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# **RESEARCH ARTICLE**

# SENSITIVE AND SELECTIVE METHOD FOR LIQUID-LIQUID EXTRACTION OF CATIONIC DYE FROM AQUEOUS SOLUTION

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# ARTICLE INFO

# ABSTRACT

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Received 14<sup>th</sup>, February, 2015 Received in revised form 23<sup>th</sup>, February, 2015 Accepted 13<sup>th</sup>, March, 2015 Published online 28<sup>th</sup>, March, 2015 A rapid, sensitive and selective method, liquid-liquid extraction has been used to remove the dye from aqueous synthetic solutions using Di (2-ethylhexyl) phosphoric acid (D2EHPA) in hexane as carrier. Malachite green (MG) was determined by using UV visible spectrophotometry at 617 nm. The percentage of extraction of dye increases with increasing pH and carrier in aqueous and organic phase respectively. The maximum percentage of extraction was obtained at pH  $\pm$ 9.0. The loaded organic dye stripped into glacial acetic acid and oxalic acid 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 pH, effect of temperature, various stripping agents, aqueous to organic phase ratio and reusability of solvent.

## Key words:

Malachite green, D<sub>2</sub>EHPA, Extraction, Stripping, Reusability.

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# **INTRODUCTION**

One of the more pressing 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 toxic (Muthuramanet *al* 2006). Large quantities of dyes are manufactured annually worldwide and are used in a variety of applications such as textiles, paper, foodstuffs, and cosmetics. Textile dyes are of environmental interest because of their wide spreaders, their potential for formation of toxic aromatic amines and their low removal rate during waste treatment (Muthuraman *et al*, 2005). When these coloured effluents enter the rivers or any other surface water systems, they upset biological activity. Ground water systems are also affected by these pollutants because of leaching from the soil (Muthuraman *et al* 2009).

Malachite green (MG) is used extensively for dyeing silk, wool, jute, leather, ceramics, cotton, and used to treat fungal and protozoan infection (Parshetti *et al.*, 2006). It is highly toxic to mammalian cells; it promotes hepatic tumour formation in rodents and also causes reproductive abnormalities in rabbits and fish (Roushdy *et al* 2011). Malachite green is high concentration causes harmful effects on liver, kidney, intestine and gonads of aquatic organisms (Garg *et al.*, 2004; Kumar *et al*, 2008; Srivastava *et al.*, 2004; Xiangliang pan *et al* 2009; Subbareddy *et al.*, 2012).

MG was tri phenyl methane dye, widely used as a strong antifungal, antibacterial, anti-parasitical agent in the aquaculture industry and as a very common multipurpose dyestuff in various dye industries. It is toxicity of exposure to cause the physiological and biochemical indexes imbalance, such as the serum, calcium, protein levels and total cholesterol level was reported (Mei jieLi *et al.*, 2012).

The photo cytotoxicity of tri phenyl methane dyes, based on the production of the reactive oxygen species, is tested intensively with the regard to their photodynamic treatment (Baptisa *et al*, 1998). (Nelson *et al*, 1980).Our reported the depositions of crystal violet and malachite green in the sediment and water of Buffalo River in New York. They found aniline dyes an aquatic environmental. From a study of (Black *et al.*, 1980) it was shown that aniline dyes colloid be mutagenic and carcinogenic to biota. The diffusive torsional dynamics of malachite green molecules in solid matrices probed by fluorescence decay was reported (Kazimonowar *et al.*, 1995).

Removal of malachite green from dye wastewater using neem saw dust was also reported (Khattri *et al*,2009).Adsorption of the MG dye was studied by bath technique and it was observed than more that 84% of the removal was achieved by using 0.025g of zeta mays dust carbon. The kinetics of adsorption developed can be used to achieve low levels of dye from aqueous solutions utilizing the chosen carbon. Almost 92% removal of dye was observed at pH of 9.0.The adsorption data

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seems to follow the Langmuir and Freundlich isotherms (Anbia et al. 2010). Chemical methods are not economically viable due to high dosage and production of large quantity of sludge (Ahmet Baban et al., 2008; Georgiou et al., 2003; KurbusTanja et al., 2003). Biodegrading (aerobic or anaerobic) of dyes is cost effective, environmental friendly and does not produce large quantities of sludge but it requires large space (Xiaoyao Tan et al., 2006). Decomposition techniques also used for removal of dyes from wastewater. For example sonolysis and Fenton process  $(H_2O_2/Fe^{2+})$ ; chemical oxidation and reduction; photo catalytic and electrochemical treatment have been considered promising efficient, cleans and greener approaches (MiHwabaek et al., 2010). Removal of methylene blue and malachite green oxalate using poly acrylic acid bound iron oxide magnetic nanoparticles and PPA-Nano clay particles were also studied (Mark and Chen, 2004; Sonawane et al., 2008).Other techniques have also used in effective treatment of the dye containing wastewater, which include conventional coagulation/flocculation precipitation, chemical ozone treatment, oxidation, ion-exchange, reverse osmosis and filtration (Wen Cheng et al., 2008). Solid membrane separation process plays an increasing role in the reduction and/ or recovery of dyestuffs fouling of membrane can be a problem (Muthuraman et al. 2010; Muthuraman et al., 2008). Aqueous bibasic system (ABS) consists of two immiscible phases that are formed when certain water soluble polymers are mixed with one another (or) with certain inorganic salts in specific concentration.

In recent years, much attention has been focused on separation techniques such as solvent extraction or liquid-liquid extraction (LLE), and liquid membrane technics 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 (Muthuraman et al, 2009). The advantages of LLE include high through put, ease of automatic operation of scale up and high purification (Lee et al., 2009). In the present study, the removal of cationic dye namely MG using D2EHPA as an extract ant in hexane was studied. Organo- phosphorous acid derivatives such as di (2- ethyl hexyl) phosphoric acid have been used by several investigators for the extraction of heavy metals and amino acids from acidic aqueous solution. Among acidic Organo-phosphorus extractant D2EHPA has been used commercially due to its good ability to extract a large number of organic and ions (Muthuraman and Teng, 2009). That is why D2EHPA has been selected as an extractant in this study. The structure (figure-1) and properties of MG dye is also given in Table -2 (MeriemDjenouhat et al., 2008; VlastaBrezova et al., 2004; Hameed et al2008).



Properties of Malachite Green (Oxalate)(MeriemDjenouhat *et al.*,2008;Vlasta Brezova *et al.*,2004;Hameed and Khaiary,2008).

Parameter	Value	
	Malachite green oxalate	
Suggested name	N,N,N,N-Tetramethyl-4,4diamino tri phenyl	
IUPAC name	carbenium oxalate	
CI Name	Basic Green-4	
CI Number	42000	
Empirical Formula	$C_{52}H_{54}N_4O_{12}$	
max	618 nm (Measured value)	
Formula weight	927.03g/mol	

# **MATERIALS AND METHODS**

# Materials and instruments

The following inorganic salts, acids, bases, and organic solvents were used in the experiments without further purification; NaCl (99%),  $Na_2SO_4$  (99%), Di (2ethyl hexyl) phosphoricacid (98%), Malachite green (99.8%), Hexane (95%), GlacialCH<sub>3</sub>COOH (99.5%),  $Na_2CO_3$  (99%) chemical were obtained from Merck.

An UV-Visible spectrophotometer (ElicoSL159.India) was used to measure the dye concentration. pH of an aqueous solution was measured by a pH meter (ElicoL1120pHmeter with probeCL51B).For cationic and anionic dyes conformation was used in systronics Electrophoresis supply-606. Deionized water was used for preparing all the aqueous solutions.

# **Extraction Method**

The organic solvent [Di (2ethyl hexyl) phosphoric acid+ Hexane] used for extraction was added to the prepared aqueous solution(MG)and pH adjusted using dilute  $Na_2CO_3$ 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 ultra-violet absorption ( $_{max}$ ) for Malachite green was 618 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 strippant were added together into a glass stoppered bottle and shaken at 100 rpm. After 5 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 95% was taken for reliable results.

The results are expressed in terms of distribution coefficient D, extraction efficiency E, and recovery efficiency R defined as

D = C  org. eq/Cf. eq	(1)
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The stripped organic solvent was reused many times for MG dye extraction.

$$E = 1 - Cf. eq/Cf. ini$$
<sup>(2)</sup>

$$R = Cs.eq/Cf.ini$$
(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 (Zhang *et al.*, 2007).

# **RESULT AND DISCUSSION**

#### Effect of pH on feed phase

The pH of the dye solution on the feed phase was varied from 6.0 to  $10.0 \pm 0.1$  with different dye concentrations was studied and the results were shown in Fig. 2. It reveals that the extraction efficiency of dye increases with increasing pH of the feed phase. The maximum percentage of extraction of dye was achieved at pH  $9.0 \pm 0.1$  as follows; 95.1% for 25mg/L, 84.0% for 50mg/L, 80.1% for 75mg/L, 76.0% for 100mg/L respectively. Above pH  $9.0 \pm 0.1$ , the appreciable amount of dye did not extracted from aqueous to organic phase. It might be at pH  $9.0 \pm 0.1$ , the OH<sup>-</sup> ion interact carrier to form anionic D2EHPA which combined with cationic MG dye to form an ion-pair complex. A (Elumalai *et al*, 2014) similar result was Recovery of dye from textile effluents using phenol as an extractant also previous reported.



#### Effect of extract ant concentration

In this experiment, D<sub>2</sub>EHPA concentration was taken with different concentrations such as 25 mg/L, 50 mg/L, 75 mg/L, 100 mg/L of MG dye solution and with varying extractant concentration from 0.03 to 0.30 mol/L at pH 9 ± 0.1 and the results were shown in Fig 3. Usually, we expect the percentage of extraction of dye increased with increasing extractant concentration. The similar results obtained: 95.1% for 25 mg/L, 84.0% for50 mg/L, 80.0% for 75 mg/L, 76.0% for 100 mg/L at pH 9.0±0.1 and 0.24 mol/L concentration of D2EHPA. This confirms that D<sub>2</sub>EHPA was effectively extracting MG from aqueous solutions. At higher dye concentration, the extraction efficiency decreased which might be due to the carrier concentration is not able to extract higher concentrations of

dye; therefore the succeeding tests the extractant  $D_2$ EHPA concentration 0.24mol/L was fixed.



Figure 3 Effect of extractant (D<sub>2</sub>EHPA) concentration (Experimental conditions: Volume of feed phase = 25 mL, extractant concentration = 0.21 mol/L at pH 9.0  $\pm$  0.1, volume of organic phase= 25 mL, A/O ratio=1:1, initial dye concentration25mg/L, equilibration time = 5 min).

#### Effect of diluents

The extractant was mixed with different diluents such as benzene, xylene, toluene and hexane were studied and the results were presented in Table 2. It indicates that the percentage of dye decreased in the order: hexane>xylene>toluene>benzene. It might be D2EHPA is less soluble in benzene, toluene and xylene. The similar extractant (D2EHPA in hexane) system was previously reported for the recovery of cationic dyes from industrial wastewater (muthuraman et al2009). The maximum percentage of extraction (95.1%) was achieved using Hexane used as diluent it might be due to dielectric constant was less compared with other diluent(T.Sekine et al 1977). Among them hexane is less toxic, hence further studies were carried out using hexane as diluent.

**Table 2** Dependence of the nature of diluents on the extraction of MG dye from aqueous solution

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Diluents	Dielectri	Percentage of extraction			
	constant(*)	25mg/L	50mg/L	75mg/L	100mg/L
Benzene	2.28	84.1	76.7	72.9	65.9
Toluene	2.24	85.1	80.2	76.9	72.5
Xylene	2.26	86.5	83.3	78.4	75.6
Hexane	2.02	95.1	84.0	80.0	76.0

# Effect of equilibrium time

The effect of equilibrium time on the extraction of cationic MG dye the aqueous solution of dye was studied in contact with the organic phase containing 0.2 mol/L of D<sub>2</sub>EHPA in hexane for 2-10 min. The extraction efficiency of dye increased with increase equilibrium time. The results were presented in Table 3. The maximum extraction efficiency was achieved in 8min and clear phase separation was also observed. Further studies were carried out at 8 min.

Equilibration time(min)	Percentage of extraction(MG)				
2	25mg/L	50mg/L	75mg/L	100mg/L	
2	80.1	76.0	72.0	60.2	
4	94.6	80.2	76.2	72.9	
6	95.0	83.3	78.4	75.6	
8	95.1	84.0	80.1	76.0	
10	95.1	84.1	80.7	76.7	

 Table3 Effect of equilibration time on extraction of MG dye from aqueous solution

# Effect of temperature

Fig. 4 shows the effect of temperature on extraction of MG dye from the aqueous solution. The extraction efficiency of MG dye remained unchanged up to  $28^{\circ}$ C. The temperature was raised from 28-75°C the extraction rate decreased from 95.1% to 54.0% for 25mg/L, 83.2% to 52.0% for 50mg/L, 80.1% to 46.2% for 75mg/L, 76.2% to 39.0% for 100mg/L respectively. Further studies were carried out at room temperature.



Figure 4 Effect of temperature (Experimental conditions: Volume of feed phase = 25 mL, extractant concentration = 0.21 mol/L at pH 9.0  $\pm$  0.1, volume of organic phase= 25 mL, A/O ratio=1:1, initial dye concentration25mg/L, equilibration time = 5 min).

# Effect of aqueous to organic phase ratio

The aqueous to organic phase (A/O) ratio was varied from 1:1 to 15:1. The organic phase was maintained at 25mL in each experiment. The A/O ratio effect was shown in Fig. 5.



The extraction efficiency of MG dye was 95% at A/O ratio for 1:1 and 2:1. Further increases the A/O ratio the extraction efficiency of MG dye has decreased. This might be due to the low volume of extractant in the organic phase was not sufficiently interact with the dye solution hence the percentage of extraction of dye decreased. At 10:1 ratio, only 72.6% of dye extracted.

Figure 5 Effect of aqueous to organic phase ratio (Experimental conditions: Volume of feed phase =25 mL at pH 9.0  $\pm$  0.1, extractant concentration = 0.21mol/L/,A/O ratio=1:1, equilibration time = 5 min).

# Effect of salt concentration

In the actual industrial textile dye bath effluent, salt like NaCl and Na<sub>2</sub>SO<sub>4</sub> are present. To study the influence of sulphate and chloride concentrations on dye extraction, dye solution with different concentrations of NaCl and Na<sub>2</sub>SO<sub>4</sub> were tested. The results are shown in fig-6a&6b which shows that the extraction efficiency of dye remained unchanged when NaCl was present in the dye solution. In contrast, the percentage of extraction decreased slightly with increasing Na<sub>2</sub>SO<sub>4</sub> concentration from 1000 mg/L to5000 mg/L in aqueous feed phase. This might be due to competitive extraction between dye and SO<sub>4</sub><sup>2°</sup> with D2EHPA.So the percentage of extraction decreased from 95.1% to 85.6% for MG dye 25mg/L initial dye concentration at pH 9±0.1 when Na<sub>2</sub>SO<sub>4</sub> was present in the dye.



Figure 6a Effect of Chloride ion concentration (Experimental conditions: Volume of feed phase =25 mL at pH 9.0  $\pm$  0.1, extractant concentration = 0.21mol/L/,A/O ratio=1:1, equilibration time = 5 min).



Figure 6b Effect of sulphate ion concentration (Experimental conditions: Volume of feed phase =25 mL at pH 9.0  $\pm$  0.1, extractant concentration = 0.21mol/L/,A/O ratio=1:1, equilibration time = 5 min).

#### Stripping studies

It is very important of back extract (strip) the dye from loaded organic phase. Various inorganic and organic acids such as sulphuric acid, hydrochloric acid, nitric acid, phosphoric acid, succinic acid, oxalic acid and glacial acetic acid were used for this purpose. Among them glacial acetic acid and oxalic acid were found to strip the dye very well from loaded organic phase into aqueous phase. The experiment was carried out with glacial acetic acid and oxalic acid concentration varying from 3 to 9mol/L and 0.07 to 0.56mol/L respectively. The stripping efficiency of dye increased with increasing glacial acetic acid and oxalic acid concentration. The maximum stripping percentage of dye (97.6%) was observed at 7mol/L glacial acetic acid and 0.56mol/L oxalic acid solutions as presented in Fig. 7a &7b. Further increase in glacial acetic acid and oxalic acid concentration showed no increase in efficiency. Hence 7mol/L glacial acetic acid and 0.56mol/L oxalic acid were selected as the stripping agent concentration.



Figure 7aEffect of stripping reagent (Glacial acetic acid) concentration (Experimental conditions: Volume of feed phase = 25 mL at pH 9.0  $\pm$  0.1, volume of organic phase = 25mL, A/O ratio = 1:1, extractant concentration = 0.21mol/L, equilibration time = 5 min stripping reagent concentration1 to 9mol/L glacial acetic acid).



Figure 7b Effect of stripping reagent (Oxalic acid) concentration (Experimental conditions: Volume of feed phase mL at pH 9.0  $\pm$  0.1, volume of organic phase = 25 mL, A/O ratio = 1:1, extractant concentration = 0.21mol/L, equilibration time = 5 min stripping reagent concentration 0.07 to 0.63mol/L oxalic acid).

#### Effect of stripping contact time

The stripping contact time was determined for the system containing 25mL of stripping agent equilibrated with varies time intervals. Stripping efficiency did not much increase with increase in contact time in the range of 1-5min. The stripping efficiency as follows: 92.5% for 1min, 94.8% for 2min, 97.6% for 3min and 97.7% for 4min. The maximum stripping efficiency was found to be 97.6% at 3 min. This indicates that the stripping reaction was very fast. Further increase in contact time did not improve stripping efficiency. Hence 3 min was selected as stripping contact time.

## **Reusability of solvent**

The stripped organic solvent was repeatedly reused for MG dye extraction under the optimized condition and the results are presented in the Table 4. The extraction efficiency (95%) of dye did not decrease up to 10 cycles. After 10 cycles, the efficiency dropped. This might be due to loss of D2EHPA during stripping.

**Table 4** Effect of reusability of solvent on the extractionefficiency, %E, of, MG from aqueous solution at 27°C.in0.21M extractant

Number	Percentage of extraction				
of reuse	25mg/L	50mg/L	75mg/L	100mg/L	
1	95.1	94.2	80.6	76.0	
2	95.0	84.0	79.3	75.3	
3	94.8	83.9	79.5	75.2	
4	94.9	83.5	79.6	75.1	
5	94.5	83.8	79.1	75.5	
6	94.9	84.1	79.9	75.7	
7	94.8	84.0	79.6	74.6	
8	94.3	83.6	79.9	74.9	
9	94.2	83.1	79.0	74.3	
10	94.0	83.0	79.1	73.1	

# Application of the developed LLE on textile waste water

The developed LLE was tested for is applicability to the real industrial textile wastewater. The wastewater alkaline and cationic in nature (pH1-10). The removal efficiency is similar to industrial sample under the optimized condition at 27  $^{\circ}$ c.

# CONCLUSIONS

The method presented offers a simple approach for selective extraction of cationic dye for removal and reused. (2-ethyl hexyl) phosphoric acid is able to extract more than 95.1% of cationic dye malachite green oxalate from aqueous solution in a short time of 5 min. The extraction efficiency of dye was not affected in the presence of salt like NaCl but the extraction efficiency of dye was slightly affected in the presence of salt like Na<sub>2</sub>SO<sub>4</sub>.Extraction efficiency of dye decreased with aqueous to organic ratio (A/O 5:1).Stripping reaction was completed within 5 min and 98% of the dye was stripped from

loaded organic phase. The solvent (D2EHPA+ Hexane) can be reused as many as 10 times without loss of efficiency.

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