

# Modified iron oxide nanoparticles as solid phase extractor for spectrophotometric determination and separation of Muroxide

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**Abstract :** This study presents a novel separation, preconcentration and determination of muroxide (MO) in an aqueous solution by sodium dodecyl sulfate (SDS)-bounded iron oxide nanoparticles (S-IONPs). It is shown that the novel magnetic nano-adsorbent is quite efficient for the adsorption and desorption of MO at 25 °C. The effect of temperature, pH of aqueous medium, and interfering ions on the recovery process were also investigated. Methanol was used for desorption of adsorbed MO. Due to the absence of internal diffusion resistance both adsorption and desorption of MO were fast and could be completed within 5 min. The results indicated that the SDS-coated IONPs could be employed in the removal of the dye from wastewater.

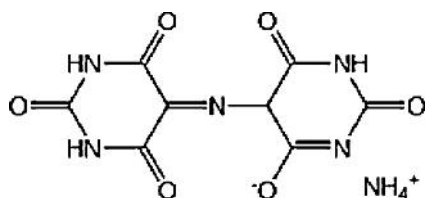
**Keywords :** Iron oxide nanoparticles; Sodium dodecyl sulfate; Preconcentration; muroxide.

## 1. Introduction

Dyes production industries and others related industries such as textile, leather, plastic, food processing, cosmetics, paper, printing, pharmaceutical and dye manufacturing industry which generally used pigments has huge amount of wastewater containing color and organic content [1,2]. Generally their waste water contains different types and categories of dyes (natural and synthetic). Among them synthetic dyes with complex chemical structure has high stability to light and oxidation and are very difficult to biodegrade and may be their degradation products have many hazards to aquatic environment [3-4]. Generally such dyes are resistant to biodegradation and cannot be removed by conventional treatment techniques, while their metabolites lead to carcinogenic, mutagenic and

teratogenic diseases for aquatic and humans lives [5]. Removal of such toxic and bioresistant pollutants from wastewater based on their transfer to the surface of non hazardous adsorbent is very significant for this purpose. The adsorption process perhaps provides an attractive and efficient dye removal procedure for the treatment of dye contaminated water samples, widely applicable because of its ability without any preliminary pretreatment before application [6-7]. Among, the various applied adsorbents those based on application of nanoparticles adsorbent with high surface area and selective and reversible binding of dye molecule has a new powerful pathway for dye removal [8-9]. Metallic nanoparticles based adsorbents have been widely applied for nanoscale devices to bind and coordinate biological materials. MO (Fig. 1) ( $\text{NH}_4\text{C}_8\text{H}_4\text{N}_5\text{O}_6$ , or  $\text{C}_8\text{H}_5\text{N}_5\text{O}_6 \cdot \text{NH}_3$ )

(ammonium purpurate or (MX) as ammonium salt of purpuric acid, may be prepared by heating alloxantin in ammonia gas to 100 °C or by boiling uramil (5- 54 aminobarbituric acid) with mercury oxide. Generally, MO applied as complexometric indicator for evaluation and determination of Ca, Cu, Ni and Co ions and some other rare earth element [10–11].



**Fig. 1. Chemical structure of basic MO.**

In this study, we used sodium dodecyl sulfate (SDS) which is an anionic surfactant and tends to interact with surface of nanoparticles and coated them. Iron oxide magnetic nanoparticles as cores and SDS as ionic exchange groups were used for recovery and determination of MO dye.

## 2. Experimental

### 2.1. Chemicals and reagents

Muroxide, methanol (99.9% m/m), ammonia solution (25% m/m), hydrochloric acid (37% m/m), FeCl<sub>3</sub> (96% m/m) and FeCl<sub>2</sub> · 4H<sub>2</sub>O (99.9% m/m) were purchased from Merck (Darmstadt, Germany) and were used without further purification processes. SDS was obtained from Sigma. All chemicals and reagents were of analytical grade purity.

### 2.2. Apparatus

The spectrophotometric measurements were carried out with a Perkin Elmer lambda25

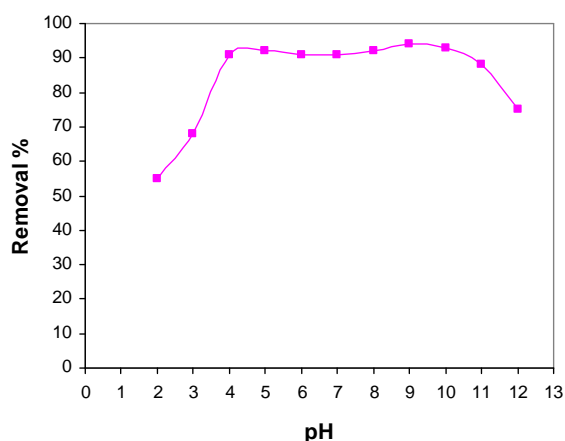
spectrophotometer. A Metrohm pH meter model 632 (Herisau, Switzerland) for pH adjustments and a magnet (1.2 T, 10 cm × 5 cm × 2 cm) for settlement of magnetic nanoparticles were used.

### 2.3. Preparation of SDS-bounded iron oxide nanoparticles (S-IONPs)

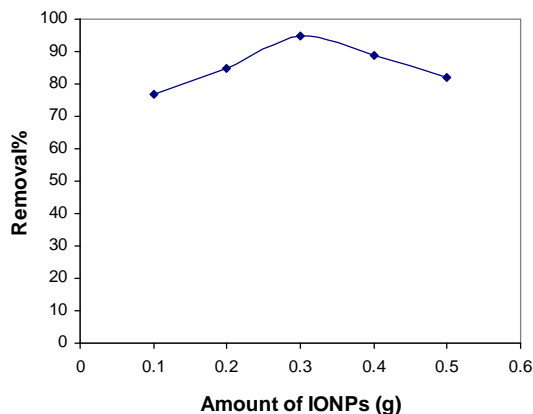
Iron oxide nanoparticles were prepared according to a previous work [12]. In order to coat the particles, 5mL of SDS solution (5% m/v) was added to about 5 g of damped nanoparticles in a beaker. The solution was stirred for 1min by a glassy rod and the beaker was then placed on the magnet. After complete precipitation of S-IONPs occurred the solution was decanted and ferrofluid was washed with distilled water for several times (3 times on average) to eliminate extra amount of surfactant from nanoparticles.

### 2.4. Adsorption and desorption of muroxide

About 0.6 g of damped S-IONPs (equivalent to 0.06 g dry SIONPs) were added to the 50mL of MO solution (0.01–1 μg mL<sup>-1</sup>) in a beaker. The pH of the solution was adjusted to 7 by addition of 2mL of 0.03M of phosphate buffer pH 7. The mixture was stirred by a glassy rod for about 100 s and the beaker was then placed on the magnet. The S-IONPs which adsorbed MO (MO-S-IONPs) were separated magnetically. The mixture was decanted and the solution above the nanoparticles was removed completely. Finally 2.5mL of desorbent solution was added to the MO-S-IONPs in a beaker. The beaker was placed on the magnet and the mixture was decanted. The absorption of the solution which has been separated from IONPs by magnet was measured spectrophotometrically at 547 nm.



**Fig. 2. Adsorption of MO on S-IONPs as a function of pH.**



**Fig. 3. Effect of different amounts of IONPs on the adsorption of MO.**

**Table 1: Effect of interfering ions on the removal of MO by S-IONPs.**

Ions	Tolerance ratio
$K^+$ , $Na^+$ , $NO_3^-$	700
$F^-$ , $CO_3^{2-}$ , $HCO_3^-$	800
$Ca^{2+a}$ , $Mg^{2+a}$	600

<sup>a</sup> After addition of 10 mL 0.1 M EDTA

### **3. Results and discussion**

The removal and preconcentration of MO by surfactant modified IONPs (S-IONPs) showed fast separation of this dye from the bulk of the water solutions. Moreover, the removal of MO reached equilibrium within about 5 min. Such a fast adsorption rate could be referred to the absence of internal diffusion resistance [13].

#### **3.1. Effect of pH variation**

The effect of pH on the adsorption of murexide ( $2.0 \mu\text{g mL}^{-1}$ ) by 0.4 g of damped IONPs at  $25^\circ\text{C}$  showed (Fig. 2) that the adsorption and removal of murexide remains constant at pH range from 3 to 9. The mean pH value (6) was used as optimum pH for further works. The volume of phosphate buffer used in 50 mL of test solution was optimized and it was found that 1 mL of buffer solution can be used as the optimum volume. Higher volumes of buffer solution decreased the adsorption and also the removal of MO.

#### **3.2. Effect of the amount of IONPs**

The amount of IONPs for complete removal of MO ( $2 \mu\text{g mL}^{-1}$ ) in a 50 mL solution at pH 6 was investigated. The results are shown in Fig. 3 and indicated that addition of 0.3 g of damped IONPs per 50 mL solution of MO ( $2 \mu\text{g mL}^{-1}$ ) lead to maximum separation of dye (Fig. 3).

#### **3.3. Effect of the SDS amount**

The results revealed that by increasing the amount of surfactant, the rate of removal of MO was nearly constant. Therefore, 0.75 mL of 0.5% (m/v) solution of SDS per 50 mL solution was chosen as optimum amount of SDS for further experiments.

#### **3.4. Effect of temperature**

The adsorption of MO ( $2 \text{ g mL}^{-1}$ ) in a 50 mL solution using 0.3 g of damped S-IONPs at pH 6 under different temperatures showed that the adsorbed amount of MO remained almost constant at different temperatures and the removal was greater than 91%.

#### **3.5. Desorption solvent (eluent)**

In this concern, desorption of the MO might be achieved using mixed methanol-acetic acid solution [25–27] or pure methanol solvent [28]. The study revealed that the desorbed MO was increased with increasing the content of methanol in the solution and the dye could be completely desorbed in the presence of pure methanol. In this study more than 98% of MO could be desorbed and recovered by pure methanol and the recovery percent from a sample solution by S-IONPs reached 97.5% .

#### **3.7. Effect of interfering ions**

The effect of common ions such as  $K^+$ ,  $Na^+$ ,  $Ca^{2+}$ ,  $Mg^{2+}$ ,  $F^-$ ,  $NO_3^-$ ,  $CO_3^{2-}$  and  $HCO_3^-$  on the

removal process under optimum conditions are presented in Table 1. These results showed that  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  cations were interfered completely even in the equal concentration with the analyte. EDTA salt was used to eliminate the interference effect of both cations.

#### **4. Conclusion**

Muroxide could be removed from an aqueous solution by the SDS-bounded iron oxide

nanoparticles. The nanoparticles can rapidly adsorbed MO and desorbed it in a suitable solution in less than 5min.

#### **Acknowledgement**

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