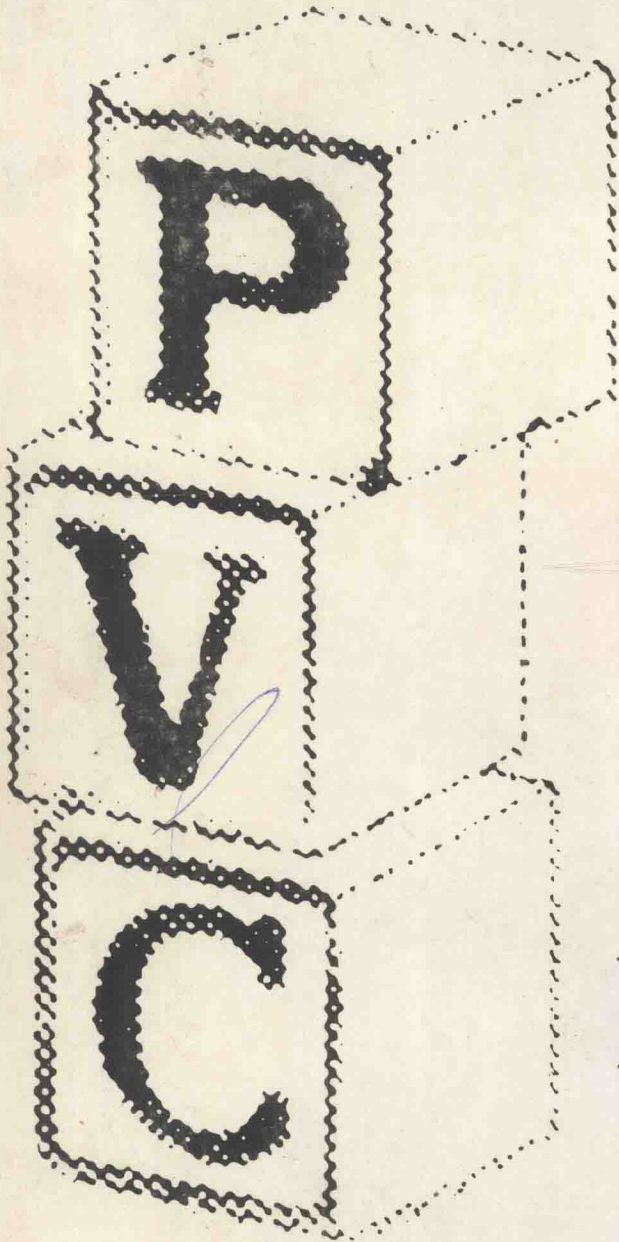




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# RETEC

## PVC

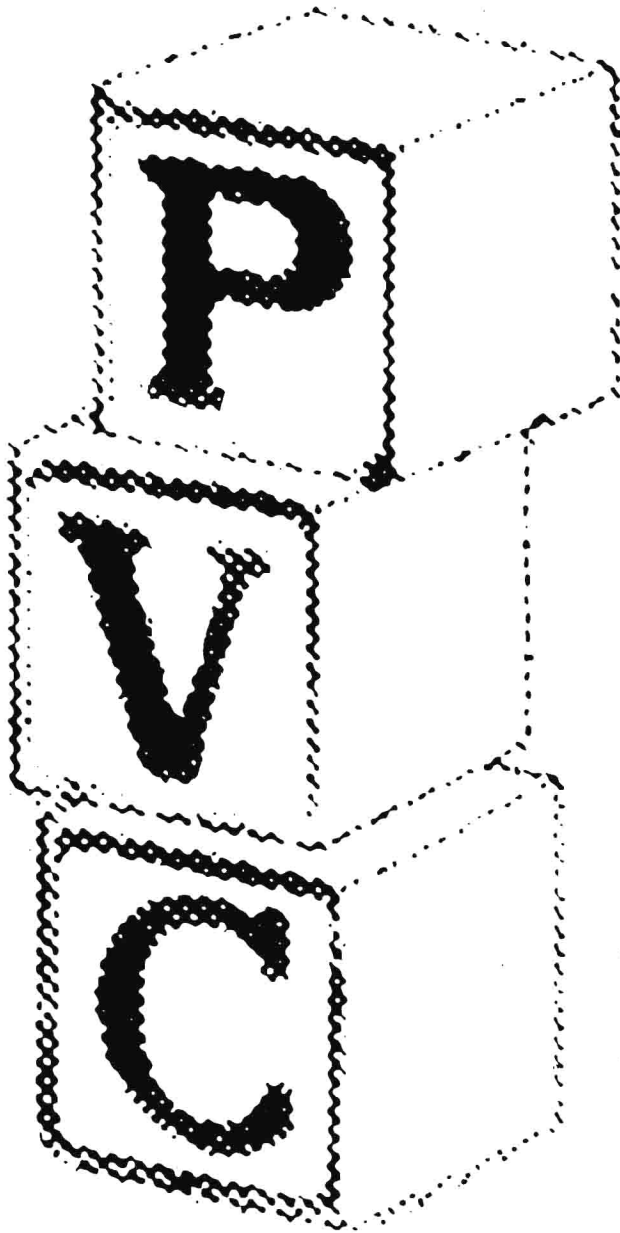
# PRIMER

OCTOBER 9 & 10, 1985  
WEDNESDAY — THURSDAY

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# TABLE OF CONTENTS

	Page
AN INTRODUCTION TO PVC: SUSPENSION AND MASS POLYMERIZATION PROCESSES .....	7
By Peter A Schwab, Vista Chemical Company	
PVC EMULSION AND DISPERSION RESINS .....	25
PART I: POLYMERIZATION	
By Bela K. Mikofalvy, B.F. Goodrich	
PART II: CHARACTERIZATION .....	41
By George R. Huddleston, B.F. Goodrich	
A REVIEW OF PROCESSING AIDS FOR PVC .....	55
By Bill Casey, Metco, M&T Chemicals, Inc.	
PVC/ABS BLENDS AND ALLOYS .....	68
By Lewis Ferguson, Borg-Warner Chemical, Inc.	
THE WHY'S, WHAT'S AND WHEREFORE'S OF ANTIMICROBIALS IN PVC .....	78
Society of Plastics Engineers	
By James F. Brophy, Morton Thiokol Corp.	
UTILIZATION OF FOAMING AGENTS FOR POLYVINYLCHLORIDE .....	99
By Raymond J. Shute, Olin Corp., PP&S-Technical Service	
UV STABILIZERS: THEIR EFFECT ON VINYL COMPOUNDS .....	113
By Gerald Capocci, CIBA-GEIGY Corp.	
COLORANT SELECTION FOR PVC .....	117
By Adelbert Bell, CIBA-GEIGY Corp.	
THE USE OF MINERAL FILLERS IN PVC: A REVIEW OF PRINCIPLES .....	131
By Michael Tapper, Pfizer, Inc.	
ORGANOTIN PVC STABILIZERS — A WHAT, HOW, WHERE AND WHY OVERVIEW .....	147
By Robert D. Dworkin, Akzo Chemie America, Interstab Chemicals, Inc.	
MIXED METAL STABILIZERS .....	161
By Dane L. Clark, Synthetic Products Co.	
LEAD STABILIZERS FOR PVC: A PRIMER .....	175
By Mary A. Berna and Gary R. Mitchener, Halstab, Division of Hammond Lead Products, Inc.	
FLAME RETARDANTS AND SMOKE SUPPRESANTS FOR PVC .....	199
By F.J. Fletcher and A. Docherty, Anzon America, Inc.	
LUBRICATION OF PVC .....	213
By Anthony Bohaczuk, Henkel Corporation	
PLASTICIZERS FOR PVC .....	238
By James T. Renshaw, Monsanto Polymer Products Company	
IMPACT MODIFICATION OF PVC .....	262
By J.T. Lutz, Jr., Rohm and Haas Company	
A COMPUTER PROGRAM TO FORMULATE FLEXIBLE PVC .....	334
By A. Don Beeler, Dean C. Finney, and Ted Douglas, Eastman Chemical Products, Inc.	
Supplement — Pound - Volume Economics, Conoco-Vista Chemical Co. ....	359

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AN INTRODUCTION TO PVC: SUSPENSION AND  
MASS POLYMERIZATION PROCESSES

BY

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# AN INTRODUCTION TO PVC: SUSPENSION AND MASS POLYMERIZATION PROCESSES

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## INTRODUCTION

Poly(vinyl chloride) (PVC) was first characterized more than 100 years ago, but did not start to gain commercial importance until the 1930s. The problems caused by its inherently poor thermal heat stability were overcome by the development of suitable stabilizer systems. Progress in PVC technology has involved a complex interaction between resin development, effects of additives, and equipment design, as well as market demands. Today, PVC is the second largest volume thermoplastic used in the U.S. and is the lowest priced of the five leading plastics. This low cost, along with its great versatility, is one of the major reasons for its large share of the plastics market.

## CHEMISTRY OF VINYL CHLORIDE POLYMERIZATION

Vinyl chloride monomer (M) is polymerized efficiently in the presence of a free radical source ( $I^{\cdot}$ ). The general reaction scheme and initial kinetics are typical of a free radical chain reaction, as shown in Figure 1. The rate of initiation ( $K_1$  and  $K_2$ ) is dependent on the type and concentration of initiator(s) and the temperature of polymerization. The rate of propagation (polymerization,  $K_3$ ) is also dependent on initiator(s) and temperature, but it becomes complex because of the presence of the precipitating polymer phase. The molecular weight of the polymerizing PVC chain is dependent on the rate of termination ( $K_4$ ) by transfer to monomer which is temperature dependent. In addition, molecular weight can also be affected by chain transfer to another chemical (chain transfer agent), and this rate of termination ( $K_5$ ) is dependent on temperature, type, and concentration of chemical additive. Because  $K_5$  is much greater than  $K_4$ , the resulting polymer chain will be of lower molecular weight.

Experimental work has shown that the free radical polymerization of vinyl chloride is thought to strongly favor a head-to-tail structure. The low order of crystallinity is attributed to the non-regular positioning of the chlorine and hydrogen atoms about the carbon atom. The degree of crystallinity of commercial PVC is estimated to be about 8-10%. Chain branching is believed to be low. What branching does exist is attributed to free radical transfer from a growing chain to the back of the same or another polymer molecule.

Three types of processes are used for the commercial manufacture of PVC: suspension, mass, and emulsion polymerization. About

83% of all PVC is produced by the suspension process, about 9% by the mass process, and 8% by the emulsion process.

### SUSPENSION POLYMERIZATION PROCESS

Figure 2 illustrates a typical suspension PVC process flow diagram. The process begins with the charging of the process ingredients, as shown in Figure 3. While being agitated, the mixture of these ingredients is brought to the desired polymerization temperature, usually by circulating hot water through the reactor's jacket. Since the polymerization of vinyl chloride is an exothermic reaction, the heat of reaction must be removed in order to maintain the desired polymerization temperature. This heat can be removed by circulating cooling water through the reactor jacket. Refrigerated water and specially designed cooling baffles can improve heat removal. Also, stainless steel-lined carbon steel reactors have an advantage over solid stainless steel or glass-lined reactors. Several producers have developed reflux condenser technology to improve the reactor's heat removal capacity.

The pressure in the reactor is the sum of the partial pressures of vinyl chloride and water at the polymerization temperature. As shown in Figure 4, the temperature and pressure remain constant during polymerization up to about 70% conversion, at which time the reactor pressure begins to drop because of the absence of liquid monomer. The rate of conversion increases throughout the polymerization, but begins to decrease at pressure drop. The reaction is then terminated at a specific conversion depending on product quality requirements. The unreacted monomer and PVC slurry are then transferred to a dump tank, and the unreacted monomer is degassed into the recovery system and stored in a separate tank to be recycled back into the reactor with the other charge ingredients.

The discovery in 1973 that exposure to vinyl chloride resulted in the increased incidence of a rare form of liver cancer (angiosarcoma) resulted in fundamental changes in the PVC industry. In addition to air emission and water discharge regulations in general, PVC producers must now comply with OSHA employee vinyl chloride exposure standards and the EPA vinyl chloride emission standards. These standards are met by additional monomer recovery in a stripping process where the slurry is heated and stripped of very small amounts of residual vinyl chloride by contact with steam. Continuous steam stripping is carried out in a trayed stripping tower by injecting steam directly into the slurry. This stripping process is carried out in such a way as to minimize heat exposure of the resin to avoid resin degradation.

The reactor is then rinsed with water. In order to meet OSHA and EPA regulations, employee exposure and reactor opening emissions are eliminated by using high pressure water cleaning equipment that operates in a closed reactor. Then the reactor's internal surfaces are treated with chemicals to prevent polymer buildup. The reactor is then ready to be recharged. Although the suspension polymerization of vinyl chloride is a batch process, after the reactor is emptied, the downstream processes can be carried out in a continuous manner.

The stripped slurry is then held in a blend tank before being centrifuged to produce a wet cake having a water content of 18-25%. A two-stage fluid bed dryer is used to dry the resin to below 0.3 wt% water. The resin is then stored in silos and is usually shipped in bulk by either rail cars or trucks, or packaged in bags or gaylords.

PVC plants before the 1970s contained many small batch reactors. Large reactor technology allowed the reactor size to be increased from 2,000-10,000 gallons to 15,000-50,000 gallons. These large reactors increased productivity, lowered plant costs, improved product quality and uniformity, and reduced vinyl chloride exposure and emissions. More and more older small reactors have been shut down and replaced with modern large reactors.

### MORPHOLOGY OF PVC

In the suspension polymerization of vinyl chloride, the bulk monomer phase is dispersed in water by vigorous agitation and the droplets produced are stabilized by the presence of a suspending agent. The correct amount of this suspending agent is used so that the monomer droplets undergo controlled coalescence during polymerization to give rise to an irregularly shaped **grain** having an average particle size of about 150 microns (Figures 5 & 6). The size, shape, and porosity of this grain is very dependent on the type and concentration of the suspending agent used and on the type of agitation (e.g. stirrer speed, agitator type, size and shape of baffles).

Microscopic examination of this PVC grain shows that it is composed of an agglomeration of **sub-grains** which are about 40 microns in average size (Figures 7 & 8). These are the stabilized monomer droplets which, during the initial phase of polymerization (about 5-15% conversion), coalesce to form the PVC grain. Further microscopic examination of these sub-grains show that they are made up of primary particle **agglomerates** which are about 5 microns in average size (Figure 9). These are formed during the early stages (2-5% conversion) of polymerization by coalescence of **primary particles**. These primary

particles are about 0.7 microns in size and are formed at very low conversion (less than 2%).

Within the monomer droplet suspended in the water, the first aggregate of polymer chains that precipitate, about 50 in number, form the smallest PVC species identified so far, the **microdomain**, which is about 0.02 microns in size. These microdomains coalesce to form a **domain**. A domain is the nucleus of the primary particle, contains about 1,000 micro-domains, is about 0.2 microns in size, and is only observed at very low conversions (less than 2%). This term is only used to describe the 0.2 micron species because it immediately starts to grow to become the primary particle. The micro-domain and domain are not features of PVC morphology at high conversions since a growth of these species with conversion obliterates all memory of them, leaving only the observable primary particle.

From this discussion, it can be shown that the formation of suspension PVC takes place through a series of interconnected aggregation steps which can be represented by the scheme in Figure 10.

Suspension PVC particles usually possess a pericellular "skin" or "membrane" which extends almost continuously over the entire outer surface of the grain. This skin is formed during the early stages of polymerization (less than 5% conversion) and has been shown to be a copolymer of vinyl chloride and the suspending agent(s). Since this skin is semi-permeable, it does contribute to the overall porosity of the resin, but not nearly to the extent as does the primary particle agglomerate.

Several different types of PVC grains can be obtained during the suspension polymerization process. The most desirable grain has a semi-permeable skin and good porosity where the pores are open and evenly distributed throughout the grain. A less desirable grain contains closed pores which are not connected to the skin. The least desirable grain is the solid grain containing little or no porosity. The production of these different types of grains is mainly determined by the choice of suspending agents.

## **REACTOR PROCESS VARIABLES AFFECTING THE PROPERTIES OF SUSPENSION PVC**

### **Molecular Weight**

The various different grades of commercial PVC are mainly determined by their molecular weight. In the absence of other active chemicals, the molecular weight of PVC is almost entirely determined by the polymerization temperature; the higher the polymerization temperature, the lower the molecular weight.

In the presence of chain transfer agents, the molecular weight is lowered depending on the type and concentration of agent. Thus, low molecular weight resin can be produced at lower polymerization temperatures using chain transfer agents. In the opposite manner, the use of chain extending agents will allow the production of high molecular weight resins at higher polymerization temperatures.

### **Particle Size**

Particle size is important for various PVC applications. For rigid extrusion, a more coarse resin is desired so that all of the grains are well fused in the extruder and no unfused fines are left to cause weaknesses in the final product. For plasticized applications (calendering, blown film), a finer resin is preferred so that there are no large unfused grains which will cause imperfections in the finished product.

Within a given reactor with a given agitation system, the particle size of PVC is dependent upon the type(s) and concentration(s) of the suspending agent(s); the higher the concentration of suspending agent(s), the finer the resin. Since the PVC grains are a distribution of sizes, particle size is usually described as an average particle size and the broadness (or narrowness) of the distribution is described as the coefficient of variation (the standard deviation divided by the average particle size); the smaller the coefficient of variation, the more narrow the particle size. Particle size can also be expressed as the percent through a set of standard mesh screens (ASTM test method D-1705-82).

### **Porosity**

The porosity of PVC is most important in controlling not only the absorption of plasticizers but also the desorption of vinyl chloride during the recovery/steam stripping process. A good understanding of the mechanism for the development of porosity in PVC is necessary to insure good quality resin. The various grades of PVC can be further divided into porous (for flexible applications) and non-porous (for rigid applications) resins.

The process of plasticizer absorption is generally believed to occur by a diffusion mechanism in which two separate mechanisms exist. The first process is a very rapid capillary filling of the intergrain (pores between the resin grains) and intragrain (pores within the resin grains) pores. The second process involves the diffusion of the plasticizer molecules into the molecular PVC chains. This second process is the rate-determining step in the absorption of plasticizer and is controlled by the size of the sorbing sphere, the primary particle agglomerate. Since it is known that one way to increase the absorption

rate of a fluid into a solid is to reduce the size of the solid particle, it can be concluded that as the diameter of the agglomerate increases, the porosity of the resin decreases. Thus, controlling the size of the agglomerate is the important step in establishing the porosity of a PVC resin.

There are several process variables that affect the porosity of PVC. These are as follows:

**Polymerization Temperature** - For a given set of polymerization conditions, reaction temperature has a very large effect on porosity; the higher the reaction temperature, the lower the porosity. This is because the agglomerate size increases as the reaction temperature increases.

**Conversion** - Conversion is also found to have a very large effect on resin porosity; the higher the conversion, the lower the porosity. Again, this is due to the agglomerate size. Resins polymerized to a higher conversion have larger agglomerates than those polymerized to a lower conversion.

**Agitation System** - Agitation is of fundamental importance in the PVC suspension process. Together with the suspending agent system, it determines the particle size of the finished product and other properties, such as porosity. Generally, the more power (e.g. higher speed) the agitation system puts into the reaction mixture, the higher the porosity. Also, different agitator blades and baffles can affect resin porosity.

**Suspending Agent System** - Given a particular type of agitation system, a particular polymerization temperature, and a reasonable conversion (75-85%), the suspending agent system has a great influence on resin porosity. The **primary suspending agent** is usually a water soluble organic polymer, such as a polyvinyl alcohol, a substituted cellulose, or a mixture of the two. Generally, the lower the surface tension of the suspending agent(s), the higher the porosity of the resin.

The primary suspending agents are chosen not only for their ability to control particle size but also for their ability to produce porous resin. However, the degree of surface activity for these chemicals is limited, thus additional **secondary suspending agents** are used to further increase porosity. A variety of materials can be used, such as non-ionic or anionic surfactants and low molecular weight polyvinyl alcohols of very low degree of hydrolysis. These secondary suspending agents increase porosity by either lowering the surface tension between the vinyl chloride and

the aqueous phase or by stabilizing the primary particle agglomerates during polymerization.

There are several ways of measuring and expressing the porosity of PVC. Among these are the following:

**Porosity** - This is measured by mercury intrusion and expressed as cc/g (ASTM D-2873-70). Also other descriptions of the porosity of the resin are available from this single measurement: void volume (cc/g), a measurement of the inter-grain pores (volume between the resin grains); average pore size (microns), a measurement of the diameter of the pores within the grain; and distribution of pore sizes.

**Cold Plasticizer Absorption (CPA)** - The amount (%) of plasticizer that a resin will absorb at room temperature (ASTM D-3367-75). This is a measurement of both inter- and intra-grain porosity and should be about the same value as the mercury intrusion porosity measurement.

**Brabender Dry Time** - Under a given set of test conditions, this measures the time (min) that it takes a given amount of resin to fully absorb (giving a dry powder) a given amount of a particular plasticizer at an elevated temperature (about 80° C) using a torque rheometer for mixing (ASTM D-2396-79). This is primarily a measure of the filling of the inter- and intra-grain pores and the rate of hot plasticizer absorption (diffusion into the resin molecules), but measures, to some extent, the capacity of the resin for plasticizer.

**Gel Test** - In this test, a plasticized milled sheet is prepared under given conditions and the number of gels (fisheyes, hard particles, glass) are counted in a given area (ASTM D-3596-77). A gel is a PVC grain which does not absorb plasticizer at the same rate as the other grains. This is usually due to the inaccessibility of the pores to the plasticizer. The number of gels will decrease to a constant number as the length of time on the mill increases.

**Microscopic Examination** - A simple technique for qualitatively examining the porosity of PVC is placing the resin on a microscope slide and immersing it in a liquid having a refractive index similar to PVC and, after equilibrium, viewing it at low magnification (100x) with transmitted light. The internal morphology of each grain can clearly be observed.

### **Bulk Density**

Generally, the bulk density (g/cc) of PVC (ASTM D-1895-69) is in opposition to its porosity; as porosity decreases, bulk density increases. Thus, all of the process variables that affect porosity also affect bulk density. The most important process variable is probably the choice of the suspending agent system.

For rigid extrusion applications, the rate of output of the extruder (especially twin-screw extruders) is directly dependent on the bulk density of the dry blend which feeds the extruder. Thus, it is important that this dry blend have a high bulk density.

### **Electrical Properties**

For wire and cable applications, the electrical conductance of the PVC resin is an important property. PVC must be produced which has extremely low conductivity (or very high resistivity). Generally, this is done by using deionized (DI) water as the process water and not contaminating the resin with non-DI water or resin made with non-DI water.

## **STEAM STRIPPING AND DRYING PROCESS VARIABLES AFFECTING THE PROPERTIES OF SUSPENSION PVC**

### **Color**

The color of PVC resin is usually measured by a colorimeter and is described by a L-, a-, and b-value. The L-value is a measure of its whiteness; the a-value is a measure of its pinkness; and, the b-value is a measure of its yellowness. In order to assure a white, non-pink, non-yellow resin, antioxidants and killing agents (to prevent further polymerization at high temperatures) can be added to the slurry before steam stripping and drying to prevent degradation.

### **Heat Stability**

Good heat stability of PVC is maintained by the proper care given to the steam stripping and drying processes. Too high a temperature or too long a residence time will decrease resin heat stability. Again, the addition of antioxidants and killing agents will aid in maintaining good heat stability.

Heat stability is usually measured in these three ways:



**Static Oven Heat Stability** - PVC is milled into a given plasticized compound and subjected to a given temperature in an air-circulating oven. Coupons (or chips) are removed from the oven over a given period of time until the milled sheet has degraded to black (ASTM D-2115-67). The resins are compared to a standard resin of the same molecular weight and similar quality. If the time-to-black is less than the standard resin, then the heat stability of that resin is poor.

**Dynamic Mill Heat Stability** - PVC is mixed into a given rigid dry blend and placed on a two-roll mill at a given speed and temperature. Coupons (or chips) are removed from the mill at given intervals of time until it turns dark or sticks to the mill. Again, the resins are compared to a standard and judged to be less stable if the milled sheet turns dark or sticks to the mill before the standard.

**Brabender Heat Stability** - PVC is mixed into a rigid dry blend and placed in a torque rheometer at a given speed and temperature. The amount of time it takes from fusion to degradation is a measure of its heat stability.

### **Percent Volatiles and Contamination**

Both of these properties are mainly dependent on the proper use of the dryer. If the dryer temperature is too low, then an excess of volatiles will be present in the resin; if the dryer temperature is too high, the amount of contamination (dark specks which are usually burnt resin) will be too high. Also, contamination can be caused by a dirty dryer.

### **Static**

Even if the PVC is dried to the proper dryness, there may be static built up on the resin which will cause poor flow properties. The static can be eliminated or reduced in the drying process by the addition of an antistatic agent (e.g. dry steam).

## **MASS POLYMERIZATION PROCESS**

Extensive research efforts were undertaken during the 1940s and 1950s to develop a successful mass polymerization process because of its potential for both cost savings (reduced raw material usage and elimination of the centrifuging and drying operations) and product improvement (reduction of chemical residues and elimination of the pericellular skin). Initial attempts followed the fairly conventional approaches of using a single reactor. A major breakthrough came in 1963 when Pechiney-