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Publisher *Taylor & Francis*

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Journal of Macromolecular Science, Part A

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713597274>

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Raymond B. Seymour^a

^a Department of Polymer Science, University of Southern Mississippi Hattiesburg, Mississippi

To cite this Article Seymour, Raymond B.(1989) 'Polymer Science Before and After 1899: Notable Developments During the Lifetime of Maurits Dekker', *Journal of Macromolecular Science, Part A*, 26: 8, 1023 – 1032

To link to this Article: DOI: 10.1080/00222338908052032

URL: <http://dx.doi.org/10.1080/00222338908052032>

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POLYMER SCIENCE BEFORE AND AFTER 1899: NOTABLE DEVELOPMENTS DURING THE LIFETIME OF MAURITS DEKKER

RAYMOND B. SEYMOUR

Department of Polymer Science
University of Southern Mississippi
Hattiesburg, Mississippi 39406-0076

Several books have been published on the history of Polymer Science [1-5], and most of the post-1899 history parallels the lifetime of Dr. Maurits Dekker, who was born in Holland on March 18, 1899.

If one refers to the Tarbell's excellent book on *The History of Organic Chemistry in the U.S., 1875-1955* [6], he will note that Chapter 20 on polymerization and synthetic polymers is dated 1914-1939. The Tarbells state that the industrial plastic called Bakelite was produced by Leo Baekeland in 1907 [7] and that Glyptal coating resins were produced by GE in 1912 [8].

According to the Tarbells, "the spectacular rise of polymer chemistry, as a separate field, was due, in a large part, to the accomplishments of Wallace Carothers at Du Pont starting in 1928" [9].

Carothers was one of the first to classify polymer synthetic reactions into those produced by condensation (step reaction) polymerization and those produced by addition (chain reaction) polymerization [10]. These concepts and those of Hermann Staudinger provided the breakthrough that was essential for the development of modern polymer science.

Of course, polymers have been part of man's environment since the beginning of civilization and long before the birth of Maurits Dekker. As stated by T. Alfrey in Chapter 1 of Stahl's *Polymer Science Overview* [5], complex and sophisticated polymer technology was developed by prehistoric man. These developments included the utilization of fibers, adhesives, composites, coatings, and leather, and this pre-19th century technology was essentially the

legacy of prehistoric and earliest historic times. However, while pre-19th century civilization was dependent on polymers for its existence, virtually nothing was known about the chemistry of these essential materials.

Hence, it should come as no surprise to learn from C. Fisher's chapter on natural fibers in *History of Polymer Science and Technology* [3] that the Swiss lake dwellers cultivated flax and wove linen into fibers 10 000 years ago. The techniques used in weaving linen and other plant fibers, such as cotton, hemp, sisal, henequen, and coir, were used in 5000 B.C. and are still in use today, and while cotton continues to be the major natural fiber, almost 1 million tons of these other plant fibers are still used annually throughout the world. Garments from wool and the hair of many other domesticated animals were woven in Babylon (the Land of Wool) as early as 4000 B.C.

Silk, like animal hair, is a proteinaceous fiber. However, unlike wool and other animal hair, silk is exuded or spun by *Bombyx mori* silkworms by a process not unlike that used for the spinning of artificial fibers. Sericulture originated in China about 3000 B.C. The silk reel is said to have been invented by Hsi Ling-chi, the 14-year-old wife of the mythological emperor Huang-to. About 50 000 tons of silk is produced annually in the orient from silkworms, and there is a renewed interest in the products of specialized silk from other spiders, tussah, and fiber-spinning worms.

Starch, proteins, including animal blood, and natural rubber have been used for centuries as adhesives and are still in use today. Over 1.5 million tons of starch and hydrolyzed starch (dextrin) adhesives are used annually in the United States. However, the use of animal glue decreased to 65 000 tons in 1987.

The principal naturally occurring composite is wood, but the ancient Israelites produced composite brick of chopped straw and clay, and the ancient Mongolians made bows by laminating wool, tendons, and silk. Concrete, which is a composite of sand and portland cement, is used today in large quantities, but the technological emphasis is on resinous composites.

As reviewed by R. Myers in *History of Polymer Science and Technology* [3], the prehistoric paintings in caves, such as those in Altamira and Lascaux, were based on aqueous dispersions of earth pigments and binders, such as egg white. These crude coatings were superseded by oleoresinous paints based on linseed oil, which cured (crosslinked) on exposure to air. The oleoresinous paints were the major coatings in the 19th century and are still used to a small extent today. However, because of the need to reduce the amount of solvents in the atmosphere, the emphasis in this \$12 billion (10^9) industry is now on water-borne and high-solids coatings.

The art of leather production antedates our knowledge of science. Leather,

which is one of the oldest commodities known to man, was originally produced by immersion of hides in aqueous solutions of tannins. Hides from over 35 million cattle and sheep are used annually to produce leather today. However, vegetable tanning has been replaced to a large extent by chrome tanning via basic chrome sulfate ($\text{Cr}(\text{OH})\text{SO}_4$), and many synthetic films are being used in place of the natural product.

Natural rubber was used by the natives in the West Indies, Central America, and South America many centuries before this unique elastomer (*Hevea braziliensis*) was imported to Western Europe. However, because of a lack of knowledge of polymer science and technology, this unique material was not utilized to any great extent until 1770, when Joseph Priestley discovered that it was a better eraser of pencil marks than bread crumbs. Accordingly, he coined the term "rubber," which is still used today in English-language countries.

Many technological breakthroughs in the 19th century enhanced the utility of fibers and coatings, but little change occurred in the technology of adhesives and leather. The catalyst for the development of man-made fibers was cellulose nitrate which was produced by Braconnet and Schönbein in 1832 and 1846, respectively, and a solution of cellulose nitrate in ether and ethanol (collodion) was produced by Audemars in 1855 [11]. Filaments of cellulose nitrate were produced and patented by Audemars, Swan, du Vivier, and Chardonnet. Samples of Chardonnet's denitrated cellulose nitrate fibers (mother-in-law's silk) were displayed at the Paris Exhibition in 1889, i.e., the year that Mau Dekker was born.

Linoleum based on a fabric-reinforced oleoresinous system is a pioneer composite which was introduced in 1860. Wilson and Green patented Collodion or Pyroxylyene solutions in 1884. This cellulose nitrate solution was used to protect injured skin (Nu Skin) and to form photographic film (Archer type) and coatings for billiard balls [12].

In spite of the unprecedented technological advances in man-made fibers and coatings, the most significant advances in polymer technology in the 19th century were the invention of celluloid and the vulcanization of rubber. The invention of celluloid by the Hyatt brothers was a technical, economic, and social activity. Both Parkes in England and the Hyatts in the United States, developed semiflexible cellulose nitrate products in the 1860s. They used the trade names of Parkesine and Celluloid to describe their plastic products [13]. Both D. Spill and the Hyatt brothers used camphor as a plasticizer for cellulose nitrate, and Hyatt and Spill were granted U.S. Patents 91,341 and 91,377 in 1867 for similar plasticized cellulose nitrate compositions.

Hyatt's Celluloid soon displaced shellac, gutta percha, and horn as plastic

materials. Tortoise shell and horn are not used much as plastics today, but the name "horner" is still used in England to describe a plastic technician [14]. As a result of the development of celluloid, Leominster, Massachusetts, became the center for the invention of plastic fabrication machinery and for the molding of Celluloid products. This phase of United States plastics history has been commemorated by the establishment of a museum of plastics at Leominster.

The contribution of Celluloid to the development of the American plastic industry was paralleled by the vulcanization process for natural rubber, which was discovered by Charles Goodyear. M. Faraday showed in 1825 that rubber had the empirical formula C_5H_8 , G. Williams obtained isoprene by the pyrolysis of rubber in 1860, and MacIntosh patented waterproof textiles (MacIntosh) by coating fabrics with solutions of rubber in naphtha (dope) in 1823, but this rubber retained its stickiness until the discovery of vulcanization with sulfur by Goodyear. Nelson Goodyear produced hard rubber (ebonite) by heating rubber with large quantities of sulfur.

Goodyear's vulcanization (crosslinking) process and the invention of the pneumatic tire by R. Thompson in 1845 resulted in an increased demand for rubber, which could not be met by the decreasing supply of wild rubber from Brazil. The cultivation of *Hevea brasiliensis* trees by Ridley was based on seeds smuggled from Brazil by H. Wickham. J. Dunlop reinvented the pneumatic tire in 1888, and these tires were used on the newly invented automobiles in France in the year 1889 [15]. In spite of the pre-1899 developments in polymers already cited, the major breakthroughs were delayed until the early years of the 20th century because of a lack of knowledge of polymer science.

It is well known that the use of many natural products, such as dyes and medicinals, stimulated the development of modern organic chemistry in the 19th century, but because of misunderstandings, these developments were not paralleled in polymer science. Unfortunately, the great organic chemists of the late 19th century, such as Baeyer (1877), ter Meer (1874), Fabrinyl (1878), Claus (1886), Kleeberg (1891), and Menasse (1894), who investigated the reaction of phenol and formaldehyde, abandoned this research in favor of crystalline products which could be readily purified and characterized.

The progress of these and other investigators was hampered by misinterpretations of the structure of these amorphous materials. In 1871, Hlasiwetz and Habermann suggested that keratin and cellulose were macromolecules [16], and in 1888 Brown and Morris reported a molecular weight of 30 000 for amyloextrin [17]. Nevertheless, the significance of these investigations was not recognized by most organic chemists who felt more at ease with the

erroneous association theory [18], and this misinterpretation delayed the development of polymer science. Most 19th-century chemists accepted T. Graham's concept that noncrystalline substances, such as starch, amylopectin, and albumen, were colloidal or glue-like, i.e., aggregates of smaller molecules [19]. This erroneous interpretation was based, in part, on the inability of the aqueous dispersions of these substances to diffuse through membranes, as was the case with solutions of crystalloid substances.

This state of confusion was enhanced by C. Nageli who maintained that materials such as starch and cellulose consisted of micelles which behaved as independent units like small crystals [20]. These pre-1899 concepts were supported by chemistry leaders such as Bergmann, Abderhalden, Freundlich, Moore, Noyes, Ostwald, Olby, Pauli, Pringsheim, and Weber. Even O. Rohn, who spent his entire life investigating acrylic polymers, described them as allotropic alterations rather than as macromolecules [21].

The progress toward an acceptable macromolecular science was further delayed by a misinterpretation of the x-ray crystallography of polymers. The pioneers in this new field maintained that a molecule must be no larger than the unit cell of the crystal. This concept was later proved to be erroneous in 1924 by I. Katz and H. Mark (1895-) [22].

In 1924, Mark, who was an expert x-ray spectroscopist, observed the x-ray diffraction pictures of a stretched rubber band which Katz had facetiously placed in an x-ray unit along with several samples of crystalline fibers [23]. H. Staudinger (1881-1965) also maintained that crystallography was not a suitable technique for the determination of the chain length of polymers [24]. Nevertheless, he was able to obtain sharp x-ray patterns for polyoxymethylene, polyethylene, and polyoxypropylene.

Staudinger spent most of his lifetime in displacing the erroneous aggregate and micelle concepts by macromolecular science. He was awarded the Nobel Prize in Chemistry in 1953 in recognition of his investigations which he had started a half century previously. After diverting his attention from classical organic chemistry, Staudinger investigated natural rubber and synthetic methyl rubber (polydimethylbutadiene), which had been produced by Kondakow in 1900 [25] and was used, without carbon block reinforcement, for making tires for the German army in World War I. Both S. Pickles [26] and Staudinger showed that derivatives of rubber retained their characteristic properties and thus discredited the aggregate theory [27].

Pickles and Staudinger disagreed on the structure of the rubber molecule. Pickles erred in suggesting a cyclic structure, while Staudinger championed linear molecules. K. Meyer, H. Mark, von Susich, Valko, and several other pioneer polymer scientists observed that the specific gravity of a stretched

rubber band was 20% greater than that of the unstretched band and attributed this effect to lateral contraction which restricts free lateral movements of the chain segments in a reversible manner [28].

Further developments of the rubber elasticity by P. J. Flory [29], improved understanding of step reaction and chain reaction polymerization techniques developed by W. Carothers [30], and extensive research on polymers by C. Marvel [31] reinforced Staudingers concepts, which are the basis of modern polymer science. It is of interest to note that Marvel (1896-1988), Carothers (1896-1937), Flory (1910-1985), and Mark (1895-) can be considered contemporaries of Dekker (1899-).

The science of polymers caught up to plastics technology in the last of the post-1899 years. However, with rare exceptions, the growth of the industry was hampered to a significant extent by the lack of knowledge of polymer science. The expansion of the rubber industry paralleled that of the automobile industry prior to World War II. However, the science of the new synthetic elastomers enhanced the growth of this industry in post-World War II years [33].

During the years prior to 1899, most rubber (*Hevea brasiliensis*) was obtained from Brazil. Since the uprooting of wild rubber trees made rubber a nonrenewable resource and plantations of *Hevea brasiliensis* were susceptible to *Dothildella ulei* fungus, most post-1899 natural rubber was obtained from plantations in Indonesia, Sri Lanka, and Malaya. The annual consumption of 23 000 tons of rubber in 1889 grew to over 4 million tons worldwide in 1988, while cloning and the use of ethylene increased the yield per acre from less than 1 to over 2.5 tons.

Small amounts of Thiokol and neoprene synthetic rubbers were produced in 1927 and 1931, respectively, but significant quantities of synthetic rubber (SR) were not produced until the late 1930s. One hundred thousand tons of SR, a styrene-butadiene copolymer (SBR), was produced worldwide prior to World War II. This volume grew to more than 10 million tons in 1987, and this SR accounts for almost 75% of all elastomer products today [32].

The growth of the fiber industry is somewhat comparable to that of the rubber industry. Natural fibers were used exclusively in the pre-1899 years and of course, cotton was king. Man-made rayon accounted for 20% of cellulosic fibers in post-1899 years but, while the per capita consumption of fibers has increased annually in both developing and developed nations, the major growth since World War II has been in synthetic fibers, such as polyesters, nylon, and acrylics. The production of rayon and synthetic fibers worldwide was equal to the production of cotton in 1984. About 15 million tons of fibers were produced worldwide in 1987, and the volume of synthetic fibers exceeded that of natural fibers.

As stated by Carl Marvel, a contemporary of Maurits Dekker, a moderate amount of thermoset plastics was produced by the German and American plastics industry prior to World War II. The principal products were phenol-formaldehyde (PF) (Bakelite) and the amino plastics (urea-formaldehyde) (UF), and melamine-formaldehyde (MF) resins [34]. The growth of alkyds [38], phenolics, and amino resins was hampered by the advent of injection-moldable thermoplastics in the 1930s. United States production of these thermosets in 1987 was 2.1 million tons [35]. A faster growth can be observed for the unsaturated esters, which were introduced by C. Ellis in the 1940s, and the epoxy resins, which were introduced by Castans in the 1950s. The annual production of these newer thermosets in the United States, in thousands of tons in 1987, was 598 and 183, respectively.

The book published by the Society of the Plastics Industry (SPI) on the occasion of its 50th birthday [1] summarizes much of the progress of the thermoplastics industry in the United States during the past 50 years. It was my pleasure to meet Dr. Mau Dekker at the First National Plastics Exposition sponsored by SPI under the leadership of Bill Cruse in 1946.

The production of a few tons of polystyrene by Dow was a milestone in 1938. I joined Monsanto as a research group leader in polymer science in 1941, and much of my effort was directed toward the production of styrene, which resulted in a production facility at Texas City. Polystyrene was produced at Springfield, Massachusetts [36]. The annual production of polymers and copolymers of styrene was 2.77 million tons in 1987.

I was fortunate in patenting moldable copolymers of vinyl chloride (Plivoc) for Goodyear in 1937, but PVC had limited growth until it was plasticized by Waldo Semon (Koroseal) and German chemists developed techniques for processing rigid PVC [37]. United States production of polymers and copolymers of vinyl chloride in 1987 was 4.14 million tons.

The commercial production of the several different types of polyethylene and polypropylene developed during the first half of the 20th century would have been heralded as a miracle if these were not plastic materials [38]. As depicted in the *History of Polyolefins* [39], the first commercial polyethylene was a highly branched polymer produced at high pressure (LDPE). A small amount of this polymer was produced by ICI in a pilot plant on the very day in 1939 that Hitler's troops invaded Poland.

The original processes for the production of high-density polyethylene (HDPE) and polypropylene (PP) were developed by J. Hogan and R. Banks in the early 1950s. They were awarded the Perkin Medal in 1987, and K. Ziegler and G. Natta, who also pioneered the commercial production of HDPE and isotactic (crystalline) PP, were awarded the Nobel prize in 1963.

The Ziegler-Natta catalyst was used to produce a commercial ozone-resistant elastic copolymer of ethylene and propylene (EPDM), and HDPE was chain extended to produce a filament that is stronger than steel. In spite of the ubiquity of these types of polyolefins, another linear low-density polyethylene (LLDPE), which is produced at ordinary pressures, was developed in the late 1970s by chemists at Du Pont, Dow, and Union Carbide [40].

Advances in the polyolefin segment of the polymer industry have included the following: Coatings for coaxial cable (1940), blow-molded squeeze bottles (1943), kitchenware (1946), Hula-Hoops (1957), trash barrels (1957), blow-molded HDPE milk bottles (1960), T-shirt grocery bags (1929), blow-molded HDPE gasoline tanks (1987), and blow-molded oil containers (1988).

As a result of these many advances in polymer science and technology, polyolefins accounted for 45% of the 26 million tons of plastic produced in the United States in 1988. The 1988 production in millions of tons for the different polyethylenes was as follows:

HDPE	3.75
PP	3.30
LDPE	2.89
LLDPE	<u>1.60</u>
Total	11.54

With the exception of some fibers, all polymers require additives or reinforcement for maximum utility [41]. In spite of their pervasiveness, general-purpose plastics have limited use as structural materials. This deficiency was overcome by the development of heat-resistant strong thermoplastics, such as nylon, styrene-maleic anhydride terpolymers (Cadon), and acrylonitrile-butadiene-styrene terpolymers (Marbon) in the 1940s. Nylon continues to be the major engineering plastic, but many other advanced plastics are now performing useful functions on land, on sea, and in space [42].

Because of the entrepreneurship of pioneers, such as Baekeland, Beetle, Debell, Delmonte, Dow, Dreyfus, DuBois, Ellis, Hyatt, Hyde, Plunkett, Scribner, Semon, Tupper, and Wyeth; the scientific contributions of Carothers, Flory, Ehlers, Fox, Goldsworthy, Mark, Marvel, and Ott; the organizational ability of Brown, Cruse, Heckman, and Morrison; and the publication efforts of Breskin, Cleworth, and Dekker, the polymer industry has grown to be the world's second largest industry.

These efforts, coupled with educational efforts in polymer science, should be continued and enhanced so that the fact that "Polymers Are Everywhere" will be recognized by all consumers of these important materials [43].

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Received November 14, 1988