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# Photochromic Dyes for Smart Textiles

*Virendra Kumar Gupta*

## Abstract

Photochromism is a light induced reversible color change phenomenon in photochromic molecule due to light and heat effect and molecular species exist in two forms which have different absorption spectra. The fascinating color change by photochromic molecules in response to specific wavelength of light produces number of applications such as U.V. protective fabrics, ophthalmic photochromic lenses, optical data storing, optical switch, sensors and display. This chapter provides a brief and conclusive review of photochromism their mechanism and application in Textiles. Although photochromic materials are in use since 1960 in lenses and sunglasses, but the development is slow due to technical difficulties and poor commercial application. Now there is renewed interest in photochromic materials which are used in nanofibers in smart textiles and in allied items.

**Keywords:** photochromic colorants, thermochromic colorants, U.V. radiation

## 1. Introduction

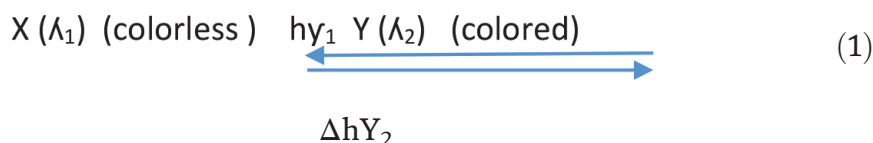
The wide and increasing application of photochromic and thermochromic colorants in different fields initiates new interest in dyes and pigments. The photochromic compounds got excited when irradiated at particular wavelength in range of 200–400 nm and few compounds in 430–455 nm range. But researchers are more interested to develop organic rather than inorganic photochromic materials because their response in 400–700 nm visible region. The use of photochromic and thermochromic colorants in making smart materials such as medical thermography, photochromic lenses [1], food packaging materials, liquid crystal alignment [2, 3], optical data storage [4, 5], non linear optics [6, 7], photo switching, molecular photonic devices and in photochromic polymers [8] are well known. There is demand for application of photochromic and thermochromic colorants in making smart textiles, which are designed to sense and respond to external environmental conditions and stimuli. Photochromic and thermochromic colorants are prone to change their colors temporarily and reversibly in presence of UV light, visible light, acids [9], alkalis, water, mechanical strain, temperature and in electric field. These dyes became colored when exposed to these environmental conditions temporarily and revert back upon disappearance of external environment. The photochromic dyes [10] are categorized as inorganic and organic molecules. In inorganic types the important are metal oxide, alkaline earth metals, sulphides, copper compounds and mercury compounds. The organic types are effective and environment friendly and they belong to the families of spiropyrans, spirooxazines, chromomenes, fulgides,

fulgimides and diarylethenes. Spiropyrans, spirooxazines and chromomenes are sensitive to thermal effect and reverse to colorless state under heat or visible light however fulgides, fulgimides and diarylethenes are thermally stable. Out of these spiropyrans are having more scientific interest than any other class.

Spiro compounds have pyran ring and linked to another heterocyclic ring through spiro group. Spirooxazines molecules contain nitrogen atom at the place of carbon in spiro group. These molecules (colorless) have non planer structure and that inhibit delocalization of  $\pi$  electrons in the molecules. In presence of UV light, molecules absorb photon energy and breaking of  $-C-O-$  bond in pyran ring takes place and there is formation of colored planar structure molecule. The planarity of molecule allow delocalization of  $\pi$  electrons and molecule become colored. This is short term phenomenon and after absorption of heat or visible light molecules convert into original structure (colorless) as shown in **Figure 1**.

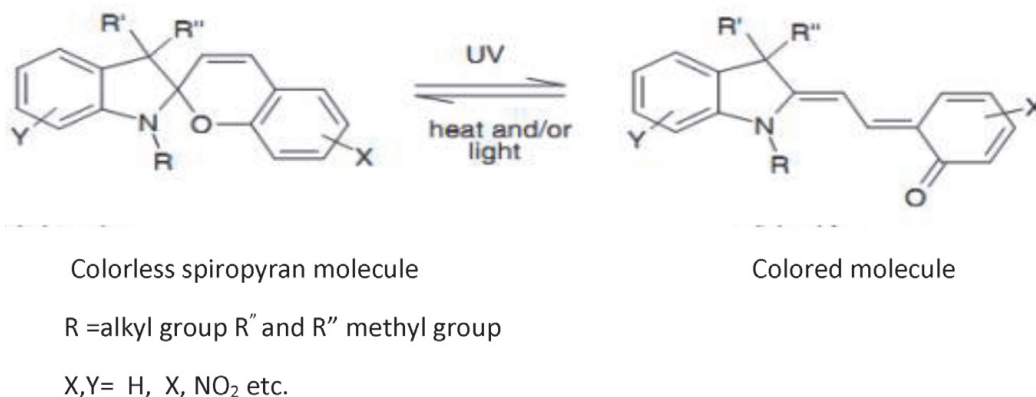
The photochromism [11, 12] may be defined as a reversible light –induced color change or reversible transformation between two different molecular structures with different absorption spectrum in reversible manner due to electromagnetic radiations. Photochromic materials are kind of chromic materials in which photochromic and thermochromic materials are of paramount importance. In photochromism the colorless molecule became colored in presence of UV Portion of light however in the thermochromic molecules heat is responsible for change of color [4].

The general physicochemical reaction of photochromic molecules are as given in equation no. 1 [13, 14].



$\lambda_1$  and  $\lambda_2$  are the wavelength of maximum absorption by corresponding molecules and  $h \nu_1$  and  $h \nu_2$  are the energy absorbed by the molecules during transformation.  $\nu_1$  is the frequency of wave in U.V. region and  $\nu_2$  is the frequency of wave in either U.V. or visible region and  $\Delta$  is heat requirement. The factors which influence reaction 1 are [15].

- Wavelength of incident light
- Speed of recovery or fatigue resistance
- Long term stability of molecule to produce high number of cycles



**Figure 1.**  
Conversion of colorless spiropyran molecule into colored molecule.

## 2. Types of photochromism

### 2.1 Positive photochromism

In this photochromism photochromic molecule absorb UV light whose  $\lambda_{\max}$  falls in UV region and colorless molecule became colored and on reversal during bleaching process in visible wavelength it become colorless.

### 2.2 Negative photochromism

It is opposite to the positive photochromism instead of coloration discoloration observed on exposure to UV light i.e. the original molecule is colored and after exposure to UV light it loss their color.

### 2.3 (c) Photo responsible materials

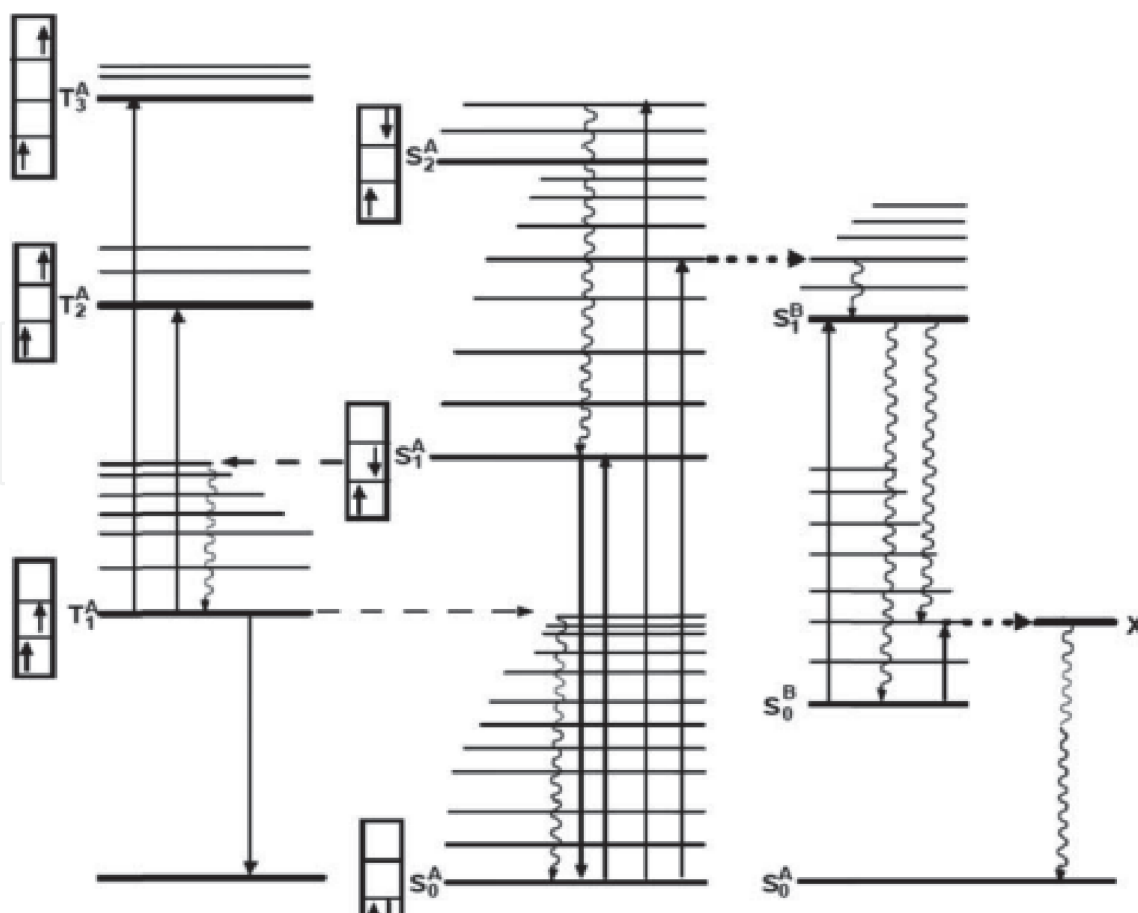
In photochromic reactions, there is conformational modifications in the structure of photochromic molecules and due to that there are change in physico-chemical properties of two form of the photochromic molecules and the change observed in physico- chemical properties of both molecules (colored and colorless) are called photo responsible materials [16].

## 3. Mechanism of Photochromism

Photochromic reaction leads to change in electronic absorption spectra of molecules. The formation of new absorption band due to transition of electrons from various vibrational levels in the excitation of colorless molecules from  $S_1A$  level to excited state  $S_1B$  after absorption of energy photon in UV region [17] and then after the colored molecules deactivated to ground state  $S_0B$ . Subsequently there is spontaneous energy release process and molecules come to original  $S_0A$  ground state. Thermodynamically the molecules in B state due to higher energy are less stable and after releasing energy became colorless and more stable. The transition from state B to A takes place via a transition state X, whose energy is higher than the triplet state of colored form  $S_0B$  and it is thermally activated. There are six mechanism which responsible for the photochromic effect and they are,

- Triplet triplet photochromism.
- Hetrolytic cleavage
- Hemolytic cleavage
- Trans-cis isomerisation
- Tautomerism
- Photodimerisation

The transition of molecules after absorption of light in different energy levels is shown in **Figure 2**.



**Figure 2.**  
Representations of electronic, radiative and non radiative transitions in photochromic materials.

#### 4. Classification of photochromic materials

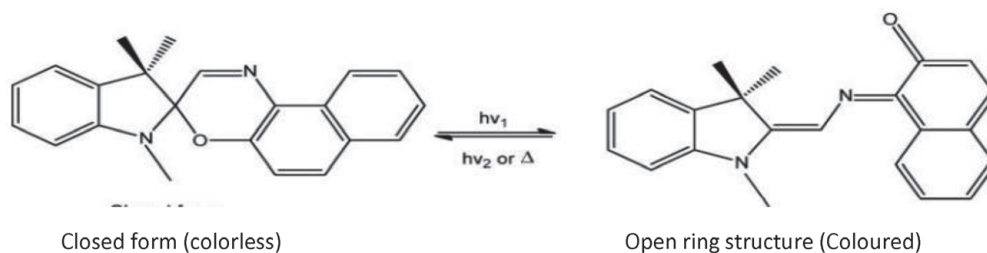
Classification of photochromic materials are based on back reaction i.e., if it from colored state to colorless state is brought in the presence of light then it is called P type photochromic materials (**Figure 1**), where as if back reaction occurred due to heat energy, it is called T type of photochromic materials.

The p type photochromic materials exist in two reversible forms upon irradiation and have good thermal stability with better fatigue resistance. In p type photochromic materials [18, 19] during exposure to UV light ring opening takes place. After ring opening, molecules absorb visible wavelength light and became colored. This state of molecule is temporary and again it became closed ring system (colorless). Most of the p type photochromic materials do not follow trans- cis isomerism and which follow they have they have open ring structure in colorless form after exposure to UV light they became closed ring structure (colored). The reverse reaction i.e. ring opening is promoted by visible wavelength [20, 21].

In T type photochromic materials photochromic reactions takes place due to thermal irradiation or by photo irradiation with visible light [ 22, 23]. It shows reversible equilibrium between trans and cis isomerism of different stability. In T type photochromism there is no breaking of bonds occur however there is rearrangement of electrons between energy levels and alteration in geometric arrangement of the molecules. The thermal reversal of molecule takes place in dark. The important T type photochromic materials are perimidinespirocyclohexadienes, spirodihydroindolizines and anils. There are some requirement in T type photochromic materials which are,

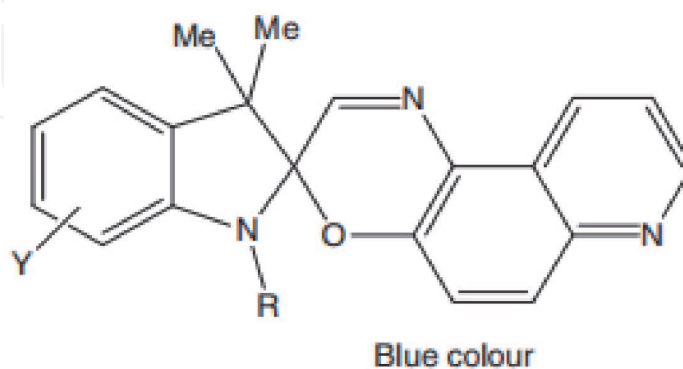
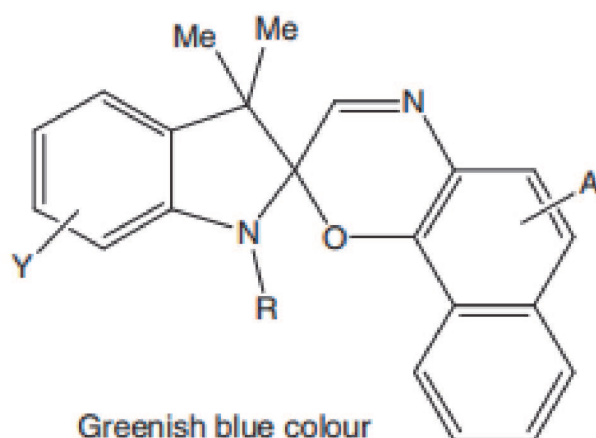
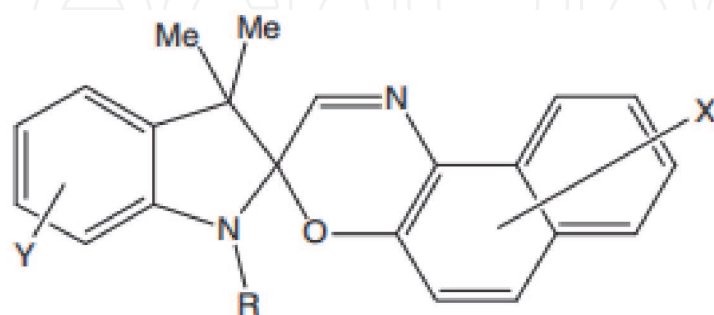






**Figure 4.**

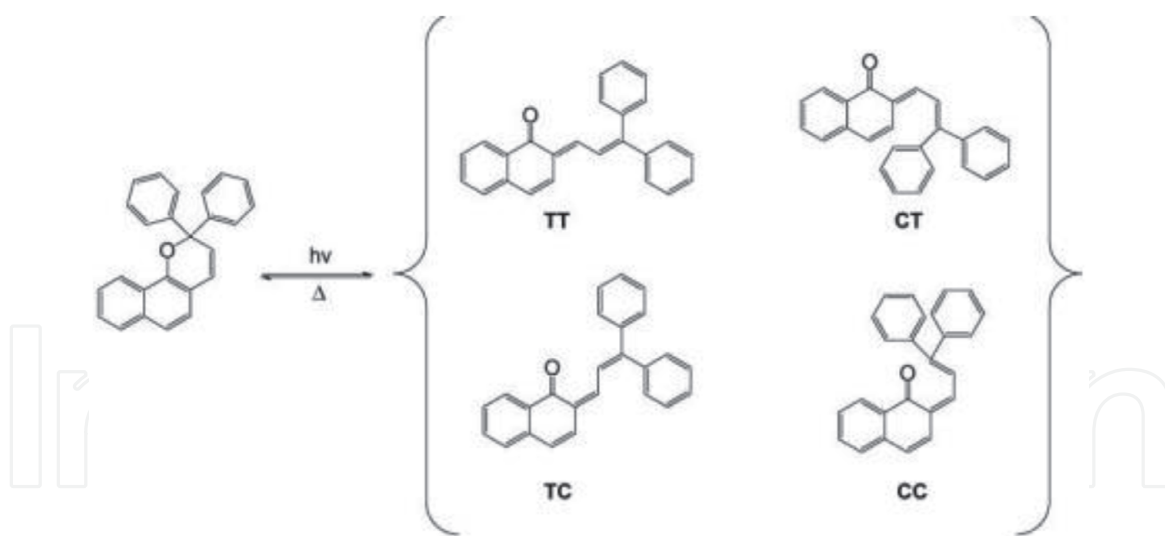
Photochromic reaction of spirooxazines (from closed ring structure to open ring structure).



**Figure 5.**

Different structures of photochromic oxazine ( $R$  = alkyl group,  $X$  = H, amino, hetaryl  $Y$  = H, halogen,  $a$  = electron acceptor).

photo coloration. The different chemical structure of spirooxazines are shown in **Figure 5**. The presence of different alkyl groups  $R$  at nitrogen atom decide the fading and color strength of molecules [32]. In 1990s, plastic photochromic



**Figure 6.**  
 Photochromic reactions of naphthopyrans (TT *trans-trans*, CT *cis-trans*, TC *trans-cis*, CC *cis-cis*).

ophthalmic lenses were manufactured using spiropyrans. Other applications are photochromic inks, dyes and various cosmetics items.

### 5.3 Naphthopyrans/benzochromenes

It has wide commercial applications such as in plastic photochromic lenses. The photochromism mechanism of benzo and naphthopyron are similar to spiropyrans [33]. They all have breaking of C—O bond in the pyron core. The photochromic reactions of naphthopyrans/benzochromenes by the light induced ring opening is shown in **Figure 6**. The ring opening of molecular structure produces more planer structure with greater conjugation of bonds. The planer conjugated structure are capable to absorb visible region wavelength and produces color. The naphthopyrans are less sensitive to temperature.

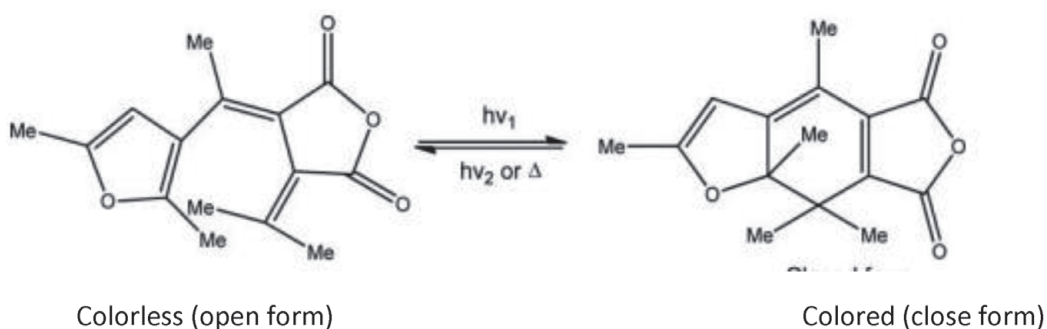
## 6. P type photochromic materials

The p type photochromic materials have applications in computing, optical circuitry, memory technology and in ultra high density storage devices. The p type photochromic materials are of two types such as fulgides and diarylethenes. In p type photochromic molecules, the open ring structures are colorless and closed ring structures are colored [34–36].

### 6.1 Fulgides

The fulgides and fulgimide family belong to P type photochromic materials (**Figure 7**). In 1905 Stobbe synthesized some photochromic fulgides named as phenyl substituted bismethylene succinic anhdides. Heller et al. [37] developed a compound succinimide called fulgimide. It exhibit good photochromic properties. It has shows absorption spectrum of both forms with efficient photoreaction, thermal and photochemical stability. The application of fulgides are In optical switches, sensors, dye inks and memory disks.





**Figure 7.**  
Photochromic reaction of fulgides.

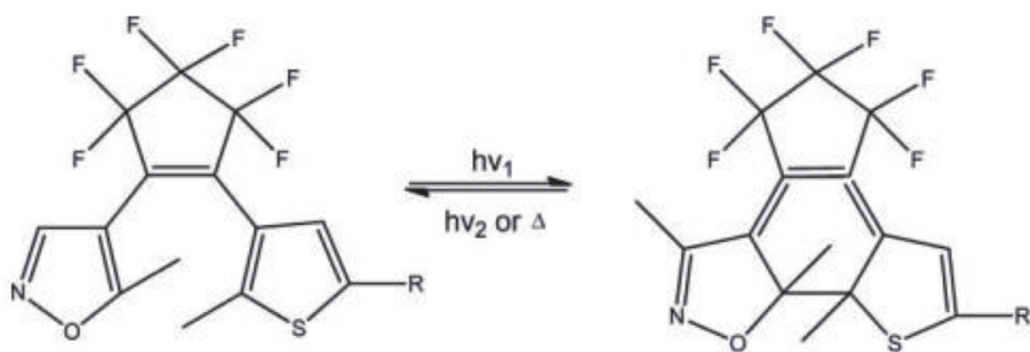
## 6.2 Diarylethenes

It has hetrocyclic five membered rings such as thiophene or benzothiophene rings and undergo thermally irreversible and fatigue resistant photochromic reactions as shown in **Figure 8**. The thermal stability of both isomers of diarylethenes are due to presence of aryl groups and when aryl groups are furan or thiophene, the closed form is thermally stable. However diarylethenes exhibit thermally reversible reactions when the aryl group is phenyl or indole.

## 7. Effect of temperature

Surrounding temperature influence the performance of photochromic dyes. The bleaching effect of photochromic dyes are accelerated by the temperature of sample [38–40]. Dulic et al. find that ring opening process of diarylethene is temperature dependent whereas ring closing process shows only slight dependence. Ortica [41] reviewed the effect of temperature on the characteristics of various photochromic materials such as spirooxazines, chromenes and arylethenes, which are as follows,

- In thermo reversible photochromic materials, thermal bleaching increase with increasing temperature.
- Specific temperature can induce spontaneous coloration in thermochromic materials however decreasing temperature will not help in reducing the complexity of photochemical reactions.



**Figure 8.**  
Photochromic reactions of diarylethenes.

- In nitro substituted chromenes, it was observed that temperature variations influenced the coloration which developed due to substitution in the molecular structure.
- A combination of photochromic and thermochromic materials possess synergistic effect and give superior performance at high temperature.

## 8. Applications in textiles

### 8.1 By exhaust dyeing

#### 8.1.1 Method 1

In this method the photochromic dyes are dispersed with dispersing agent [42] and dissolved in water keeping the M:L ratio of dyeing 1:50. The dyebath pH is maintained between 4.5–5.5. The dyebath temperature raised from 40 °C to 60 °C with 2 °C/min gradient and then after it is reduced 1 °C/min and final dyeing temperature is maintained at 90 °C and dyeing is continued at this temperature for 60 min. After completion of dyeing, soaping, rinsing and washing are done to improve fastness properties.

#### 8.1.2 Method 2

In this method the photochromic dyes can be applied as a disperse dye on polyester fabrics by exhaust method of dyeing [43]. The dye is pasted with acetone and then stirred in water with dispersing agent (1%) keeping the M:L ratio of dyeing 1:50. The pH of the dyebath is maintained between 4.5–5.5. The aqueous dyebath is boiled to evaporate acetone, subsequently temperature is raised to 120 °C and dyeing is done at this temperature for 45 minute. After dyeing reduction clearing treatment is given at 70 °C for 20 min. and finally sample is soaped, rinsed and washed.

### 8.2 By continuous dyeing

In this method photochromic dyes are dissolved in acetone and then mixed with binder solution and padded with padding mangle at appropriate pressure. After padding fabric is dried at 80 °C and cured at 140 °C for 3.0 minute in hot air oven or stenter machine [44].

#### 8.2.1 As a disperse dyes in printing

Photochromic dyes can be used as a disperse dyes which are insoluble in water. The photochromic dyes are disperse with dispersing agent and wetting agent of anionic nature. The dye dispersion is milled on a roller mill by using ceramic balls in glass jar. The dye dispersion is mixed with sodium alginate thickener solution to get printing paste. The polyester or nylon fabric can be printed with printing paste. The fabric is dried at 100 °C and cured at 140 °C for 5 minute. After reduction clearing treatment, printed samples were soaped with nonionic detergent and finally neutralization is done.

### 8.3 Application of thermochromic materials

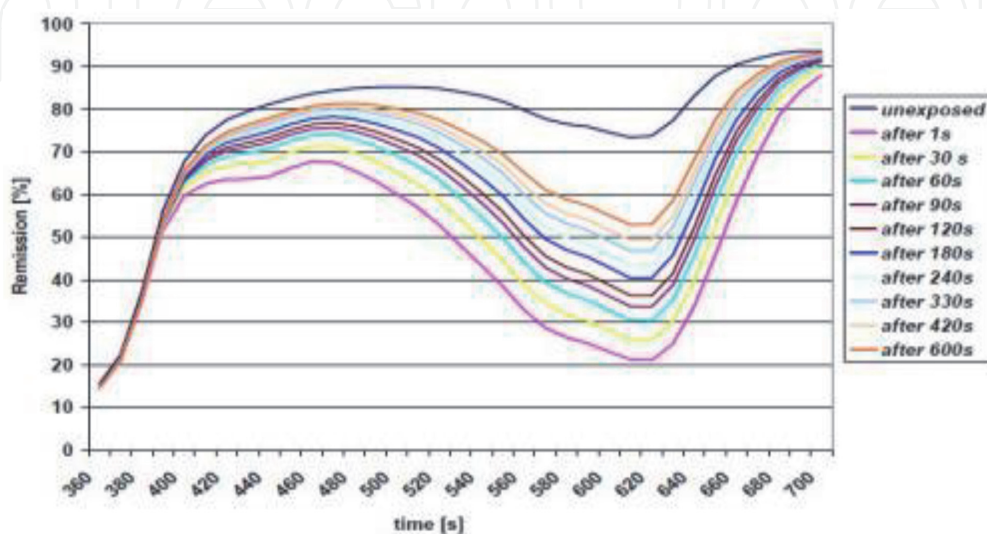
Photochromic materials have applications in both textiles and non textiles. In the textile field new fashionable colors in T-shirts, hand bags and caps are produced by dyeing with photochromic dyes. There is application of photo colorable textured yarn in knitting, weaving and embroidery. Polypropylene threads are produced by mass coloration by adding photochromic dyes in melt polymer solution, which on exposure to U.V. light produces different colors. Photochromic colorants are also used in developing camouflage patterns for military protective clothing. The patterns change their colors on exposure to sunlight and match with surrounding environment. Photochromic microencapsulated fabrics are produced which change their color on exposure to sun light. In non textile field photochromic materials are used in ophthalmics, surface coating applications and dye lasers.

### 8.4 Color measurement

Due to reversible color changing properties of photochromic dyes, it is very difficult to measure color value of the shade produced due to photochromic effect. For measuring the color value, it is essential to control several parameters such as temperature and time interval between U.V. irradiation of sample and measurement. A.F. Little et al. [45] developed a technology to measure the color value of photochromic textiles using independent U.V. irradiation with traditional spectrophotometer. The temperature of sample measuring cabinet was controlled by localized air heating system. The temperature of sample cabinet is maintained at 24 °C and time interval of 30 sec. is kept between irradiation and measurement of sample which can be seen in **Figure 9**.

### 8.5 Washing fastness test

Due to dynamic color change properties of photochromic dyes, it is difficult to measure the fastness properties. The traditional assessment method of color fastness using gray scale standards [46] are not appropriate, therefore it is measured by comparative test method. In this method we measure the color difference of sample before and after wash and compare with color difference before U.V. irradiation and after 1 min. Exposure to U.V. irradiation. The level of photo coloration



**Figure 9.**  
Color measurement (color bleaching) of photochromic dye fabric samples in 30 sec. Time interval.

developed by U.V. irradiation varies with photochromic colorant classes. It was revealed that in selected spirooxazine colorants the degree of photo coloration increases with initial washing and subsequently decreases. In case of naphthopyrans, the degree of photo coloration decreases continuously with successive number of washings. In case of printing the washing fastness more depends on binder quality.

## 8.6 Light fastness/photostability

The conventional method of exposing the sample to accelerated fading instruments (Xenotest or MBTF) is not applicable to photochromic colorants. In photochromic colorants due to dynamic color change properties, for light fastness measurement a normalized value of color value to be calculated. The normalized value is defined as the degree of photo coloration after a particular time of exposure on the xenotest instrument to the fraction of initial degree of photo coloration i.e.,  $\Delta E/\Delta E_0$ .

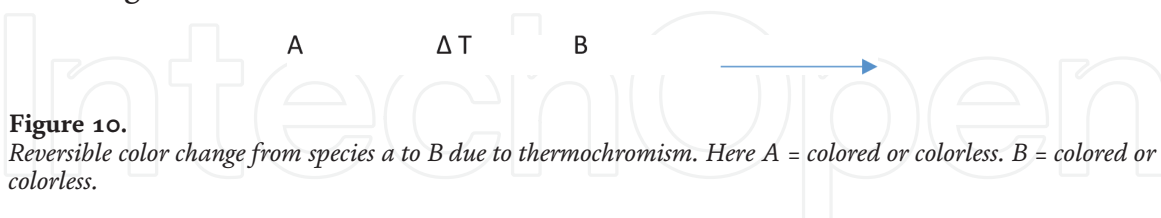
$\Delta E_0$  = color difference before and after U.V. Irradiation without exposing to xenotest instrument.

## 9. Thermochromism

Thermochromism may be defined as the reversible change in the color of compound due to temperature change [47]. The phenomenon of thermochromism may occur even in small temperature interval. The thermochromism can be depicted as shown in **Figure 10**.

The following type of materials can exhibit thermochromism properties [48].

- Organic compounds
- Inorganic compounds
- Polymers
- Sol gels

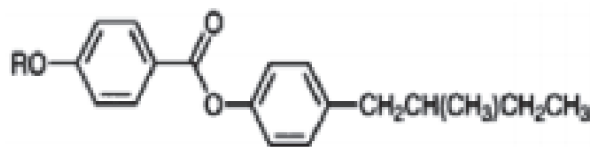


## 10. Organic compounds

It may occur as a result of equilibrium between molecular species such as acid – base, keto-enol and different crystal structure. The organic thermochromic materials have application in fibers, optics and optical sensors. The organic thermochromic compounds show sharp color change due to temperature variation. The different organic thermochromic compounds may be classified as follows.

### 10.1 Liquid crystals

Some organic materials when pass from crystalline solid to isotropic liquid state, they form stable intermediate phases (mesophase). Transitions between phases are



**Figure 11.**  
*Chiral molecules in cholesteric mesophases form.*

brought either by influence of temperature or solvent respectively [49]. The cholesteric (chiral nematic) are the most important type of liquid crystals for thermochromic systems. Here molecules are arranged in helical form. The reflection of light by liquid crystals are influenced by temperature. The pitch of the helical arrangement of the molecules decides the wavelength of reflected light [50]. The liquid crystals are microencapsulated to get the thermochromic effect. They are applied to the fabric with the help of binder (**Figure 11**).

## 10.2 Stereoisomerism

Organic molecules which possess stereoisomerism, show thermochromism especially ethylenes. When change in temperature takes place, molecules of these compounds switch between different stereoisomers. Generally the required temperature for thermochromism to take place is more than 150 °C. So they can not be used for textiles application [51].

In another case, the crystal violet lactone is pH dependent rather than temperature dependent. At pH above 4.0 crystal violet lactone is colorless and below 4.0 pH, it is violet.

### 10.2.1 Rearrangement

Due to molecular rearrangement of organic compounds more conjugated structure resulted and formation of new chromophores take place. Such type of molecular arrangement occurs due to temperature variation, change in polarity of the solvent or pH of the solution.

## 11. Inorganic thermochromic system

Thermochromic behavior is exhibited by solid or liquid inorganic molecules. In such type of molecules thermochromic properties are due to following mechanism [52],

- Phase transition
- Change in ligand geometry
- Equilibrium between different molecular structures
- Change in the number of solvent molecules in the co-ordination sphere.

These compounds show thermochromic properties at high temperature (150 °C), therefore they are not suitable for textile application.



## 12. Microencapsulation

In reversible thermochromic compounds a colorless dye precursor and color developer both are dissolved in hydrophobic non volatile organic solvent and resulted solution is encapsulated [53]. On heating, melting of organic solvent occurs and there is appearance of color in thermochromic compound. On cooling solvent solidify and system comes to original color. Microencapsulation has some advantages that it protect sensitive coloring agents from external environment and allows several thermochromic colorants to be combined together and produces several narrow color ranges.

The organic solvents used in microencapsulation are alcohols, hydrocarbons, ester, ketones, thiols and alcohol –acrylonitrile mixture. The important thermochromic colorants are N-acyl leuco-methylene blue derivatives, fluoran dyes and diphenylmethane compounds. There are large number of compounds work as color developers such as phenol derivatives specifically bisphenol A and bisphenol B. In some recent work there are use of 1,2,3 triazoles such as 1,2,3 benzotriazole, dibenzotriazole, thioureas and 4 hydroxy coumarin derivatives.

## 13. Application of thermochromic pigments on textiles

### 13.1 By exhaust method

- Cationic agent 5–8% (owf)
- Thermochromic Pigments 10–15% (owf)
- Non ionic leveling and dispersing agent 10–15% (owf)
- Acrylic Binder 10–15%

Thoroughly pretreated fabric taken in water keeping M; L ratio 1:20 and we add cationic agent at temperature of 60 °C and run in the aqueous media for 15 min to get positive charge on fabric. After treatment fresh water is taken in the bath and thermochromic pigments are added, temperature is maintained at 70 °C run fabric for 10–15 min. During dyeing non ionic dispersing agent and leveling agent are added. Finally acrylic binder is added and we run the fabric at 70 C for 15 min. The fabric sample is soaped and washed. In microencapsulated fabric the melting temperature of solvent control the temperature at which decolonization/colorization of thermochromic colorant occur.

### 13.2 Continuous method (In solution)

- Cationic agent 10–15%
- Thermochromic pigment 50%
- Nonionic dispersing and leveling agent 10–15%
- Acrylic binder 20%



### 13.3 Application technique

Pad  $\longrightarrow$  Dry  $\longrightarrow$  Cure  
At room temperature (expression 70%) 80 C (3 min.) 140 C (2–3 min.)

### 13.4 Printing recipe

- a. Thermochromic Colorant 20 part
- b. Emulsion thickner 76 part
- c. Acrylic binder 4 part

## 14. Thermochromic cellulose fiber

Marcin Rubacha et al. [54] developed a method to get thermochromic pigment added cellulosic fiber called Lyocell. 1–10% chromicolor AQ-INK magenta type 27 pigmentation was used as a thermochromic modifier.

## 15. Photochromic polymers

A photo chromic polymer has photo chromic, chromophoric groups inside the polymer backbone chain. The chromophoric group respond to external radiative stimuli during the photo-irradiation of the polymers, there are change in physical chemical and optical properties of polymer in reversible manner. In 1967 Lovrien attempted to produce a polymer chain with photo irradiation sensitive properties by incorporating azo chrysophenine into polymethacrylaic acid. In 1970 Agolini and Gay [55] investigated photo chromic polyamides incorporating azo benzene. The potential application of photo chromic polymers are in photo chromic glasses, UV sensors, halographic recording media, non linear optics and memory devices.

## 16. Textile printing

Feczko et al. [56] printed cotton fabric by using photo chromic dyes based on Ethyl cellulose –spirooxazine nano particles with light absorbers. They use micro-encapsulation technique to incorporate photochromic colorants. Vikova [57] applied photo chromic pigments on different fabrics such as cotton, polyester and poly acrylonitrile by screen printing using pigment printing method.

## 17. Sol –gel coating methods

A sol is dispersion of solid particles in liquid where particles are sufficiently small (0.1–1.0  $\mu\text{m}$ ). Due to inorganic nature [58] of layers formed by the sol gel process it possess strong wear resistance and very thin nanometric sized layers. The preparatory materials in preparing sols are inorganic metal salts or organometallic compounds. The preparatory materials are submitted to series of hydrolysis and polymerization reactions to produce colloidal suspension or sol, once the polymerization is completed the colloidal form of the sol developed. Cheng et al. [59–61]

prepared silica as a matrix material for fixing the photo chromic dye 5 Chloro 1,3 dihydro-1,3,3 trimethyl spiro on the surface of wool fabric through sol-gel process.

## 18. Mass coloration

Mass coloration or dope dyeing is method in which colorants are added in the spinning composition before extrusion of filaments. Photo chromic polypropylene [62] thread was prepared by this method. Vikova et al. [63, 64] prepared polypropylene multi filaments by adding photo chromic pigments during dope preparation. Concentration of photo chromic pigments was 0.25,0.5,1.5 and 2.5% by weight.

## 19. Conclusion


Photochromic and thermochromic materials presenting a new field of research which are yet not fully explored in field of textiles and other allied fields in context of application and durability. The photochromic and thermochromic colorant occupied a niche position in colorant industry. Their application mainly concerned with fashion, leisure and sports garments. Incorporation of photochromic colorants in nanofiber based photochromic textiles can be made use of in smart textiles because of their ability to react with external stimuli, which may work as chromic sensor. Efforts can be done to improve properties such as light fastness and simplistic application procedure. Presently two types of thermochromic system liquid crystals and molecular rearrangement types have successful commercial applications. Japan and U.S, A had developed wide range of thermochromic and photochromic materials, but innovations are required for commercial applications. The factors influencing the equilibrium between colored and colorless form of colorant has to be further explored. Presently only small number of commercial organizations are engaged in research and development work of improved compositions. There is need of study with respect to formulations, encapsulations and application of photochromic and thermochromic materials in textiles.

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