CHAPTER - 2
GENERAL EXPERIMENTAL

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2.1 Experimental procedure

The method adopted for the present work is as similar as that described by Kipling [1]. Essentially a certain volume of dye solution is kept in contact with a known amount of the adsorbent at a constant temperature to attain equilibrium. At definite intervals of time, portions of dye solutions were taken out and the concentration was determined spectrophotometrically from which the amount of dye adsorbed was calculated.

2.2 Preparation of the adsorbent

All the chemicals used were of analytical grade except otherwise stated. In the latter case, wherever necessary, the materials were purified with the help of reported methods [2-4]. The adsorbents selected for the present work were alumina (neutral and basic), silica gel and graphite. The method followed for the preparation and the necessary processing is described below for each of these adsorbent separately. As the extent and behaviour of adsorption mostly depends on the method of preparation and the pretreatment given to the adsorbent [5-7], the adsorbent sample once prepared was used throughout the series. In case if it is necessary to prepare the sample again, the same procedure was followed. Further, particles of the same mesh size range were used for all the experiments with a given sample. The range of the particle size selected was as narrow as possible. This was different with different substances and was actually determined by trial experiments. The particle size of the adsorbent selected for these experiments were on the basis of their settlement on the bottom of the system, so that the portion of the solution could be taken out conveniently from the supernatant liquid. A brief description about the preparation and the prior treatment given to adsorbents are given below.

2.2.1 Silica gel

Chromatographic grade silica gel (Sisco Lab., Bombay, India) of 60 - 120 mesh size was treated with concentrated hydrochloric acid to remove surface impurity if any. It was then washed with boiling distilled water repeatedly till free from chloride ions and dried at 80 - 85 ^{o}C for 12 hours.

2.2.2 Graphite

Nuclear pure graphite pieces supplied by Bhabha Atomic Research Centre, Bombay were powdered, washed with boiling distilled water and then dried at 85 ^{o}C for about 16 hours, cooled and stored in a desiccator at room temperature.

2.2.3 Neutral alumina (Active)

Chromatographic grade neutral alumina (active) was supplied by Glaxo Laboratories (India) Ltd., Bombay, India. It was sieved for the particles of 100 - 140 mesh size and washed with boiling distilled water to remove the surface impurity if any. It was then dried at about 85 ^{o}C for 16 hours, cooled in a desiccator at room temperature.

2.2.4 Basic alumina (Active)

Chromatographic grade basic alumina (active) was supplied by Indian Drugs & Pharmaceuticals Ltd., Hydrabad, India. It was sieved and particles of 100 - 140 mesh size were selected for the present work. It was then washed with boiling distilled water and dried using the same procedure stated above and stored in a disiccator at room temperature.

Prior to the use of any adsorbent, it was heated at ~ 85 ^{o}C for overnight and then allowed to cool in the desiccator. Finally a fixed amount of the adsorbent was used for the purpose.

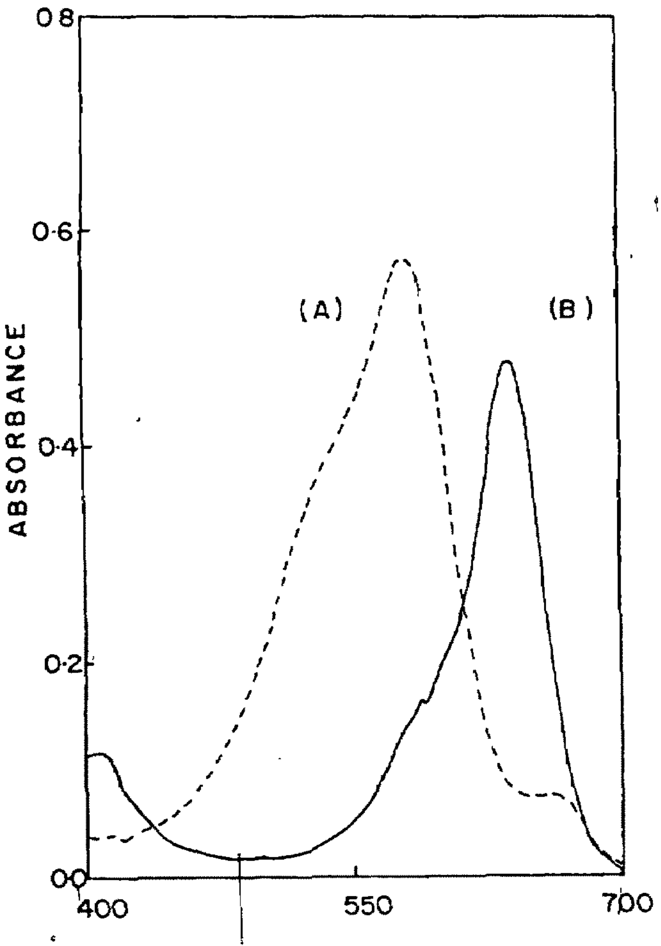
2.3 Preparation of adsorbate solutions

The dyes used in the present work were Acid Green g (Acid Green 25, Madhavdas Manilal & Co., Bombay), Acid Black 10 BX (Acid Black 1, Atul India), Ketone Blue A (Ciba of India Ltd.), Bismark Brown R (Basic Brown4, Ciba of India Ltd.), Astrazone Blue BG (Basic Blue 3, Chicka Ltd., India), Methyl Violet 6B (Basic Violet 1, BDH, England) and New Fuchsin (Basic Violet 2, Loba - Cheme IndoAustranal Co., Bombay). All the above mentioned dyes except Basic Violet 1 and Basic Violet 2 were the commercial textile dyes and were purified by dissolving in ethanol solution and recrystallised [8,9]. Stock solutions (1.0×10^{-3} M) were prepared by dissovling weighed quantities of dyes in deinoized water prepared from closed system glass apparatus. The experimental solutions were obtained by successive dilutions. In order to avoid the staining of the glass container of the stock solution [4], the container was washed before use, with a dilute solution of a suitable surface active agent (such as CTAB, SDS, etc.) and then rinsed with deionised water. Moreover fresh stock solutions were prepared every week.

2.4 Measurement of amount of dye adsorbed

A known amount of the adsorbent was kept in contact with a certain volume of the dye solution and the mixture was kept in a thermostated bath to attain equilibrium at a certain temperature. The ratio of actual amount of adsorbent and the volume of the dye solution (convenient for any experiment) was pre-determined by trial experiment. Thus 1.0g of neutral alumina was found suitable for 300 ml of Ketone Blue A ,and 200 ml of Acid Green 25 respectively whereas 0.3 gm of same adsorbate was suitable for 300 ml of Acid Black 1. In case of graphite adsorbate system 2.0g of graphite was suitable for 200 ml of dye solution. Due to higher adsorption capacity of silica however, a lesser quantity was needed. In actual experiment with 300 ml of dye solutions 0.3or0.5 g silica was found sufficient. These amounts were selected in experiments for the measurement of time growth of adsorption. In other experiments the quantity was adjusted appropriately according to the need. The system i.e., the dye solution in contact with the adsorbent was kept in a thermostat to maintain a constant temperature was shaken continuously using an

Fig.2.1a Absorption spectra of (A) Basic Violet 1 and (B) Acid Green 25 from aqueous solution.



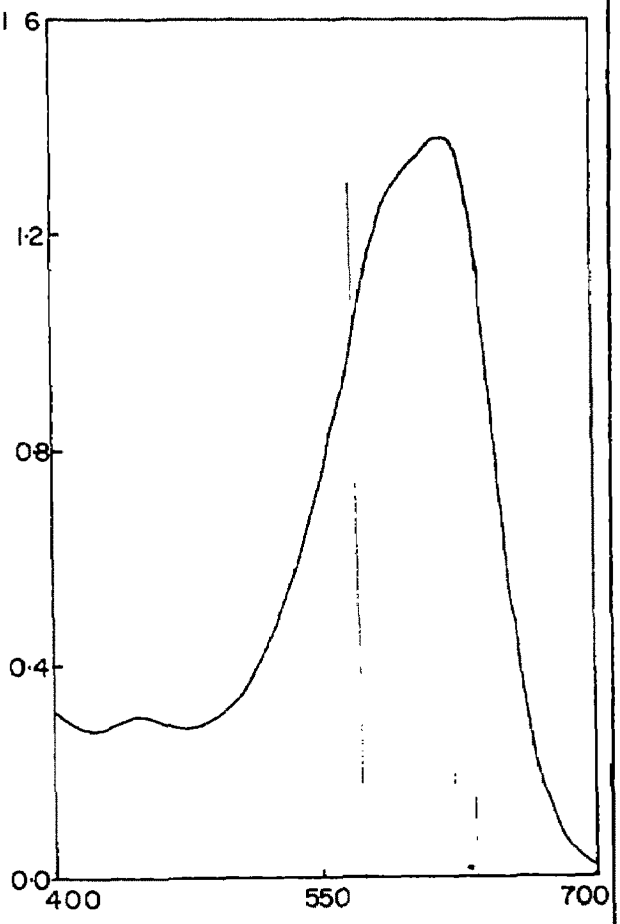


Fig.2.1b Absorption spectra of Acid Black from aqueous solution.

electrical device. At suitable time intervals shaking was stopped momentarily and the sample solutions were withdrawn for the measurement of the absorbance. On settling down of the solid adsorbent a fixed volume (3ml) of the solution was pipetted out from the supernatant liquid. Precaution was taken to avoid any solid particle coming out along with the solution. These samples afterwards were used for the determination of the concentration from absorbance measurements.

2.5 Determination of the concentration of the solution

The measurement of absorbance of the sample solutions was determined by the use of spectrophotometer (Spectronic-20, Bausch and Lomb, USA). For this the frequency of maximum absorption of the dyes were obtained from corresponding spectra determined by UV-VIS spectrophotometer (Shimadzu UV-240, Japan). The values of the frequency of maximum absorption (λ max.) selected for the dyes used in the present work are given below:

(The corresponding plots are given in Figs. 2.1a and 2.1b.)

Name of the Dye	λ max (nm)
Astrazone Blue BG (Basic Blue 3)	652
Ketone Blue A	635
Acid Green g (Acid Green 25)	640
Bismark Brown R (Basic Brown 4)	465
Brilliant Green Crystals (Basic Green 1)	625
Methyl Violet 6B (Basic Violet 1)	582
New Fuchsin (Basic Violet 2)	553
Acid Black 10BX (Acid Black 1)	615

The concentration of the dye solution was obtained by the use of Lambert Beer's Law. The expression is as follows:

$$A = \log I_o/I_t = \varepsilon CT$$

Where A is the absorbance, Io and I_t are the intensities of the incident and the transmitted light respectively. ε is the extinction co-efficient, C is the concentration of the dye and T is the thickness of the cell.

The absorbance of the initial dye solution (i.e., before adsorption) of known concentration was measured in order to determine the extinction coefficient of the dye solution which was used to determine the actual concentration of the dye remaining in the solution at the moment. Thus from the difference in the concentration of the dye remaining in the solution and that present initially, the amount of dye adsorbed at any stage of adsorption was calculated.

The main aim during these experiments as indicated already, was to determine the time growth of adsorption of dyes from aqueous solution as a function of the chief determinant. The process was observed to be fast in the case of dye adsorption by the use of the silica gel and alumina in comparison to the graphite used as adsorbent. In the case of graphite-dye systems the equilibrium was attained within 2 hours while in other cases it was established within a short interval of time (30 - 40 minutes).

Initially more readings were taken within a short span of time (each reading after two minutes) as the initial stages of the process is considered to be responsible for the actual rate of the time variation. Later this time intervals were increased gradually upto 20 to 30 minutes depending on the system. As the volume of the experimental adsorbate solution was large (200 or 300 ml) any expected alteration in the actual amount of dye adsorbed due to changes in the volume as a result of removals for test samples was considered to be negligible. Also at equilibrium, the concentration remained unaltered; therefore any volume correction for this factor being of little importance, were not made in order to avoid the complications.

2.6 References

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