

**Experimental study on Solvent Extraction of Crystal Violet dye**Pranali R. Sonawane¹, Harshada G. Bhostekar², Sona R. Moharir³, Mahesh A. Suryawanshi⁴, V.B. Mane⁵^{1,2}U.G. Students, Department of chemical engineering, Bharati Vidyapeeth College of Engineering, Navi Mumbai, Maharashtra, India¹^{3,4,5}Assistant Professor, Department of chemical engineering, Bharati Vidyapeeth College of Engineering, Navi Mumbai, Maharashtra, India

Abstract: Many industries use dyes extensively in different operations such as textile, paper, plastic, dyeing, pharmaceutical, cosmetics, leather tanning etc., for coloring their final products. Some dyes are toxic, mutagenic and carcinogenic. Thus the removal of dyes from the waste water is necessary before it gets mixed with the non-polluted water. Various methods have been used to remove dyes from aqueous solutions. Liquid-liquid extraction (LLE) is a significant potential to remove the dyes from wastewater. In the present study, we selected a cationic dye namely methyl violet for liquid-liquid extraction using Tri butyl phosphate (TBP) as an extractant and 1-Dodecanol as a diluent. The influencing parameters such as effect of extractant concentration, dye concentration and pH on extraction performance have been studied. It is seen that as the pH increases extraction of dye increases, dye extraction increases with decrease in extractant concentration and extraction increases with increases in dye concentration.

The influence of the parameters such as effect of extractant concentration, diluents, dye concentration and pH on extraction performance will be studied.

Keywords : Liquid Liquid extraction, Tri Butyl Phosphate, Methyl Violet,.

1. Introduction:

Synthetic dyes are essential refractory organic compounds and are often found in the environment as a result of their wide industrial usage. Dyes are broadly used in numerous industries such as textile, paper, artificial manufacturing, cosmetics and leather, for tinting and colouring the products. The discharge of dye wastewater from these industries contains harmful and toxic materials and is considered as a potential source of pollutants in environment which may accumulate to a toxic concentration and cause serious problems. Based on mentioned facts, the dye removal from water and as the main source, industrial wastewater is very important and essential treatment is somehow required before contaminated industrial waste discharge. Typical and popular wastewater treatment methods are not very effective for dye removal, particularly because the dye stability against light oxidation and also being resistant to aerobic bio-oxidation.

The dye stuff lost in the textile industry poses a major problem to wastewater sources. Indeed, textile industry produces high levels of dye and floating solid materials. It is estimated that 5000 tons of dyeing materials are discharged into the environment every year. These poisonous materials absorb the oxygen of the water. This has raised much as it threatens human life and the environment. Industrial wastewaters contain various kinds of toxic substances such as cyanides, alkaline cleaning agents, degreasing solvents, oil, fat, and metals.

Dye pollutants are generally resistant to biological degradation and a number of chemical and physical processes such as chemical precipitation, coagulation, electrocoagulation. Physical methods such as precipitation, flocculation or adsorption using bone char and activated charcoal do not degrade the pollutants but only transfer them from the liquid phase to the solid phase, thus causing secondary pollution or requiring regeneration that is a costly and time-consuming process. Chemical methods including oxidative degradation by chlorine, hydrogen peroxide and ozone, reductive degradation by sodium hydrosulfite, photocatalysis and electrochemical treatment require high dosage of chemicals and produce large quantity of sludge, and thus have been proved to be expensive. Moreover, the resulting by-products by chemical degradation may be coloured themselves or/and even toxic. Biodegradation (aerobic or anaerobic) of dyes is cost effective, environmental friendly and does not produce large quantities of sludge, but it is very selective and thus not suitable for most dyes. For most of these decolorization methods the expensive dyes cannot be recovered because of the destruction of dyes. The non-destructive techniques seem to be more attractive since these covered dyes are more valuable than that of the purified water. Solvent extraction method is another effective way to remove dyes from wastewater.

Liquid-liquid extraction (LLE) is a significant potential to remove the dyes from wastewater. LLE is based on the principle that a solute can distribute itself in a certain ratio between immiscible solvents, and the extraction process depends on its mass transfer rate.

Methyl violet (MV) is chiefly used in copying papers, in heterograph and printing inks. It imparts deep violet colour in paints and printing inks. It is also used to obtain shades of deep colours that can be applied for the dyeing of cotton, silk, paper, bamboo, weed, straw and leather. It is also widely used in Gram's Stain for the demonstration and primary classification of bacteria, as an anti-allergen and bactericide, the Flemming triple stain with iodine and Newton's crystal violet technique. Methyl violet is metachromatic and is sometimes used to demonstrate amyloid. The release of coloured

waste water from industries may present eco toxic hazards and may eventually effect human through food chain. The coloured compounds are affecting aquatic eco system. Some dyes are toxic, mutagenic and carcinogenic. Thus the removal of dyes from the waste water is necessary before it gets mixed with the non polluted water. Textile industries produce large amounts of liquid wastes that contain organic and inorganic compounds. The effluents generated from the textile industry are of utmost concern because of their high volume and pollution potential.

The pharmaceutical, food, and biotechnology industries abound with examples where there is a need to remove organic compounds from aqueous effluents or fermentation broth. Organo-phosphorus compounds and long chain tertiary amines are effective extractants to recover and separate organic acids from dilute solutions.

Dyes can be of many different structural varieties like acidic, basic, disperse, azo, anthraquinone based and metal complex dyes among others. The textile industry is the largest consumer of dye stuffs. During the coloration process a large percentage of the synthetic dye does not bind and is lost to the waste stream. Approximately 10-15% dyes are released into the environment during dyeing process making the effluent highly colored and aesthetically unpleasant. In addition to being toxic, dye effluents also contain chemicals that are carcinogenic, mutagenic or teratogenic to various organisms, this is especially serious because many chemicals can cause damage to genetic material without being expressed immediately.

Normally one of the two phases is an organic phase while the other is an aqueous phase. Under equilibrium conditions the distribution of solute over the two phases is determined by the distribution law. After the extraction the two phases can be separated because of their immiscibility. Component is then separated from the extract phase by a technique such as distillation and the solvent is regenerated. Further extractions may be carried out to remove more components. Liquid-liquid extraction can also be used to remove a component from an organic phase by adding an aqueous phase. Solvent extraction is used in nuclear reprocessing, ore processing, and the production of fine organic compounds, the processing of perfumes, the production of vegetable oils and biodiesel, and other industries.

2. Materials and Method:

Materials: Distilled, Crystal Violet Dye, Tributyl phosphate, 1-Dodecanol, HCL (Hydrochloric acid).

Apparatus: A UV visible spectrophotometer was used to find out λ max and measure dye concentration in the raffinate phase. pH meter was used to measure pH of aqueous solutions. Vigorous Agitation of solution was done manually. Centrifuge to let the two phases set.

Preparation of dye solutions

A stock solution of 1000 mg/L of MV was prepared by dissolving an appropriate quantity of dye which was diluted in a liter of distilled water. The working solutions were prepared by diluting the stock solution with distilled water to give the appropriate concentration of the working solutions. Further the dye solution of various ppm's (20-100 ppm) were prepared from stock solution (1000 ppm).

Procedure: Liquid- liquid extraction

The extraction process was carried out by mixing an equal volume (2 mL) of aqueous solution of dye and organic solution of (Tributyl Phosphate + 1- Dodecanol) in a sample bottle and the sample bottle was shaken vigorously for 30 minutes manually for proper mixing. These solutions were then centrifuged at 1000 RPM for 30 minutes to obtain 2 phases (Extract and raffinate phase) after equilibrium was established, the phases were separated using Micro pipette or syringe. Sample of aqueous solution at the bottom was taken for absorbance measurement and dye concentration measurement. The same procedure was carried out in each dye concentration (viz. 20 ppm, 30 ppm, 50 ppm, 75 ppm and 100 ppm). The wavelength of maximum absorption (λ max) for MV was 590 nm. The distribution ratio (D) and percentage of extraction (E) were calculated as per the following equations..

$$D = [\text{dye}]_{\text{org}} / [\text{dye}]_{\text{aq}}$$

$$E = 100 \times \{ [\text{dye}]_{\text{aq}0} - [\text{dye}]_{\text{aq}} \} / [\text{dye}]_{\text{aq}0}$$

Where $[\text{dye}]_{\text{org}}$: dye concentration in the organic phase (mg/L);

$[\text{dye}]_{\text{aq}0}$: initial dye concentration in the aqueous phase (mg/L);

$[\text{dye}]_{\text{aq}}$: dye concentration of aqueous phase after extraction (mg/L)

3. Result and Discussion

Effect of pH in the feed phase

The pH of the extraction solution is considered one of the most important factors affecting the extraction process. In MV below pH 2 changed colour from dark violet to light violet and the original dark violet colour was changed above pH 3 when the solution was made acidic using concentrated HCL. Hence the effect of pH of solution was studied between 1.0 to 3.0 (Acidic) shown in Fig. 1. At pH 1 the extractant becomes positively charged, concentration of H^+ was high and they compete with MV. So the percentage of dye extraction decreases.

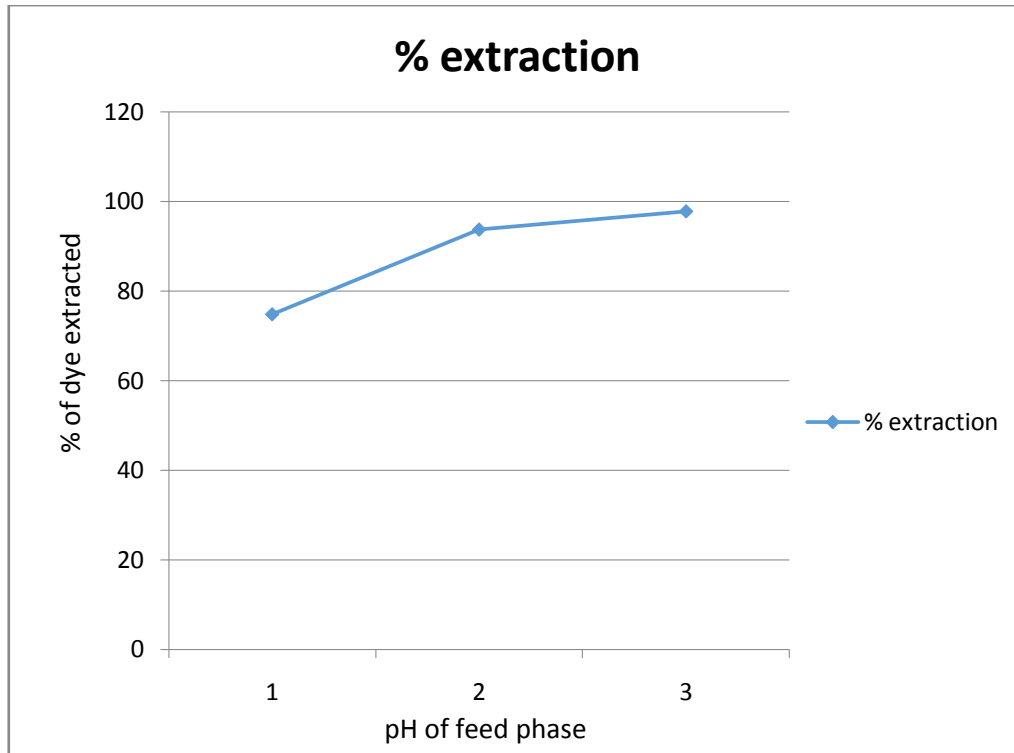


Fig: 1 Effect of pH on % extraction

Effect of extractant concentration:

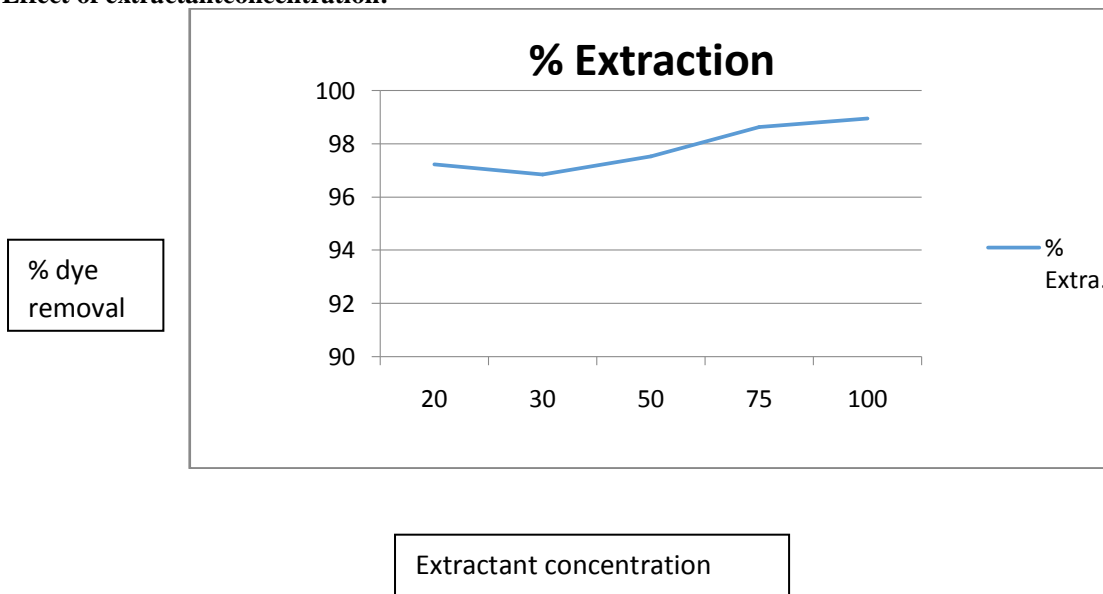


Fig. 2. Effect of extractant concentration

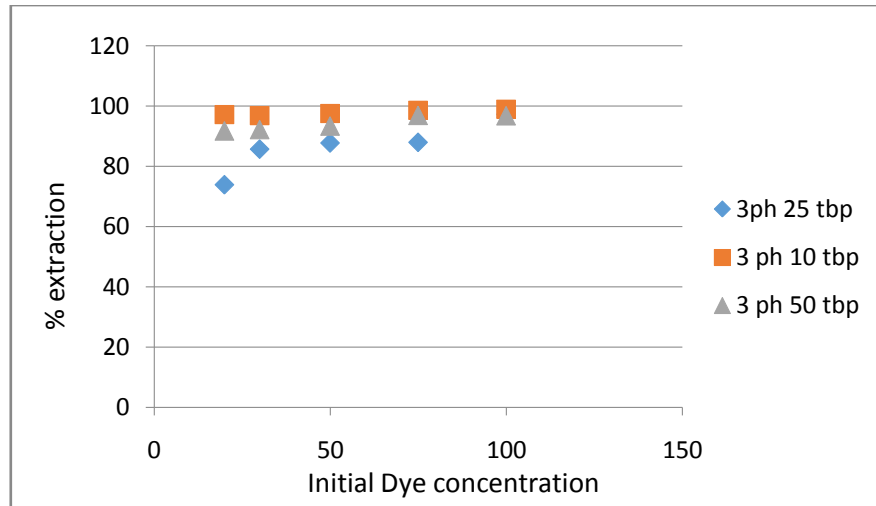
Extractant concentration is an important parameter because it determines the capacity of an extractant for a given initial concentration of dye. The effect of extractant concentration was studied with MV dye removal keeping all the experimental conditions constant. The extraction of dye by Tributylphosphate at different extractant concentrations from 10-75 concentration at pH 3.0 was studied. The results are shown in Fig. 2. The result (Fig. 2.) shows that As extractant concentration increases % extraction decreases. Further increase in extractant concentration, did not show significant increase in % removal of dye.

Effect of dye concentration

The effect of dye concentration on the extraction process was tested at extractant concentration. The extraction percentage of various initial concentrations of cationic dyes in the range 20 mg/L to 100 mg/L was studied and the results are shown in Fig. 3. At initial dye concentration of 20 mg/L, 98.95% of extraction was obtained. Further increase the

initial dye concentration in the aqueous phase, the percentage of extraction decreases. Because, in higher concentration of dye has higher energy, and high in color. So, the extractant cannot able to extract the dye completely. Hence, the subsequent extraction studies were conducted using 20 mg/L of dye concentration.

% extraction vs initial feed conc



4. Conclusion

Methyl violet was extracted from aqueous solution using liquid liquid extraction. Equilibrium study was carried out for Methyl violet dye using different organic extractant like TBP in 1-dodecanol. Extraction of dye from aqueous solution was carried out using TBP as extractant, the percentage extraction of dyes increased with increasing pH in the aqueous feed phase. The equilibrium isotherms showed that the distribution coefficient KD was found to be increased from 82 to 96 for 2 pH at 10% TBP concentration and 90% 1-Do decanol. It is studied that the extraction increases with increase in the pH and decrease in extractant (TBP) concentration.

5. References

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