PIGMENTS AND DYESTUFFS

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Keywords: Chemical engineering, color, dyeing, organic pigments, inorganic pigments, organic dyes, inorganic dyes, production, engineering aspects and application of pigments and dyes, equipment.

Contents

Summary

Dyes and pigments are coloring substances, i.e. substances imparting a specific color to the substance (substrate) being colored. Dyes, among which organic compounds predominate, dissolve in and fuse or react with the substrate being dyed. Pigments are insoluble and they color the substrate by coating its surface. Finely ground inorganic substances predominate among pigments. Dyes are organic compounds, which contain in their molecules color imparting chromophoric groups and acid or basic auxochromic groups responsible for dyeing ability due to the auxochromes, dye molecules can be permanently bonded with fibers or other materials). Pigments are coloring substances, which do not enter into chemical reactions with polymers and are insoluble in them. Depending on their chemical and crystalline structure and particle sizes, pigments can cause the surface reflection or absorption of light. Chemical engineering aspects of pigments and dyes synthesis made essential part in development applied in chemical technology operations and constructions of specialist apparatuses. Besides their traditional use in textile, leather, paper, as well as the paint and varnish industries, dyes and pigments have become indispensable in other fields such as microelectronics, medical diagnostics and information recording techniques and they continue to be intensively developed.

1. Introduction

Coloring and the use of dyes and pigments for this purpose belong to the oldest human skills. Initially, natural substances obtained from plants and minerals were used. The development of industrial methods of manufacturing fabrics brought about a rapid increase in the demand for coloring substances. This was one of the main causes of the development of chemical technology, chemical engineering and organic chemistry, which quickly became the basis for dyes and the precursors used in their production. As a result, the production became independent of raw materials used for producing natural dyes. Today’s dyes are part of a major group of chemical products. Their importance has grown in almost every area of an economic activity. Besides traditional products, such as fibers and leather goods, dyes are currently indispensable for the production of newspapers and magazines, books, plastic products, decorative materials, films, food, everyday equipment, and other products in which color plays a significant role in satisfying human aesthetic needs.

1.1. History

The first dyes were *Turkey red* (produced from common madder) and *Indigo blue* (obtained from plants belonging to the family *indigofera*). A breakthrough event in the production of dyes was the chemical synthesis of *Aniline purple* carried out by William Henry Perkin in 1856. For the purposes of the synthesis, benzene was nitrated to nitrobenzene and then reduced to aniline, which was further oxidized. This process became the basis for the first industrial production of a synthetic dye. Another discovery based on the use of aniline as a raw material led to the synthesis of several triarylmethane dyes (*Magenta*, 1859). In 1862, Chrysaniline (an acridine dye) was synthesized by Nicholson. Diazotization allowed the production of azo dyes (*Bismarck brown*) in the 1870s. In the same period Grabe and Liebermann synthesized a derivative of alizarin (the main component of the natural dye present in *Turkey red*) while Adolf Bayer synthesized and determined the structure of *Indigo*, for which, among other things, he was awarded the Nobel Prize. In 1889, mordant dyes appeared on the market and in 1893 relatively inexpensive sulfur dyes began to be manufactured and used. At the turn of the 19th and 20th centuries the manufacture of dyes was already a significant field of economic activity, developing mainly in Germany, Switzerland, the United Kingdom, USA, and France. Several dozen companies (including Bayer, Hoechst, CIBA, BASF), which later contributed greatly to the development of the whole chemical industry, already operated in those countries. Competition between chemists in the search for new dyes in the second half of the 19th century and in the first half of the 20th century often led to accidental discoveries. Phthalocyanine dyes, the first of which was isolated as an impurity produced in the synthesis of phthalmide, were a major discovery of this kind. Also the beginning of the production of reactive dyes, capable to form a chemical bond with the fiber, belongs to the breakthrough events in the development of dye technologies. The main events in the development of the dye industry are shown in the table below (Table 1).

Year	Event	Ref.
4000 BC	Use of natural dyes: Turkey red, indigo blue	1
1856	First synthetic dye: aniline purple – azine dye (W.H. Perkin)	2
1857	First industrial production (Mauveine – UK)	2
1858	Diazotization reaction (P. Griess)	3
1859	Synthetic triphenylmethane dye: Fuchsine patented (Verguin, France)	3
1862	Chrysaniline – acridine dye synthesized (Nicholson)	4
1863	First azo dye: Bismarck Brown (C. Martius)	3
1868	Derivatives of alizarine (main component of Turkey red – anthraquinone dye) synthesized (C. Graebe, C. Liebermann, W. H. Perkin)	3
1876	Industrial production of alizarine	1
the last decade of 19th century	Indigo synthesis developed (A. Baeyer, T. Sandmayer, K. Heumann)	3
1879	Meldon blue – oxazine dye developed (Doebner)	3
1884	Tetrazine – yellow acid dye belonging to group of acid dyes (Ziegler)	5

1893	First sulfur dyes	6
1897	Industrial production of indigo (Germany)	3
1901	Polycyclic vat dye synthesized (Bohn)	1
1906	Thioindigo – red vat dye synthesized (Friedlander)	3
1915	Production of metal complex dyes of 1:1 type (Switzerland, Germany)	1
1923	Dispersed dyes (UK)	1
1929	Industrial production of copper phthalocyanine (UK)	1
1951	Metal complex dyes of 1:2 type (Switzerland)	1
1956	Reactive dye (UK, Germany, Switzerland)	2

1 Czajkowski, 2006
2 Christie, 2001
3 Gordon and Gregory, 1987
4 Gardner, 1915
5 Richter, 1939
6 Krasuski, 1904

Table 1. The historical development of dyes' production. [Czajkowski, 2006; Christie, 2001; Gordon and Gregory, 1987; Gardner, 1915; Richter, 1939; Krasuski, 1904].

1.2. Definitions

Coloring agents are substances able to impart color to substrates. Traditionally, coloring agents have been divided into soluble dyes and insoluble pigments. Pigments have stable physical and chemical properties during dyeing, whereas both natural and physical dyes are soluble in water or organic solvents. Dyes are classified according to their chemical constitution or depending on the methods and fields of their application. The chemical classification of dyes is primarily based on the presence of a chromophore, i.e. a chemical group which determines dyeing power. The principal dyes here are azo, anthraquinone, indigoid, arylmethane, and phthalocyanine dyes. A classification of dyes according to their practical use is shown in Table 2. Because of the large number of dyes used in the textile industry another classification of dyes and pigments is specific to this industry, which demands dyes in a wide range of fast colors and brilliant shades (harmless after dyeing) and that the dyeing process be simple and inexpensive and the dyed fabrics be resistant to: light, atmospheric changes, washing in a warm solution of soap and detergents, boiling, ironing, sweat, mechanical action, and the chemicals pre sent in domestic detergents.

Dye group name	**Description**
Acid (acid-chrome)	Dyes with built-in hydroxyl and carboxyl groups, showing power to form: stable complex compounds with metals and ionic and hydrophobic bonds; azo dyes used to dye protein fibers (wool, silk) and polyamide fibers
Basic (cationic)	Dyes with built-in amino groups, used in the form of hydrochlorides and oxalates; form ionic bonds; azo, diphenylmethane, triphenylmethane, thiazine, oxazine, azine, xanthene and acridine dyes; used to dye protein and synthetic (polyacrylic, polyamide and polyester) fibers

Direct	Dyes fixed without mordants; acting by forming hydrogen and vane der Waals bonds; azo and stilbene dyes for cellulose fibers
Mordant	Dyeing consists in prepadding of the substrate with metal salts and then applying dyes forming insoluble complexes with the metals; anthraquinone, nitroso, xanthene, oxazine, triphenylmethane and azo dyes
Vat	Dyes converted into a soluble form through chemical reduction to a leuco compound (vat) and after substrate saturation subjected to oxidation to the originally insoluble form; anthraquinone, indigoid, azine, acridine, indamine and thiazine dyes for cellulose and animal fibers
Ice	Dyes formed directly on the substrate in a process in which the coupling component is used to saturate the substrate while the diazo (2-dizanio) component in the bath at a temperature of ~0°C causes a component coupling reaction, forming insoluble dyes for plant fibers
Dispersed	Water insoluble dyes, used in the form of a suspension of highly dispergated particles penetrating into the substrate structure; azo, anthraquinone and nitroso dyes for synthetic (polyester, polyamide, acetate, acetate rayon) fibers
Reactive	Dyes containing reactive (chlorotriazine, epoxy, ethyleneimide) groups having the ability to form stable covalent bonds with the groups ($-NH_2$, $-OH$, $-SO_3H$) present in the substrate structure; for cellulose and protein fibers
Pigments	Insoluble dyes used during the throwing of fibers

Table 2. A classification of dyes according to their use

Kind of pigment	**Pigments**
Inorganic pigments, white	$\mathrm{Tio_2}$, zns, lithopone ($\mathrm{zns+baso_4}$), $\mathrm{baso_4}$, zno, $\mathrm{pbso_4}$, $\mathrm{2pbco_3{\cdot}pb(oh)_2}$, $\mathrm{zn_3(po_4)_2{\cdot}4h_2o}$
Inorganic pigments, black	$\mathrm{Fe_3o_4}$, carbon black,
Complex inorganic colored (cic), black	Spinel systems – $\mathrm{cucr_2o_4}$, $\mathrm{(co,fe)(fe,cr)_2o_4}$, $\mathrm{(fe,mn)(fe,mn)_2o_4}$, $\mathrm{fe_2tio_4}$, $\mathrm{(fe,co)fe_2o_4}$, $\mathrm{(ni,fe)(cr,fe)_2o_4}$, $\mathrm{(ni,mn)(cr,fe)_2o_4}$; hematite/corundum pigments – $\mathrm{(fe,cr)_2o_3}$,
Inorganic pigments, red	$\alpha\mathrm{-fe_2o_3}$; (cds+cdse), $\mathrm{pb_3o_4}$, hgs, $\mathrm{pb(cr,mo,s)o_4}$
Inorganic pigments, orange	$\mathrm{2pbo{\cdot}pbcro_4}$, ass
Complex inorganic colored (cic), orange	Rutile systems - $\mathrm{(ti,sb)o_2}$;
Inorganic pigments, yellow	α-feooh, γ-feooh, cds, ($\mathrm{cds+baso_4}$), $\mathrm{pbcro_4}$, pbo, $\mathrm{as_2s_3}$, $\mathrm{zncro_4}$, $\mathrm{bivo_4}$, $\mathrm{pbcn_2}$,
Complex inorganic colored (cic), yellow	Rutile systems – $\mathrm{(ti,cr,sb)o_2}$, $\mathrm{(ti,ni,sb)o_2}$; zircon pigments – $\mathrm{(zr,pr)sio_4}$; baddeleyite – $\mathrm{(zr,v)o_2}$, cassiterite – $\mathrm{(sn,v)o_2}$, pyrochlore – $\mathrm{pb_2sb_2o_7}$
Inorganic pigments, brown	$\gamma\mathrm{-fe_2o_3}$,
Complex inor-	Rutile systems - $\mathrm{(ti,cr,w)o_2}$, $\mathrm{(ti,cr,w)o_2}$; spinel systems –

ganic colored (cic), brown	$zn(fe,cr)_2o_4$,; $fe(fe,cr)_2o_4$, $(fe,mn)(fe,cr,mn)_2o_4$, $(zn,fe,mn)(fe,cr,mn)_2o_4$, $nife_2o_4$, $(zn,fe)fe_2o_4$, $(zn,mn)cr_2o_4$; hematite/corundum pigments – $(fe,cr)_2o_4$;
Complex inorganic colored (cic), grey	Rutile systems - $(ti,v,sb)o_2$; zircon pigments – $(zr,pr)sio_2$; perclase – $(co,ni)o$, cassiterite – $(sn,sb)o_2$,
Inorganic pigments, blue	$2cuco_3 \cdot cu(oh)_2$, $na_2al_2si_6o_{24}s_2$
Complex inorganic colored (cic), blue	Spinel systems – $coal_2o_4$, $(co,zn)al_2o_4$, $co(al,cr)al_2o_4$, co_2sno_4, $(zn,co)(cr,al)_2o_4$; phenacite – $(co,zn)_2sio_4$, olivine – co_2sio_4,
Inorganic pigments, green	$3cu(aso_2) \cdot cu(ch_3coo)_2$, $crpo_4$
Complex inorganic colored (cic), green	Spinel systems – $cocr_2o_4$, $(co,ni,zn)_2tio_4$; hematite/corundum pigments – cr_2o_3; garnet – $3cao:cr_2o_3:3sio_2$,
Complex inorganic colored (cic), pink	Spinel systems – $zn(al,cr)_2o_4$; zircon pigments – $(zr,fe)sio_4$; hematite/corundum pigments – $(mn,al)_2o_3$, $(cr,al)_2o_3$; sphene – $cao:sno_2:sio_2:cr_2o_3$,

Table 3. Types and classification of inorganic pigments. [Völz, 1995; Kirk and Othmer, 1950].

1.3. Notion of Color

Newton (1672) was the first to split sunlight by means of a prism, obtaining a spectrum of rainbow colors from red to violet. He showed that the sunlight is a mixture of monochromatic colors. Color is directly bound with the organs of the sense of sight, equipped with proper light sensation receptors, rods, and cones. The retina, converting luminous energy into current pulses (transmitted by the optic nerve) which stimulate brain activities producing visual sensations of color**,** plays a critical role. The functional and defect-free sense of sight enables complex and widely varied description of the perceived color sensations. The main color sensations include: shade – a qualitative feature of a color, dependent on the monochromatic light wavelength, color saturation – the fraction of a given color in the coloring, and lightness – the fraction of achromatic colors (from white to black). The range of the sensations is often made complete with such characteristics as: brightness, size, shape, structure, luster, transparency, depth and many others. Color sensations change depending on the illumination of an appraised object (a source of light), spectral characteristics of the reflected light and the observer’s relevant individual characteristics.

1.4. Physical Basis of Color Theory

Color is the property of electromagnetic radiation with a wavelength between 400 and 800 nm. A mixture of light waves (photons) with a wavelength of 400-800 nm and a vibration frequency of $0.75 \cdot 10^{15}$-$3.75 \cdot 10^{14}$ s^{-1} produces the sensation of white color (white light). The color of individual chemical substances derives from their interaction with white light (e.g. sunlight, quartz lamp light, or electric bulb light). Light beams can be shone through a substance (if it is transparent), reflected from it or completely absorbed. If the total radiation falling on a chemical compound passes through it or is reflected from it, the substance has white color. If white light on contact with a substance is completely absorbed, the latter appears to be black. Most often, however, chemical compounds selectively absorb certain radiation ranges and the color of a given compound is complementary to the absorbed radiation, i.e. it depends on the mixture of the waves which pass through the compound or are reflected from it (Table 4).

Wavelength Of Absorbed Mono-chromatic Light, Nm	Color Of Absorbed Light	Complementary Color
400	Violet	Green-Yellow
460	Blue	Yellow
520	Green	Purple
580	Yellow	Blue
640	Red	Blue-Green
720	Purple	Green

Table 4. Relationship between color and wavelength of monochromatic light.

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Biographical Sketches

Józef Hoffmann graduated from Wrocław University of Technology (WUT), the Department of Chemistry in 1978. In 1982 he finished his doctoral studies in the Institute of Inorganic Technology and Mineral Fertilizers, WUT. He defended his PhD dissertation entitled: “Waste-free Technology for Producing Feed Phosphates from Extraction Phosphoric Acid” and he obtained the Doctor of Technical Sciences degree. In 2002, he received habilitation on the basis of his monograph entitled: “New Directions in Urea Phosphate Utilization in Phosphorous Compounds Technology". Since 2004, he has been working as a WUT professor. Professor Józef Hoffmann's main research fields are: inorganic technology, particularly phosphoric acid production through the decomposition of phosphoric raw materials, extraction of phosphoric acid, purification techniques (the wet phosphoric acid process), methods of obtaining impurity-free phosphorous compounds meeting food specifications, methods of obtaining condensed phosphates, unit operations in technologies of producing solid and liquid phosphatic fertilizers, mixed micronutrient fertilizers, as well as and suspension fertilizers. An important aspect of Professor Hoffmann's research is the reduction of the impact of technological processes on the environment.

Since 2004, Józef Hoffmann has been also working as the Director of the Institute of Inorganic Technology and Mineral Fertilizers at WUT. Prior to that, he was the Deputy Director for Cooperation with Industry and of the Institute Physical Chemistry of Mineral Fertilizers Laboratory. Currently, he is the head of the specialization: "Management and Quality Systems in the Chemical Company" in the Faculty of Chemistry at WUT. In this program he lectures on chemical technology, low-tonnage chemical production and manufacturing process economics. During the past several years, he visited and cooperated with various institutions, including: WChTI Sofia, Hogeschool Zeeland in Vlissingen, Universite Louis Pasteur in Strasbourg, Philips Analytical in Cambridge and Instituto Superior do Engenharia in Porto. He is appointed as an expert in the Ministry of Science and Higher Education and the coordinator of the Technical Work Group for Chemical Industry Affairs in the area of the Best Available Technologies (BAT) for Speciality Inorganic Chemicals (SIC). He was the supervisor to two doctoral students who already defended their dissertations. He also supervised many graduated masters of science students, including five graduate students from Portugal and Belgium. He is the author and co-author of 127 publications, 33 granted patents and over 100 unpublished research documents and expert opinions for the industry and various institutions. He has implemented 25 technological solutions for mainly fertilizer products (liquid micronutrient fertilizers applied to leaves, fertilizers granulated by crushing, solid fertilizers), and domestic detergents. He has received many awards, including the Prime Minister's Award for research-implementation achievements, the Great Poland Business Award for implementing fertilizer technologies, the Technical Associations Award for scientific-research achievements and several Chemistry Faculty Dean and WUT Presidential awards.

Andrzej Puszyński graduated from the Wrocław University of Technology (WUT), the Department of Chemistry, in 1961. His MSc thesis was entitled: "Polyisobutylene Chlorination". In 1969 he defended his PhD dissertation on "Research on Nitraminopyridines" in the Department of Chemistry, WUT and received the Doctor of Chemical Sciences Diploma. In the years 1961-1971 he worked as a university teacher in the Faculty of Engineering and Economics, the Academy of Economics in Wrocław. In the years 1971-1973 he held the same position in the Faculty of Mathematics, Physics and Chemistry, the College of Pedagogy in Opole. In the years 1973-2004 he was working as a lecturer in the Institute of Organic and Polymer Technology, the Faculty of Chemistry, WUT. Currently he is a retired employee of WUT but still active in his professional field. He performs, among other things, responsible functions in the Association of Chemical Industry Engineers and Technicians. He is the author of 66 scientific publications, including 22 editions of a textbook on organic technology, polymers, and plastics.