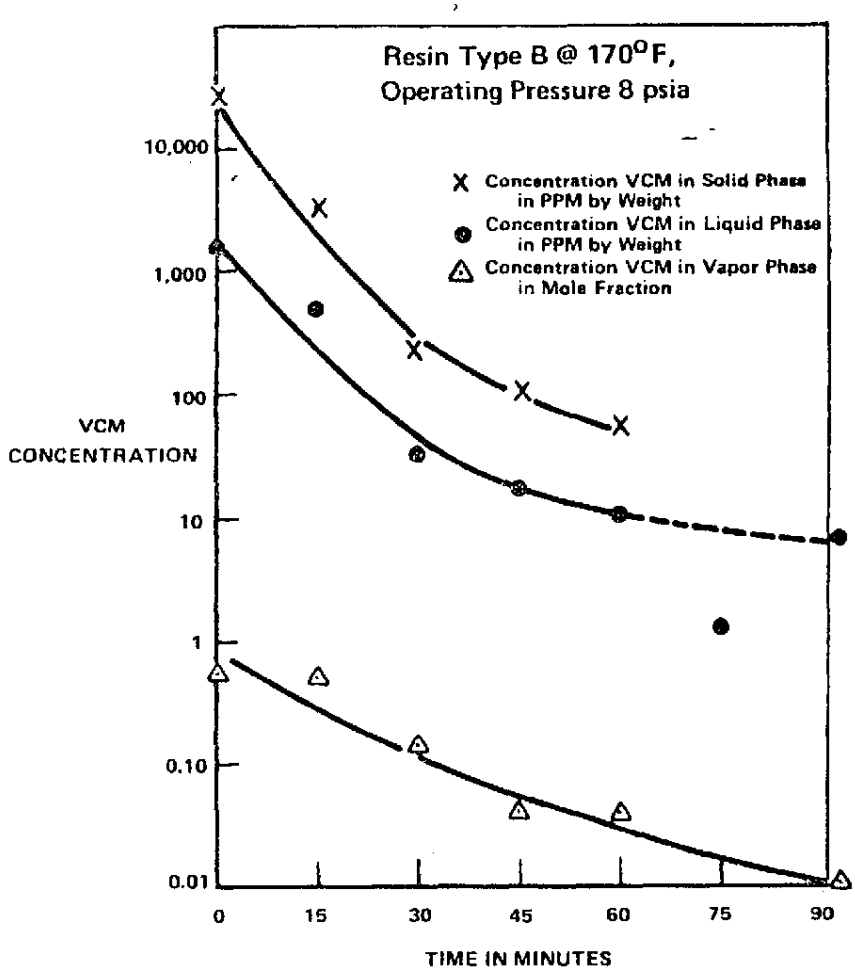

FIG. 2. VCM CONCENTRATION IN SOLID, LIQUID AND VAPOR PHASES AS A FUNCTION OF TIME DURING PLANT STRIPPING

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TABLE I

APPROXIMATE MONOMER LOSSES AFTER STRIPPING

IN MANUFACTURE OF RESIN TYPE A

Process Step	VCM Content (PPM) End Step	Percent Loss in Step
Leaving Stripper	2,000	--
Slurry Blending	800	60
Centrifuging	720	10
Drying	290	60
Storage	215	40
Shipping	105	50

Changes of Temperatures, Pressure and VCM Content in Resin with Time.

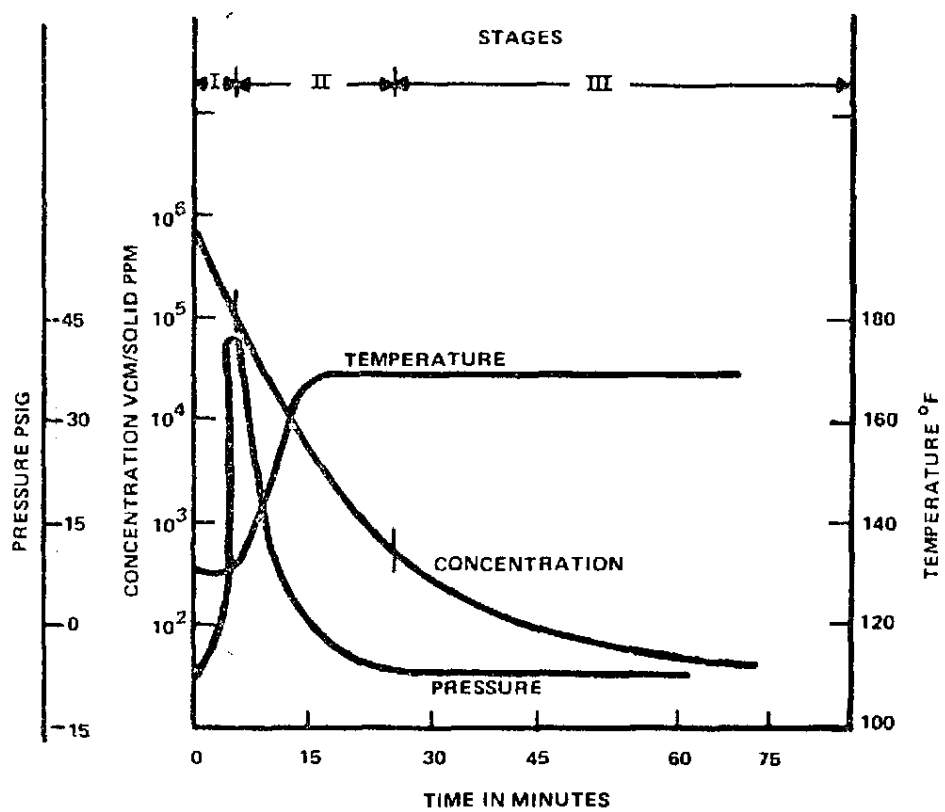

FIG. 3. STAGES OF PLANT SLURRY STRIPPING

TABLE II

LOSSES OF MONOMER IN THREE STAGES OF STRIPPING

OF BATCH FROM SLURRY OF 10,000 LBS. MONOMER CHARGE*

STAGE	I	II	III
PPM of VCM In Resin			
Initial	180,000	30,000	500
Final	30,000	500	1
Pounds Lost**	1,275	251	4

*Approximate Conversion = 85%

**Approximate Weight Ratios of Monomer in Vapor, Liquid and Solid Phases are 1:100:1000 during stripping.

TABLE III

PVC HOMOPOLYMER AND COPOLYMER RESIN TYPES

USED IN THIS STUDY

Resin	Composition	Description	IPTU* Range	% Accessible Porosity**	Inherent Viscosity dl/gm
A	PVC	General Purpose Low Porosity	19-21	10 - 11	0.88-0.92
B	PVC	General Purpose Medium Porosity	26-28	17 - 18	0.94-0.98
C	PVC	Specialty No Pericellular Membrane High Porosity	30-32	25 - 26	0.80-0.88
D	Vinyl Acetate Copolymer	---	---	22	0.46-0.50
E-1	Propylene Copolymer	---	16-20	8.5- 10	0.70-0.76
E-2	Propylene Copolymer	---	16-20	---	0.57-0.63

*Irreversible Plasticizer Take Up By Method Reference (1)

**By Mercury Porosimeter According to Reference (2)

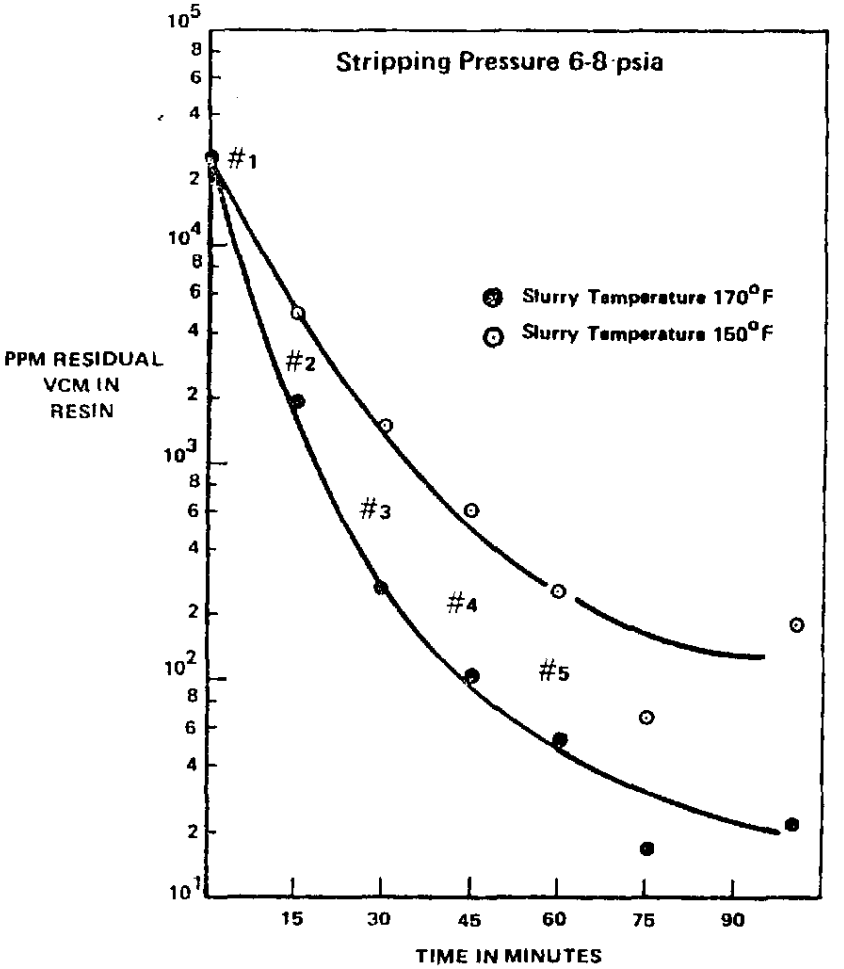

FIG. 4. EFFECT OF STRIPPING TEMPERATURE ON VCM LOSS IN TYPE B RESIN

TABLE IV

TEMPERATURES OF SLURRY AND VAPOR FOR RESIN TYPE B

IN PLANT STRIPPING STUDY*

Sampling Point*	Time (Minutes)	High Temperature Run		Low Temperature Run	
		Slurry Temp. °F	Vapor Temp. °F	Slurry Temp. °F	Vapor Temp. °F
1	0	136	136	132	130
2	15	170	170	152	150
3	30	168	172	151	152
4	45	168	172	150	154
5	60	170	172	150	156

*See Figure 4

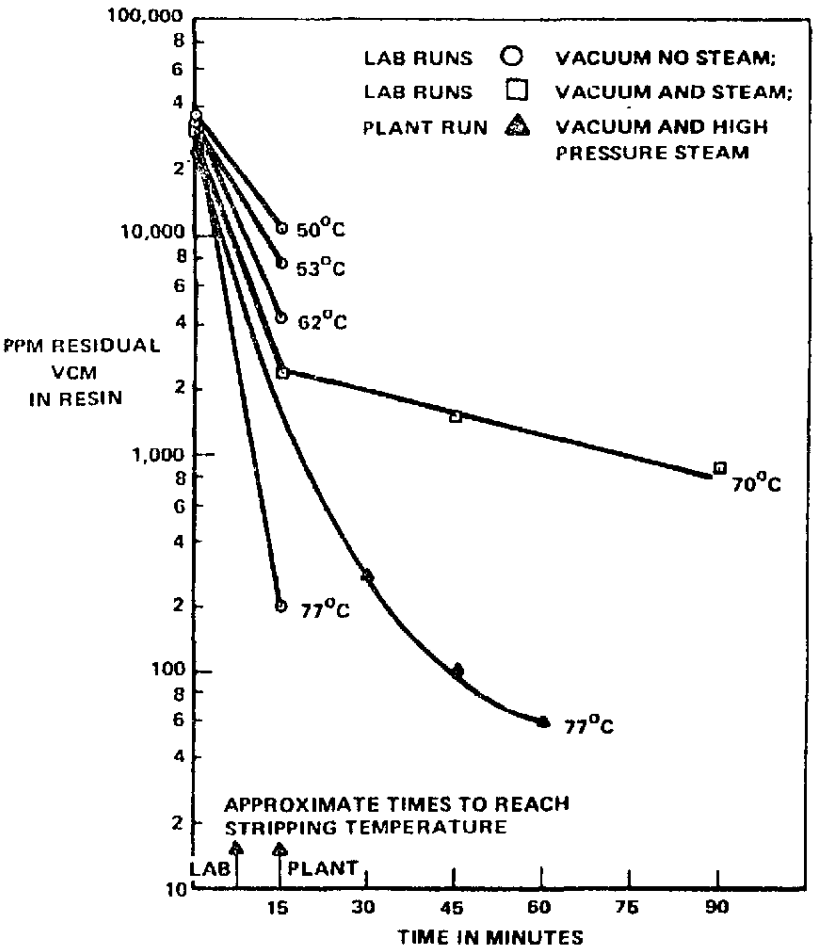

FIG. 5. LABORATORY VERSUS PLANT SLURRY STRIPPING OF TYPE B RESIN AND TEMPERATURE EFFECTS

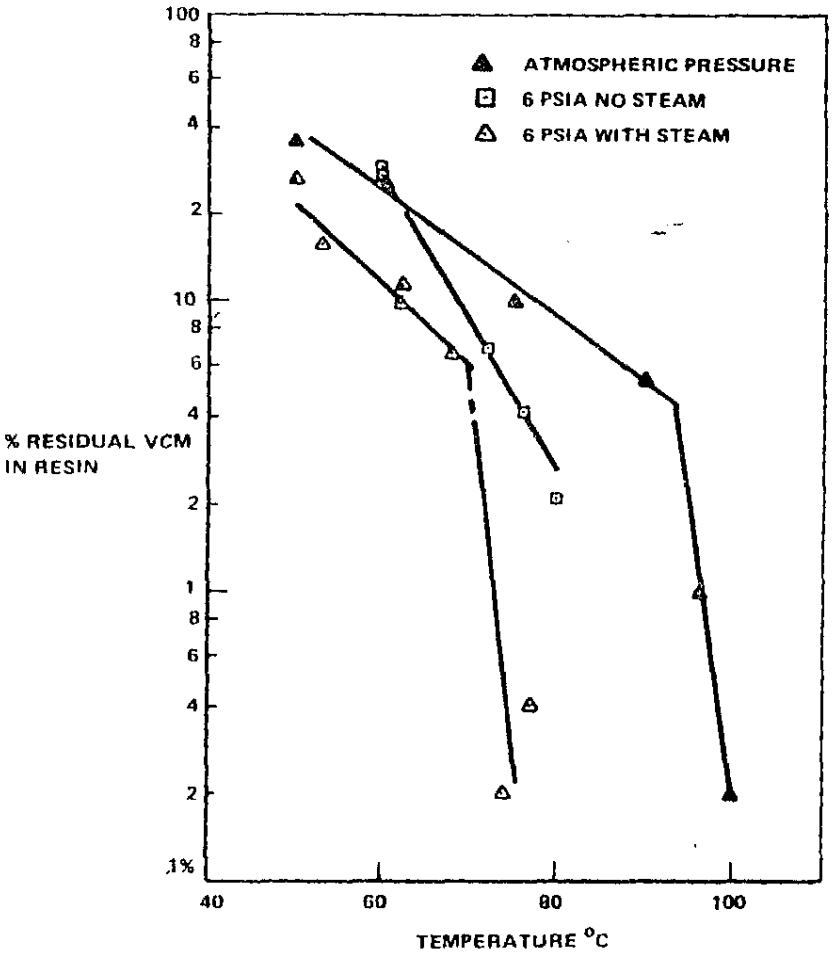

FIG. 6. EFFECTS OF PRESSURE AND STEAM PURGE ON VCM REMOVAL IN LABORATORY STRIPPING OF TYPE B RESIN (VCM 45,000 PPM) STRIPPING TIME 15 MIN.

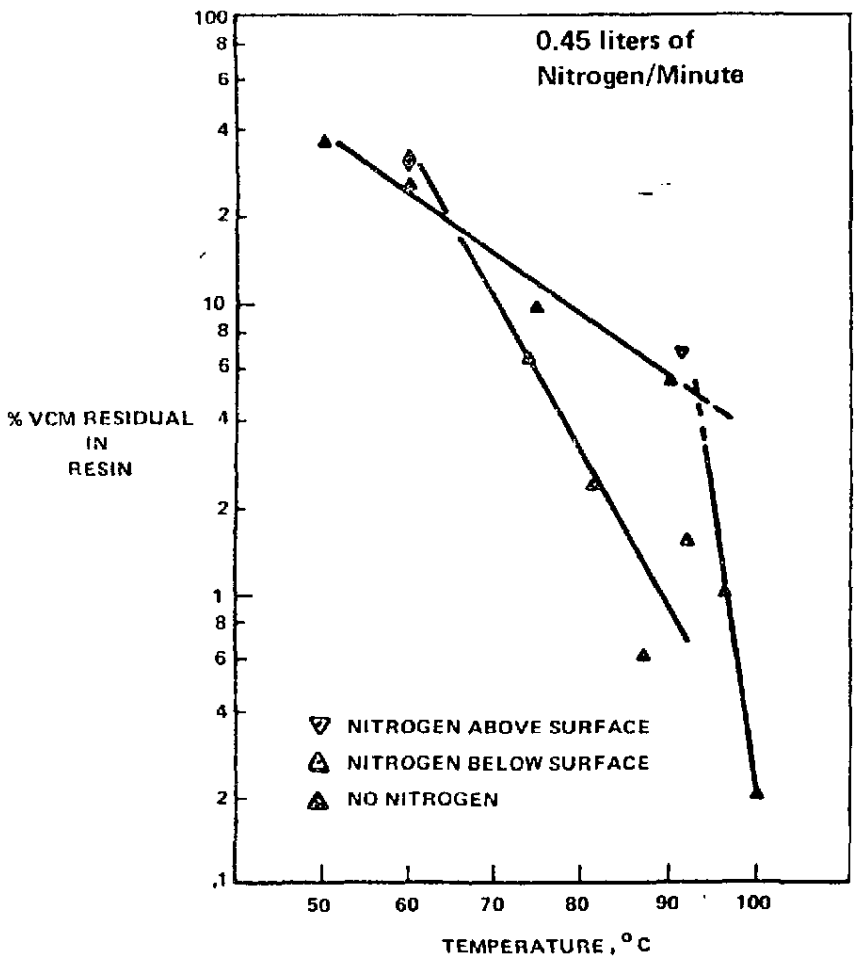

FIG. 7. EFFECTS OF NITROGEN SPARGING ON VCM REMOVAL IN LABORATORY TYPE B RESIN (VCM 45,000 PPM) STRIPPING TIME 15 MIN.

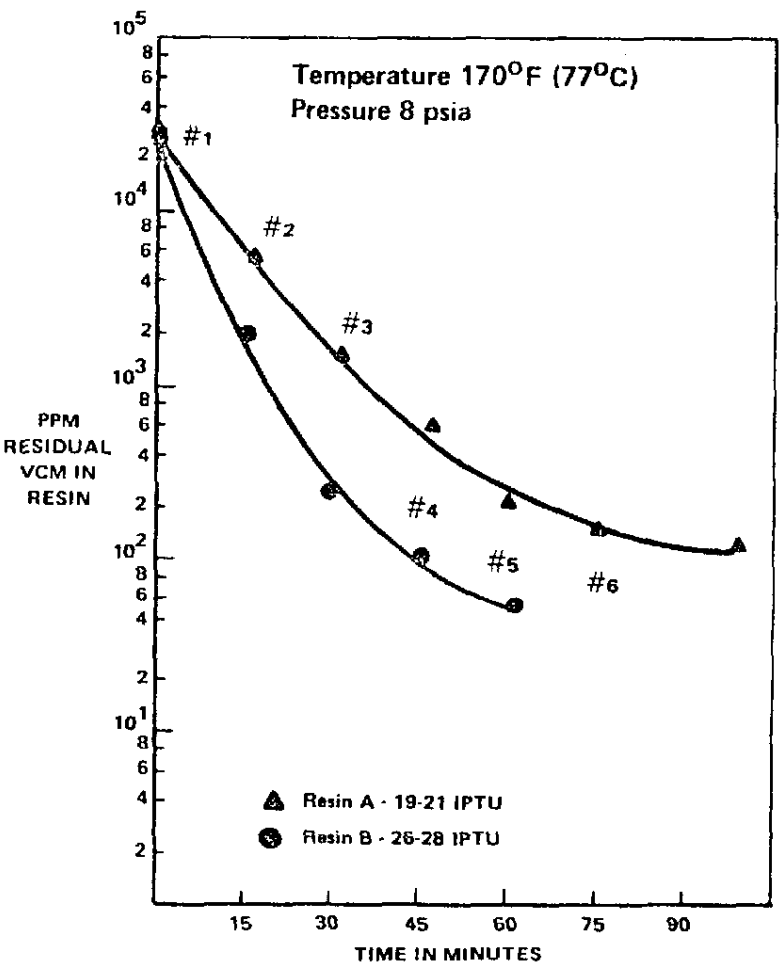

FIG. 8. EFFECT OF POROSITY ON VCM REMOVAL IN PLANT STRIPPING

TABLE V

TEMPERATURES OF SLURRY AND VAPOR FOR RESIN TYPES A AND B IN PLANT STRIPPING*

Sampling Point*	Time (Minutes)	Slurry Temperature °F		Vapor Temperature °F	
		Resin A	Resin B	Resin A	Resin B
1	0	133	136	136	136
2	15	167	170	170	170
3	30	167	168	172	172
4	45	170	168	174	172
5	60	170	170	175	172
6	75	170	---	175	---
7	100	170	---	175	---

*See Figure 8

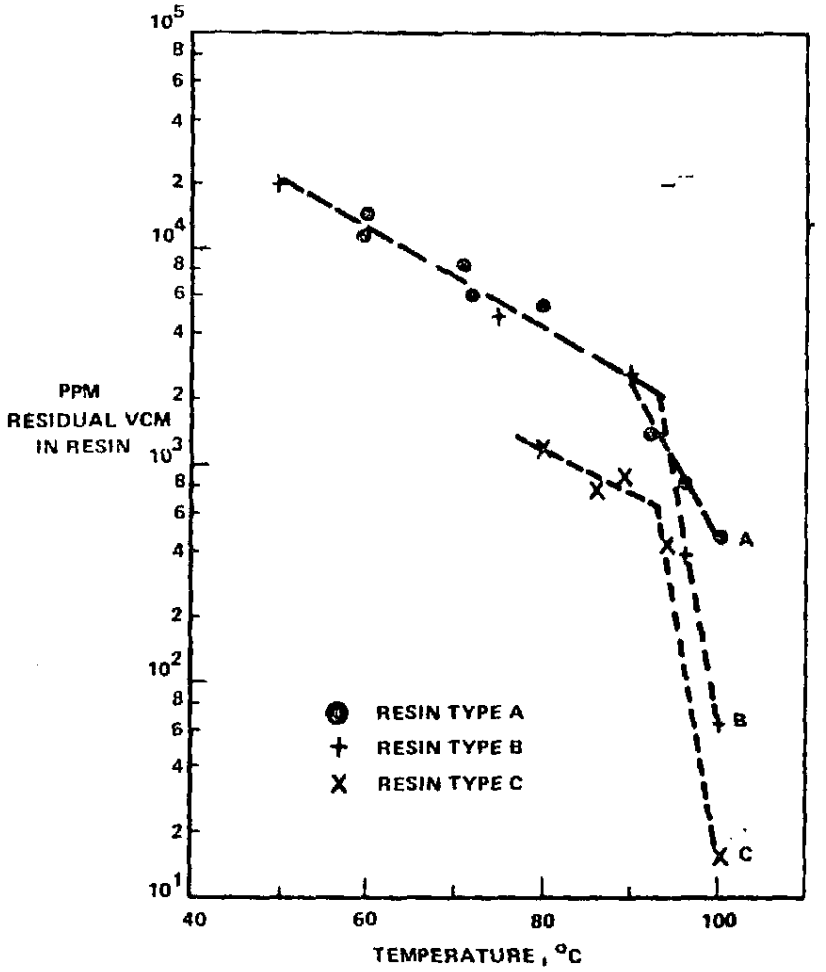

FIG. 9. LABORATORY STRIPPING OF RESIN TYPES A, B AND C AT ATMOSPHERIC PRESSURE (VCM 43,900, 47,000, 23,400 PPM, RESPECTIVELY) STRIPPING TIME 15 MIN.

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TABLE VI

EFFECT OF PARTICLE POROSITY ON RESIDUAL LEVEL OF VCM
IN RESINS STRIPPED IN LABORATORY AT
100°C, ATMOSPHERIC PRESSURE, 15 MINUTES

Resin Type	A	B	C
Porosity - IPTU*	20	26	30
PPM Of VCM in Resin			
Initially	43,900	47,000	23,400
After Stripping	450	60	15
% Original VCM Remaining	1.05	0.13	0.06

*Irreversible Plasticizer Take-Up Procedure Reference 1.

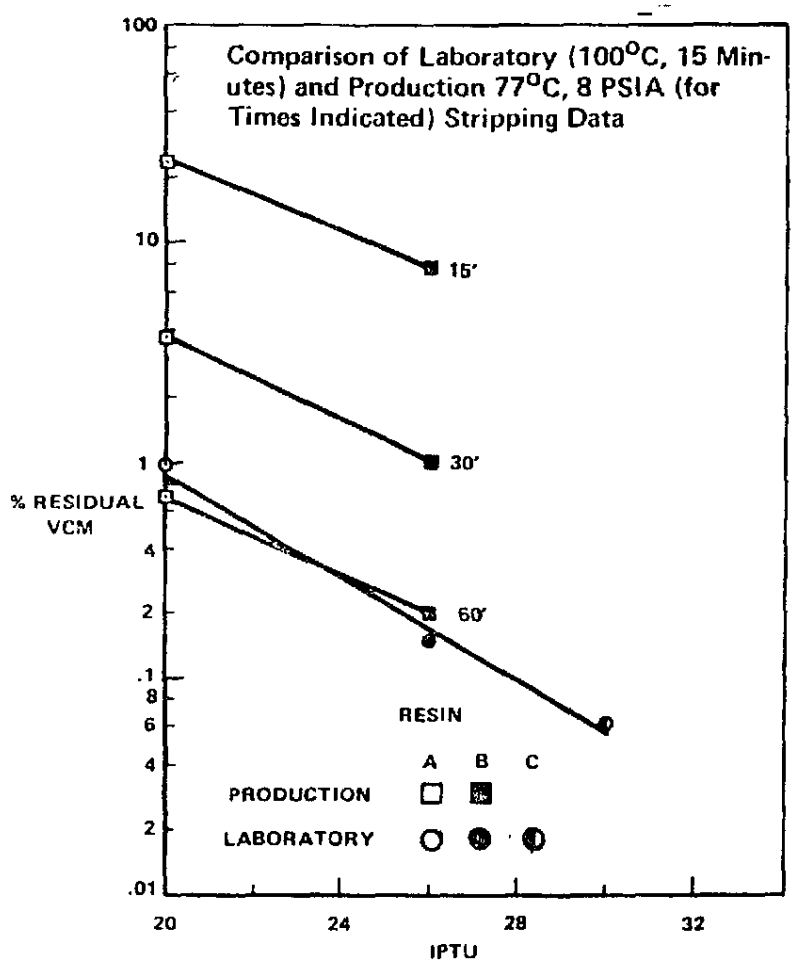


FIG. 10. EFFECT OF RESIN POROSITY ON RESIDUAL VCM

TABLE VII

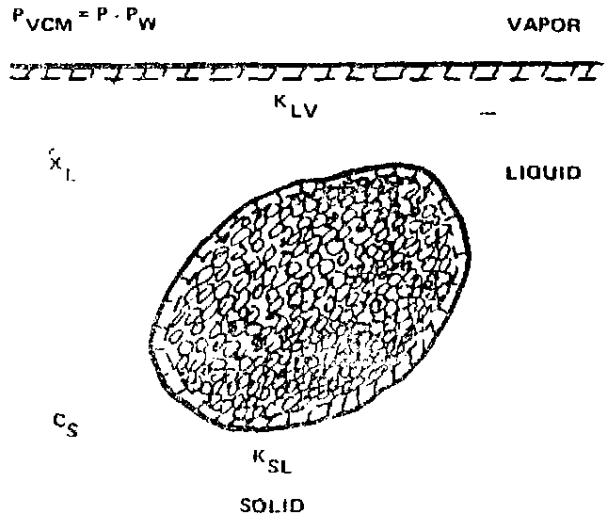
INFLUENCE OF STRIPPING TEMPERATURE AND PRESSURE
ON PARTICLE POROSITY IN LABORATORY STRIPPING STUDIES
TYPE B RESIN

Resin*	Stripping Conditions 15 Minutes Temp. °C	In. Vacuum	Resin VCM Content After Stripping	IPTU***
Control	---	---	45,000**	26
a	50	Atm.	18,900	24
b	60	Atm.	14,000	27
c	75	Atm.	4,700	29
d	100	Atm.	40	28
e	53	14	7,400	29
f	62	14	4,100	30
g	68	14	1,000	30
h	77	14	100	31

*Resin in all experiments filtered by suction and dried at 40°C (105°F) in a vacuum oven.

**Original VCM content of master batch slurry.

***Irreversible plasticizer take up according to reference (1).



FLUX VCM $\propto$ (CONDUCTANCE) (DRIVING FORCE)

$$F_{SL} \propto (A_S K_{SL}) \left(C_S - \frac{X_L}{H_{SL}} \right)$$

$$F_{LV} \propto (A_L K_{LV}) \left(X_L - \frac{P_{VCM}}{H_{LV}} \right)$$

FIG. 11. MODEL FOR SLURRY STRIPPING

C_S, X_L, P_{VCM} are concentrations or partial pressure of VCM; K_{LV}, K_{SL} are mass Transfer coefficients; H_{LV}, H_{SL}, are Henry Law constants; A_S, A_L, are areas at S/L and L/V interfaces.

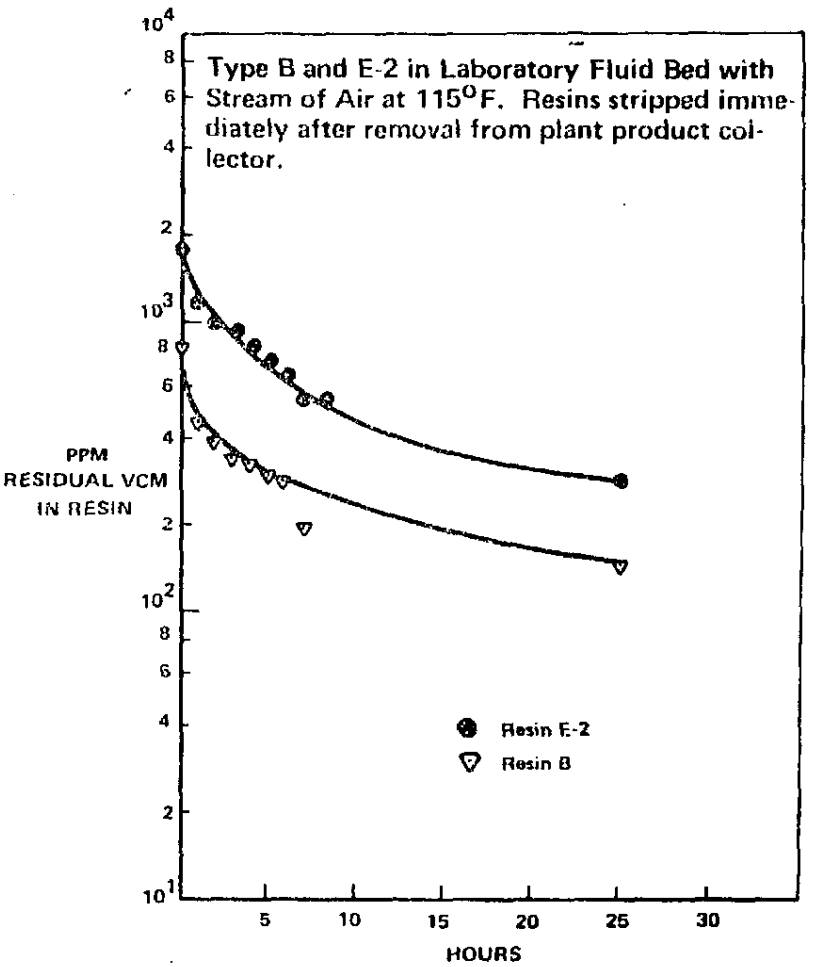


FIG. 12. STRIPPING OF VCM FROM DRY PVC RESINS

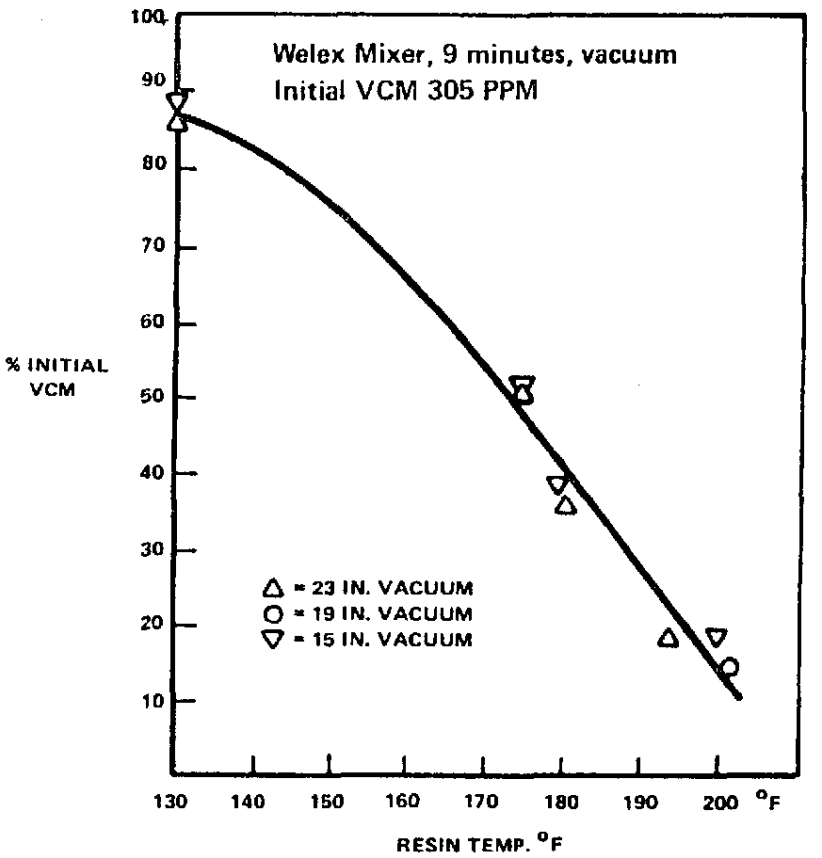


FIG. 13. STRIPPING VCM FROM SOLID RESIN TYPE E-1 RESIN (VCM 305 PPM)

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TABLE VIII

INFLUENCE OF TEMPERATURE IN PLANT DRYING

OF TYPE A RESIN

Inlet Air Temperature (°F)	VCM Residual in Resin		% VCM Remaining
	Drier Inlet**	Drier Exit**	
280	850	200	24
260	850	350	41
220	1050	500	48

*Rotary drier with capacity of 5,500 lbs./hr; Residence time 15 minutes; Air exit temperature approximately 150°F

**Average of several samples