Synthesis, Characterization and Dyeing Performance of Reactive Dyes

The present thesis comprises of four chapters as arranged in following manner.

Chapter 1 – Introduction and literature survey of reactive dyes

Chapter 2 – Experimental work

Chapter 3 – Characterization of synthesized dyes

Chapter 4 – Application of Reactive dyes

Chapter 1

Introduction and Literature survey of Reactive dyes

Dyes are often a coloured organic chemical or blend that can be used to colour a substrate such as cloth, paper, plastic or leather. Until the middle of the twentieth century, most dyes were derived from plants or animals. Indigo, Tyrian Purple, Alizarin, Cochineal, and Logwood were utilized as natural dyes. The dyes and textiles sector is growing at an extremely rapid rate. The development of the dyes sector and chemical intermediates plays an important role for market growth. The development of reactive dyes is something of a turning point in the history of synthetic dyes. Reactive dyes are the only dyes that form a kind of covalent bond with material during the dyeing process. Several researchers develop and modify structure of reactive dyes.

Chapter 2

In this chapter six series of reactive dyes were synthesized, Phenyl urea derivatives were used for preparation of series I to IV, 2-amino-5-naphthol-7-sulfonic acid(J-acid), 8-Amino-1-naphthol-3,6-sulfonic acid(H-acid), 2-amino-8-naphthol-6-sulfonic acid (Gamma acid) and 1- hydroxy-6-methylamino-3-naphthalenesulphonic acid(methyl J acid) are used as coupling component. For the synthesis of series V 4-amino-4'-hydroxy benzylidene acetophenone(4-amino-4'-hydroxy chalcone was used and K- acid, H acid,J—acid, N methyl J-acid, N-phenyl J-acid, Gamma acid , and peri acid were employed as coupling materials. In series VI mono azo reactive dyes of hot brand were prepared with diazotized o-anisidine was coupled with various cyanurated 4-amino-4'-hydroxy benzilidene acetophenone, cyanurated coupling component including H-acid, K-acid, Gamma acid, N-phenyl J-acid, J-Acid and Peri acid. To determine the purity of the dyes, thin layer chromatography was used. Each dye was assessed using elemental analysis and some of the chosen dyes are assessed using IR and ^{1}HNMR spectrum characteristics.

Chapter 3

In this chapter characterization of synthesized dyes was described. For characterization of reactive dyes following methods were used.

(i) Thin Layer chromatography(TLC):

In Thin Layer chromatography (TLC) the basic equipment is simple and exorbitant and time of evolution is short. Following solvent were used.

Solvent Composition: DMF + Benzyl alcohol + Water (2:3:3)

The spotting solvent: DMF

Temperature: 31-32 ºC

(ii) Ultraviolet (UV) and Visible spectroscopy:

The wavelength (λmax) was measured on primer Spectrophotometer SS 5100. It is observed that the wavelength λmax. was recorded around 420 to 590 nm of synthesized dyes.

(iii) Infra-Red (IR) Spectra:

The IR spectrum data are used for dyes characterization. On the careful observation of all synthesized dyes it is observed that for -OH stretching vibration found around 3300-3400 CM^{-1},-NH_2 stretching vibration found around 3200-3300 cm^{-1}, C-Cl stretching vibration found around 600-800 cm^{-1},N=N stretching vibration found around 1543-1564 cm^{-1}, triazine stretching vibration found around 1498-1499 cm^{-1},C-H stretching vibration found around 2928-2936 cm^{-1},-SO_3Na stretching vibration found around 1035-1260 cm^{-1},C=O stretching vibration found around 1675-1684 cm^{-1},-NO_2 stretching vibration found around 1360-1370 cm^{-1}.

(iv)Nuclear Magnetic Resonance Spectroscopy:

The ^{1}H-NMR spectrum data are used for structural characterization of the dyes. On the careful observation of all synthesized dyes, it is observed that the δ ppm values of all dyes are in good agreement with the corresponding structures.

Chapter 4

All synthesized dyes were applied on silk, wool and cotton fabric. Dyed fabric of cotton, wool and silk were mounted on shade cards 1 and 2 for cotton, 3 and 4 for wool, and 5 and 6 for silk. Series-I to VI gives orange, golden yellow, red and several red tone colours. In series-I as a coupling component J-acid was used gives orange colour, in series-

II as a coupling component H-acid was used gives red colour, in series-III as a coupling component Gamma acid reddish orange, in series-IV as a coupling component orange to reddish brown colour. In series V as a coupling components J- acid, N-methyal -J-acid, N-phenyl-J-acid, K-acid, Peri acid were used gives orange to reddish brown colour. In series VI as a coupling components H-acid, Gamma acid, J-acid, N-phenyl J-acid, K-acid and peri acid gives purple red to marron colors.

All synthesized dyes were applied on Cotton, silk and wool, the fixation value of dye on cotton fabric range between 76% to 89%. The several parameters like temperature effect, salt concentration effects, dyeing time effects and pH effects on exhaustion and fixation were studied. From the study, 60 ^{o}C temperature is more suitable for getting higher fixation value on cotton fabric. 100 g L^{-1} Salt concentration gives higher fixation value of dyes. 60 Minutes of time for dyeing gives highest fixation value of dyes. Dyeing at 10 pH gives best fixation value of dyes on cotton fabric.

Dyes have good to excellent lightfastness characteristics, good to very good washing fastness properties on cotton, silk and wool. The dyes have good penetration and affinity of the synthesized dyes to the cotton, silk and wool fabric.This is because reactive colors strongly cling to the fabric and have an affinity for it. Good light fastness may be attributed to the conjugated systems better resonance stability and the dye structure within the fabric being converted from a scattered form to an aggregated or crystalline one. Synthesized dyes Z1-Z10 have good antibacterial activity.

The reactive dyes have good heat stability, according to the TGA study data. They can be used in high-tech applications like colour filters because of their excellent thermal stability.

Dyes are tested with multifibres, no staing observed on several fibers like wool, acetate, nylon and polyester. Therefore advisable for to be used to all kind of fibers. The stability of reactive dyes also evaluated at 40 ^{0}C. Stability of dyes gives good results, indicates that it has good stability at 40 ^{0}C.