

# Practical Polymerization for Polystyrene

by Richard B. Bishop

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## Foreword

With the excellent and thorough book by Boyer and Boundy on styrene, it is surprising to find a book published on a similar subject. However, the author feels that there exists a need for a new book on styrene. Even though the ACS monograph was very complete, the fact is that most of the work for that book was completed before 1950.

In this volume there will be no attempt to correct or even to modify what the passage of time has clarified among our concepts, theories, or practices as they were enumerated in the earlier book. Rather, an attempt will be made here to treat the subject matter of styrene and its polymerizations in as practical a manner as possible so that it can be understood by the technician or others who might be interested in or responsible for operations in the polystyrene field.

It is certainly with some fear and with crossed fingers that the author dares to hope that this book will produce the same satisfaction and degree of usefulness to the reader that was established by its predecessor.

The author wishes to say that he owes much to the previous book by Boyer and Boundy, and he only hopes that he can add to the storehouse of information and understanding in this field with his practical approach to the polymerization of styrene.

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# 1

## Historical Background of Polystyrene

### 1.1 HISTORY

The earliest reference to the work in styrene can be traced back to a storax gum which was obtained from a tree. If distilled with water, it gave an oily material of unpleasant odor and had carbon and hydrogen in the same ratio as benzene has. In general, Bonastre is credited with the initial isolation of styrene monomer in 1831, and, since this is an early date, you might substantiate this finding in *J. Pharm. Chim.* 17:338. The study of the polymerization, however, began when E. Simon in 1839 (*Annalen* 31:265-277) described his experimentation in which the styrene monomer was changed into a material of higher order, and he interpreted this material to be a styrol oxide. Most of this study of Simon indicated that a jelly was promoted on contact with air and by heating.

Other early investigators (in 1837 D'Arcet, G. J. Mulder in 1839, G. H. Gerhardt in 1841, and in 1844 A. Glenard and C. H. Boudault by various hot-tube methods) were all able to prepare a styrene type monomer from various ingredients, such as camphor, cassia oil, calcium cinamate and a resinous body called dragon's blood.

In 1845, two English chemists Hoffman and Blyth (*Annalen* 53:314) heated styrene monomer in sealed glass tubes at 200°C and obtained a solid product which they called metastyrene. Following this along in 1851, Berthelot produced styrene by a pyrolysis of several hydrocarbons. Again this was the red-hot tube method and it was the basis of this hot tube, dehydrogenation, that actually became the basis which led to the production of styrene in commercial practices.

In the patent field, Kronstein in 1900 filed a patent (German 17,378) and later in *Ber.* 35:4153 in 1902 described the processes dealing with polymerization in a closed vessel in the absence of oxidizing agents. In 1911, F. E. Matthews filed British patent 16,277, which described the



thermal and catalytic processes for polymerizing styrene monomer to yield a substance for the production of articles formerly made from celluloid, glass, hard rubber, and wood.

Lemoine did similar early work with styrene in sunlight and in blue light and near ultraviolet and found them to be photometrically effective between 3°C and 35°C.

All of these polymers were of low grade and suffered greatly from the purity of the monomer. The work of Stobbe and Posnjak, measuring relative viscosities using Oswald apparatus, reported in *Annalen* 409(1), 1915, and *Ber.* 47:2703 in 1914, reported earlier uses of peroxide.

Because styrene monomers were very reactive, the storage and refining and purification of this material was very difficult. It was not until 1922 when Moureau and Dufraisse in *Compt. Rend.* 174:259 found that inhibitors of aromatic amines and phenols retarded the polymerization of styrene monomer. Immediately with this, the ability to improve distillation stages and to improve storage opened up somewhat the styrene market.

Ostromislensky reported in the *Journal of Russian Physical Chemistry Society*, 44:204, that as far back as 1911 he started to study the mechanism of this reaction, and his work carried on through the 1920s. Typical of the work of Ostromislensky is a patent of January 11, 1927, (U.S. 1,613,673) to Naugatuck Chemical Company entitled "The Process for Manufacturing Plastic Compositions and Products Obtained Thereby." This describes very early work with styrenes and rubbers. This patent and earlier patents describe the products that could be obtained by different polymerization processes, say at 100°C and 200°C, and states that the lower temperature had the better resins and, although polystyrene was obtained more quickly at the higher temperature, it had inferior properties, especially brittleness.

The first commercial production of styrene monomer was undertaken in about 1925 by the Naugatuck Chemical Company, but this was continued only for a short time. The I. J. Farben Industry in Germany had started a high commercial and successful operation not only in monomer but in polymer, and this resulted in the early 30s of having almost 6,000 tons a month produced by alkylation with aluminum chloride, purification followed by distillization, and then dehydrogenation and purification by distillation of the resulting monomer. This is very well discussed in *German Plastics Practices* by DeBell, Gloggins, and Glore (a 1946 publication of DeBell and Richardson) and is also reviewed in Chapter 19, A Survey of Styrene and Polystyrene in Germany, in *Styrene, Its Polymers, Copolymers and Derivatives* by Boyer and Boundy.

Staudinger (an American chemist) contributed significantly to fundamental polymer research in his studies of styrene monomers and the linkings through the vinyl chains. He is thought to have named the prod-

# Chain reaction "polystyrene"

Tel. 612-2111 3

## 1.1 HISTORY

uct of this chain reaction "polystyrene," and the basic chemistry of the vinyls and styrene particularly is a subject of a great amount of work through the 30s and 40s by Staudinger and his students.

If anything derogatory could be said of the materials produced in the early 20s and 30s, it was their tendency to crazing or cracking. "After a week or two of commercial use, they had the appearance of shattering into tiny fragments with only little pressure, and they would so shatter if pressured by hand." Mechanically, therefore, these materials were almost useless. This particular quotation is from Teach and Kisling's book on polystyrene (Reinhold Publishing Company, New York, 1960), page 5.

In 1937 the Dow Chemical Company introduced a domestic polystyrene called Styron. This utilized the catalytic pyrolysis process but, with the developments of inhibiting and distillation founded by others, they had made it commercially feasible. Monsanto Chemical Company and the Bakelite Company also soon followed, supplying polystyrene to the plastic trade. However, in 1938 only 200,000 pounds of polystyrene was produced.

In Germany, however, in the 30s, quite successfully, at least six or seven varieties of a material called Trolitul were being offered to the trade. These had a variety of molecular weights and, in some instances, copolymers with acrylates.

Early polymerization techniques disclosed the continuous tower process but very often fed by prepolymerization reactors in units of two which were used alternately to feed the towers their 33% polystyrene, 67% monomer. The towers then were operated at a series of increasing temperatures, and the polymer was then extruded on a cooling roll and was devolatilized.

In this country Dow found that they could control the heat of the reaction by placing the styrene monomer in a can usually rather larger than a gallon in size and immersing this material in successively hotter and hotter water until the polymerization was complete. Poor quality in final product was the result until a degree of sophistication in the polymerization of styrene was accomplished in that all the monomer was either polymerized or was completely removed from the polymer that resulted. With the advent of emulsion polymerization and suspension polymerization and the ability to control the polymerization reaction by the mass of water that surrounded the small droplets of styrene, better products started to result from these polymerizations. At the same time, the polymerizations in the tower process were greatly improved so that, beginning with approximately the years 1942 or 1943, this material has had a phenomenal growth, although progress in polymerization of polystyrene was hindered as a commercial entity by the war.

However, the greatest impetus that polystyrene received was from

the demands for synthetic rubber brought about by World War II. A group of companies consisting of Carbide, Koppers, Monsanto, and Dow pooled all their resources to manufacture the 200 million pounds per year of monomer that were required in 1944 for the synthetic rubber program. This program was so effective that, by the end of the war, GRS represented 86% of the total production of synthetic rubber and this was probably primarily a mixture of 75% butadiene and 25% styrene. The historical development of styrene is summarized in Table 1.1.

**Table 1.1** HISTORICAL HIGHLIGHTS OF STYRENE DEVELOPMENT

Year	Who	What	References
B.C.	Unknown	Storax gum	
1831	Boastre	Isolation styrene monomer	<i>J Pharm Chim</i> 17:338
1839	E. Simon	Polymerization to "Styroloxide"	<i>Annalen</i> 31:265-277
1837-44	D'Arcet; Mulder; Gerhardt; Glenard & Boudault	Hot tube polymerizations	Various
1845	Hoffman & Blyth	Sealed glass tubes	<i>Annalen</i> 53:314
1900	Kronstein	Closed vessel	German Patent 17,378
1911	Matthews	Thermal & catalytic poly for commercial products	British Patent 16,277
1914	Stobbe; Posnjak	Viscosity measurements	<i>Ber.</i> 47:2703
1922	Moureaux; Defraisse	Inhibitors	<i>Com. Rend.</i> 174:259
1911-30	Ostromislensky *	Mechanism	<i>J Russ Phy Chem Soc</i> 44:204
1925	Naugatuck	Commercial product monomer	
1940	I. G. Farben Industries	Commercial product monomer 600 tons/month	<i>German Plastics Practices</i> DeBell
1930-40	Staudinger	Chain-reaction mechanism	
1937	Dow	U.S. Polystyrene Styron	Dow Publication
1938	War Production Board	Styrene monomer for synthetic rubber	U.S. Government publication

\* U.S. Pat. 1,541,175-6; 1,613,673; 1,683,401-2,3,4,5.

It was stated earlier that only 200,000 pounds of polystyrene were made in 1938, whereas, by the end of World War II, the polystyrene production was approaching 50 million pounds, an increase of approximately 250 times. By 1961 this had reached approximately one billion pounds, and by 1966 the production of polystyrene and its varieties was over two billion pounds (Table 1.2).

**Table 1.2 POLYSTYRENE PRODUCTION IN THE UNITED STATES**

Year	Type or Maker	Form	Lb/Yr
1938	Monsanto, Dow Union Carbide	Crystal	200,000
1946	Crystal and blend impact		50,000,000
1961	Various types and makers	Crystal, graft Impact blends ABS	1,000,000,000
1966	Various types and makers		2,000,000,000
1969	ABS, Impact, Crystal		3,000,000,000

In the monomer area, it must be stated that most of the ethyl benzene was manufactured by an aluminum chloride alkylation process of benzene and ethylene which was started by Friedel-Crafts in 1884 and carried forward by Balsohn in 1879, Radziewanowski in 1894, Reid et al. from 1923 to 1927, and Natelson in 1933. The practice of alkylation by aluminum chloride was practiced until the late 1950s, when alkylation by  $\text{BF}_3$  received some push by a process sold by the Universal Oil Products Company. The consumption of catalyst in these  $\text{BF}_3$  alkylations was much higher than had been expected, and this process did not receive the favor that had been hoped.

More recently, Zeolite alkylation catalyst and improved aluminum chloride, ethyl chloride promoter systems have been receiving a lot of attention because of the clarity of the product from the alkylation reactor and the selectivity and yields of this process.

The cracking of the ethylbenzene to the styrene in the processes that were developed in the 30s and 40s, using iron-promoted catalyst with a small amount of potassium, gave conversions per pass of approximately 35% to 40%. More recently, these conversions have been upped considerably to the area of 80% to 90% with catalysts that have much longer life and require less regeneration. Most of this operational know-how is available in the Badger, Union Carbide, and Cosden process which has recently licensed eight of the newer units that have been installed throughout the world.

Cosden must also be mentioned for its extremely foresighted action in a distillation system which distilled out of  $\text{C}_8$  hydrocarbons the ethyl-



benzene present, using approximately a 300-plate tower. Now approximately 10% of the ethylbenzene used in the industry is directly distilled from petroleum.

In the fields of polymerization, some remarkable progress has been shown in the suspension system for the manufacturing of polystyrene, impact polystyrene, and certain grades of ABS. The work of Monsanto, patented by Stein and Waters for the suspension polymerization of styrenes and rubbers in the 1958 to 1959 era is typical of this. Also, along this line, the work of Amos, McCurdy and MacIntyre of Dow, the so-called stirring patent, has indicated that it is necessary to stir the prepolymer solution during the initial phases of polymerization in order to guarantee the quality of the impact material for both its appearance and physical properties.

The work of the acrylonitrile-butadiene-styrene tripolymers by Marbon in the emulsion area and the emulsion polyblending techniques of similar materials practiced by Monsanto and the very early progressive and stepwise work of U. S. Rubber in the ABS field should also receive rapt attention for their ever improving properties of engineering materials that have resulted from improved techniques.

Continuing research in new monomers and polymerization of these monomers with the old tried-and-true styrenes, using ever improving systems, makes it difficult to predict what next step on these advancing fronts will open up new areas of application and what will be the final use of the ever expanding types of material that grow out of our experiments of today.

Although the methylstyrenes made by American Cyanamid from the diaryl ethane process initially started by Dominion Power were not successful commercially, the area at which they are aimed, namely higher-temperature resistant polymers, still is an attractive and an elusive area for today's research. Certainly materials like paracyanostyrene, when turned into polymers with its heat-distortion temperature of 130°C, are much better than the commercial products that we have today. The chloride derivatives of styrene, although commercially exploited earlier, also should be given a second look, not only for their higher heat distortions, but also because of the fact that they are flame-resistant and fire-proof materials which are improved over those existing in the market today.

With today's knowledge of how to control molecular weight, what changing molecular weight and structure mean in physical properties, and with new techniques of controlling polymerization reactions and with new processing equipment to handle polymers that are more difficult to form, it is most apparent that we are standing on a threshold of a plastic age.



Recent statistics indicate that plastics are approximately  $\frac{1}{8}$  the production of steel and with steel having an advantage in density of approximately seven times, it is fairly readily apparent, on a cubic-inch or cubic-foot basis, that plastic is coming close to steel. It is predicted by 1975 that plastics in general, being figured on a ton basis, might out-produce steel. By 1980, they may outproduce all of the ferrous and non-ferrous metals. With the alloying potential of all of the plastics, barely touched by 1967, it would not be surprising to see plastics continue their accelerated growth, which in certain instances may be better than in the past five to ten years.

## 1.2 POLYMERIZATION TECHNIQUES

Most of the early works of Ostromislensky, Lemoine, and Kronstein were done with mass polymerization techniques, primarily by heat alone.

With the discovery that peroxides could also initiate radical polymerization, the field opened up considerably in the formation of polystyrene. In the mid 30s, a tremendous amount of work was started in many of the nations on emulsion polymerization, using a variety of techniques which allowed one to make synthetic rubber which by 1938 was approaching the best natural material in properties and wear. It was inevitable with this tremendous push brought on by World War II that other techniques for the polymerization of polymers and especially polystyrene would be developed.

As the size of the mass reactions grew, it was obvious to almost everyone that it was impractical to produce large amounts of polystyrene in any single unit. The first U.S. commercial application was the so-called can process of Dow Chemical Company where the polymerization was carried out in a series of baths increasing in temperature from about 70°C to 170°C. Cans were successively moved to a higher and higher temperature. After the polymerization was complete, the tin was stripped from the polystyrene block, and this was crushed, devolatilized by being heated, and passed under a vacuum to remove the residual low volatile materials.

In the late 30s and early 40s, the Germans practiced a polymerization technique which advanced the polymerization in rather large stirred vessels to approximately 35% polymer. Then the materials were run down through towers which had heating and tremendous cooling capacities built into them. The very viscous liquid materials were run off into a movable belt which passed them under heat and vacuum to remove the volatiles of this product line. Each of the two mentioned processes obvi-

ously implied that the greatest deterrent to the polymerization was the removal of heat from the mass product. Since the emulsion polymerization techniques did not suffer from this particular difficulty, because of the water available to remove excess heat, immediately attempts were made to polymerize styrene in workable formulations by modification of the rubber techniques that were then available. However polystyrene made in this way had a very high molecular weight and did not prove to be usable in the machines or the applications that were then being tried out for the infant polystyrene business.

Persistence, however, along these lines finally developed suspension polymerization and one of the early publications of this work is in *Indian Rubber World* where, by using finely divided tricalcium phosphate, Hohenstein was able to produce a bead or sugar polymer which had excellent molding characteristics. In suspension, the prime problem that exists in mass polymerization, that of heat removal, was not encountered. When the suspension system was also coupled with the use of a peroxide, the polymerization could be run below the boiling point of water. This made for much easier operation than had been the practice in the mass polymerization technique, which used cans or a continuous tower in combination with kettles for polymerization.

An early mass polymerization technique passed the prepolymerized polymer into a tray or press where the platens served to cool the reactants. As long as a large area per unit volume was built into these press processes, some fair control was experienced. However, the press reactors as they were run through the 40s and early 50s, because of local overheating, left much to be desired from a safety viewpoint and also for the preparation of the best types of materials.

As techniques improved, the mass polymerization and the suspension polymerization proved to be the most economically attractive methods of preparing good polystyrenes. Along with the improvements in the polymerization system, came improvements in the feed mechanisms and the ability to degas or devolatilize many materials that were high in volatility. The advent of the vented extruder helped greatly in this area.

Along with the development of the techniques of polymerization for polystyrene which gave good and acceptable commercial types of material, also came many advances in the understanding of the actual mechanism of the reactions and what one must do in the polymerization in reference to time, temperature, and other variables to make the most acceptable products.

A great deal of time and effort through the 40s and early 50s went into the study of the physical properties of the polymers and the various techniques for the manufacturing of these polymers. It was early evident that the crystal material, because of its shattering nature, had to be im-

proved in impact qualities and many attempts were made at polymerization of styrene with rubber and of mixing styrene with various rubbery materials.

Table 1.3 gives the processes and production of polystyrene recently in the United States as indicated by a number of sources.

**Table 1.3** POLYSTYRENE STATISTICS FOR 1969, MILLION POUNDS PER YEAR

<i>Producer</i>	<i>Process *</i>	<i>Crystal</i>	<i>Impact</i>	<i>Foamable &amp; Board</i>
Dow	BC	350	330	30
Monsanto	S,FP,e&g	200	200	
Bakelite	BC	90	110	
Foster Grant	S	80	130	50
Rexall (Dart)	S,BCb	60	80	50
Cosden Petroleum	S		160	
Hicksville	S	10		
Hammond (Gordon)	FP,g	20	20†	
Ticonderoga (Amoco)	FP,g	20		
Brand (Amoco)	BC	20	60	
Standard of Indiana (Amoco)				
Richardson	new reactors	30		
Haskell N.V. Macromol				
Dart		30	30	
Shell	BC	50	20	
Solar	FP,e	50	20	
Koppers	S	100		100
United Cork (New BASF)				85
Miscellaneous		15	15	
Totals		1,075	1,215	315
U.S. Steel, announced for 1970-71		100	100	

SOURCES: *Chem. Week* Feb. 10, 1962, p. 69; Jan. 7, 1961, p. 31; *Mod. Plastics*, Oct. 1960, p. 212; *Chem. Marketing, Res. Co.*, 1961 Statistics; DeBell & Richardson, *Consolidated Search Styrene Resins*; "Polystyrene Today & Tomorrow," *Plastic World*, Oct. 1968.

\* S = Suspension; FP = Filter Press; BC = Bulk Continuous; e = extruded; g = ground; b = batch.

† This was low grade.

### 1.3 TYPES OF MATERIALS

As the sophistication of the end user developed and the knowledge acquired by the material manufacturers became clear, it was obvious that no one type of material would fill the needs of all of the polystyrene uses of the early 50s. It was also apparent that the crystal clear nature of polystyrene was an immediate advantage but that the shattering or brittleness of the material was one of its major disadvantages (Tables 1.4 and 1.5).

**Table 1.4** POLYSTYRENE END USE, 1966

Million Pounds

Packaging	340
Refrigeration	74
Major appliances	22
Small appliances	15
Radio and television	54
Housewares	150
Lighting and signs	42
Toys	168
Combs, brushes	32
Pipe	22
Monofilament	5
Miscellaneous (including export)	429
Total	1,365

SOURCE: *Chemical Week*, March 18, 1967**Table 1.5** ESTIMATED MARKETS AND PROCESSES FOR POLYSTYRENE, 1965-1980

Markets—millions lbs/yr	1965	1970	1975	1980
Building	250	320	390	505
Transportation	16	25	35	45
Packaging	340	745	1,270	1,950
Electrical	95	135	190	275
Housewares	180	440	690	1,050
Furniture	40	140	380	900
Appliances	160	280	510	750
Process				
Powder	2	31	75	150
Compression transfer	None	None	None	None
Coating	180	350	540	770
Calendering	None	None	None	None
Wire insulation extruded	None	None	None	None
Blown container extruded	180	380	680	1,000
Film-sheet extruded	320	810	1,250	1,800
Injection	1,150	1,740	3,100	4,500
Totals	2,000	3,400	5,000	7,500

SOURCE: Chart in recent *Plastic World*.

Immediately blends of polystyrene and rubber began to appear on the market in the late 40s and early 50s, and, although these materials left much to be desired in the actual blending, they were immediately accepted by the trade as impact materials. As their use grew, so did the knowledge of polyblending techniques. It was not long before it was immediately obvious that, if a person wanted to mix materials to the best advantage, he should somehow mix them at a point where their viscosities were somewhat equal. This immediately started people running vis-