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Chapter

Dyeing Chemicals

Shekh Md. Mamun Kabir and Joonseok Koh

Abstract

Dyeing auxiliaries play an important role in the determination of the final dyeing quality. The formation of additional complexes with dyes and auxiliary agents enhances the exhaustion of dyes on textile substrates. For aqueous-based dyeing, dye auxiliaries such as chelating agents, dispersing agents, leveling agents, electrolyte, pH control agents, and surfactants form complexes with the dye on natural and synthetic fibers. A growing awareness of the impact of industrial pollution on the environment became crucial for the dyeing industry in the closing decades of the twentieth century. These chapters discuss the characteristics of dyeing chemicals and how auxiliary substances can assist in achieving outstanding dyeing performance.

Keywords: chelating agent, dispersing agent, leveling agent, electrolyte, pH control agent, surfactant, antifoaming agents

1. Introduction

The textile dyeing industry is constantly increasing because of the growing consumption of fabrics and garments; moreover, till the next decade, a billion consumers will be added to the global market [1]. The processing of such a large volume of fabrics and garments is conducted through dyes and chemicals. A variety of factors can influence the quality of dyeing and its complex mechanisms in batch reproduction. The degree of levelness and reproducibility of dyeing depends on not only the dyes and chemicals used but also the control of temperatures and pH conditions in the dye bath [2]. To achieve any progress in such studies, it is necessary to control the parameters in order to determine their effect on the dyeing system. To study the performance of dyeing in an aqueous dye bath system, it is essential to alter the dye concentration while maintaining, and all other conditions constant so that the changes in the chemical structure of the solvent and the nature of the dye species can be analyzed [3].

Natural and synthetic dyes play an important role in the process of dyeing textile fabrics and garments. Different classes of dyes are used for coloring different textile materials with the aid of auxiliaries, which facilitate the homogenization of the mixtures [4]. The method of determining the equilibrium constant of the dye–auxiliary complex can be constructed.

\[
\text{Dye} + \text{Auxiliary Agent} \rightarrow \text{Addition complex}
\]

\[
D + AA \rightarrow AC
\]

The association between the dye and the auxiliary agent may proceed as far as the colloidal particles are dispersed in dyes. Apart from this, there exists an equilibrium between the dyes and the textile auxiliaries, which have been added to the
dye bath [5]. The auxiliaries exist in a state of association equilibrium [6]. A new era of dyeing chemical research began in 1930 when soap was replaced by synthetic surfactants [7]. Since many researchers have described the use of various textile auxiliaries in dyeing [8]. Thermodynamic studies of dyeing make useful contributions to the general theories of intermolecular forces while diffusion processes are also influenced by the dye concentration, temperature, nature of dyeing auxiliaries, and polymer structure [9]. A major challenge for textile wet processors is addressing the increasing cost and demand associated with the use of different auxiliaries in industrialized countries. However, in recent years, there has been significant demonstration of a lack of knowledge about the usage of dyeing chemicals. In this study, the potential for dyeing auxiliaries was explained.

2. Dyeing chemicals

This chapter demonstrates the characteristics of different auxiliary agents, which tend to form colloidal flocculation and dye–auxiliary complexes with dyes. A chelating agent is used to remove the hardness of water by bonding with calcium and magnesium ions and other heavy metal ions in hard water, by forming a stable complex compound that does not decompose over a prolonged processing period [10]. Dispersing agents, pH, and temperatures affect the changes in shade and fastness of disperse dyes [2, 11]. A greater degree of levelness is linked with greater retarding effect, which means a longer dyeing time [4]. In the presence of electrolytes, dyeing of cotton fabric with anionic dyes is to suppress negative charge at the fiber surface and to promote increased dye exhaustion [12]. If the dye bath pH is adjusted prior to the dyeing process, it may affect the absorption of dye on the fiber [13]. Synthetic surfactants have been extensively studied by many researchers [14]. After World War II, fabric softeners emerged through the introduction of the synthetic surfactant [15].

2.1 Chelating agent

The word “chelate” is obtained from the Greek word “chel,” which means crab’s claw. Chemically, a chelate is an organic compound that can form a ring structure by bonding with metal ions. Chelating agents are mostly used in dye baths, as they remove the hardness of water by bonding with heavy metal ion. Chelating agents form a stable complex compound that does not decompose over a prolonged processing period [10]. The coordination of water with metal ions enhances the acidity of the dye solution, which is dependent on the physical and chemical characteristics of the metal ions [16]. Inorganic chelating agents are also used as detergent by suspending and dispersing agents. They require less than the stoichiometric quantity predicted to keep ions in solution (threshold effect). They will dissociate to sodium phosphate in water over time losing their ability to chelate, especially in hot water. Polyphosphates are derivatives of phosphoric acid and are made by reacting phosphorous pentoxide with phosphoric acid. Important polyphosphates are tetra sodium pyrophosphate (Na₄P₂O₇), sodium phosphate (Na₅P₃O₁₁), and sodium hexametaphosphate (Na₆P₆O₁₈) [17] (Scheme 1).

Scheme 1. Formation of sodium polyphosphates.
Organophosphonic acids also act as chelating agents and aid detergency by dispersing and suspending soil. They are more stable than inorganic polyphosphates in hot water and exhibit threshold effect (Figure 1).

Amino carboxylic acids form very stable complexes, when reacting with metal ions. They react stoichiometrically and can be formed quantitatively to determine calcium and magnesium by titration. Disodium-ethylenediaminetetraacetic acid (EDTA) and nitrilotriacetic acid (NTA) are categorized in these groups [17] (Figure 2).

Several hydroxyl groups containing organic compounds precipitate bi- and trivalent metal cations in alkaline medium and also acts as effective sequestering agents; however, they are not effective for calcium and magnesium. Some of the well-known products in this category are glycolic acid, gluconic acid, and citric acid (Figure 3).

Ethylenediaminetetraacetic acid also prevents or removes scales by forming water-soluble complex compounds, which vigorously react with metal ions [18]. Gluconic acid is obtained from fruit, honey, and wine. In an aqueous solution at neutral pH, gluconic acid forms gluconate ion, which is used in cleaning products in which it dissolves mineral deposits through the formation of ligand with metal compounds [19]. Citric acid also strongly interacts with active metal components. This interaction helps metal dispersion and decreases the specific surface area and pore volume of the metal components. The addition of citric acid increases the amount of metal ion adsorption. Increasing the adsorption depends on the formation of more complex structures (Scheme 2).
Glycolic acid exhibits better chelation power in all metal ions because of the lower molecular weight, which helps to remove all metal ions. Glycolic acid, which is a naturally occurring organic agent, exhibits more penetration of metal ions. Natural chelates are biodegradable and non-toxic to the environment. Under natural conditions, EDTA is converted into ethylenediaminetriacetic acid, and then forms a ring structure with other metals, creating persistent organic pollutants [20]. Kabir and Koh [21] have demonstrated that glycolic acid provides effective chelation efficiency for a moderate acid donor. Consequently, glycolic acid exhibited a higher percentage of dyebath exhaustions and better dye ability than other organic chelating agents.

2.2 Dispersing agent

Disperse dyes are nonionic chemicals that are barely soluble in water and often crystallize with varying particle size [22]. These characteristics are inadequate for dispersing dyes of water and cause unleveled dyeing. To achieve the required particle size and distribution, the disperse dye is milled, usually in the presence of a dispersing agent [23]. Generally, the dispersing agents are anionic, e.g., ligninsulfonates, or polycondensates of arylsulfonic acids with formaldehyde, which facilitate milling. Dispersing agents have shown a dual function role: breaking down aggregated dye particles and dispersing dyes in the dye liquor. Dispersing agents consists of high-molecular weight or polymeric compounds in which polar or ionizing groups alternate with nonpolar groups along the chain. The backbone of a dispersing agent is nonpolar, while the polar or ionizing groups are located in the side chains. Johnson [24] demonstrated that the adsorption of dispersing agent is oriented parallel to the surface, so that the nonpolar groups adjoin to the surface and the polar groups turn outward.

The application of disperse dyes on polyester fabric mainly occurs via the inclusion of dispersing agents. The hydrophobic tails of the dispersing-agent molecules are oriented toward the center of the dye micelle, which facilitates micellar solubilization of the disperse dye molecules, thereby conferring higher dye solubility. The disperse dyeing mechanism has been categorized in four stages: (i) dissolution of dye in water by the formation of the dye micelle with dispersing agents; (ii) transference of dye molecules from the solution to the surface of the fiber; (iii) replenishment of dyebath by the dissolution of solid material from the dispersion; and (iv) diffusion of dye into the fiber (Figure 4).
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Murray and Mortimer [25] mentioned sodium dinaphthylmethane sulfonate and lignosulfonates as agents used for disperse dyes. A combination of dispersing agents and water-soluble polymers, for example, poly (vinyl alcohol), sodium polyacrylate, and the maleic anhydride-styrene copolymer, in admixture with anionic surfactants, would be useful in processing the pigments. Wolf and Bauer [26] suggested the use of dissolved disperse dyes in dimethylformamide and formed dispersions by pouring these solutions into aqueous solutions of the dispersing agents. Dispersing agents formed the dispersion by varying particle size of disperse dyes (Figure 5).

Heimanns [27] explained that a high-concentration of dispersing agent acts as a barrier to diffusion and reduces the dye yield in thermo-fixation. For better color yield, liquid brands of disperse dyes contain a smaller amount of dispersing agent. To ensure stability, the amount of dispersing agents must be maintained in the dye bath [8].

### 2.3 Leveling agents

To achieve uniform dyeing on fabric, it is essential to add a suitable leveling agent in the dye baths. However, it is quite difficult to explain the functions and actions of leveling agents. Werner [4] explained that the effects of textile auxiliaries, such as leveling agents, would appear at first sight to contradict the very nature of physical methods. Figure 6 shows that equilibrium in dyebath-fiber systems is caused by a large number of processes that play an important part in producing good dyeing. Leveling agents enhance the force in a state of association equilibrium in the dye bath. Werner [4] investigated that the formation of dyes and leveling agents represents bonding similar to what occurs in the dyeing of fibers. The effects of leveling agents are: (i) leveling agents tend to decrease the absorption of dyes by forming a dye-auxiliary

![Figure 4. Sodium dinaphthylmethane sulphonate and sodium lignosulphonate.](image)

![Figure 5. Mechanism for dyeing of polyester with disperse dye and dispersing agents.](image)
complex with free dyes maintaining equilibrium; (ii) leveling agents act as retarding agents; and (iii) dye migration proceeds slowly, leads better leading to improved leveling of dye on the fiber. A cationic polyethoxylated amine can perform strong leveling action. The greater cationic character a strong complex formation, pronounced retardation of dyeing, and higher risk of precipitation (Figure 7).

The polyethoxylated chain is longer ($n > 50$), and the dye auxiliary complex is dispersed by the cationic leveling agents. Amphoteric leveling agents have both anionic and cationic groups, so its activation depends on the dye bath pH [20].

2.4 Electrolyte

During dyeing in the dyebath, electrolyte serves as three important roles—driving dye into textiles causing, maximum exhaustion of dye molecules through the presence of salt, and fixing dyestuff to the cellulose material [28]. The dyeing mechanism of reactive dye can be classified into two phases, exhaustion and fixation. The process is lengthy because considerable time is spent on the controlled heating of the dyebath and the portion-wise addition of salt and alkali to avoid unleveled dyeing and maximize the exhaustion and fixation [20]. A colorless crystalline solid NaCl composed of inorganic compound of sodium and chloride, a salt in which ionic bonds hold the two components together in the familiar water-soluble white crystals, has a key role in the textile dyeing process in maintaining the electrolytic balance in the textile dyeing process. Glauber's salt is the common name for sodium sulfate dehydrates $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$; it occurs as white or colorless monoclinic crystals, which are most commonly used in the dyeing industry [29]. Vacuum salt is manufactured by recrystallization of purified brine solution. In the vacuum crystallization process, raw salt is dissolved in water to make a saturated solution and clarify the impurities from the bottom. A vacuum is generated by using a suitable vacuum pump [30].

Figure 6. Equilibrium in dyebath-fiber systems containing leveling agents.

Figure 7. Different leveling agents.
When cotton/bast fibers are immersed in water, its surface due to the hydroxyl ions also becomes anionic; hence, the dye particles and the cellulosic fiber tend to repel each other. So, the level of substantivity is reduced. The addition of salt creates an electrical positive double layer, which hides the negative electrostatic charge (Donnan Potential) of the cellulose surface. This allows the dye to approach the fiber, allowing better interaction of Van der Waals forces as well; this improves the substantivity. Neale et al. [31] proposed the existence of the Donnan equilibrium. The idea of the Donnan equilibrium was employed for the modern electrochemical theories of the dyeing of cellulose from aqueous solutions of direct dyes [32].

NaCl enhances the diffusion of dye and its adsorption onto fiber. Bicarbonate and carbonate increase the dye bath pH and perform dye fixation through the formation of covalent bonds. “Bolaform” electrolytes contain anionic and cationic organic groups, which are separated by large distances. The word ‘bola’ means a long cord with heavy balls. The alkyl chains whose lengths are sufficiently long act as electrolyte to become surface-active compounds. Hamada et al. [33] extensively studied various physical properties of bolaform electrolytes or amphiphilic colloids. The bolaform electrolytes or amphiphilic colloids contain a single positive or negative site (Figure 8).

2.5 pH control agents

Dyebath pH is adjusted prior to the dyeing process; otherwise, it may affect various factors, such as the absorption of dye into the fiber, by increasing or reducing alkalinity [34]. The pH controlling agents play three important roles: (a) maintain a high degree of acidity; (b) control the pH within narrow tolerances; and (c) slide the pH in acidic conditions [35]. The pH controlling agents are usually based on two chemicals, a weak acid and its salt, with a stronger base such as acetic acid, sodium acetate, or phosphoric acid-sodium phosphate. When the dyeing temperature increases, pH control agents release more acidic compounds. Ammonium sulfate decomposes gradually, producing ammonia and sulfuric acid, which is a strong acid that subsequently lowers the pH when ammonia escapes, at boiling temperatures. However, enclosed or partially enclosed machine, such as a winch, it is not very efficient because ammonia is prevented from escaping into the dye bath (Scheme 3).

Organic esters are also used as an alternative method for obtaining a pH that slides in the direction of acidity under the conditions of processing. In 1953, Brotherton Co. Ltd. introduced the Estrocon process, which is based on the addition

\[
\text{(NH}_4\text{)}_2\text{SO}_4 \;+\; 2\text{H}_2\text{O} \;\xrightarrow{\text{H}_2\text{SO}_4 \;+\; 2\text{NH}_4\text{OH}}
\]

\[
\text{NH}_4\text{OH} \;\xrightarrow{\text{NH}_3 \;+\; \text{H}_2\text{O}}
\]

\[
\text{H}_2\text{SO}_4 \;\xrightarrow{2\text{H}^+ \;+\; \text{SO}_4^{2-}}
\]

Scheme 3. Dissociation of ammonium salt.
of diethyl tartrate or ethyl lactate for dyeing wool with acid milling dyes and chrome dyes by the single-bath method [36] (Scheme 4).

Almost 35 years ago, Sandoz introduced the Sandacid V process, in which $\gamma$-butyrolactone undergoes hydrolysis to produce butyric acid [37] (Scheme 5).

Koh et al. [37] found that hydrolysable organic esters achieved a relatively wider range of pH sliding than ammonium sulfate or sodium di hydrogen phosphate, which can produce good exhaustion with satisfactory dyeing in the closed dyeing process.

### 2.6 Surfactants

Surfactant molecules should have surface active properties with the chemical structure of a hydrophilic (water loving) and hydrophobic (having little attraction for organic molecules) balance [38] (Figure 9).

These molecules get preferentially oriented at the interface between air and water, which lowers the surface tension of water substantially when dissolved in the concentration range of 0.1–10 g/l. The driving force for the activation of surface active agents is the formation of micelles, in which the lyophobic tails are associated with themselves, and the hydrophilic heads are surrounded by water molecules. Surfactants are classified into different ways such as use, ionic charge, and chemical structure. According to the ionic structure, surfactants can be classified into four categories: anionic, cationic, amphoteric, and nonionic [39]. Anionic surfactants are surfactants that are ionized into anions and cations but the anion is the dominating ion in the solution. Examples are alkyl benzene sulfonate and sodium lauryl sulfate (Figure 10).

An ionic surface active agent that produces cation as the dominating ion when dissolved in water is called a cationic surfactant. For example, dodecyl dimethyl ammonium chloride and distearyl dimethyl ammonium chloride (Figure 11).

Nonionic surfactants that does not show any ionic behavior when dissolved in water are called nonionic surfactants. Examples are Lanolin ethoxylate and glycerol monostearate (Figure 12).
Surfactants that ionize and produce large segments (these segments are called Zwitter ions) carrying both anionic and cationic ions when dissolved in water are called amphoteric surfactants. Lauryl dimethyl betaine and cocamidopropyl betaine are the most commonly used (Figure 13).

Surfactants are widely used as wetting agents, emulsifiers, detergent, lubricants, or dispersing agents [40].

2.7 Anti-foaming agents

The dyeing process can lead to the formation of macro and micro foams during circulation of dyes and auxiliaries in the dye bath. A macro foam is displayed with large bubbles, which are visible on the surface of the system and produce cosmetic imperfections [41]. A typical list of anti-foaming agents is as follows: 2-ethyl hexanol (EH), tributyl phosphate (TBP), poly (dimethyl siloxane) (PDMS) amides, mineral oil, fatty acids, and their derivatives [42] (Figure 14).
Silicone-based anti-foaming agents consist of small polar groups and hydrophobic polymer chain. The hydrophobic polymer chain is typically a permethylated siloxane. The PDMS chain gains its hydrophilic character by modification with poly (ethylene glycol) (PEG) and poly (propylene glycol) (PPG). PEG and PPG are water-soluble polymers that allow materials to retain good water swellability [43]. A decrease in foaming occurs with an increase in the hydrophilicity of the copolymers. When the hydrophilicity of the copolymers increased, the micro foams were removed more effectively. Kekevi et al. [43] demonstrate that the uniqueness of these anti-foaming agents is caused by two properties—flexibility, which enhances the acquired conformations that result in efficient packing at various interfaces and lower cohesive energy, which is derived from the cross-sectional area of the siloxane at the interface (Scheme 6).

![Scheme 6. Silicone-based anti-foaming agents.](image)

2.8 Reducing agents

Beginning of the nineteenth century, sodium dithionate (Na$_2$S$_2$O$_4$) was introduced as a reducing agent for the vat dyeing process [8]. Effective sodium dithionate, also called hydrose, provides an effective reduction in indigo dyes [44]. The amount of Na$_2$S$_2$O$_4$ used in the reduction of indigo dyeing, formed a large amount of by products and sulfite (SO$_3^{2-}$) and sulfate (SO$_4^{2-}$) ions- (Scheme 7).

![Scheme 7. Oxidation-reduction of indigo.](image)
Stripping methods, mostly used in textile finishing, can remove dye from colored fabric. The process is named as ‘back stripping’ or ‘destructive stripping’. The depth of shade is changed by back stripping; on the other hand, dyes are chemically altered by destructive stripping. Fono and Montclair [45] showed that dyes containing azo groups that can be chemically reduced to an almost colorless amine by using chemical reducing agents. The mechanism of reductive stripping depends on the structure of dyes and fibers and the chemical nature of reducing agents. Chavan [46] explained that different chemical combinations of reducing agents and stripping assistants, which are being used to strip the dye from fabric (Scheme 8).

Scheme 8.
Stripping process of azo dyes.

\[
\text{NaOH} + \text{Na}_2\text{S}_2\text{O}_4 \rightarrow \text{NH}_2 + \text{NH}_2
\]

Sodium formaldehyde sulfoxylate (CH$_3$NaO$_3$S) (Rongalite C) and Rongalite FD are also used as reducing agents in the dyeing industry (Scheme 9).

Scheme 9.
Rongalite FD salts.

As sodium dithionate is highly toxic, many researchers [47] have investigated to replacing it with ecological reducing agents such as glucose, fructose, inverted sugar, and molasses (Figure 15).

Figure 15.
Ecological reducing agents.

Many studies have been devoted to catalysts that accelerate the reduction of vat dyes when added to certain reducing agents. BASF-marketed a catalyst, bis-(dimethylglyoximato-diamminocobaltinitrite) for use as a reducing agent.

2.9 Softeners

A softener is a chemical used to make the touch of fabric more pleasing. Softened fabrics are fluffier and have better drape ability. In addition to esthetics, softeners improve abrasion resistance, improve tearing strength, and reduce needle cutting when the garments are sewn. Softeners are divided into three major chemical categories: anionic, cationic, and nonionic. Anionic softeners have a negative charge on the molecule, which comes from the carboxylate group (-COO-), sulfate group (-OSO$_3$-), or phosphate group (-PO$_4$-). Fatty alcohol sulfates are made by the reaction of a hydrophobe with sulfuric acid [17] (Scheme 10).
Fewer sulfate groups result in better softeners. Lightly sulfonated oils are sometimes called self-emulsifying because they form turbid water solutions (Scheme 11).

![Scheme 11. Formation of sulfated triglycerides.](image1)

Cationic softeners have a positive charge on the large part of the molecule. The amine becomes functionalized under a pH of 7. Quaternary ammonium salts are activated at all pH levels. The ionic interaction causes complete exhaustion from baths and orientation on the fiber surfaces, which cause good slipperiness and reduction in the static charge on the fabric surface. There are several cationic softeners that are usually used in the textile industry, such as fatty amines, fatty amino esters, fatty amino amides, and quaternary ammonium salts (Scheme 12).

![Scheme 12. Synthesis of quaternary ammonium salts.](image2)

The nonionic softener has three subcategories: ethylene oxide derivatives, silicones, and hydrocarbon waxes based on paraffin or polyethylene. Polyethylene emulsions are hard, waxy films, which serve to reduce the coefficient of friction. Silicones are polysiloxane polymers. Silicon resembles carbon in which it is tetravalent and forms a covalent bond with other elements (Scheme 13).

![Scheme 13. Reaction of dimethylpolysiloxane.](image3)
2.10 Anti-creasing agents

Crease is formed in cotton fiber because of the intermolecular hydrogen bonding of primary and secondary hydroxyl groups of the polymer chains. The amorphous regions of the hydrogen bonds can easily break down by folding. In the folding state, the broken hydrogen bonds stabilize. Different durable press finishes have been used for many years for use in cotton fabric. N-methylol compounds such as dihydroxyethylene urea react readily with the hydroxyl groups of cellulose chains [48] (Figure 16).

![Figure 16. Cross-linking agents based on N-hydroxyethyl and N-alkoxymethyl compounds.](image)

N-Hydroxyethyl acrylamide is also used as a cross-linking agent for cellulosic fibers (Figure 17).

![Figure 17. N-(hydroxymethyl)acrylamide](image)

Activated vinyl groups or divinyl sulfone and N, N-methylene-bis-acrylamide are also used cross-linking agents. Divinyl sulfone precursors are used for the cross-linking reaction with cellulose. Cross-linking reactions with epoxides such as epichlorohydrin are mostly used on cellulosic fibers. In addition, aziridine derivatives such as tetramethylene-bis-(N, N-ethylene urea) and trisaziridinyltriazine are effective cross-linking agents [49] (Figure 18).

![Figure 18. Cross-linking agents based on aziridine derivatives.](image)
3. Conclusion

Although considerable research has been conducted regarding dyeing auxiliaries, chemical and other related industries have faced many problems with respect to environmental issues and cost effectiveness for consumers. Although thermodynamic studies of dyeing can make useful contributions to the general theories of intermolecular forces, diffusion processes, and the influence of different parameters such as dye concentration, temperature, dyeing time, and polymer structure remain largely unexplained. When equilibrium studies are carried out and assessed in conjunction with the growing amount of information on the structure of dyeing chemicals and aqueous solutions, their relevance to the dyeing theory should not be underestimated. Due to the lack of chemical knowledge of dyers, quality dyeing is deteriorating with a significant effluent load. This study clearly shows that the different dyeing chemicals used in the dye houses also need investigation to determine the procedures in which the chemicals are used to dye natural and synthetic textiles. This may be a good first step in presenting different chemicals used in the coloration industry.

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