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Research Article

DYE REMOVAL AND SEPARATION EFFICIENCY OF CYANEX 301FROM THE DYE MIXTURE

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ABSTRACT

A selective, sensitive and efficient liquid-liquid extraction (LLE) of industrial waste water containing dyes namely Methylene Blue (MB) and Eriochrome Black T (EBT) has been studied using Bis(2,4,4-trimethyl pentyl)dithiophosphinic acid(Cyanex 301) in dichloromethane, as an extractant. Dye free aqueous phase was obtained and the dye mixture was extracted into organic phase. With the lowering of pH there was an increase in efficiency of extraction. The dyes could be separated from each other by treating the organic layer, with sodium hydroxide solution. Methylene blue remained in the organic phase and Eriochrome black T got stripped into the aqueous phase. MB & EBT were later determined spectrophotometrically at 654nm& 530 nm respectively. The method was optimized by varying experimental parameters such as feed source pH, effect of diluent, cyanex concentration, extraction time, aqueous to organic ratio, effect of salt concentration, and effect of concentration of stripping agent. The dyes were totally extracted, separated and determined at the optimum conditions.

INTRODUCTION

Aquatic ecosystem is largely polluted by the discharge of colored wastewater from dye industries. Even trace amount of dyes (less than 1ppm for some dyes) in water is highly visible and is generally termed as contaminant which is undesirable (Crini G,2006). This released waste water leads to eco-toxic hazard,which eventually will affect human life through food chain and also pose a threat to the safety of the environment (Lin, J. et al, 2008; Ashraf, S. *et al*, 2006).Numerous industries like textile, food, paper printing, plastic, leather and cosmetics, contribute largely to this type of contamination. Therefore, there is a serious need for removal ofthese dyes from wastewater. Removal procedure is the most tedious, due to chemical stability, high chemical oxygen demand (COD) and toxicity of some of these dyes. Also some dyes are suspected to be carcinogens and mutagenic (Holme, I., 1984).

Azo dyes are the largest group of dyes used in industry (Zhang, C. *et al*, 1995). Methylene blue (MB) is one of the most commonly used dyes for dyeing cotton, wood and silk. Though MB is not highly hazardous and has medicinal uses, it can cause some harmful effects. Acute exposure of MB will lead to increased heart rate, vomiting, shock, cyanosis, jaundice, and quadriplegia and tissue necrosis in humans (Kumar, K.*et al*, 2005)

Another dye, Eriochrome Black-T (EBT)is also hazardous as such and its degradation products like Naphthaquinone arecarcinogenic (Dave, P. *et al*, 2011).

Therefore, it was thought worthwhile to develop a cost effective and efficient method of extraction for removal of these dyes from wastewater before it enters main stream water source thereby preventing the damage to the environment.

Several methods have been used for removal of dyes from wastewater. The widely used methods include, electrocoagulation (Alinsafi, A. *et al*, 2004),coagulation and precipitation (Liu, R. *et al* 2003), adsorption (Sanghi, R. *et al*, 2002), adsorption onto agricultural solid waste (Namasivayam, C. *et al*, 2002), micellar enhanced ultrafiltration (Ahmad, A. *et al* 2006), nanofiltration (Purkait, M. *et al*, 2003; Zhong, P. *et al*, 2012; Patel, T. *et al*, 2012; Cheng, S. *et al*, 2012), several oxidation processes (Neamtu, M. *et al*, 2004), electrochemical degradation (Martinez-Huitle, C. *et al*, 2009), ozone based processes (Zhang, J. *et al*, 2009, photo catalytic degradation (Bali, U, *et al*, 2004; Siboni, M. *et al*, 2011; Li, Y. *et al*, 2001; Nagel-Hassemer, M. *et al*, 2012; Kim, Y. *et al*, 2008; Lorena, L. *et al*, 2006; Lakshmi, S. *et al* 1995), different bentonites (Arvanitoyannis, I. *et al*, 1989), various types of activated carbon (Mezohegyi, G. *et al*, 2012; Kannan, N. *et al*, 2001), biological treatments (Khataee, A. *et al*, 2010; Oller, I. *et al*,

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2011; Sarayu, K.*et al*, 2012) and using $NiFe_2O_4$magentic nanoparticles (Moeinpour, F.*et al*, 2014)

Use of bulk liquid membrane (BLM) system for selective extraction of methylene blue is also reported (Madaeni, S. *et al*, 2011). The system not only requires a carrier but also needs a nonionic surfactant. Removal of Methylene blue and Eriochrome black T using biosorptiontechnique on Scolymushisponicus was reported but requires thebiosorption kinetic data to fit with pseudo kinetic model (Barka, N. *et al*, 2011).The chemical methods require high dosage and produce large quantities of sludge, hence are not very economical (Baban, A. *et al*, 2003; Georgiou, D. *et al*, 2003; Kurbus, T. *et al*, 2003).The physical methods merely transfer the pollutants from one medium to another and are non-destructive, thus requiring secondary treatment (Bes-Pia *et al*, 2003).Fouling of membrane reduces flux during ultrafiltration (UF) and nanofiltration (NF). Biodegradability of dyes limits the use of conventional biological wastewater treatment method (Muthuraman, G. *et al* 2005)

In recent years, much attention has been focused on a separation technique such as liquid–liquid extraction (S.Z,-E,*et al*, 2015; Sathya, M. *et al*, 2015; Chen, X. *et al* 2013; Muthuraman, G. *et al* 2012; Muthuraman, G., *et al*, 2009_a;Muthuraman, G., *et al*, 2009_b; Machado, A. *et al* 2011; Ueda, A. *et al* 2011; Gharehbaghi, M. *et al* 2012; Talebi, A. *et al* 2013). Liquid–liquid extraction (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 (Woo, L. *et al*, 2000).Liquid–liquid extraction method is used for the purificationenrichment, separation and analysis of various compounds in mixtures. Therefore, the selection of both a diluent and an extractant determines equilibrium for a given system and the efficiency of extraction process.

The structure of the dyes is shown in Fig. 1 and Fig 2.

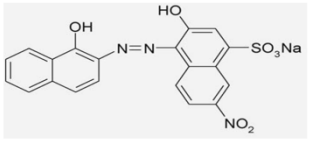

Fig 1 Structure of Eriochrome Black T

Eriochrome Black T (Mordant Black 11); MW 461.38; λmax 530nm

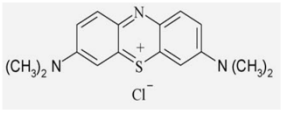

Fig 2 Structure of Methylene Blue

Methylene Blue (Basic Blue 9); MW 319.85; λmax 654nm

Advantages and disadvantages of some methods of dye removal from wastewater are tabulated in Table 1 (Robinson, T. *et al*, 2001).

extractant was studied. The operating conditions for dye extraction and its determination were optimized. Separation and recovery of dyes could be achieved using back stripping agent.

Experimental

Materials

Cyanex 301 was procured from Cytec, Canada. MB, EBT, sodium hydroxide pellets, hydrochloric acid, sulphuric acid, dichloromethane were of AR grade.

A UV-visible spectrophotometer (1800 Shimadzu) was used to measure the absorbance of the dye and to establish its λmax and its concentration. pH of an aqueous solution was measured by a pH meter (PANH Labindia). Wrist action shaker was used for extraction by shaking.

Cyanex 301 was diluted in dichloromethane. The dye solution was prepared in milli Q water. Hydrochloric acid and sodium hydroxide were used to adjustpH. Sodium hydroxide solution was used for dye separation and as a stripping agent. Further for recovery and reuse of dichloromethane, organic phase containing MB was stripped with sulphuric acid. Industrial waste water was used as the real sample.

Procedure

Extraction method

In a glass-stoppered bottle, the organic solvent 25mL [Cyanex 301 in dichloromethane) (V_o mL)] was added to 25 mL (V_a mL) of the synthetically prepared aqueous dye mixture solution and pH was adjusted to 1.0 using 2M HCl. The glass-stoppered bottle was shaken for 5minutes time on a wrist action shaker at about 100 rpm. The mixture was then transferred into a separating funnel and allowed to settle for a minute until the two layers separated. The aqueous layer at the top was dye free solution and the lower organic layer consisted of dye mixtures. The aqueous solution was measured at 654 nm for MB traces and at 530 nm for EBT. The loaded organic layer was further treated for separation of the two dyes from each other.

Separation and Stripping Method

In separation and stripping, the loaded organic extractant was transferred to glass bottle and treated with 25mL 0.01M sodium hydroxide solution. The glass-stopper bottle was shaken for 2minutes on a wrist action shaker at about 100 rpm and then allowed to stand for 5minutes. EBT layer was observed at the top and the lower organic layer consisted of MB which was stripped for organic phase reusability.

Table 1

Physical/chemical methods	Advantages	Disadvantages
Fenton's reagent	Effective decolourization of both soluble and insoluble dyes	Sludge generation due to Fe^{2+} used
Ozonation	Applied in gaseous state no alternative of volume	Short half life (20 min) of ozone
Photochemical	No sludge production	Formation of by products
Sodium hypochlorite	Initiate and accelerates azo bond cleavage	Release of aromatic amines and adsorption of organic halides
Electrochemical destruction	Breakdown compounds are non hazardous	High cost
Activated carbon	Good adsorbent due to cellular structure	Very expensive and disposal of spent non feasible
Peat	Good adsorbent due to cellular structure	Specific surface areas of adsorbent are lower than activated carbon
Wood chips	Good sorption capacity for acid dyes	Requires long retention time
Silica gel	Effective removal for basic dyes	Prevent commercial application
Ion exchange	Regeneration, no adsorbent loss	Not effective all types of dyes

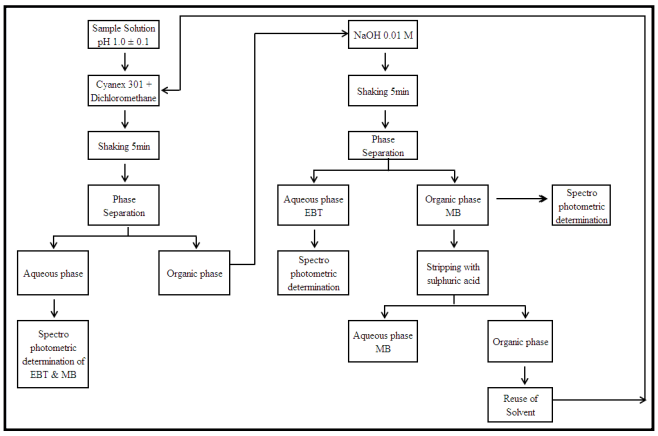

Fig 3 Schematic experimental setup for liquid-liquid extraction for removal of dye

RESULTS AND DISCUSSION

Effect of pH

To evaluate the effect of pH on the efficiency of extraction, pH of source feed solution was investigated at different values of pH ranging from 0.5 – 8 with different dye concentrations. pH adjustments were done using 2MHCl and dilNaOH solution. The results showed that the extraction efficiency decreased with increasing pH. The maximum extraction of dye was observed at pH 1± 0.1 as shown Fig.4a.and Fig. 4b.The optimum pH 1.0±0.1 was selected for further studies. The studies indicated that the aqueous phase obtained after extraction was free from dyes.

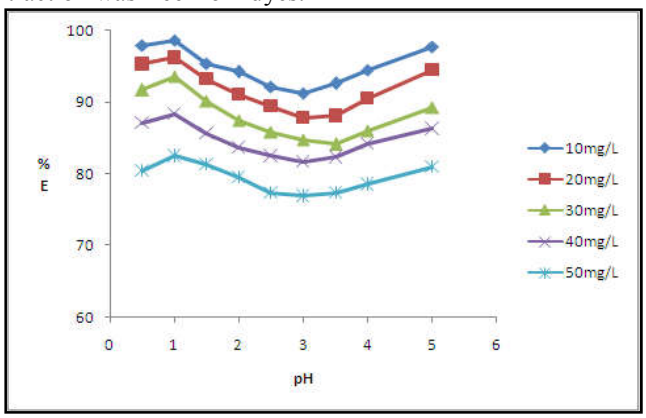

Fig. 4a Effect of pH on Methylene blue (Experimental conditions Volume of feed phase = 25 mL, extractant concentration = 0.005 M, volume of organic phase= 25mL,Room temperature 27^{0}C).

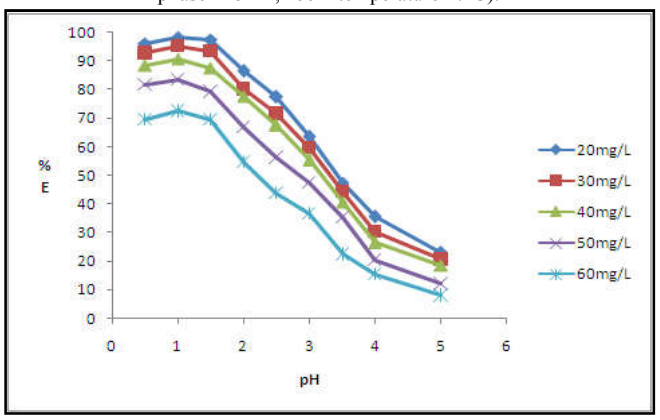

Fig 4b Effect of pH on Eriochrome Black-T (Experimental conditions Volume of feed phase = 25 mL, extractant concentration = 0.005 M, volume of organic phase= 25mL, Room temperature 27^{0}C).

Effect of diluents

Various solvents such as chloroform, xylene, toluene, n-hexane, dichloromethane, ethylacetate and diethyl ether were tried for the extraction study. Among them, chloroform and dichloromethane extracted the dyes quantitatively; percentage extraction is tabulated in Table 2. There was negligible extraction observed in n-hexane. Dichloromethane was used for all further studies, since it is less toxic as compared to chloroform and phase separation was faster.

Table 2 Effect of diluents on dye extraction efficiency

Diluent	MB % Extraction	EBT % Extraction
Chloroform	98.6	98.6
n-Hexane	36.0	45.4
Dichloromethane	98.5	98.6
Ethylacetate	98.0	96.3
Diethylether	10.5	15.6
Toluene	40.2	70.6

Effect of Cyanex 301 concentration

In this experiment, cyanex concentration was varied from 0.001 to 0.01M in dichloromethane along with different concentrations of dyes. The concentrations of MB taken were 10mg/L,20mg/L,30mg/L,40mg/L,50mg/L and the Eriochrome Black T concentrations taken were 20mg/L,30mg/L,40mg/L,50mg/L 60mg/L. The results are shown in Fig 5a and Fig 5b.The percentage of extraction increased with increasing extractant concentration and beyond 0.005M the extraction efficiency remained unchanged. Hence, 0.005M cyanex in dichloromethane was used for further extraction studies. Extraction of the dyes in dichloromethane alone was negligible thereby confirming the roll of cyanex in the formation of adduct with the dye.

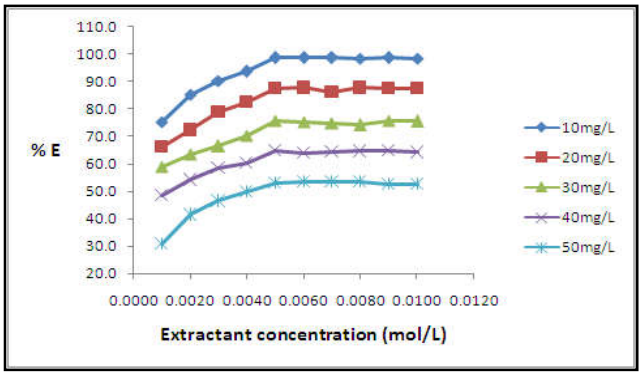

Fig 5a Effect of extractant concentration on Methylene blue (Experimental conditions: Volume of feed phase = 25 mL, pH 1.0 ± 0.1, volume of organic phase= 25mL, Room temperature 27^{0}C).

Effect of shaking time

The organic and aqueous phases were shaken for a period of 1–15 min. The results are presented in table 3a & 3b. The maximum amount of dye was extracted at 5 min. Beyond 5 min, the extraction efficiency remained unchanged. For further studies, the shaking time was fixed at 5 min.

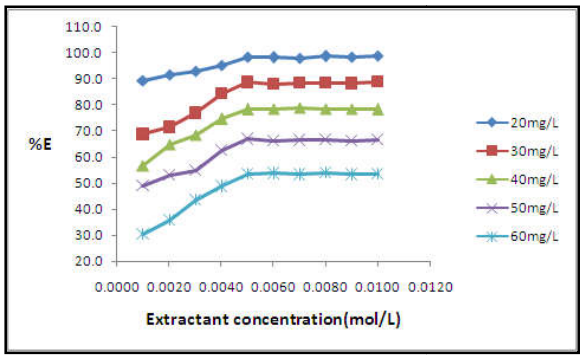

Fig 5b Effect of extractant concentration on Eriochrome Black T (Experimental conditions: Volume of feed phase = 25 mL, pH 1.0 ± 0.1, volume of organic phase= 25mL, Room temperature 27^{0}C).

Table 3a Effect of shaking time on MB

On MB	10 mg/L	20 mg/L	30 mg/L	40 mg/L	50 mg/L
1	90.2	82.0	78.0	70.1	65.0
2	91.0	85.0	84.0	76.2	68.0
3	93.0	90.0	86.0	81.5	74.0
4	95.0	94.0	91.7	87.0	80.0
5	98.6	96.4	94.0	90.0	85.0
6	98.4	96.7	94.2	90.1	85.6

Table 3b Effect of shaking time on EBT

On EBT	20 mg/L	30 mg/L	40 mg/L	50 mg/L	60 mg/L
1	96.0	85.0	83.0	82.0	75.0
2	96.8	87.0	84.0	86.0	78.0
3	97.0	90.0	90.0	89.0	81.5
4	97.0	93.0	92.2	92.0	86.7
5	98.6	96.0	95.0	93.0	90.0
6	98.2	95.8	95.2	93.2	90.5

Effect of extraction phase ratio

The volume phase ratio of aqueous to organic phase (A:O) was studied at 27^{0}C ±0.5. The extraction efficiencies at different volume ratios are presented in Fig.6. About 98% extraction efficiency was achieved from 1:1 to 5:1 A:O ratio; beyond 5:1 A:O ratio, the efficiency of extraction decreased.

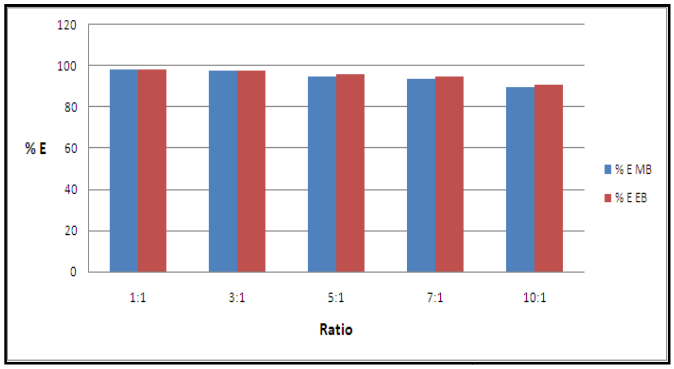

Fig 6 Effect of extraction ratio

When the ratio was increased to 10:1, the extraction efficiency dropped from 97% to 90%. The A:O ratio from 1:1 to 5:1 yielded a higher percentage of dye removal from aqueous solution. This could be due to the higher free concentration of organic phase when the aqueous to organic phase ratio (A:O) is lower.

Effect of salt concentration

In the actual textile dye bath effluent, salt like sodium chloride and sodium sulphate are present. To understand the effect of salts on dye extraction, dye solution with different concentrations of sodium chloride and sodium sulphate were tested. The results are shown in Table 4a and 4b. which shows that the percentage removal of dye remained unchanged with increase in sodium chloride and sodium sulphate concentrations.

Table 4a Effect of salt concentration on MB

Concentration of anions (mg/L)	Percentage of MB extraction				
	10	20	30	40	50
Chloride					
1000	98.7	96.2	98.7	95.2	90.4
2000	98.6	96.1	95.1	94.1	90.2
3000	98.3	97.4	94.8	94.3	89.7
4000	98.2	96.6	95.1	94.1	90.1
5000	98.1	96.2	95.3	93.8	90.0
Sulphate					
1000	98.8	96.4	95.2	95.1	90.2
2000	98.4	96.3	95.1	94.3	90.1
3000	97.8	96.1	94.8	94.1	89.6
4000	98.0	94.6	95.0	94.3	89.3
5000	98.2	96.3	94.4	94.5	89.1

Table 4b Effect of salt concentration on EBT

Concentration of anions (mg/L)	Percentage of EBT extraction				
	20	30	40	50	60
Chloride					
1000	98.6	96.5	95.0	94.4	90.0
2000	98.2	96.2	95.4	94.0	90.1
3000	98.3	96.0	95.2	94.2	90.0
4000	98.9	96.4	95.3	94.5	90.3
5000	98.5	96.1	95.1	94.1	90.3
Sulphate					
1000	98.7	96.1	95.1	94.3	89.9
2000	98.2	96.3	95.2	93.9	90.0
3000	98.1	96.0	95.1	94.2	90.2
4000	98.3	95.0	95.3	94.1	89.5
5000	98.5	96.3	94.9	94.4	89.3

Separation and Stripping of organic phase

In the present study, the dye mixture was extracted into the organic phase. It was important to separate the two dyes by stripping one of the dyes into aqueous phase. Sodium hydroxide was used as the stripping agent. The effect of concentration of stripping agent was studied and is presented in Fig 7. The results show that 0.01M sodium hydroxide was required to strip Eriochrome black T, efficiently.

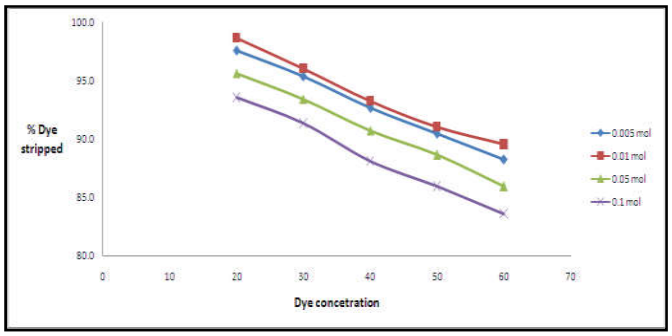

Fig 7 Effect of stripping reagent

Reusability of solvent

In order to reuse the organic solvent, dye needs to be removed. As, Methylene blue dye which remains in the organic phase and could be stripped using sulphuric acid using the same experimental set up with shaking time about 10 min. The organic phase so obtained was distilled and could be reused.

Application of the developed LLE on textile waste water

The developed LLE was tested for its applicability to the real industrial waste water containing the dyes. The removal efficiency is similar to industrial sample under the optimized conditions.

Conclusion

The method presented offers a simple approach for selective extraction and separation removal of a dye mixture. Clean dye free aqueous phase could be achieved and the extracted dyes viz. Methylene blue and Eriochrome Black T were also separated from each other. The extraction efficiency was not affected in the presence of salt like sodium chloride and sodium sulphate. Time required for extraction as well as separation was very less. The concentration of extractantCyanex 301 measured was also low i.e. 0.005M. Under optimized conditions, real textile wastewater was treated, and it was found that extraction efficiency did not deviate much from that of synthetic solution.

Acknowledgement

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