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Azo dyes: Synthesis, Classification and Utilisation in Textile Industry

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In Loving Memory of Late Professor Doctor ""Mohamed Refaat Hussein Mahran""

Abstract
In this work, we have presented a review of azo dyes which are the most important synthetic colorants that have been widely used in textiles, Azo dyes are organic compounds, that contain the coloring azo function (N=N)and are used in the production of colored materials. Azo compounds have vivid colors and comprise about two-thirds of all synthetic dyes. azo dyes, such as monoazo, diazo, and triazo dyes, are defined by the presence of one or more azo groups (-N=N-) connected to the –OH or –NH2 type auxochrome groups. Then we pointed out the synthesis of azo dyes using the traditional method(diazotization/coupling reaction) and prepared a new azo dye using natural polyphenols found in plants, which are used as coupling agents. Finally, we indicate the Dyeing process of cotton fabric with azoic dyes as used in the textile industry.
Keywords: Azo dyes, synthesis of azo dyes, types of azo dyes.

1. Coloration

For the past century, coloring has been an important vision factor for humans, especially in the textile industry, coloring is defined as an important factor for the textile. [1-8] There are two methods to impart color to the textile materials, first is dyeing, and the second is printing. [9-14] In addition, there are several dye classes used in the textile sector. One of the most commonly used dyes is azo dye.

2. Azo Dyes

Azo dyes make up the biggest category of colorants, accounting for 70% of all organic dyes manufactured globally[15, 16] and 60% of the usage[16, 17]. Azo Dyes are grouped into around twenty-five distinct categories due to the varied chemical structures of chromophores[17, 18]. The success of azo dyes is a result of their easy synthesis processes, extensive structural variety, high molar extinction coefficient, low cost, and medium to high fastness characteristics concerning light and moisture[19]. They are used for a variety of purposes, including the coloring of natural and manmade materials, ink, cosmetics, food, leather, and painting solutions [20-24].

Azo compounds are chemically represented as R–N=N-R', where R or R' can be either aryl or alkyl compounds, and –N=N- is the azo group. [25]

The majority of azo dyes are made by diazotizing an aromatic primary amine and then coupling it with one or more electron-rich nucleophiles, such as hydroxy and amino. Additional techniques for creating azo dyes as reducing nitroaromatic derivatives in an alkaline medium; oxidising primary amines with lead tetraacetate or permanganate potassium; condensing hydrazines and quinones; and condensing primary amines with nitroso derivatives.[26, 27]

2.1. Definition of azo dyes

The dyes are synthetic aromatic organic compounds, as is widely known, and are often employed to color or dye various sub-standard materials for aesthetic and other functional purposes. The textile sector is a significant user of various synthetic dyes and contributes significantly to the nation's economic development.[21]

2.2. History of Azo Dyes

For the past few years, Azo Dyes have been in use. The simplest Azo dyes to use are direct dyes. These direct dyes have chemical components that enable them to dissolve in water and be readily absorbed by various substances. The first Azo dye, Congo red, was employed in 1884; however, more chemically synthesized dyes that are more heat- and

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light-resistant have since taken their place. Azo pigments are the most common and ancient type of food coloring. In 1858, Peter Griess discovered them.[28, 29]

2.3. Organic chemistry of azo dye

Azo dyes have an azo group—-N = N—[23, 30-32]that joins two hybridized carbon atoms, these carbons frequently, but not always, are a component of aromatic systems.[33] Azo dyes are comprised of an amine or phenol linked to a diazotized amine.[34]

A single azo group makes up the majority of azo dyes, although others include two (disazo), three (trisazo), or even more[31, 35]

Based on the exact structure of the molecule, azo dyes can produce a full spectrum of colors. Azo dye compounds may be found in the colors yellow, orange, red, brown, and blue[36]. Different replacements for aromatic rings lead to differences in the system's degree of conjugation in the azo dye, which affects color. As is well-known, a molecule's ability to absorb visible light at a certain wavelength relies on the size of its conjugated system [37-39]. For example:

(Shortest π system) → yellow → orange → red → green → blue (longest π system).

2.3.1. Azo groupe

A double-bonded azo linkage makes up the chromophore group, the portion of an azo-colorant molecule that creates color. The azo colorants' chromophoric group changes a substrate's color by either selectively absorbing or dispersing visible light, or light with wavelengths of around 400-750nm[40]

Two nitrogen atoms make up the azo linkage, and they are also connected to carbon atoms. One or more of these carbon atoms are part of an aromatic carbocycle, an aryl moiety (often found in derivatives of benzene or naphthalene), or a heterocycle (pyrazolone, thiazole, etc.). The azo group's second around carbon may also be a component of an aliphatic derivative, such as acetoacetic acid.

An azo-colorant molecule can be summed up as follows in general

aryl - N = N - R

where R may be an aryl, heteroaryl, or -CH = C(OH) - alkyl derivatives

2.3.2. Azo dye formation and structure

Figure 1 illustrates the general chemical structure of an azo dye, which is made up of a backbone, auxochrome groups, chromophoric groups, and solubilizing groups [26, 35].

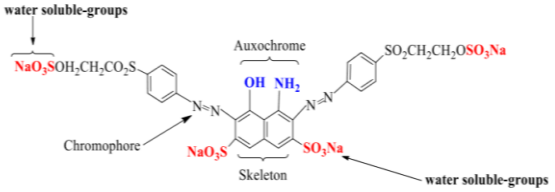


Figure 1. Structure of the azo reactive dye.

The azo bonds together with the chromophores and auxochromes they are linked to, define the color of the azo dyes[30]

2.4. Classification of dyes according to azo grouping number (Classification of azo dyes in Color Index)

According to how many azo links are present in a single dye molecule, azo dyes are classified as monoazo, disazo, tri-sazo, polyazo, and azoic[41-43]. A value between 11,000 and 39,999 is given to azo dyes in the Color Index (CI) system to match their molecular composition (Table 1). The organization of dyers and colorists developed the color index number, which is used to classify dyes [26, 44-46]

Chemical Class	CI no.
Mono azo	11000-19999
Bis-azo	20000-29999
Tris-azo	30000-34999
Polyazo	35000-36999
Azoic	37000-39999

2.4.1. Mono azo dyes

Mono Azo dyes are dyes with only one azo group in their chemical structure[47].

These dyes can be classified into

- Dyes do not contain dissolving groups
- Dyes that contain dissolving groups:[48]

2.4.2. Dyes that do not contain -SO3H, -COOH groups

These groups include dyes: Solvent, Pigment, Mordant, Basic, Disperse and reactive

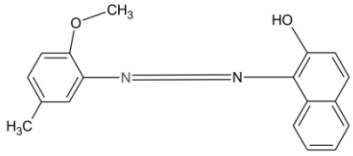
2.4.2.1. Solvent Mono Azo Dyes

A small number of mono-azo dyes are employed as solvent dyes. This set of dyes is incapable of forming a solubilizing group.

Example: Sudan Red R or Solvent Red R CI-12155

By diazotizing cresidine and combining it with B-Naphthol, Sudan Red R is made.

Cresidine→β-naphthol

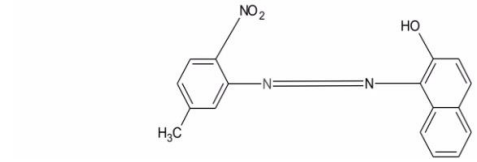


2.4.2.2. Pigment Mono Azo Dyes

The following is an important example of this family of insoluble mono-azo dyes that are utilized as pigment[49]**.**

ex: pigments orange 5 CI-12075

• 2,4dinitro aniline → β- naphthol



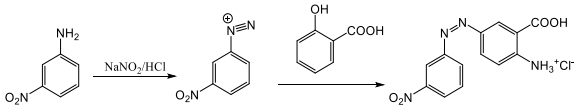
2.4.2.3. Mordant Mono Azo Dyes

This dye cannot be applied directly to the fiber; instead, a basic mordant must be applied to the fiber

They can create a complicated metallic compound called a lake

The presence of an o-hydroxyl group that reacts with the azo linkage to produce metal complexes makes azo dyes mordant

Because of their high light and washing fastness, mordant dyes are important.[50]



C.I. Mordant Yellow1

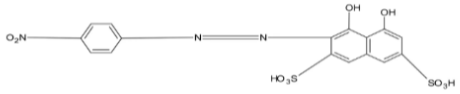
2.4.2.4. Basic (cationic) Mono Azo Dyes

Basic dyes have an auxochromic group of -NH2 or -NR2 and a chromophore system of -N-N**.**

The light fastness and washing fastness are often quite poor. Because of these factors, basic colors are rarely employed in the textile business

Example: Chromotope 2

P-nitro Aniline → chronotropic acid



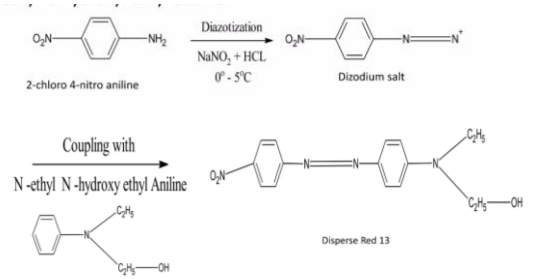
2.4.2.5. Disperse Mono Azo Dyes

Disperse dyes are mostly used to give polyester fiber a yellow-to-blue tint. Additionally, natural fibers like wool and silk are utilized with it

Disperse mono-azo dyes have a simple structure and small molecules. As a result, it enters the fiber structure with easy

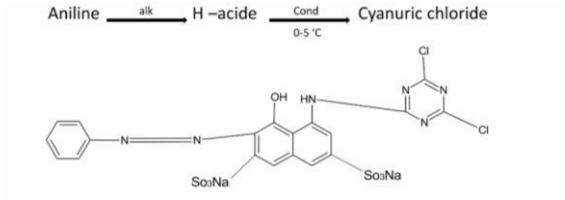
For instance, Cellition scanet B or Disperse Red 13

The compound is made by diazotizing 2-chloro-4-nitro aniline and coupling it to N-ethyl N-hydroxyl ethyl aniline



2.4.2.6. Reactive Mono Azo Dyes

Monoazo reactive dyes using a dichloro triazine reactive system, Example: Reactive Red B [51]



2.4.3. Dyes that contain -SO3H, -COOH groups is called

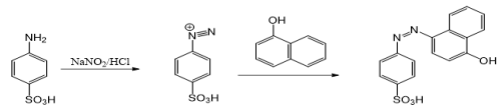
The major soluble mono-azo dyes are listed below.

- Acid dyes
- Mordant dyes
- Direct dyes

2.4.3.1. Acid dyes

Acid azo dyes are relatively cheap dyes that are used in dyeing wool from an acidic medium, as they give moderate to good degrees of fastness, especially against light.

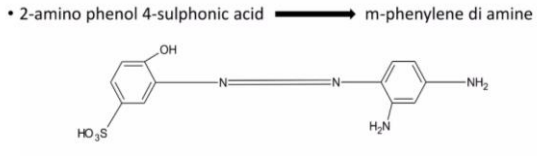
For example, C.I. dye. Acid Orange 20



2.4.3.2. Mordant dyes

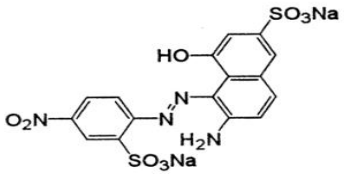
The soluble -SO3H group is also present in several mordant dyes. They have an ortho -oH group to the azo linkage, which can form a complex with the mordant

Ex-mordant brown 13



2.4.3.3. Direct dyes

This group of dyes is used to directly color leather, silk, and other porous fibers like wool. Ex Acid Violet: 1.



2.4.4. Bis (Dis) Azo Dyes

Bisazo dyes are those that have two azo groups in their chemical composition[52] This category includes a lot of azo dyes. There are four kinds of bis azo dyes available. The following general formula is given to each class.

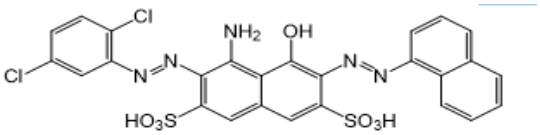
- 1. D → Z ← D'
- 2. C ← T → C'
- 3. D → CN → C
- 4. D → ZXZ ← D

Where, D = Diazo component, T = Tetrazo component, C = coupling component coupled with one molecule of a diazo component, CN = an aromatic which after coupling with a diazo component provides an amino group for further diazotization, and Z = a coupling component coupled with two molecules of diazo components

In ZXZ, X May be -NH, - NHCONH-, or more complex Linkages

2.4.4.1. Disazo dyes of the type D → Z ← D'

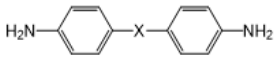
There are a small number of soluble dyes in this group, and the soluble ones are either acidic or mordant, and they are often blue, red, brown, and black colors. An example of these dyes is: C.I. Acid Green 19



2.4.4.2. Disazo dyes of the type C ← T → C'

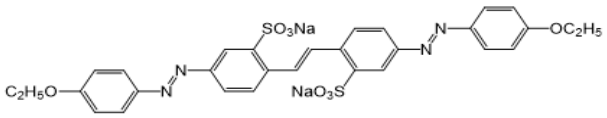
This group is very significant in the pigment industry and is regarded as one of the biggest groups of di-azo dyes. There are a lot of direct dyes in it, and there aren't many acidic or fixative dyes. Usually, the Tetrazo component defines whether the color produced is acidic or direct.

There are a small number of important direct dyes that can be prepared from the diamine compound, and the general dye follows the following:



Where X is -NHCONH-, -NH-, -CH=CH-, -CONH-, -S

An important example is C.I. Direct Yellow 12

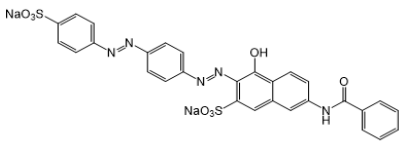


It is then followed by an ethylation process and used as a direct dye

2.4.4.3. Disazo dyes of the type D → CN → C

More than 250 dyes are known from this group, about half of which are direct dyes and the rest include acid dyes and some Mordant dyes.

As an example of direct dyes, C.I. Direct Red 81, uses a mono-azo dye containing an amine group that can be azotized again and is considered a diazo compound.

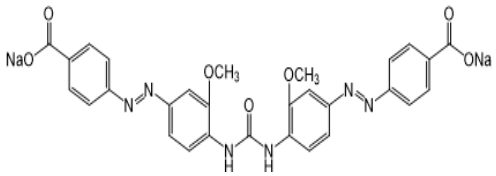


2.4.4.4. Disazo dyes of the type D → ZXZ ← D

This important group includes direct dyes, which can be divided into two types

2.4.4.4.1. *Dyes include urea derivatives – NHCONH*

As an example, C.I. Direct Yellow 49



The reaction is carried out by passing phosgene gas through an alkaline solution of the dye, where the reaction takes place as follows:

2R-NH2 + COCl2 → R-NHCONH-R + 2HCl

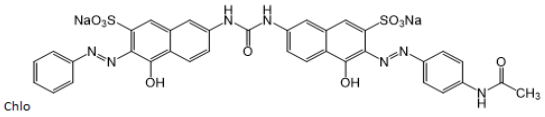
The resulting acid reacts with the increase in alkali. Two different mono-azo dyes can be used and the phosgene binds them together to produce an asymmetric dye.

2.4.4.4.2. *Dyes that contain Carbonyl - J acid as a coupling compound in the middle*

- J acid urea: as an example

The dyes derived from this compound are widely used

C.I. stain Direct red 23 (chlorazol scarlet 4BS)

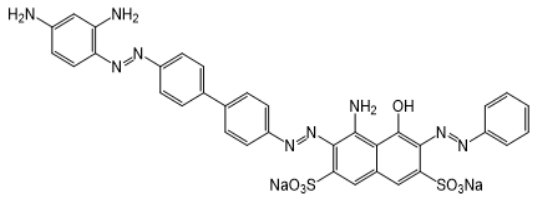


2.4.5. *Tris Azo Dyes*

These dyes divide into several subgroups, three of which are especially important. These groupings frequently include polyazo dyes as well as direct dyes.

2.4.5.1. *Trisazo dyes of types D → Z ← T → C*

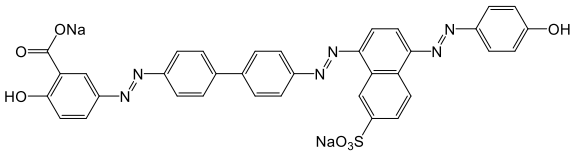
This group's pigments are frequently colored black or brown. For instance, we obtain C.I. Direct Black 38 by the following three duplication operations: -



2.4.5.2. *Trisazo dyes of the type C' ← CN ← T → C*

The pigments of this group include the colors brown, olive blue, and black, and they are the largest of the groups in terms of the number of pigments belonging to them.

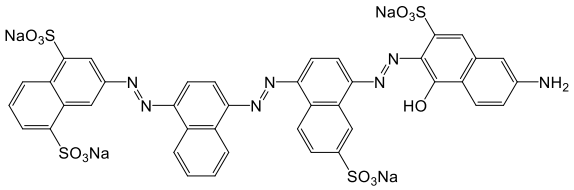
As an example, the C.I. pigment. Direct Brown 54



2.4.5.3. *Trisazo dyes of the type D → CN → CN' → C*

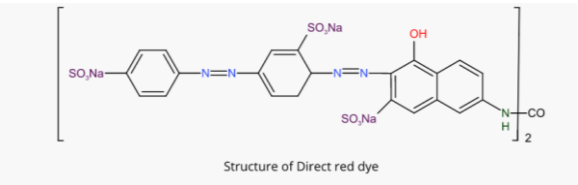
They include blue and gray direct dyes, and when C is the compound J acid or N-phenyl- J acid, the resulting dyes have strong color shades and good adaptability to cellulose fibers. Examples of frequently used dyes are the following:

C.I. Direct Blue 71 (Chlorantine Fast Blue GLL)



2.4.6. *Poly azo dye*

Complex dyes are called polyazo dyes. They have a molecule with three or more azo groups. They are included in the category of direct dyes. They use black, brown, and red dyes to color leather.

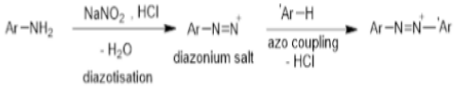


Direct red is the most popular dye for this type of color(figer)[53, 54]. The latter can be produced by phosgenating the suitable diazo dye. After N-acetyl-J is acid coupled, 6-amino-3,4′-azodibenzene-sulfonic acid can be diazotized to accomplish this. The hydrolysis of the acetyl group occurs before the phosgenation stage[18].

2.5. Synthesis of azo dyes Chemically by the diazotization/coupling reaction

Azo dyes are man-made substances with an azo link, -N=N[55], and are often made from the

aromatic amine substrates nitro and nitroso. The employment of an adequate oxidizing/reducing reaction or a diazotization/coupling reaction is essential to the synthesis procedures. One of the most significant reactions in the growth of industrial organic chemistry is this one. A coupling component and a diazonium salt are needed for the synthesis



Scheme 1. Diazotization and azo coupling

Scheme 1 depicts a two-stage reaction pathway that includes diazotization and azo coupling[56]. A primary aromatic amine (ArNH2), also known as the diazo component, is treated with sodium nitrite in the first stage of diazotization to create a diazonium salt (ArN+Cl-), under-regulated acidity and at fairly low temperatures. The more unstable diazonium salt is therefore reacted with a coupling component, such as a phenol, an aromatic amine, or a -ketoacid derivative, in the second stage of the process, known as azo coupling, to create the azo dye [37]

The majority of diazonium salts are explosive and unstable when they are dry. They are always synthesized at 0 °C with continuous stirring in an acid medium to reduce contact with water and create phenol, and they are then employed directly in the coupling reaction.

The diazonium salt interacts as an electrophile with a coupling component that is rich in electrons (a phenol or an aniline) which gives different colors of azo dye[37]when used to finish the production of the azo color

2.5.1.1. Factors affecting the rate of diazotization reaction (Dye formation)

Diazotization happens between 0 and 5°C in temperature. The diazonium salt breaks down when the temperature rises.

- Temperature
- a concentration of acid -
- pH of Sodium nitrite's (NaNO2)

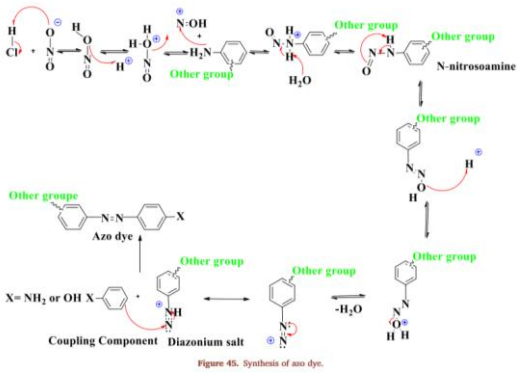


Figure 45. Synthesis of azo dye.

2.6. Synthesis of azo dyes by Gewald reaction

Using the Gewald process to create azo dye

Applications of this reaction include peptide analogs, dyes, and electronic materials; moreover, conjugated carbohydrates, agrochemicals, medicines and biomedicine, cane masters, etc.

As per Ram W. Sabnis **[29, 57]**, azo dyes 26 (Figure), were produced by condensation of benzo-thiophene-3 (2H) -one-1,1-dioxide with ethyl cyanoacetate. This was followed by diazotization using nitrosyl sulfuric acid and coupling with aryl amines substituted with N, N-dialkyl (acid coupling).**[29]**

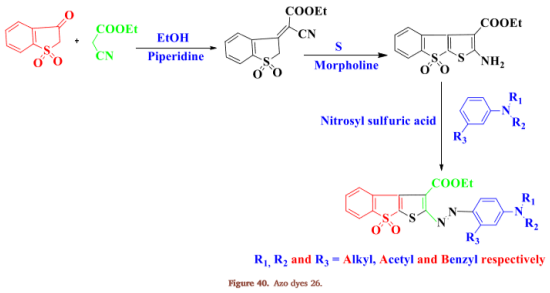


Figure 40. Azo dyes 26.

other methods for the synthesis of azo dyes

- Ther are other techniques for making azo dyes include
- Reducing nitroaromatic derivatives in an alkaline medium, reducing nitrosated compounds with AILIH4, oxidizing primary amines with potassium permanganate or lead tetraacetate, condensing hydrazines and quinones, and condensing primary amines of nitrosated derivatives[26, 27].
- Various synthetic techniques have been used to create new generations of azo dyes[58, 59]. hydrazone, chromene, and derivatives of 2-amhothiophene and 2-aminothiazoles, for

instance, are chemical groups found in the azo dye syntheses[60, 61].

Synthesis of azo dyes naturally by using polyphenolic compound in plants as a coupling agent

Focus has been placed on developing non-toxic or less hazardous dyes through health, safety, and environmental requirements during the past 20 years in the synthetic dye sector[62]. However, businesses that produce colors that cause cancer or those that include harsh chemicals still exist. Today, several studies have been conducted to rediscover the delight of producing color using sustainable, non-toxic, natural sources[63]. Generally speaking, azo dyes are not found in nature and can only be created chemically. To lessen the harmful effects, however, numerous azo dyes have lately been made utilizing certain natural sources.[50, 64, 65]

plants have vast quantities of Polyphenolic compounds as figer (Phenolic acid, flavonoids, lignans, and stilbenes) which are used as possible substitutes for synthetic phenols that can be reacted as a coupling agent with diazonium salts to create azo compounds[66]

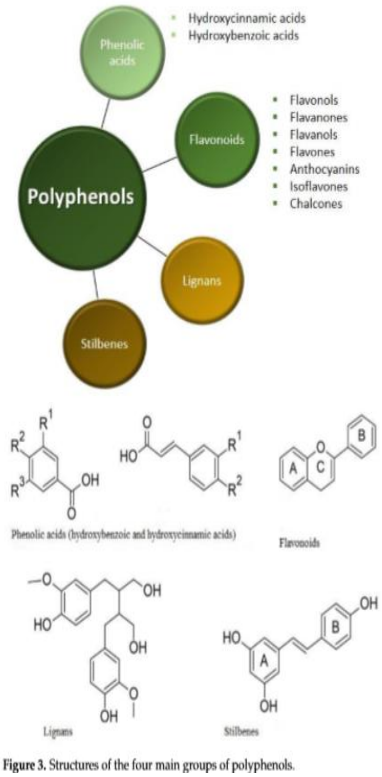


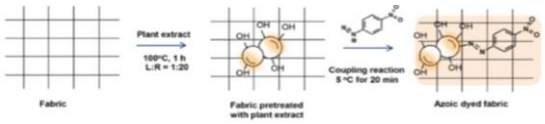
Figure 3. Structures of the four main groups of polyphenols.

innovative azoic dyeing technique, in which polyphenol-containing natural dyes successfully increased the color strength and fastness characteristics of colored various textiles. The method for azoic dyeing with natural dyes is the same as for conventional azoic dyeing; however, natural coupling components or plant polyphenols are used in place of the synthetic coupling component.[66, 67]

Ex:-

- Dyeing of Polyester and Nylon with Semi-synthetic Azo Dye by Chemical Modification of Natural Source Areca Nut[66] in this paper using three distinct primary amines—p-nitro aniline, p-anisidine, and aniline—that had been diazotized to create their corresponding diazonium salts which coupled with an areca nut extract (epicatechin)[66].
- Preparation of Azo Dye from Acacia catechu and its Application on Silk Fabrics[64]

The two parts of our azoic dyeing procedure are shown in Figure 1: first, the coupling component was absorbed into the fabric, and then, in situ, coupling was performed with the diazotization of a primary aromatic amine to create azo colors inside the fiber [66, 67].



Advantages of azoic dyeing

1- Textile materials can be dyed in a variety of physical shapes using a variety of machinery, or even in small quantities with household items in the absence of machinery [68]

2- It is easy for synthesis processes, high solubility, and high substrate absorption.[31, 69]

3- It is feasible to get dark blue, black, orange, and red hues economically

4- The reproducibility of dyes is quite good.

5- Commercial economic sustainability and low cost[40].

6- Dischargeable combinations are the majority.

7- Overall, the fastness characteristics are good

Disadvantages of azoic dyeing

1- The application process is difficult and drawn out

2- Several chemicals must be used and preserved. difficulties handling the constituent compounds under common chemical processing applications particularly the diazotization of aromatic amines' rapid bases

3- available hues are a few

4- It has always been difficult to make azoic-colored material fast to rubbing. To produce good rubbing fastness, it is crucial to effectively remove surplus naphthol from the surface of the treated material, have a high coupling component substantivity, and thoroughly soap the material after development

5- Since there would be cross-coupling between different naphthols and diazonium salt, compound hues as generated with other dye classes by mixing three or four dyes are difficult to make with azoic dyes and the combined shades are unpredictable and non-reproducible

6- Only after the coupling stage is the final shade apparent. As a result, any unevenness that results from naphtholation may be unnoticed and only become apparent after coupling, when shade correction is hard

Textile coloration with azo dye

Azo dyes offer an enormous range of colors with high fixing qualities and stability. It is noted that of the approximately 3,000 different dye types available on the market, approximately 50% belong to the category of azo dyes[42]. In cotton fabric bright colors like orange, crimson, scarlet, navy blue, and black are produced using azoic dyes.

There are several azo dye varieties and categorization schemes in use. Disperse dyes, metal-complex dyes, reactive dyes direct dyes, and acid dyes are types of azo dyes applied in textiles. Direct dyes are used on cotton and other cellulose-based fabrics. The non-electrostatic forces cause the dyes to attach to the fabric. The acid azo dyes are applied using a process that is nearly identical to that of the direct class and has an affinity for wool and silk. Yellow acid azo color tartrazine was developed in 1884 and is still widely used today. acid azo dyes showed fair to very good fastness against rubbing, perspiration, and washing. They can be applied to wool as dyes.[70, 71]

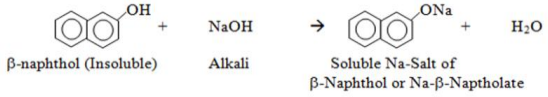
The two most frequent metals utilized in these colors are chromium and copper. These dyes' characteristics are changed by adding other metals, which allows them to serve a variety of functions. These eco-friendly, economical, and long-lasting dyes are the best available, offering a large range of options for many sectors.[71]

Dyeing process of cotton fabric with azoic dyes

It is not possible to find these dyes premade. There are three primary steps in the azoic dyeing process. [33, 34, 72] They are a) Naphtholation, b) Diazotisation, and c) Coupling

Naphtholation

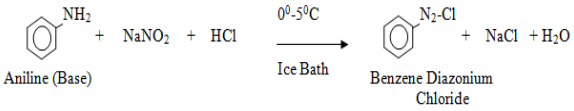
Naphthols are insoluble in water and require the treatment of an alkali to become soluble in water.[73]



Fabric is impregnated with this salt solution.

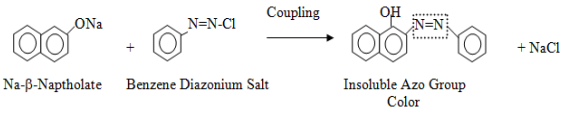
Diazotisation

At a temperature of–5 C, a base having an amino group (-NH2) combines with sodium nitrite (NaNO2) to generate a diazonium chloride solution of that base in the presence of enough HCl. [48, 73]



Coupling

The impregnated material requires treatment in a diazonium solution bath to facilitate coupling, which produces color inside the fabric. Maintaining the PH is important.



Summary

Azo dyes now have the highest dye chemistry production volume, and in the future, their relative importance could further increase. Azo dyes are often utilized in many different industries, including the food, pharmaceutical, paper, cosmetics, textile, and leather sectors. Azo compounds have vivid colors and have several types of monoazo, diazo, and triazo dyes There are most methods to synthesize azo dyes as the Gewald reaction, Synthesis of azo dyes Chemically by the diazotization/coupling reaction (More than 60 % of the synthesized dyes are produced by this reaction)and Synthesis of azo dyes naturally by using the poly phenolic compound in plants as coupling agent this method increased the color strength and fastness characteristics of colored various textiles. The Different dyes which have an azo group in chemical structure and give good properties and fastness when applied to fabric

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