

## Research Article

# Spectroscopic and Thermogravimetric Investigation of Cd(II) Dinonyldithiophosphate: Removal of Cadmium from Aqueous Solutions

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Dinonyldithiophosphoric acid (HDDTP) was synthesised from the reaction of phosphorus pentasulphide and nonyl alcohol. Dinonyldithiophosphate complex of cadmium [Cd(DDTP)<sub>2</sub>] was prepared by mixing solutions of Cd(II) with HDDTP in ethanol at room temperature. The acid and its complex were characterised by elemental analysis and spectroscopy. The thermal behaviour of Cd(DDTP)<sub>2</sub> was investigated by thermogravimetric analysis. Removal of Cd(II) from aqueous media by HDDTP solution was also studied. The optimum conditions for removal of Cd(II) were investigated for effects of solvent, pH, contact time, concentration, and inorganic anions. Cd(II) was quantitatively removed from aqueous solutions at the pH range of 0.5 < pH < 6, under the conditions that the stoichiometric ratio of HDDTP/Cd(II) ≥ 2/1. It can be stated that contact of the Cd(II) with HDDTP was sufficient for quantitative removing of cadmium from acidic aqueous solutions.

## 1. Introduction

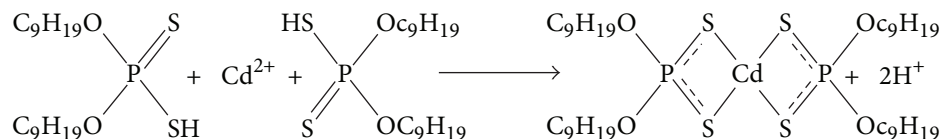
Organodithio compounds are widely used in chemical and industrial applications [1–3]. Due to the synthesis and purifying challenges of long chains dialkyldithiophosphoric acids, their use in experiments was rare [2, 4], although long chains dialkyldithiophosphoric acids are more easily transferred into the organic phase [5] and more stable against hydrolysis than the short-chain ones [6]. The well-known lubricant zinc dialkyldithiophosphate (ZDDTP) has been extensively investigated [7], but only a few articles have been published on the thermal properties of the other heavy metal dialkyldithiophosphates [8, 9]. Similarly, there are only a few studies on Cd(II) dialkyldithiophosphates for its removal from aqueous solutions [10–14], but there are no articles on Cd(II) dinonyldithiophosphate.

Cadmium is one of the most toxic heavy metals for living organisms. One of the important natural heavy metal pollution sources is phosphate rocks/phosphorus fertilizers. During the production of phosphorus fertilizers, dangerous amounts of heavy metals pollute the environment. The main

source of cadmium accumulation in the agricultural lands is the phosphorus fertilizers and its restriction is almost impossible [15]. Thus, cadmium is one of the common environmental contaminants and widely distributed around the world.

The other important pollution sources are different industrial activities such as plating of metal, mining, pigments, polyvinyl chloride plastics, petrochemicals, alloy and steel industries, and municipal discharge-wastewater [16]. According to the international standards the threshold level of Cd(II) concentration should be lower than 0.01 mg/L in wastewater used for irrigation [17]. Therefore it is necessary to eliminate Cd(II) from contaminated waters.

Because of its high mobility in soil and plant system and easy accumulation, cadmium may cause toxicities such as cancer (lung and prostate), kidney damage, and bone diseases [18]. To reduce or remove Cd(II) from wastewater or contaminated waters, various methods have been developed including adsorption [19–22], biosorption [23, 24], ion exchange [25], hydrogel [26], and chemical precipitation-extraction [27].

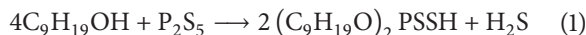


SCHEME 1

In the present study, dinonyldithiophosphoric acid and its complex of Cd(II) were prepared and characterised by spectroscopic techniques and thermal analysis. The removal of Cd(II) from aqueous solutions by using dinonyldithiophosphoric acid was investigated.

## 2. Materials and Methods

**2.1. Preparation and Characterisation of HDDTP and Cd(DDTP)<sub>2</sub>.** HDDTP was prepared from the reaction of phosphorus pentasulphide and nonyl alcohol by using a microwave oven (CEM-MDS 2000, Matthews, USA) according to a previously published method [5] and, to make a sufficiently pure compound, the method described in the literature [6] was modified. The obtained mixture was cooled and the unreacted solid part was filtered. The oily acid was converted to calcium salt by neutralisation with lime slurry at room temperature. The salt was separated and washed with hexane and acidified with 3 M H<sub>2</sub>SO<sub>4</sub>. The free dinonyldithiophosphoric acid floating on top of aqueous phase was separated as a yellowish-green viscous liquid with 98% purity:



An adequate amount of 10<sup>-2</sup> M solutions of viscous oil HDDTP and Cd(II) in ethanol is mixed at room temperature, and Cd(DDTP)<sub>2</sub> was obtained as white precipitate with melting point 28.5°C (see Scheme 1).

The prepared acid and its complex were characterised by elemental analysis (EA), ultraviolet-visible (UV-vis), infrared (IR) spectrophotometers, and inductively coupled plasma-optical emission spectrometry (ICP-OES), and the thermal behaviour of Cd(DDTP)<sub>2</sub> was investigated by thermogravimetric analysis (TGA). Elemental analysis was carried out using a CHNS-O elemental analyser (Carlo Erba EA 1108, Milan, Italy). UV-vis spectra of the samples dissolved in CCl<sub>4</sub> were recorded in the range 200–900 nm by a UV/visible spectrophotometer (Unicam-UV2-100, Cambridge, UK) using a 1 cm quartz cell. The IR spectra were recorded on a Fourier transform IR (FTIR) spectrometer (Midac Co. M Series, Costa Mesa, CA, USA) as neat liquid, using a NaCl cell in the range 400–4000 cm<sup>-1</sup>. The concentrations of Cadmium were determined by an ICP-OES (Perkin Elmer 2100 DV, Massachusetts, USA) at 228.802 nm. The TG measurement was performed using a thermogravimetric analyser (Shimadzu TGA-50, Kyoto, Japan) with a temperature range from room to 800°C at the rate of 20°C min<sup>-1</sup> under nitrogen (20 mL min<sup>-1</sup>) atmosphere.

**2.2. Experiments on Cd(II) Removal from Aqueous Solutions.** We have prepared solutions of HDDTP (2·10<sup>-2</sup> M) in the different solvents such as kerosene, benzene, *n*-hexane, petroleum ether, and carbon tetrachloride. The stock solution of cadmium (1·10<sup>-2</sup> M) was also prepared in distilled water. All of the chemicals were of analytical reagent grade. Removal of Cd(II) from aqueous solutions to the organic phase was investigated as a function of pH for various anions, solvents, extractant concentration, and extraction times. Extraction or contact experiments related effect of time and pH were performed between 1 and 5 minutes and pH: 0.5 and 6.0, respectively. Removal of Cd(II) using HDDTP was carried out from different 0.1 M aqueous solution of NaClO<sub>4</sub>, NaCl, NaBr, NaNO<sub>3</sub>, and CH<sub>3</sub>COONa salts by stirring equal volumes (25 mL) of 10<sup>-3</sup> M organic and aqueous phases at room temperature in a conventional separation funnel at different times (min). The pH values were adjusted by adding sulphuric acid or sodium hydroxide, and the pH values were measured with a pH meter (Jenway 3010, London, UK). After separation of the phases, an aliquot of the aqueous part was analysed by an ICP-OES for the particular Cd(II) ion concentrations at 228.802 nm.

The recovery percentage (*R*%) of Cd(II) can be calculated as follows:

$$R(\%) = \frac{[C^{n+}]_0 - [C^{n+}]}{[C^{n+}]_0} \times 100, \quad (2)$$

where [C<sup>n+</sup>]<sub>0</sub> is initial concentration of the Cd(II) in the aqueous phase and [C<sup>n+</sup>] is the concentration in the aqueous phase after extraction.

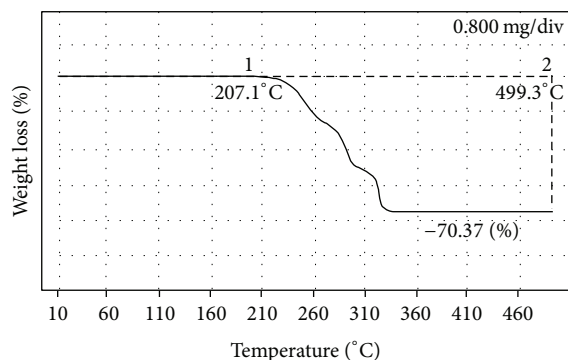
## 3. Result and Discussion

**3.1. Characterisation of HDDTP and Cd(DDTP)<sub>2</sub>.** Potentiometric titration of the acid with 0.1 M NaOH showed that the product contained 98% HDDTP, which is a yellowish-green viscous oil. The elemental analysis results for (C<sub>9</sub>H<sub>19</sub>O)<sub>2</sub>PSSH; Analytical calculated (%): C, 56.5; H, 10.2; S, 16.7, and found values (%): C, 56.8; H, 10.3; S, 16.5. Elemental analysis of the recovered white solid Cd(DDTP)<sub>2</sub> (melting point 28.5°C) was C, 49.3; H, 8.7; S, 14.6; P, 7.1; and Cd, 12.8 for analytical calculations and the found values were found to be C, 49.5; H, 8.2; S, 14.4; P, 7.0; and Cd, 12.6, and the results are in agreement with each other.

The UV-vis absorption spectrum of dinonyldithiophosphoric acid in CCl<sub>4</sub> using a 1 cm quartz cell showed a peak at 267 nm can be attributed to *n* → π\* electronic transition related >P(=S)SH group (Table 1). The UV-vis absorption

TABLE 1: The UV-vis. ( $\lambda$ , nm) and IR ( $\bar{\nu}$ ,  $\text{cm}^{-1}$ ) data of HDDTP and Cd(DDTP)<sub>2</sub>.

Compound	$\lambda_{\text{max}}$ nm	(P)-O-C $\bar{\nu}$ , $\text{cm}^{-1}$	P-O-C $\bar{\nu}$ , $\text{cm}^{-1}$	(P=S) $\bar{\nu}$ , $\text{cm}^{-1}$	(P-S) $\bar{\nu}$ , $\text{cm}^{-1}$	(S-H) $\bar{\nu}$ , $\text{cm}^{-1}$
HDDTP	267	1300–940	900–820	740–660	540	2550
Cd(DDTP) <sub>2</sub>	294	1251–930	838–718	677–649	570	—

FIGURE 1: Thermogravimetric analysis of Cd(DDTP)<sub>2</sub> at a heating rate of 20°C min<sup>-1</sup> under atmosphere of nitrogen.

spectrum of Cd(DDTP)<sub>2</sub> was obtained at the same conditions and relevant band was found 294 nm (Table 1) and can be attributed to the metal-ligand electronic transition.

The recorded IR data of the acid and complex (Table 1) showed a good agreement with earlier studies carried out on other dialkyldithiophosphates [1, 2]. The IR spectral data of cadmium dinonyldithiophosphate have been recorded at the range of 4000–400  $\text{cm}^{-1}$  (Table 1) and it was observed that the characteristic band of the dinonyldithiophosphoric acid at 2550  $\text{cm}^{-1}$  related to (P)S–H stretching vibration was vanished with the formation of Cd(DDTP)<sub>2</sub>, and there was no absorption record in the region. Classically, the strong intensity band present in the region 740–660  $\text{cm}^{-1}$  has been attributed to P=S double bond and the absorption peak at ~540  $\text{cm}^{-1}$  has been assigned to P–S single bond (Table 1). By formation of the complex, the absorption values of the bands were significantly shifted, and P=S peak was shifted down, while P–S peak was shifted up. Thus, P=S double bond strength was decreased, while P–S single bond strength was increased. This result was in agreement with the previous studies on the other dialkyldithiophosphates [9, 10, 13]. This approach can be easily acceptable for simple M<sup>+</sup> salt of dithiophosphoric acids; for example, these two bands (P=S and P–S) of potassium butyldithiophosphate can be seen at 703  $\text{cm}^{-1}$  and 550  $\text{cm}^{-1}$ , respectively [13].

According to the obtained thermogram (Figure 1), Cd(DDTP)<sub>2</sub> is stable at temperatures up to ~207°C and does not contain water. As shown in Figure 1, Cd(DDTP)<sub>2</sub> decomposes in three steps, and the main weight loss of the complex takes place between 207 and 332°C because of the decomposition of the organic part. The total weight loss percentage of the Cd(DDTP)<sub>2</sub> is 70.4% up to the temperature 499°C. The weight change is lower than 3% over 332°C temperature. The results are in good agreement with

our earlier studies on heavy metal dialkyldithiophosphate complexes [9].

**3.2. Cd(II) Removal from Aqueous Solutions.** The molar ratio of dinonyldithiophosphoric acid/cadmium has main influence in the extraction process (Table 2(a)). Cd(II) was quantitatively recovered from the acidic aqueous solution under the conditions that the stoichiometric ratio of HDDTP/Cd(II) was >2/1. When the experiment was performed with lower molar ratio, such as equal volumes of 1·10<sup>-3</sup> M HDDTP, and 1·10<sup>-3</sup> M metal solution was mixed, the removal ratio of Cd(II) was found to be 60%. A favourable result was obtained, and Cd(II) was quantitatively extracted from the aqueous solution to organic phase when molar ratio of HDDTP/Cd(II) increased to 3 (Table 2(a)). The pH experiments on the extraction of Cd(II) showed that (Table 2(b)) it is possible to suggest Cd(II) can be quantitatively extracted between pH 0.5 and 6. The contact time of metal-ligand on the removal of Cd(II) was examined (Table 2(c)), and it was found that 1-minute stirring time can supply quantitative removal of cadmium.

The experiment conducted on different solvents such as kerosene, benzene, *n*-hexane, petroleum ether, and CCl<sub>4</sub> implies that there is no difference between solvents used (Table 2(d)). Therefore, all the investigated solvents can be used in the removal of Cd(II). We have also investigated the effect of common inorganic anions such as NaClO<sub>4</sub>, NaCl, NaBr, NaNO<sub>3</sub>, and CH<sub>3</sub>COONa on the extractions of Cd(II) from aqueous solution. We have found that there were no differences except for CH<sub>3</sub>COONa salt (Table 2(e)). Cadmium acetate was also a stable complex and its solubility in water was very high. In addition, our primary results showed that the Cd(II) could not be recovered back by 1–5 M H<sub>2</sub>SO<sub>4</sub> from organic to aqueous phase.

Our results were in agreement with the studies related to extraction of Cd(II) by diethyl-, dipropyl-, and dibutyl dithiophosphates [10–14]. In these studies they showed that pH 3–5 is suitable for Cd(II) removal, and they form different Cd(II) complexes in the basic pH in order to regenerate from organic phase to aqueous phase. The compounds containing long chain alkyl or more carbon are easily soluble in organic solvents and their solubility in aqueous media decreases. If the results were compared with our findings, the effect of dinonyl group can easily be seen. Cd(DDTP)<sub>2</sub> with long nonyl group is insoluble in water and very soluble in organic solvents and 1 minute is sufficient for quantitatively removing of cadmium from aqueous media. There was an acceptable agreement between the previous results and our findings on removing Cd(II) from aqueous solutions using alkyldithiophosphates as extractants.

TABLE 2: Determination of removed cadmium from aqueous solutions by dinonyl dithiophosphoric acid. (a) Effect of HDDTP concentration, (b) influence of pH of aqueous phase, (c) influence of contact time, (d) effect of solvents, and (e) effect of salts ( $\lambda$ : 228.802 nm).

(a) Effect of HDDTP concentration (pH: 3; time: 3 min.; solvent: kerosene)						
HDDTP conc. ( $10^{-3}$ M)	0.5	1.0	2.0	3.0	4.0	5.0
Extraction rate (%) Cd <sup>++</sup>	28	60	100	100	100	100
(b) Influence of pH of aqueous phase (HDDTP: $3 \cdot 10^{-3}$ M; time: 3 min; solvent: kerosene)						
pH of aqueous phase	0.5	1	2	3	4	6
Extraction rate (%) Cd <sup>++</sup>	100	100	100	100	100	100
(c) Influence of contact time (HDDTP: $3 \cdot 10^{-3}$ M; pH: 3; solvent: kerosene)						
Time (Min.)	1	2	3	4	5	
Extraction rate (%) Cd <sup>++</sup>	100	100	100	100	100	100
(d) Effect of solvents (HDDTP: $3 \cdot 10^{-3}$ M; pH: 3; time: 3 min)						
Solvents	Kerosene	Benzene	<i>n</i> -Hexane	Petroleum ether	Carbon-tetrachloride	
Extraction rate (%) Cd <sup>++</sup>	100	100	100	100	100	
(e) Effect of salts (HDDTP: $3 \cdot 10^{-3}$ M; pH: 3; time: 3 min; solvent: kerosene)						
Salts (0.1 M)	NaClO <sub>4</sub>	NaCl	NaBr	NaNO <sub>3</sub>	CH <sub>3</sub> COONa	
Extraction rate (%) Cd <sup>++</sup>	100	100	100	100	0.0	

Present results showed that Cd(II) could be easily removed from the acidic aqueous solutions by HDDTP/any organic solvent.

#### 4. Conclusions

Dinonyldithiophosphoric acid instantly reacts with Cd(II) to form a white solid complex at room temperature. The Cd(DDTP)<sub>2</sub> is stable up to  $\sim 207^\circ\text{C}$  and insoluble in water but very soluble in organic solvents such as kerosene, benzene, *n*-hexane, petroleum ether, and carbon tetrachloride and these are suitable solvents for using HDDTP or its salt as extractants.

The experiments concerning the influence of the concentration of HDDTP/any solvent on removal of Cd(II) from the acidic aqueous solutions show that cadmium was quantitatively removed, under the conditions that the stoichiometric ratio of HDDTP/Cd(II)  $\geq 2/1$ . The experiments related with pH of aqueous media show that Cd(II) can be removed completely from the acidic solutions at pH range of  $0.5 < \text{pH} < 6$ . When the technique is applied on any acidic wastewater or sludge, it does not require any pH adjustment and the method can supply low costs. Experiments related to effect of contact time for removal of Cd(II) showed that contact of the HDDTP with Cd(II) was sufficient for its quantitative removing. Cd ions were quantitatively transferred to organic phase after 1 min. mixing of Cd(II) and HDDTP. Because the potential ecological risk of cadmium is relatively high, it is necessary to remove or reduce Cd(II) levels in the municipal or industrial discharges. It can be concluded that HDDTP is an appropriate substance for removal of Cd(II) from acidic aqueous solutions.

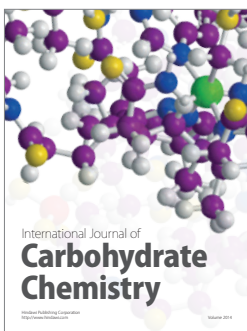
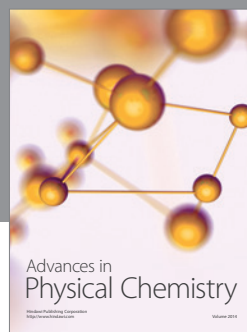
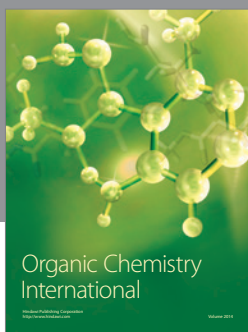
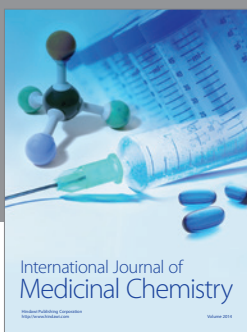
#### Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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