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Removal of Acid Yellow 36 from aqueous solution by Solvent
Extraction method using tri-octyl amine as a carrier

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Abstract : The environmental challenge of synthetic azo dyes is of increasing concern due to the serious health effects on animals and human being. Metanil Yellow (Acid Yellow 36) is a highly water soluble dye is extensively used for the coloring of soap, spirit lacquer, shoe polish, bloom sheep dip, for the preparation of wood stains, dyeing of leather, manufacture of pigment lakes and for staining paper. Though Metanil Yellow is a non-permitted colorant, but still it is widely used as a colorant in sweet meat, ice creams, soft drinks and beverages. Due to its orange yellow color the dye is also extensively used for coating turmeric. Recovery of Azo dye compounds of Acid yellow 36 (monosodium salt of 4-m-sulphophenylazodiphenyl amine) from aqueous solution by tri-octyl amine (TOA) in kerosene was studied. The UV-visible absorption range of acid yellow 36 λ_{max} is 434 nm. The extraction efficiency of Acid Yellow 36 dyes decreased with increasing pH and TOA concentration in aqueous and organic phase respectively. The loaded organic dye maximum stripped into NaNO_3 , NaOH and KOH basic solution. The extraction and stripping of dye does not varied in presence of various inorganic salts. The other parameters examined were; effect of extractant concentration, effect of diluents, effect of pH, various stripping agents.

Key words: solvent extraction, Acid yellow 36, stripping agents, effect of diluents.

Introduction:

One of the more serious environmental problems associated with the textile industry is the removal of colour from textile effluent prior to discharge in to environment. Textile wastewater may be highly coloured and toxic¹. Dyes or dyestuffs are essentially coloured substances capable of imparting their color to other substances. Today, the majority of dyes are synthetically produced². They cause severe headache, profuse sweating, mental confusion and similar health hazards. Therefore removal of dyes from waste water is most desirable³. Azo dyes constitute an important class of synthetic, colored, organic compounds, which are characterized by the one or more azo bonds $-\text{N}=\text{N}-$ ⁴. It is used to 60% of total dyes.

Acid yellow 36 an acidic azo dye is widely used in dyeing of silk, paper, soap, leather etc. as an effective coloring agent. Hence, it may be found in wastewaters of many industries and hazardous to environment⁵. It is chemically designated as 3-(4-anilinophenylazo)benzenesulphonic acid sodium salt, chemical formula is $\text{C}_{18}\text{H}_{14}\text{N}_3\text{NaO}_3\text{S}$, representing molecular weight of 375.38 mg/l.⁶

Acid azo dyes are high environmental concern due to their high solubility and potential to contaminate ground and drinking water supplies⁷. Solid membrane separation process plays an increasing role in the reduction and/ or recovery of dyestuffs fouling of membrane can be a problem^{8,9}.

In recent years, much attention has been focused on separation techniques such as solvent extraction or liquid-liquid extraction (LLE) and liquid membrane techniques etc. LLE is based on the principle that a solute can distribute itself in a certain ratio between immiscible solvents and extraction process depends on its mass transfer rate^{10,11}. The advantages of LLE include high through put, ease of automatic operation of scale up and high purification include high selectivity, flexibility, and adaptability to remote operation¹².

Tertiary amines are known to be powerful extract ants for organic acids due to their physical properties; amine must always be used in the form of solutions in organic diluents¹³.

In the present study, the solvent extraction of anionic dyes namely Acid yellow 36 from acidic aqueous solutions using TOA in kerosene as an extractant. The various parameters influencing on extraction such as pH in the feed phase, TOA concentration in the organic phase. That is why back stripping (back extraction) of dye from loaded organic phase was also carried out in this work. The influencing parameters such as various stripping reagent were studied.

Experimental

Reagents and instruments

The following inorganic salts, acids, bases, and organic solvents were used in the experiments without further purification; sodium chloride(99%), sodium chloride(99%), Tri octyl amine (98%), Acid yellow 36 (99.8%), Hexane (95%), sodium hydroxide(99.5%) and potassium hydroxide(99%) chemicals were obtained from Merck.

An UV-Visible spectrophotometer (Elico SL 159 India) was used to measure the dye concentration. The pH of an aqueous solution was measured by a pH meter (Elico LI 120 with probe CL 51B). Systronics Electrophoresis 606 was used to find out whether the dye is cationic or anionic.

Extraction method

The organic solvent [Tri octyl amine + Kerosene] used for extraction was added to the prepared aqueous solution (AY 36) and pH adjusted using 0.05N HCl solution. Then taken into glass stoppered bottle was shaken at 100 rpm for 5min in a shaker. The solution mixture was then transferred into a separating funnel. Sample of aqueous solution at the bottom of the separating funnel was taken for absorbance measurement of the dye to determine dye concentration. The wavelength of maximum absorption (λ_{max}) for Acid yellow 36 was 434 nm. The dye concentration in the organic phase was calculated on the basis of mass balance.

Stripping method

In stripping, the loaded extract ant and the aqueous stripping were added together into a glass stoppered bottle and shaken at 100 rpm. After 10 min the content was transferred into a separating funnel. The aqueous strip pant was taken for dye concentration measurement. All the experiments were run in duplicate and analytical parameters were performed in triplicate for each run.confidence limit of 93% was taken for reliable results.

The results are expressed in terms of extraction efficiency (E) and recovery efficiency (R) defined.

$$E = 1 - C_f \cdot \frac{eq}{C_f} \cdot ini \quad \text{----- (2)}$$

$$R = C_s \cdot \frac{eq}{C_f} \cdot ini \quad \text{----- (3)}$$

Where C represents dye concentration; the subscripts f, org, s, eq, and ini represent the feed phase, organic phase, stripping aqueous phase, at equilibrium, and initial respectively^{14, 15}.

Results and discussion

Effect of extractant concentration

The effect of extractant concentration was first carried out in the absence of extractant. It is interesting to note that in the absence of tri octyl amine no extraction of dye occurred in the organic phase. The experimental data for the percentage of extraction versus extractant concentration are plotted in Fig.1. The percentage of extraction decreased with increase in concentration of TOA. This confirms that TOA is effective in extracting acidic azodye. Maximum extraction of 95.1% for 40 mg/L, 84.0% for 80 mg/L, 80.1.0% for 120 mg/L, occurred at TOA concentration of 1×10^{-2} to 5×10^{-2} mol/L. Further increase of TOA concentration decreases the extraction efficiency. Hence, the subsequent extraction studies were conducted using 1×10^{-2} mol/L of extractant.

The reaction for extracting in acidic medium and stripping the Acid Yellow 36 dye is as follows.

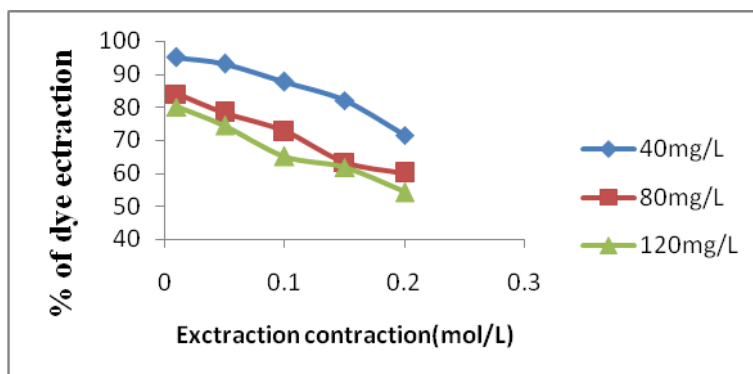
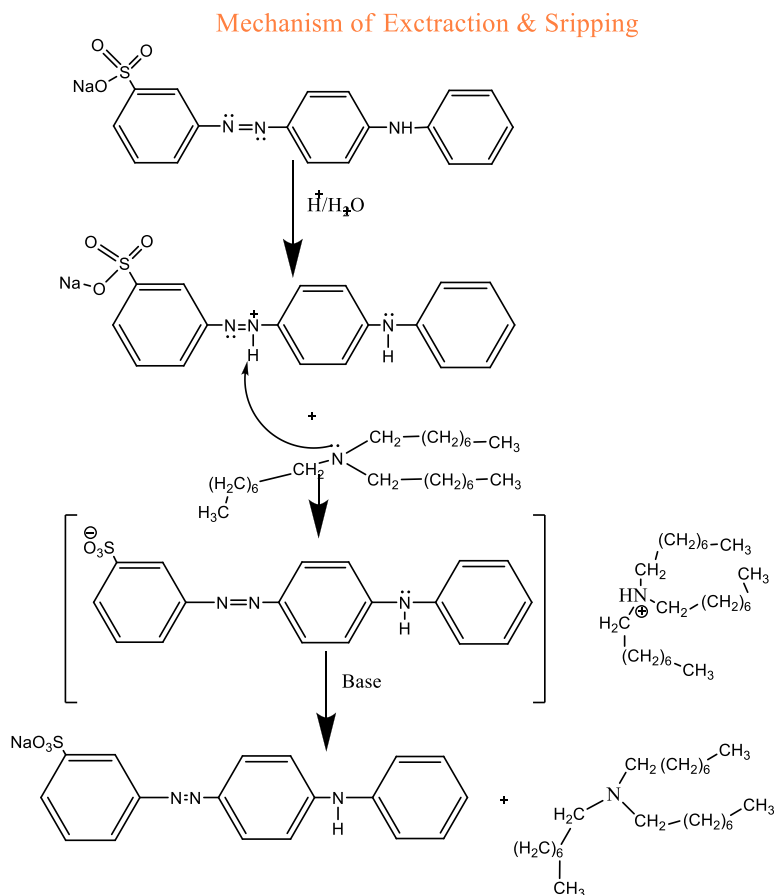


Figure 1 Effect of extractant (TOA) concentration (Experimental conditions: Volume of feed phase = 25 mL, extractant concentration = 1×10^{-2} mol/L at pH 2.0 ± 0.1 , volume of organic phase = 25 mL, initial dye concentration = 40 mg/L).

Effect of diluents

The extraction was carried out in different low density diluents aromatic compounds such as benzene, xylene, toluene and aliphatic compound such as hexane, kerosene were studied presented in the Table 2. TOA solubility in benzene, toluene and xylene were partially. The percentage of dye decreased in the order: kerosene> hexane>benzene>toluene>xylene>. Among them kerosene is less toxic, hence further studies were carried out using Kerosene as diluent.

Table 2 Dependence of the nature of diluents on the extraction of AY 36 dye from aqueous solution

Diluents/solvents	UV-Visible absorbance (nm) (aqueous solution)	Percentage of dye extraction
Hexane	0.172	87.8
Toluene	0.232	82.7
Benzene	0.258	85.7
Xylene	0.394	71.2
Kerosene	0.161	91.1

Effect of pH of source phase

The removal of AY36 was highly influenced by the pH factors. The anionic dyes were extracted from aqueous solution with different pH from 1 to 5 \pm 0.1 was studied. The effect of pH of the source phase on the efficiency of dye extraction is shown in Fig 2. The maximum percentage of extraction was achieved at pH 2 \pm 0.1 as follows; 95.1% for 40mg/L, 84.0% for 80mg/L, 80.1% for 120mg/L, respectively. It reveals that the percentage of dye extraction decreased with increasing pH of aqueous solution¹⁶. It might be at lower pH, H⁺ ion concentrations is high and allows the anionic dye to forms an ion-pair complex with cationic TOA¹⁷. Hence the pH of the feed solution, pH 2 \pm 0.1 recommended for further studies. Hence further studies were carried out at pH 2.0 \pm 0.1.

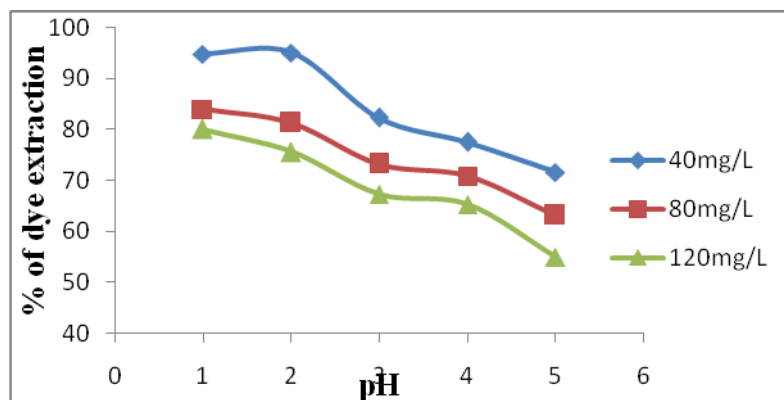


Figure 2 Effect of pH and TOA concentration(Experimental conditions: Volume of feed phase = 25 mL, various pH and extractant concentration= 4×10^{-2} mol/L, volume of organic phase=25mL,Initial dye concentration40mg/L).

Effect of stripping reagents

In any extraction processes, it is very imperative to back extract the extracted dye from the organic phase. Various inorganic bases such as sodium nitrate, sodium hydroxide, sodium carbonate, sodium bicarbonate, potassium hydroxide and sodium acetate have been tried as stripping agents. Maximum extraction efficiency of 98.1% for 0.001 to 0.015M NaOH, 96.5% for 0.01M KOH, 82.1% for 0.01M NaNO₃, 79.0% for 0.01M NaCO₃, 76.4% for 0.01 M NaHCO₃, 75.9% for 1M CH₃COONa was achieved at 40 mg/L initial dye concentration were used in this experiment and the results were presented in Table 3. Generally, we expect that the percentage of stripping increases with increasing stripping reagent concentration. The maximum percentage of stripping (98.1%) was achieved at 0.010mol/L potassium hydroxide. Further Sodium acetate is a weaker base hence it required higher concentration (1mol/L) to strip the dyes from loaded organic phase. It concluded that, the proton was abstracted by the base from protonated tri-octylamine of the ion pair complex.

Table3. Effect of stripping agents on percentage of dye stripping from organic phase

Stripping agents(mol/L)	% of stripping
NaOH	
0.001	82.3
0.005	89.6
0.010	93.5
0.015	98.1
KOH	
0.010	96.5
NaNO ₃	
0.010	82.1
NaCO ₃	
0.010	79.8
NaHCO ₃	
0.010	76.4
CH ₃ COONa	
1	75.9

Conclusions

In the solvent extraction method offers a simple approach for selective extraction of anionic azo dye for removal and recovery. The extraction efficiency of dye decreases with increasing the concentration of dye. The extracted dye was successfully stripped into 0.010 mol/L NaOH solution from loaded organic phase. The maximum extraction efficiency in acidic medium AY 36 was obtained at $\text{pH} = 2.0 \pm 0.1$.

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