

Organic Coatings:
Their Origin and Development

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Organic Coatings: Their Origin and Development

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Foreword

After the development of the art of painting, and the use of lacquers and oleoresinous paints, many centuries past before the introduction of a host of synthetic coatings. The Monk, Theophilous, in his "De Diversis Artibus," summarized coatings science, prior to the 12th century. In a series of books on paints, edited in the 1940's, Dr. Mattiello also supplied some up to date information on the History of Coatings. Fortunately, a group of modern science historians presented reports at the ACS Symposium in order to update all aspects of the history of coatings.

The book on the origin and development of high performance polymers, which was coedited with Dr. Kirshenbaum, served as a valuable addition to our knowledge of history of that phase of polymer science. Hopefully, this treatise on the history of coatings will be as well received by those scientists who are applying the knowledge developed by macromolecular giants in previous years.

Preface

Since the drying of oleoresinous paints involved man's first controlled polymerization, Nobel Laureate Lord Todd's Statement, "I am inclined to think that the development of polymerization is perhaps the biggest thing chemistry has done," is an appropriate opening sentence for a preface on the History of Coatings.

The statement made by Nobel Laureate Hermann Staudinger's antagonists to support their polymer science incredulity viz, "we are shocked, like zoologists would be, if they were told somewhere in Africa was found an elephant which was 1500 feet long and 300 feet high," might also be applied to some more modern scientist who overlook history and coatings science. Yet, the 10 billion dollar US coatings industry, which is the nation's major consumer of organic chemicals, also contributes to the prevention of degradation of our modern stationary and mobile structures and to our artistic lifestyle.

Through its establishment of the Center For History of Chemistry, the American Chemical Society has demonstrated the need to recognize past contributions to science. The funds supplied by firms such as, Arco, Dow, du Pont, Exxon and GE for the establishment of this center have been augmented by a generous gift from Dr. Arnold Beckman.

Fortunately, this center chose the history of polymer chemistry as its first project. This book on the proceedings of the International Symposium on History of Coatings, is published in support of the center's first project.

The world history of painting techniques, which will be published in 1990 by the Federations of the Associations of Technicians from Continental Europe, in the field of paint, varnish, enamel and printing inks and The Coatings Handbook to be published in 1990 by Elsevier will also be in tune with the first project selected by the Center for History of Chemistry.

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The editors wish to thank the many authors who presented reports at the International Symposium on The History of Organic Coatings at the National Meeting of The American Chemical Society at Miami Beach, FL, September 11-14, 1989. The editors appreciate the assistance of Ms. Machell Haynes who aided in the organization of the symposium and the typing of many of the chapters in the book.

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Origin and Development of Polymeric Coatings

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Abstract

Prior to the mid 19th Century, the only polymer technology available was in the field of organic coatings. The art of decorating the walls of caves was practiced many thousand years ago. The early materials were aqueous dispersions of earth colors and resinous additives, such as crushed berries, eggs, milk or tree sap. Aqueous dispersions, such as distemper, and white wash were used for many centuries but were replaced, in part, by oleoresinous paints. The recipe for the production of these primitive paints was published by Theophilus in the 12th Century. While vegetable oil-modified paints, such as alkyls continue to be used, they were replaced, to large extent, by the introduction of cellulose nitrate lacquers in the 19th Century. While all of the various types of coatings continue to be used, the trend is toward water-borne and high solids coatings which meet environmental specifications.

Waterborne Coatings

As a result of regulations, which limit the amount of volatile organic solvents in coatings, there has been a renewed interest in water borne coatings, which were the earliest types of paints used by the caveman. He used earth colorants as pigments and crushed berries, animal blood, egg whites, and sap from dandelions, milkweed and trees as adhesives in these crude paints.

The paintings of the grand bison at Altamira, Spain and the Chinese horse at Lascaux, France are believed to be 15,000 years old. The Obiri rock paintings in Arnhem Land in Northern Australia also date back to prehistoric times. Some 5000 years ago, the Egyptians improved their coatings by adding other adhesives, such as casein and the term "distemper" from the Latin *temperare*, meaning to mix, was used to describe these water borne paints.

Pigments

The principal pigments, used by the Palaeolithic artisans were charred wood (black), chalk (white) and iron and manganese oxide for red and yellow colors. About 3 or 4 thousand years ago, the Egyptians supplemented these basic pigments with lapis lazuli (blue), azurite (green), ochres (red and yellow), orpiment (yellow), malachite (green), gypsum (white) and lamp black.

The Egyptians also developed vegetable colorants such as those from the madder root. The ancient Romans used red lead as a pigment in coatings but the first synthetic pigments were Egyptian blue and white lead (cerussa). White lead was made by Pliny by the reaction of vinegar on lead sheets in the atmosphere over 2500 years ago. Egyptian blue was produced by the calcination of a mixture of lime, alumina, silica, soda ash and copper oxide at least 10 thousand years ago.

Other classic white pigments were zinc oxide, zinc sulfide, lithopone and basic white lead sulfate which were introduced in 1770, 1783, 1847, and 1855, respectively. Titanium dioxide, which was produced from black ilmenite in 1924, became the major white pigment and is the world's most widely used pigment.

Ancient Binders

Shellac, which was erroneously called "Indian Amber" by Pliny is one of the few resins obtained from insects. This resin, is secreted by a coccid insect (*Laccifer lacca*) which feeds on the lac trees in India and Thailand. It was used for making lac sticks over 3000 years ago. These sticks were used to coat rotating objects on a lathe by pressing the lac stick which was softened by the friction developed during rotation.

Lac, which is the hard resinous secretion is dissolved in ethanol and the residue of the insects and twigs is removed by filtration. The color which ranges from light yellow to dark orange is dependent on the type of host tree used as a source of the shellac and the extent of refining. Since shellac is insoluble in aliphatic hydrocarbon solvent such as "mineral spirits" it continues to be used as a stain suppressant seeker under other solvent based coatings shellac is also used for coating pills, candy and fruit.

The word lacquer is derived from the word lac. However, the first true lacquer was Japan lacquer was obtained from the sap of a sumac tree (*Rhus Vernicera*) during the chow dynasty about 3000 years ago. The use of Japan lacquers was expanded and improved during the ming period (1362-1644). The resin which was obtained by thermal evaporation of the aqueous solution was mixed with pigments, and used as a high gloss coating. It was not uncommon to build a thick coating by the application of as many as 250 layers of lacquer.

Varnish based on solutions of amber were used as early as 250 BC but the formations were not until a monk named Theophilus described the production of an oil varnish by dissolving resin in hot oil. Amber was one of the resins used at that time. The name varnish is derived from vernix which is the latin word for amber. Subsequently, natural resins were obtained from trees in tropical regions. The resins were classified as ancient or fossil, semi-fossil and recent, depending on when they were separated from the trees. These resins were also named after their geographic source, such as Manila, Batu Dammer, Congo, and Kauri.

Oleoresinous Paints

The term paint is usually applied to a coating consisting of pigments dispersed in a drying oil such as linseed oil but through misuse, this term has been expanded to include many other coatings, including water based coatings. There is some evidence that drying oils were used in ancient Egypt and more definite reports that linseed oil was used for coating in the 4th Century A.D.

Linseed oil, which is obtained from the seeds of flax (*Linum usitatissimum*) was the first vegetable oil binder for making paints. This oil is a glyceryl ester of unsaturated acids, such as linoleic and oleic acids. Linseed oil forms a film when exposed to the atmosphere and the rate of this "hardening" is accelerated when white lead is mixed with the oil. It has been suggested that a trace of free acid in the oil reacts with the lead salts to produce an oil soluble salt which is called a drier or siccative. Detailed recipes for producing oleoresinous paints were supplied by Theophilus.

Presbyter Theophilus, who was also called Rugerus, in cooperation with another monk, called Roger, wrote "De Diversis Artibus" which described painting and other practical arts used in church decoration. Neither of the coauthors showed any scientific talent but they did record unpublished recipes which had been used for centuries.

Linseed oil continues to account for more than 50 percent of the drying oils used in paints but its share of the market which was over 90 percent in 1900 has been decreased by the use of soybean oil, tung oil, dehydrated castor oil, oiticica oil and fish oil. The hardening (drying) of these glyceryl esters in air is dependent on the extent of unsaturation and is accelerated by the addition of driers.

Driers

Lead salts of organic acids, which were the original driers, function as polymerization catalysts throughout the entire film depth. In contrast, cobalt salts function as surface driers. Hence, until recently, it has been the practice to use combinations of lead and cobalt driers in order to achieve a uniform drying rate. Manganese and zirconium salts may be used in place of lead. Many organic acids have been used to form these heavy metal salts but the most widely used salts are naphthenates which are based on naphthenic acid which is a residue of petroleum resinous. Other acids used for the production of driers are oiticica, tallic, rosin and linolenic acids.

Drying of Paints

It has been recognized for many centuries, that film formation from oleoresinous paints, was dependent on atmospheric exposure of the unsaturated oils. It is now recognized that crosslinking between the polymer chains occur and that an insoluble polymer network is produced after the absorption of oxygen in this "drying" reaction.

The mechanisms in the drying of nonconjugated acids involves the formation of hydroperoxide groups on the allylic carbons, i.e., carbon atoms of the methylene groups adjacent to the double bonds. Polymerization occurs via a radical chain mechanism, called autoxidation. This theory also applies to conjugated acids, such as linolenates, but the "drying reaction" is much faster with these polyunsaturated oils.

Cellulose Nitrate (Pyroxylenes)

Solutions of shellac in ethanol and sumac sap were used as coatings centuries ago but the first lacquer from a manmade polymer was not available until the middle of the 19th Century. Braconnet nitrated starch in 1833 and Pelouze nitrated cellulose 5 years later. The names of xyloiding and pyroxylenes were used by the inventors to describe their products.

In 1846, Schönbein improved the nitration process by using a mixture of nitric and sulfuric acids and patented this cellulose trinitrate, which he called guncotton. Schönbein observed that his guncotton was soluble in a 50:50 mixtures of ethanol and ethyl ether but credit is usually given to Maynard, who announced the availability of collodion as a waterproof coating for wounds in 1847. In 1882, Stephens used amyl acetate as a solvent for cellulose nitrate and Wilson and Green patented pyroxylenes coatings for carriages and automobiles in 1884.

The availability of huge stockpiles of surplus guncotton, after World War I, catalyzed peacetime uses for this polymer. In 1923, cellulose nitrate (CN) lacquers, erroneously called nitrocellulose lacquers, were used as automobile finishes, under the trade name of Duco. After the introduction of competitive resins for automotive coatings, catalyzed lacquer and a multicolored CN were developed in the 1950's. "Super lacquers based on cellulose nitrate-isocyanate resins were introduced in the 1960's when CN continued to hold second place, next to alkyds, among the industrial finishes. Alkyd-CN blends and CN-blends with copolymers of vinyl chloride and vinyl acetate are also used as commercial

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coatings. Additional information of the history of cellulosic coatings is provided in subsequent chapters by Dr. Fisher.

Alkyds

The first polyester resin was produced by Berzelius by the condensation of glyceryl tartrate in 1847. W. Smith made Glyptal coatings in 1901 by the controlled esterification of glycerol by phthalic anhydride. This technology was improved by Friedburg a few years later and by Kienle in 1921.

Kienle recognized that the condensation of difunctional

reactants produced linear polymers and that trifunctional reactants produced crosslinked, infusible network polymers. Accordingly, he reacted ethylene glycol and phthalic anhydride, in the presence of drying oils and obtained linear polymers, which could undergo autoxidation polymerization, like that described for oleoresinous paints. He used parts of the words of the reactants alcohol and acid to coin the word alkyd. A profile on Kienle is provided in a subsequent chapter by Dr. Kauffman and an entire subsequent chapter is also devoted to the history of alkyds.

Kienle also classified his alkyd resins as short, medium, and long oil on the basis of their having an oil content of less than 40, less than 50 and over 50 percent oil, respectively. The Kienle patent was declared invalid by the U.S. Patent Office in 1935 but the name alkyd is still used to describe the major coating resin used worldwide.

Phenolic Resins

Phenolic resins, which were noncrystalline and lacked a precise melting point, were produced and discarded as undesirable "goos and gunks" by the leading organic chemists of the late 19th Century. As a result, many of the first and second generation students of these professors, avoided these and other polymers and concentrated their efforts on crystallizable and distillable compounds.

In 1872, Bayer condensed phenol with methylal and, at the suggestion of Fischer, Kleeberg repeated this experiment, using formaldehyde, instead of methylal in 1891. Smith patented phenolic resins for use as electrical insulators in 1899 and Swinburne produced these products commercially in 1904.

Baekeland, a visitor from Belgium, who decided to remain in the USA, was aware of the importance of functionality and of the mistakes made by his predecessors in their investigations of phenolic resins. Fortunately, he had been awarded enough money by Eastman for his Velox photographic paper patent to enable him to establish his own laboratory and to chose his research projects.

In 1907, he produced an ethanol-soluble novolac, which was used as a substitute for shellac, by the condensation of an excess of phenol with formaldehyde, under acid conditions. In 1908, he produced resole prepolymers by the condensation of phenol and formaldehyde, under mild alkaline conditions. These prepolymers called A-stage, could be converted into infusible C-stage insoluble castings or coatings, in the presence of strong acids. Nevertheless, neither the novolac or resole resins could be used as substitutes for natural resins in oleoresinous varnishes.

However, Albert heated the phenolic resin (PF) with a large excess of rosin and produced a product which could be used with tung oil to produce "four hour enamels." In

1928, Turkington patented oil soluble varnishes which were produced by the alkaline condensation of formaldehyde with para-substituted phenols, such as p-phenylphenol. Additional information on the history of phenolic coatings is provided in a subsequent chapter.

Amino Coatings

Tollens described resins produced by the condensation of urea and formaldehyde in 1884 and John patented this polymer. (UF) in 1918. In 1936, Henkel patented comparable resins (MF) based on melamine instead of urea. Both resins, which were described by the term amino resins, are insoluble in common solvents and are used as additives for curing other coatings. For example, the addition of MF reduces the curing time of alkyd resins by 50%. Soluble amino resins, which can be used as coatings, are produced when they are modified by etherification with butanol. Additional information on amino resins is provided in a subsequent chapter by Dr. Albrecht.

Vinyl Chloride Polymers and Related Resins

Regnault described polyvinyl chloride (PVC) in 1835, but since no solvents were available for this polymer, no PVC coatings were produced. In 1920, Reid of Union Carbide, and Voss and Dickhauser of I. G. Farbenindustrie filed for patents on a more soluble copolymer of vinyl chloride and vinyl acetate. This copolymer, which was produced, under the trade name of Vinyllite, was used for coating beer cans in 1936.

The versatility of this copolymer, copolymers of vinyl chloride and vinylidene chloride and PVC was increased, in 1944, by the development of plastisols by suspending a resin, produced by emulsion polymerization, in a liquid plasticizer and then fusing the mixture at elevated temperatures.

While polyvinyl alcohol (PVAL), obtained by the hydrolysis of polyvinyl acetate (PVAC) is insoluble in organic solvents, the acetal, produced by the condensation of butyraldehyde and PVAL is soluble and is used as a base for "wash Primers" or metal conditioners.

Polyvinyl acetate (PVAC) was patented by Klatte and Rollet in 1914. PVAC is used as an adhesive and the major resinous component of a widely used water borne coating. Additional information on the history of vinyl coatings is provided in a subsequent chapter.

Acrylic Esters

While acrylic acid was polymerized in 1847, its esters remained as laboratory curiosities until the 1900's when Rohm wrote his Ph.D dissertation on acrylic esters. He continued the investigation of these products for several decades. In 1956, General Motors Company replaced some of its cellulosic automotive finishes by acrylic coatings. Some of these were thermoset by the incorporation of melamine resins or by the introduction of additional functionality in the acrylic monomers. Additional information on acrylic latex coatings is provided in a subsequent chapter by Dr. Harren.

Silicones

Polysiloxane was synthesized by Kipping, in the early 1900's. Since he believed that these polymers were ketones, he called them silicones. While he was pessimistic about their commercial use, these water and temperature-resistant coatings were commercialized in the 1930's by Rochow of General Electric Company, McGregor of Union Carbide and Bass of

Dow Corning. Additional information on silicone coatings is provided in a subsequent chapter by Drs. Finzel and Plueddemann.

Epoxy Resins

Ethoxyline resins, which are now called epoxy resins, were patented by Schlack in 1939. Many of the prepolymers were versatile resins produced by the condensation of a diphenol (bisphenol A) and epichlorohydrin. Since these resins possess terminal oxirane (epoxy) groups, they can be crosslinked by reaction with polyamines at ordinary temperatures. These resins may be also be crosslinked by esterification of the pendant hydroxyl groups by cyclic anhydrides at elevated temperatures. Additional information on epoxy resin coatings is provided in a subsequent chapter by Dr. Reddy.

Polyurethanes

The original resins, which Bayer produced in 1937, by the condensation of aliphatic diisocyanates and diols were elastomers and foams. However, both one and two component polyurethane coatings are available. The two component systems are produced by the addition of aromatic or aliphatic diisocyanates to an ester or ether with terminal hydroxy groups.

The room temperature-one component systems contain unreacted terminal isocyanate groups, which react with moisture in the atmosphere to produce an amine which undergoes an additional crosslinking reaction. Baked polyurethane coatings are produced by heating phenol-capped isocyanates to remove the phenol and permit the isocyanate group to react with diols present in the coating system. Additional information on the history of polyurethane coatings provided in a subsequent chapter by Dr. Edwards.

Other Coatings and Profiles

As described in a subsequent chapter by Dr. Skolnik, rosin and other naval stores have been used for many years in coating. Actually, turpentine was one of the first non-alcoholic solvents used by paint artisans.

As outlined in a subsequent chapter by Dr. Deanin, the advancement of the science of coatings has been impeded by a lack of adequate educational facilities. Hopefully, his report will encourage other universities to provide courses in coatings science.

In addition to the profile by Dr. Kienle, provided by Dr. Kauffman, Drs. Fisher and Mattson have provided profiles on Drs. Doolittle, Mattiello and Long. Dr. Tarbell, who was the recipient of the Dexter award at the Miami Beach meeting, has published many joint reports on history of chemistry, with his wife. The profile on Dr. Long is also written by a husband and wife team.

The chapter on History of Plasticizers is written by Dr. Craver, who could be called "the father of plasticizers." He, like the Tarbells and Mattsons is also a member of an energetic husband and wife team. The chapter on the history of block copolymers is provided by Dr. Hsieh who is also a pioneer in this field. Likewise, the chapter on the history of polyvinylidene chloride coatings is provided by Dr. Gaylord who is another well known polymer science pioneer.

The chapter on membrane coatings was provided by Dr. Fritsch who is also recognized as a pioneer in his field. Other chapters on the history of polystyrene coatings, power coatings, diagnostic coatings, container coatings, automotive coatings, photo-cured

coatings and radiation-cured coatings have been provided by Drs. Stevens, Usmani, Robinson, and Wu, respectively.

Hopefully, these reports will prove the inaccuracy of the phrase, "ignorance in action" applied to coatings science by IUPAC.

Application of Coatings

Coatings were originally applied by hand but this technique was upgraded by the use of feathers and brushes. Brushing continues to be the most widely used technique for the application of paint. Rollers were used to apply paint in the 1940's and this technique is still used. Spray guns were developed in the 1920's and paints continued to be applied by high pressure spraying.

The spraying technique has been upgraded by hot spray application and by airless spray in which atomization occurs as a result of a pressure drop as the paint leaves the nozzle of the gun. More uniform thickness of coatings and higher efficiency are obtained when the paint is electrically charged or when the atomized spray passes through an electric field before it is deposited on the surface to be coated.

In the early 1970's, Brewer developed techniques for the electrodeposition of water borne coatings. In this technique, the resin particles carry positive or negative charges that are deposited by anionic or cathodic techniques. Coatings may also be deposited as powder which is then fused by heating and the deposited coatings may be cured by electron beam (EB) or ultraviolet radiation in the presence of a sensitizer, such as benzophenone. Information on powder coatings, electrodeposition, photocuring and electron beam curing is supplied in subsequent chapters by Drs. Gribble, Usmani, Hoyle and Kinstle, respectively.

Future Trends

The art of coatings, which is one of man's oldest arts, is gradually becoming transformed to a science. In spite of the application of many competitive materials, coatings continue to be used at an unprecedented rate for both decoration and protection. Over 1 billion (10^9) gallons of coatings are produced annually in the US at a selling price of \$10 billion (10^9). This volume will continue to increase and may increase dramatically as new resins and new application techniques are developed.

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