## ABSTRACT

Electrochemical and spectroscopic techniques have been used to investigate the behaviour of azo dyes, particularly the reactive azo dyes. The azo dyes studied were Methyl Orange (4-[4-(dimethylamino)phenylazo]benzenesulphonic acid), Orange II (4-(2-hydroxy-1-naphthylazo)benzenesulphonic acid) and Procion Red MX 5B, (Reactive Red 2) viz.5-[4,6-dichloro triazin-2-yl amino)-4-hydroxy-3-phenylazo, 2,7-naphthalene disulphonic acid. Reactive azo dyes are coloured organic compounds that contain one or two cyanuric chloride groups that can form a covalent bond between a dye molecule and a cellulosic fibre. These dyes are known pollutants in textile waste water and their colour persists notwithstanding conventional waste-water treatment. The polluted water then enters local water bodies. Procion Red MX 5B in particular is one of the more intensely coloured water soluble reactive dyes used in the textile industry.

Azo dyes can undergo two electron reduction giving a hydrazo derivative, or they could undergo 4e -reduction to give the corresponding aromatic amines, thereby decolourising the dye. The aim of the study was to electrochemically decolourize the reactive azo dyes in solution, and to this end the electrochemical reduction of Procion Red MX 5B dye was studied.

Cyclic voltammetry (CYV) is one of the techniques used in this investigation of the electrochemical reduction of Procion Red MX 5B. A three electrode system was used to study the electrochemistry of Procion Red MX 5B in the aqueous solution buffered with phosphate buffers of pH range 3-11. The working electrode was a glassy carbon disc, saturated calomel was the reference electrode and the counter electrode was either a spiral platinum wire or graphite rod electrode. The effect of pH on the reduction peak of the dye was studied and it was found that the reduction peak potential of the dye shifts to negative potentials as the pH increases. The effect of a second voltammetric cycle, and the scan rate, on the first reduction peak that appeared was studied. The interdependence of the peaks was studied by varying the potential window and changing the direction of the sweep. This was done in acidic media (pH 3 and 6), basic media (pH 8) and neutral media (pH 7).

Results obtained suggested that the dye was undergoing electroreduction into corresponding amines.

Bulk electrolysis on the dye at a potential indicated by the CYV studies at pH 7.06 yielded one of the aromatic amines expected from the electrodegradation of Reactive Red 2 which proved unequivocally that the electrochemical reduction of Reactive Red 2 leads to corresponding aromatic amines. The working electrode used for bulk electrolysis was a graphite rod. The degradation of the dye was monitored by UV-vis spectroscopy where it was found that the  $\lambda_{max}$  of the dye decreased systematically as electrolysis progressed. The voltammogram run on the completely electrolysed solution did not have the peak responsible for the reduction of the dye, confirming the UV evidence that the dye was completely electrolysed.

It was concluded that Procion Red MX 5B undergoes 4e reduction in phosphate buffer at pH range 3-11 at glassy carbon electrode. Isolation and identification of aniline by NMR from the completely electrolysed solution proved unequivocally that the dye Procion Red MX 5B can be electrodegraded into the corresponding aromatic amines. It is clear that decolourisation can be performed electrochemically and industrial applications are obvious. UV-vis spectroscopy complements the electrochemical and NMR evidence of decolourization.