Summary

Synthesis and application of new reactive dyes containing ureido cross-link.

In this work seven homobifunctional, homopolyfunctional ureido reactive dyes (Disazohomobifunctional & polyazohomobifunctional) were synthesized. These dyes based on synthesis of ureido diamine intermediate (DADPU), which synthesized by using harmless and economical method. These dyes divided into four types according to their reactive groups such as follows.

- 1- Bis dichlorotriazine (DCT) ureido reactive dyes.
- 2- Bis monochlorotriazine (MCT) ureido reactive dyes.
- 3- Bis monochlorotriazine/sulphatoethylsulphone (MCT/ SES) ureido reactive dye.
- 4- Bis sulphatoethylsulphone (SES) ureido reactive dye.

The strategies of the synthesis of these dyes were discussed as in **Schemes (3.1 – 3.5)**. From which different cyanorated coupling components were initially prepared by the substitution reaction of cyanuric chloride with (γ acid/ or H acid) coupling components followed by diazotization with bisdiazonium salt of ureido diamine intermediate.

The characterization of these dyes was recorded by using different methods as

C, H, N, and S microanalysis, IR, ¹HNMR, and UV/vis Spectral data are recorded in Experiintal part of the thesis and indicated in table (3.1) and figures (3.1 -3.14).

Dyes structures: -

The Chemical structure of ureido diamine intermediate and synthesized reactive dyes 1-7 are given as follows:

Dye NO.	Dye Structure
1	CI NH OH OH OH HN CI NaO ₃ S SO ₃ Na NaO ₃ S SO ₃ Na
2	CI N N OH N=N-W-N-N-N-N-N-N-N-N-N-N-N-N-N-N-N-N-N-
3	NaO ₃ S — NH OH OH NH N=N — N=N — NaO ₃ S SO ₃ Na
4	NaO ₃ S — NH NH NH NH NH NH SO ₃ Na NaO ₃ S
5	NaO ₃ SOCH ₂ CH ₂ O ₂ S Nh Nh OH OH NH NH C - NH - N=N - NaO ₃ S SO ₃ Na NaO ₃ S SO ₃ Na
6	NaO ₃ S NaO ₃ S
7	NaO ₃ SOCH ₂ CH ₂ O ₂ S Na NaO ₃ S SO ₂ CH ₂ CH ₂ OSO ₃ Na NaO ₃ S SO ₃ Na

The ureido reactive dyes were applied to 100% cotton fabric to evaluate their dyeing parameters, exhaustion and fixation values. The exhaust dyeing method was used as an application method. The effect of salt concentration and the temperature of the neutral exhaustion stage were initially examined. Also, the effect of variations in alkali concentration, the temperature of the fixation stage and dye concentration on the dye uptake and total fixation yield were investigated.

The results obtained are summarized in the following points:

- 1- All dyes shows high percentage of primary exhaustion at low salt concentration (20 g/L) the slight increase in primary exhaustion on increase the Sodium Sulphate concentration from 40 60 g/L reflected the high affinity of these dyes for cotton. This is due to the linear and planar stricture of the dye molecules, which enables close alignment with the chains of cellulose, resulting in increase the dye substantivity.
- 2- The different in exhaustion values between these dyes depend on the molecular weight and the planarity of the dye structure and the number of anionic group in the dye molecule. Primary exhaustion values were in the range (37-65) %.
- 3- Increasing the neutral exhaustion temperature from 40 to 60 °C, at any level of salt concentration led to a further increase in the primary exhaustion. This result indicated that the ureido reactive dyes are likely to present better substantivity on increasing temperature of the neutral exhaustion stage.
- 4- The results for the alkaline fixation stage in the presence of different concentration of Sodium Carbonate (5-25 g/L) were subsequently assessed. From the exhaustion and total fixation yield of bis(MCT) dyes 3, 4 and 6 increased by increasing of

sodium carbonate concentration, reaching maximum values at 25 g/L Sodium Carbonate and at 80 °C.

- 5- However the bis(DCT) dyes 1, 2 shows the lowest exhaustion and fixation yield when applied at the typical fixation temperature of 40°C, the decrease in exhaustion and fixation values on increasing alkali concentration possibly due to the hydrolysis reaction of the DCT groups.
- 6- Increasing the fixation temperature from 50 to 90°C for bis(SES) dye 7 resulted in a significant decrease in the dye exhaustion and fixation yield probably due to hydrolysis reaction of SES groups.
- 7- The bis(MCT/SES) dye 5 was less sensitive to variation in fixation temperature and exhibited highest exhaustion and fixation yield at 60°C, presumable due to its MCT and SES reactive group combination, which enable good fixation and resistant to hydrolysis reaction.
- 8- The extent of the exhaustion and total fixation of dyeing cotton fabric with different dye concentrations [1-5% on the weight of the fiber (owf)] was investigated. It was observed that the lower dye concentration exhibited a higher extend of exhaustion and total fixation than the higher depth of shade. This is believed to be due to the fact that increasing dye concentration would led to an increase in dye aggregation, which in turn reduces the dye penetration in the fiber. Additionally, at high dye concentration the number of available dye sites on the fiber decrease, resulting in a lower extent of exhaustion and fixation yield.
- 9- The fastness properties of (2% owf) dyeing on cotton using the synthesized uriedo reactive dyes were evaluated. All dyes exhibited good fastness to washing and perspiration depending

on the amount of dye fixed. The dry rubbing fastness was better than the wet rubbing fastness. However the light fastness of the dyed cotton was slightly inferior to that of typical reactive dyes, probably due to high molecular size and their lack of light fastness conferring substituents of electron withdrawing groups and or electron donating groups.