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History of Vinyl Chloride Polymers*

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ABSTRACT

In 1926 Semon tried to dehydrohalogenate high molecular weight poly(vinyl chloride) (PVC) in a high boiling solvent to get an unsaturated polymer which might bond rubber to metal. Unexpectedly, he obtained plasticized PVC, a flexible product inert both electrically and chemically. This discovery opened the door to the commercialization of PVC, a plastic with an annual United States production now exceeding 6 billion pounds. Special PVC's and PVC products have been developed taking advantage of the many favorable properties. Rigid structural products from house siding to pipes are becoming of increasing importance. Two main types of polymers have been utilized: 1) one prepared by suspension polymerization, and 2) a special variety prepared by colloidal polymerization and spray drying. This latter material has been especially useful for making plastisols. Plasticizers and stabilizers were developed to maximize useful and nontoxic properties. Vinyl chloride monomer (VCM) production and copolymerization evolved as lower cost processes, higher quality products, and greater manufacturing safety were introduced. Recent challenges for the industry have included pollution and carcinogenic hazards which have been overcome by imaginative new technologies. The rate of growth of the industry is shown graphically.

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*Based, in part, on W. L. Semon's recollections and upon a presentation before the Cincinnati, Ohio Regional Seminar of the Vinyl Division of the Society for Plastics Engineering, October 2, 1973.

INTRODUCTION

The early development of the industry based on polymers of vinyl chloride (PVC) differs from the development of most polymer industries. First, the discovery of the useful properties of plasticized high-molecular-weight PVC was accidental. It resulted from a serendipitous observation by a young scientist. Second, the PVC industry in the United States, was not developed by a plastics manufacturer but through efforts in the rubber and chemical industries.

Nevertheless, the growth of the industry has been phenomenal. As shown in Fig. 1, in 1955, PVC production was slightly more than 500 million pounds. In 1979 it topped 6 billion pounds. Along with this growth there has been a decrease in the price of PVC resin. The price since 1955 is shown in Fig. 2. More recently, inflation and increased raw materials and energy costs have increased the price for PVC.

The PVC industry is still growing rapidly. Each new use as it develops adds a fresh start on the growth curve. Consequently, the overall curve for the industry has not yeat started to level off.

The current energy situation makes PVC even more of a bargain. PVC is one of the lower priced polymers today when viewed on a cost-per-cubic-inch basis. One reason is that while most other plastic materials are entirely dependent on hydrocarbon-based feedstocks, the PVC molecule is more than half chlorine.

PVC offers economic advantages and possesses high potential for penetration into markets normally served by traditional materials such as wood and metal in construction. PVC competes with low cost plastics in volume applications but, because of its versatility in being compounded, it can also serve in applications requiring so-called "engineering thermoplastic" uses in extrusion and molding.

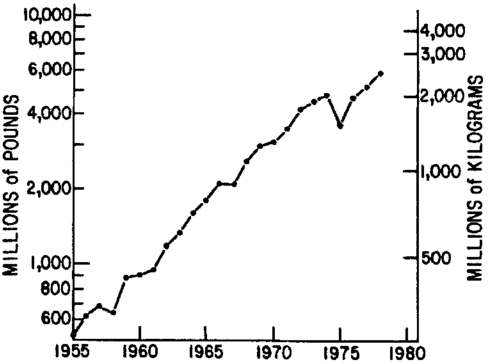

FIG. 1. United States production of PVC and PVC copolymer resins. (Source: Society of the Plastics Industry, Inc.).

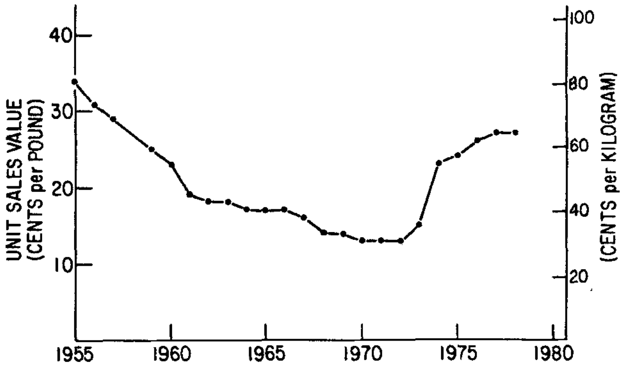

FIG. 2. The unit sales value of PVC, 1955-1978. (Source: U.S. International Trade Commission.)

TABLE 1. Technical Factors

1. Reliable source of monomer
2. Improved PVC manufacture
3. Development of improved compounding ingredients
4. Improved processing machinery
5. Better understanding of PVC molecule

Several important factors have contributed in PVC's phenomenal growth. Some are technical (Table 1); others are service in nature (Table 2).

I (G.A.S.) feel it is appropriate at this point to preface the remainder of this paper with a few comments. As you will note, the principal author of this paper is Dr. Waldo L. Semon (Fig. 3), now retired from the BFGoodrich Co. Inherently then, the presentation will reflect primarily the BFGoodrich view of the development of the industry

TABLE 2. Service Factors

1. Quality control
2. Improved testing methods
3. Consideration for worker and consumer safety
4. Promotion of economic advantages

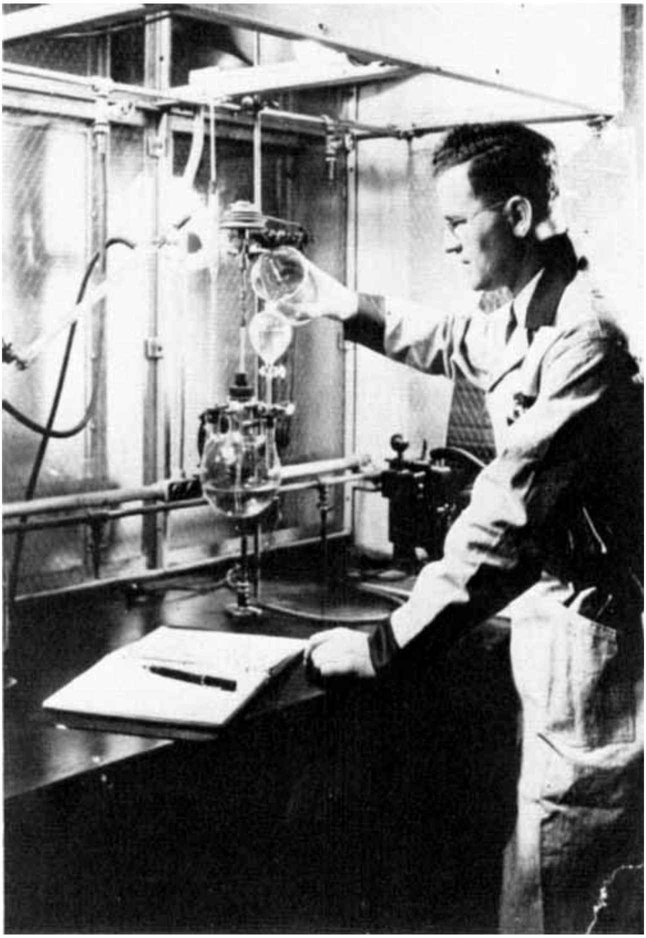

FIG. 3a. Dr. Waldo L. Semon, research chemist, in 1937 working in the Research Laboratory.

based on polymers of vinyl chloride. In particular, it will stress the development and growth of the PVC industry as related to plasticized high-molecular-weight PVC.

Any gaps in chronology are unintentional. Dr. Semon and I prepared this report—and particularly that section covering the period 1926 through World War II—based on Dr. Semon's recollections. Dr. Semon is credited with discovering the means of plasticizing high-molecular-weight PVC and thus opening the door to commercialization of this highly versatile thermoplastic.

FIG. 3b. Dr. Waldo L. Semon, retired research professor, Kent State University, in 1980.

DISCOVERY AND EARLY HISTORY

In the early 1920s, BFGoodrich was investigating the use of rubber as a chemical raw material. One BFG scientist, Dr. Harry L. Fisher, found that raw rubber could be isomerized (cyclized) to give a number of polymers varying in properties from gutta percha-like to shellac-like [1]. One of the resulting materials was developed and served as the basis for the Vulcalock process for bonding rubber to metal [2]. The process was used extensively for making rubber-lined tanks or tank cars. Goodrich's Dr. Howard E. Fritz (Fig. 4), sales manager for rubber-lined tanks, hoped that a nonthermoplastic bond could be discovered. With this in mind and with the idea of broadening the patent protection, Dr. H. L. Trumbull (Fig. 5), manager of the company's research laboratory, hired Waldo L. Semon in 1926 to search for a synthetic adhesive.

Semon thought that he might start with some material other than crude rubber and convert it into a polymer having adhesive properties. He succeeded with both vulcanized scrap rubber and German methyl rubber. Not satisfied, Semon hoped to make an adhesive from a more simple synthetic organic polymer.

FIG. 4. Dr. Howard E. Fritz, manager of Tank Lining Sales, BFGoodrich Co., 1926.

He began to look at a substance, the monomer vinyl chloride, which had been reported as early as 1838 by the French chemist Regnault. He found that Baumann [3] in 1872 had described the preparation of vinyl chloride (VCM) which, when exposed to sunlight, was converted to an insoluble amorphorus mass. In 1916 Ostromislensky [4], working with VCM, had obtained polymers which he named cauprene chloride. He considered the insoluble gamma form of cauprene chloride to be completely nonprocessable and intractable. Semon thought that this so-called gamma form might serve as an adhesive if he could remove

FIG. 5. Dr. H. L. Trumbull, manager of Chemical Research Department, BFGoodrich Co., in 1926.

the HCl, but he found it to be insoluble in practically everything at room temperature. Consequently he prepared the polymer and succeeded in solvating it at a high temperature in a high boiling solvent. Although he was unsuccessful in dehydrohalogenating it, when he examined the cooled "solutions" he found them to be flexible and elastic. Moreover, on further examination he found them to be chemically nonreactive and quite resistant to ordinary solvents.

For a time, Semon forgot about his main assignment and investigated this interesting new product. He molded a golf ball from one highly elastic composition. He then tried it for other products, including molded shoe heels and as insulation on the handles of pliers and a screw driver and wire—using a hot solution of the plasticized PVC in chlorobenzene. Since his main problem dealt with lining tanks and this new material did not adhere directly to metal, he spot-welded metal gauze on the interior of a small vessel, coated the gauze with a paste of powdered PVC polymer and plasticizer, and then heated it to obtain a fused, mechanically bonded PVC lining. The tank was surprisingly corrosion resistant to hydrochloric, nitric and sulfuric acids and concentrated sodium hydroxide. These early experiments were the foundation for plasticized PVC [5] and of the plastisol process [6].

Semon, a rubber chemist, was intrigued by the possibility that he had found a substitute for rubber, and was astute enough to sense the potential, unique commercial applications of the material. As long as PVC was viewed as a substitute for rubber, there was resistance to its acceptance because of economics and lack of technology necessary for its application, but Semon saw that the material had special propeties and potential uses which made it superior to rubber in many applications.

But any product development program required a reliable source of high-molecular-weight PVC polymer. Mass polymerization of VCM was unpredictable due to difficulties in initiation and the inability to remove the heat of polymerization from even medium-sized batches. Polymerization of VCM dissolved in methanol or other common solvents gave polymers of unsatisfactory quality.

Semon and his co-workers learned in the 1920s that Carbide and Carbon Chemical Corp. (now Union Carbide Co.) was developing lacquers and rigid molded articles from copolymers of VCM and vinyl acetate. They obtained samples of these resins, but found these did not give flexible rubberlike materials when used in the place of high-molecular-weight PVC. Carbide and Carbon agreed to produce the special high polymer which BFG sought. The polymer was reported to be made by partial polymerization of liquid VCM in a mass polymerization process.

So the initial phase of a product development program for high-molecular-weight PVC was underway at BFG in the early 1930s.

FIRST PRODUCTION ITEMS

As Dr. Semon recalls, BFG's first PVC production items were shock absorber seals made in a mold such as those used for molding rubber. These seals were highly satisfactory since they did not swell in the shock absorber fluid. Rather, as a result of partial extraction of the plasticizer, the seals shrank onto the plunger during use.

Semon knew the seals were a start but other technical problems for using high-molecular-weight PVC were still to be resolved. The PVC was variable in particle size, porosity, acetone extract and stability, and was also expensive. Few suitable plasticizers were available. Tricresyl phosphate was good in many respects. It gave, for example, flame-resistant properties, but had too much odor. Dibutyl phthalate was low in odor, but was volatile. Heat and light stability of the plasticized product left much to be desired.

These were but a few of the problems to be considered in the development program. In order to get management to justify further expenditures for additional development, products were prepared from hot PVC solutions. Coated electroplating racks and thin film coated fabrics were soon developed. Somewhat effective stabilizers, sodium silicate and white lead, were also added.

In the early 1930s the name Koroseal, which stands for sealant from corrosion, was coined as BFG's trade name for the products made from its high-molecular-weight PVC.

Dr. Semon remembers an incident wherein he demonstrated vividly to his vice president of sales the waterproofing ability of plasticized PVC. Dr. Semons' wife had just made new cretonne curtains. He took some of the brilliantly colored scraps and solution coated them with Koroseal material. He showed the samples to a vice president and confidently claimed that they were waterproof. Seing doubt in his superior's eyes, Dr. Semon placed a sample of the coated cretonne on top of the papers in the vice president's in-out mail basket. Then, before the executive could stop him, Dr. Semon poured water from a water decanter onto the material. It didn't leak.

"If it had leaked, I don't know what would have happened to me or to the Koroseal project. But it tickled the vice president's interest. And, shortly, we added new impetus to the development program," Dr. Semon says.

One of the early customers, General Electric, started the manufacture of their Flamanol insulated wire in which a flame-resisting insulation of PVC plasticized with tricresyl phosphate was applied to copper wire by a typical extrusion process. The next product was PVC coated fabric (silk coated with a cement of plasticized PVC in methyl ethyl ketone). Insulated wire, raincoats, and shower curtains were really the products which started the business based on high-molecular-weight PVC.

The increasing consumption of PVC emphasized the need for an improved process for preparing the polymer, a better and less expensive source of monomer, and better plasticizers. First, a project was initiated to develop a better way for preparing PVC. One was developed for polymerizing VCM under pressure in an emulsion. In this program a number of different types of glass-lined pressure vessels were made and tested. Ultimately, a satisfactory vessel and stirrer seals were produced and scaled up for semiproduction use.

Second, VCM was being produced by reacting ethylene dichloride with sodium hydroxide in methanol solution. Since an improved method for making PVC was desired, research was conducted on alternate techniques. The catalytic reaction of acetylene with HCl was eventually selected.

Third, a BFG scientist, T. L. Gresham, was assigned the development of improved plasticizers. He tested more than a thousand compounds, and eventually selected di-2-ethylhexyl phthalate (dioctyl phthalate, DOP) as the best general purpose plasticizer [7].

Semon's and his co-workers' efforts with plasticized high-molecular-weight PVC had not gone unnoticed. In 1937, Semon—in visiting a German exhibition in Dusseldorf, Germany—was told that one of his publications [8] had a profound effect on German development. He saw flooring, wall covering, insulated wire, piping, upholstery, and other items. While Semon was impressed with the progress, he noted difficulties in the quality of some of the products. However, it didn't dampen his enthusiasm; he just knew that more development work needed to be done, and he reported that to his management upon his return.

In the years immediately preceding World War II, a wide variety of products were made from plasticized PVC and sold in the United States under the Koroseal trademark. Packings and gaskets had been sold from the very beginning of 1935, and in 1937 a volume of business was realized from the sale of Koroseal beverage tubing. Coated cloth, trouser belts, suspenders, and wristwatch straps were made and sold under the Koroseal name. Raincoats, shower curtains, and other film products were also popular.

Practically all of the BFGoodrich Koroseal products, including those enumerated above, met with such tremendous success that it became necessary to construct commercial PVC plants at Niagara Falls, New York, in 1940, followed by a larger plant at Louisville, Kentucky, in 1942.

In early 1941 the Chemical Division of the BFGoodrich Co. had been organized for the sale of polyvinyl chloride resins and plastics in the raw form. The trade name Geon (from geo for earth and eon, meaning long lasting) was adopted to cover the entire series of "polyvinyl" materials.

In November 1943 the Chemical Division was expanded to include all functions of a separate company unit with its own manufacturing, technical, and sales departments.

Operating independently of all other divisions of the parent organization, the Chemical Division moved to Cleveland, Ohio, in July 1944. A completely equipped sales-service laboratory was constructed adjacent to the sales office. This was a unique move inasmuch as a laboratory was readily accessible to the individuals of the division who were aiding customers in their manufacturing problems.

During World War II the Chemical Division, and later the Chemical Company, devoted its entire effort to war production. Geon resins and plastics had proved excellent for wire insulation, and the U.S. Navy used vinyl-insulated wire almost exclusively in those instances where fire-retardant insulation was required. Both United States producers of PVC, BFG and Union Carbide, were involved in supplying the needs of the armed forces. Total production of PVC resins and plasticizers increased phenomenally during the war years.

By 1950, five companies were producing PVC in the United States and this number quadrupled by the mid 1960s. In 1979, 20 United States companies produced the material.

When the war ended, the industry was faced with finding civilian outlets for the substantial production of PVC. This was done by extensive product application work with potential customers to develop and use the excellent properties of PVC–durability, versatility, and greater resistance to ignition and flame spread than other polymers.

Expansion of applications and markets were made possible by development of new materials such as PVC latex, copolymers of vinyl chloride, plastisols, and easy processing resins.

Where solvents were required previously in coating applications, the latexes provided a number of advantages. The dispersion of PVC resin in water eliminated the use of solvent and its subsequent recovery, it eliminated the danger of potential fires and explosions, it eliminated toxic solvent fumes, it permitted higher solids loading, and it assured better adhesion to the finished products.

These advantages provided an entry for latex usage in the fabric coating and binding applications, such as in boots and inflatable structures.

Research on latex resin preparation led to an important BFGoodrich patented invention. This preparation depended on maintaining, during the polymerization, a fixed and definite relation between the amount of dispersing agent in the reaction mixture or latex and the surface area of the polymer particles being formed. By doing this a very flowable latex is produced.

In our early work we developed a vinyl resin which can be dispersed in a plasticizer to give a very flowable mixture which can be coated on fabric or poured into molds. By heating this mixture a clear, flexible, tough product is formed. This technique was covered by a Semon patent granted in 1940 [6]. These are called paste resins.

With the cooperation of our then British Geon associate in Great Britain, the first BFGoodrich paste resin was produced in Louisville in 1948. The introduction of these improved paste resins opened up new applications in spread coating, dip coating, embossing, dip molding, and cavity and rotational molding such as are used in making flotation buoys, sponge and foam products, and in printing and painting techniques.

During the 1950s, several patents were granted BFGoodrich chemists for new methods to speed up the polymerization process.. Some of these techniques have now been replaced with more modern ones, but at the time they helped increase equipment productivity and thus reduce PVC cost.

To meet the increasing demands of the building and construction industry, we needed a PVC with improved processing and physical properties. In 1951, BFG introduced a hard, tough, and rigid thermoplastic PVC composition which could be easily processed by milling, molding, calendering, and extruding, and which by virtue of unique thermoplastic characteristics possessed the ability to be forced and shaped into final products by techniques such as extruding, molding, deep drawing, and embossing. For example, siding made of PVC can be embossed with a wood grain pattern.

However, the new rigid PVC composition, while tough and hard, was not impact-resistant enough to meet the demands of applications requiring impact resistance. Research led to the development of a very special processing aid and rubber combination which led to an extremely high-impact PVC. These mixtures give rigid compositions which are highly impact-resistant and are easily processable by usual techniques to give products with excellent chemical, physical, and electrical properties. This contribution was instrumental in our entry into the building industry in supplying PVC for use in siding, window frames, guttering, spouting, piping, trim, paneling and ceiling grid systems, and other applications. Today, rigid vinyl accounts for more than half of the general purpose PVC business.

There was a need in the electrical and construction business for a material with the outstanding properties of PVC, such as corrosion, weather, light, and chemical resistance, and electrical and mechanical properties which could be used at higher temperatures.

A patented process has been developed for chlorinating high-molecular-weight PVC which increases the second-order transition temperature from 75°C to as high as 130°C depending on the extent and method of chlorination. The added chlorine is predominantly in the 1,2-ethylene configuration in contrast to the less stable 1,1-ethylene configuration produced earlier by others. Soon after this development it was discovered that this high temperature material functions as a processing aid, an impact improver, and gives a more stable product.

These discoveries opened up new applications and extended the range of products available from PVC. As a result, a satisfactory higher temperature application had been achieved over previous thermoplastics. One application is in hot water pipes for both domestic and industrial use.

PROCESS IMPROVEMENTS

Prior to the early 1960s vinyl chloride monomer was produced from acetylene and hydrogen chloride. In the late 1950s BFG developed an improved commercial process for thermally cracking ethylene dichloride (produced by the addition of chlorine to ethylene). The ethylene dichloride cracking produces VCM and hydrogen chloride. The hydrogen chloride was reacted with actylene to make more VCM. In the early 1960s we were using this half-ethylene, half-acetylene route to make vinyl chloride. At the same time BFG research scientists were investigating the production of ethylene dichloride from ethylene, hydrogen chloride, and air or oxygen. It was recognized that if this could be accomplished efficiently, we would have an all-ethylene route to vinyl chloride and could eliminate the more expensive acetylene completely.

As a result, research scientists were successful in finding the catalysts and conditions of what is called the oxyhydrochlorination reaction. Because BFGoodrich developed microplant techniques, we were able to take the discovery from the laboratory through microplant and pilot-plant development to a commercial plant in a relatively short time.

In this process ethylene is reacted with chlorine to produce ethylene dichloride which is cracked to give VCM and hydrogen chloride. The hydrogen chloride is returned to react with more ethylene and oxygen to give more ethylene dichloride and continue the cycle to more VCM.

Within 2 years from the start of the development work in 1964, a plant was on stream at Calvert City, Kentucky, based on an all-ethylene route to VCM. It was producing 300 million pounds of VCM per year. Since then many other VCM producers, worldwide, have licensed the process.

As to the PVC polymer, much development work has evolved over the past 50 years.

The two major pioneer United States producers of PVC pursued different paths: Union Carbide went the PVC copolymer route; BFG went the plasticized homopolymer PVC route.

Early on, BFG recognized that PVC granule size and uniformity were keys to successful use of the polymer. Cubers were developed to assure uniformity of granules.

Other elements had to be overcome. Stabilizers, pigments, and lubricants were essential in using the plasticization method to convert the heat-sensitive PVC into a workable material.

Almost all early work on uses for high-molecular-weight PVC was carried out on rubber processing equipment. For the industry to grow, new machinery had to be developed. And as the new machinery appeared, the PVC material had to be adapted so as to be converted into end products at higher rates and lower costs.

The size of PVC reactors has grown spectacularly. Some in use today are estimated to be in the 30,000 gal category.

As new fundamental information was gradually developed, the art of formulating PVC grew into a science. Scientists researched and developed the fundamentals needed to develop and control PVC properties which extended PVC usage into new applications.

Melt flow properties were studied. Molecular weight, molecular weight distribution, and resin particle size and shape were, and are, being investigated.

Such fundamental studies led to development of easier processing resins which absorb plasticizer faster. Another key step was to produce resins which could be mixed with a plasticizer in a dry mixer, thus saving processing time.

Another area of primary importance, understanding the molecular structure of PVC, has drawn increased attention in recent years.

Essentially, two reasons had stymied study in this area. First, proprietary interests inhibited or prevented publication of much good work carried out in industry. Second, and perhaps more important, PVC has been regarded as a difficult material to study and has not been a favorite polymer for academic study.

More recently, however, there has been a surge of interest in PVC as evidence by the acceleration in publications and increased activity in academic institutions. This may simply reflect the fact that PVC researchers, both industrial and academic, are now building on a more solid foundation based on understanding of polymers in general.

MARKET DEVELOPMENT

The size and vigor of the PVC industry today has been a result of innovation in technology, product, and process improvement. There has been another factor equally important–an aggressive, responsive marketing effort to demonstrate the product performance of PVC.

A prime example is the use of PVC in piping–in drain-waste-vent (DWV), in water supply, and in irrigation [9]. Standards had to be met, long-term testing accomplished, and the, literally, years of effort expended to prove that PVC piping can and does perform well within the standards established in local, state, and regional plumbing codes. As a result of such effort, more than 2 billion pounds of PVC went into piping in 1978.

RESPONSE TO ENVIRONMENTAL CONCERNS

As we approached the fiftieth year of our association with PVC, we faced another type of problem. In January 1974 it was found that three BFG employees who had worked for many years in our Louisville plant where the gaseous vinyl chloride monomer is polymerized to PVC had died from a rare form of liver cancer, angiosarcoma. Since then additional cases have been identified among PVC workers in plants of various companies in the United States, Canada, and Europe.

Since the angiosarcoma finding, our actions to safeguard the health of our workers and reduce VCM exposure has received top priority. We reported all available facts to our employees, to appropriate government and medical agencies, and to the public. We set up task forces to reduce exposure to the lowest possible level using all available and most advanced technology.

We undertook the most intensive research and development program in our history to develop new technology. We committed capital expenditures of tens of millions of dollars over a 3-year period to reduce VCM exposure at our plants and in our products. One hundred thirty-five scientists and technicians spent over a year working on solutions. Their findings led to numerous changes in our PVC manufacturing process and products. The decisions of our management reflected concern for worker safety and for the requirements of the large industries and customers who are dependent on PVC products for which there are limited feasible substitutes [10].

These actions have resulted in remarkable progress. Workers are being protected from VCM exposure in compliance with rigid standards established by the government. We are operating within the standard of 1 ppm. The loss of VCM to the surrounding environment also meets rigid government emission regulations. Vinyl chloride monomer is usually trapped in the resin during polymerization. Removing the last traces of this monomer from the resin particle is very difficult. BFG has developed new polymerization recipes which give uniformly porous resin particles which allows easier removal of the monomer. In addition, this makes higher quality PVC resin with little or no hard particles, thus reducing or eliminating undesirable imperfections in the finished products. The residual monomer content in PVC resins and compounds leaving our plants is now so low that we are confident processors and users will meet all of today's health and government standards.

This progress has been due in substantial measure to new technologies we have developed and are licensing to other PVC processors. One such technology package is an improved method of continuously stripping and recovering VCM from the slurry of resin produced by suspension polymerization. Also, our high pressure reactor cleaner technology eliminates or reduces the number of times polymerization reactors must be manually cleaned. Another development is our "clean reactor" technology which virtually eliminates worker exposure to VCM when preparing reactors between charges. Our objective is to eliminate vinyl chloride monomer exposure or reduce it to the very lowest possible level, and, indeed, we are well on our way to reaching that goal.

Where do we go from here? We will never be satisfied until we know everything about PVC. We want to know further how the structure contributes to the properties; how structure modification can improve processing, properties, and stability; and how to tailor-make polymers for specific applications. As our fundamental studies continue and more sophisticated instrumentation is developed, we are gaining a greater understanding of the nature and problems of PVC chemistry and technology.

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