



Interaction of 2, 4-Dinitrophenol (2, 4-DNP) and 2, 4, 6-Trinitrophenol (2, 4, 6-TNP) with Metal Hexacyanoferrate (II) Complexes

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Author's contribution

The sole author designed, analyzed and interpreted and prepared the manuscript.

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ABSTRACT

Antimony, cadmium and zirconium hexacyanoferrates (II) were synthesized and characterized by elemental and spectral studies. Interaction of 2, 4-dinitrophenol (2, 4-DNP) and 2, 4, 6-trinitrophenol (2, 4, 6-TNP) with antimony, cadmium and zirconium hexacyanoferrates (II) have been studied at neutral pH (7.00 ± 0.01) and a temperature of $30 \pm 1^\circ\text{C}$. The progress of adsorption was followed spectrophotometrically by measuring the absorbance of substituted phenols at their corresponding λ_{max} . The nature of adsorption has been interpreted from the shape of adsorption isotherms. The Langmuir type of adsorption is followed in the concentration range of $10^{-3} - 10^{-4}$ M of 2, 4-DNP and 2, 4, 6-TNP solutions. The 2, 4, 6-TNP was found to have greater affinity for the antimony, cadmium and zirconium hexacyanoferrates (II) than 2, 4-DNP. High adsorption capacity has been observed for cadmium hexacyanoferrate (II) while it is minimum with zirconium hexacyanoferrate (II), which indicate highly porous characteristics of cadmium hexacyanoferrate (II) in comparison to other metal hexacyanoferrates (II) studied. Removal of toxic phenol is necessary for the protection of our environment. Phenols react with soil to reduce their fertility therefore its removal from soil is also important for our food security.

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1. INTRODUCTION

It is assumed that transition metal ions, abundant in the primeval sea, would have formed complex compounds with simple and readily available molecules [1,2]. It is therefore, reasonable to assume that transition metal ions could have easily formed several soluble and insoluble complexes with the abundant cyanide ion present in the primeval sea. The insoluble double metal cyano complexes thus formed could have settled at the bottom of the sea or on the sea shore and catalysed a number of oxidation, condensation-oligomerization reaction on their surfaces. The Arrhenius [3] had proposed existence of metal ferri-and ferrocyanides on primitive earth. Insoluble double metal hexacyanoferrates (II) are an important class of compounds and have been widely used as adsorbents [4,5] photosensitizers [6] and ion-exchangers [7,8]. Phenol compounds are strictly regulated compounds worldwide because of their extreme toxicity. In the receiving bodies themselves both flora and fauna are adversely affected. Phenols react with soil to reduce their fertility. Removal of phenol is important for both agricultural and environmental point of view. Phenol compounds are utilized and also found in the effluents of industries such as coke plants, paint and dye formation, automobile manufacturing, explosive manufacturing, chemicals, synthetic fibre and resin manufacture etc.

Lawal et al. [9] reported removal of phenol from water by using carbon adsorbents prepared by pyrolysis of sorghum and millet straws in orthophosphoric acid. Adsorption characteristics of natural zeolites as solid adsorbents for phenol removal from aqueous solutions kinetics, mechanism and thermodynamics studies was investigated by Yousef et al. [10]. Kiran et al. [11] demonstrated simultaneous adsorptive removal of cyanide and phenol from industrial wastewater. Enzyme - based processes for removal of phenol from wastewater current status and future challenges are described by Narayan et al. [12]. Ardelean et al. [13] reported adsorption characteristics of some polymeric materials with olefin groups used for removal of phenol and p-nitrophenol from aqueous solutions. Adsorption of phenol from wastewater using locally available adsorbents from agricultural waste materials was investigated by

Girish et al. [14]. Literature survey showed adsorptive interaction of phenols with a number of adsorbents viz: potato polyphenol oxidase [15], natural clay [16], activated neem leaf [17], yeast [18] and zeolites [19].

A search of the literature indicated that several reports are available on adsorptive interaction and removal of phenols using different types of adsorbents, but little work has been reported on the adsorption and removal of phenols using metal hexacyanoferrates (II) as adsorbents. In view of this attempt has been made to determine the surface properties of metal hexacyanoferrates (II) and phenol system. In addition, current work describes a removal of 2, 4-DNP and 2, 4, 6-TNP through adsorptive interaction with antimony, cadmium and zirconium hexacyanoferrates (II). The 2, 4-DNP and 2, 4, 6-TNP were chosen as representative phenols due to their two and three electron withdrawing groups, respectively.

2. METHODOLOGY

2.1 Chemicals

Antimony chloride, cadmium chloride, zirconyl chloride, 2, 4-DNP, 2, 4, 6-TNP and potassium ferrocyanide were obtained from BDH, Poole, United Kingdom. All chemicals used were of Analytical Reagent Grade. All solutions were prepared in doubly distilled water.

2.2 Synthesis of Metal Hexacyanoferrate (II) Complexes

Antimony, cadmium and zirconium hexacyanoferrates (II) were prepared by adding metal chloride (500 ml, 0.1 M) and potassium hexacyanoferrate (II) (167 ml, 0.1M) solution with constant stirring [20]. The reaction mixture was heated on a water bath for 3 h and kept as such at room temperature for 24 h. The precipitate was filtered under vacuum, washed several times with distilled water and dried in an air oven at 60°C. The dried product was grounded and sieved to 125 µm particle size.

2.3 Characterization of Metal Hexacyanoferrate (II) Complexes

All three metal hexacyanoferrates (II) were characterized by elemental and spectral studies. Percentage composition of metals and carbon,

hydrogen, nitrogen were determined with the help of IL 751, atomic absorption spectrophotometer and a CEST – 118 CHN Analyzer, respectively. The values are given in Table 1.

These metal hexacyanoferrates (II) are found to be insoluble in water and amorphous in nature and give no XRD pattern. Antimony hexacyanoferrate (II), cadmium hexacyanoferrate (II) and zirconium hexacyanoferrate (II) complexes were found to have light blue, light green and gray colours, respectively. Antimony, cadmium and zirconium hexacyanoferrates (II) revealed infrared spectral peaks around 3600 cm^{-1} and 1600 cm^{-1} characteristic of water molecules / OH group and HOH bending, respectively. Two sharp bands at around 2000 cm^{-1} and 580 cm^{-1} are characteristics of cyanide and Fe-C stretching, respectively [21]. Another sharp band at 490 cm^{-1} probable shows the presence of metal – nitrogen band due to polymerization [22]. The infrared spectral data are given in Table 2.

2.4 Adsorption Studies

Effects of pH on adsorption of substituted phenols on metal hexacyanoferrate (II) complexes

The adsorption of substituted phenols on metal hexacyanoferrates (II) as a function of pH range (1.0-10.0) and concentration (10^{-4} M) of adsorbates was studied at room temperature $30\pm 1^\circ\text{C}$. The desired pH was maintained by dilute NaOH or HCl solutions. The pH of each solution was verified using a pH meter (Lamotte Chemical Company). A series of 50 mL test tubes were employed for the adsorption studies. 10 mL of substituted phenol was placed in into test tubes and adjusted to desired pH. Metal hexacyanoferrate (II) (25 mg) was added to each test tube. The test tubes were then stoppered and agitated for 24 h. The equilibrium was attained in about 6 h. The contents were then centrifuged at 3000 rpm for 15 minutes, decanted and the absorbance of 2, 4- DNP and 2,4,6- TNP solutions after adsorption was measured spectrophotometrically at their corresponding λ_{max} 360 nm and 382 nm, respectively.

Effects of concentration on adsorption of substituted phenols on metal hexacyanoferrate (II) complexes

The adsorption of 2, 4 -DNP and 2, 4, 6 -TNP on metal hexacyanoferrates (II) as a function of

phenol concentrations ($10^{-3} - 10^{-4}\text{ M}$) was studied at room temperature ($30\pm 1^\circ\text{C}$). The final adsorption studies of phenols at various concentrations was studied at neutral pH (7.0 ± 0.01), this is due to physiological significance of neutral pH because most of the reactions in biological systems takes place at neutral pH range. A series of 50-mL test tubes were employed. Each tube was filled with 10 mL of phenol solution of varying concentration and adjusted to neutral pH (7.0 ± 0.01). Metal hexacyanoferrate (II) (25 mg) was added to each test tube and agitated for 24 h. Equilibrium was attained in about 6 h. The equilibrium time and concentration range were however decided after a good deal of preliminary investigations. The absorbance of 2, 4-DNP and 2, 4, 6-TNP were measured spectrophotometrically at their corresponding λ_{max} 360 nm and 382 nm, respectively. The amount of substituted phenols adsorbed was calculated by the difference in concentration before and after the adsorption.

3. RESULTS AND DISCUSSION

The percentage uptake of 2, 4-DNP and 2, 4, 6-TNP as a function of pH and was studied over a pH range of 1.0 – 10.0 (Figs. 1 & 2). The percentage uptake was calculated by equation

$$\% \text{ uptake} = \frac{\text{Difference in concentration of substituted phenols before and after adsorption}}{\text{Concentration of substituted phenol before adsorption}} \times 100$$

The values of percentage uptake of 2, 4-DNP and 2, 4, 6-TNP on different metal hexacyanoferrates (II) are given in Tables 3 & 4, respectively. The maximum pH value for the uptake of 2, 4-DNP and 2, 4, 6-TNP were found to be 4.0 and 2.0, respectively. It seems that uptake of both 2, 4-DNP ($\text{pK}_a = 3.96$) and 2, 4, 6-TNP ($\text{pK}_a = 0.38$) are maximum near their respective pK_a on all three metal hexacyanoferrates (II) studied. Above the pK_a value of the phenols, the phenolate ion would be predominant. With the negative charge on phenolate ion seems to be repelled by surface charge of metal hexacyanoferrates (II), causing less adsorption than in the case of dissociated phenols at pH values lower than pK_a [23]. The 2,

4 - DNP and 2, 4, 6 - TNP both substituted phenols have pKa values below neutral pH (7.0 ± 0.01), so high adsorption occur at acidic pH range.

It is observed from Tables 3 & 4 that 2, 4, 6-TNP is strongly adsorbed on all three metal hexacyanoferrates (II) in comparison to 2, 4-DNP. Electron density of aromatic ring is strongly influenced by the nature of the substituent's present in it. The nitro group is electron withdrawing and reduces the overall electron density in the pi- system of benzene ring. Since the presence of OH group is common to both 2, 4-DNP and 2, 4, 6-TNP, higher adsorption in case of 2, 4, 6-TNP is due to presence of one more withdrawing nitro group.

Adsorption isotherm of 2, 4-DNP and 2, 4, 6-TNP on metal hexacyanoferrates (II) are shown in Figs. 3 & 4 and values of maximum adsorption are given in Table 5. It is observed from Figures 3 & 4 that adsorption is fast in all cases and isotherms are regular positive and concave to the concentration axis. Slow adsorption gradually leading to constancy takes place at higher adsorbate concentration. In general the adsorption curves were characterized by a gradual rise and flattening at higher adsorbate concentration. The affinity of metal hexacyanoferrates (II) towards adsorption for both 2, 4-DNP and 2, 4, 6-TNP follows the order:

Cadmium hexacyanoferrate (II) > antimony hexacyanoferrate (II) > zirconium hexacyanoferrate (II)

Table 1. Element analysis of antimony, cadmium, and zirconium hexacyanoferrates (II)

Metal Hexacyanoferrates (II) ^a	Percentage (%) found				
	Metal	Iron	Carbon	Hydrogen	Nitrogen
SbFc	47.50	12.50	15.25	0.89	17.79
CdFc	41.59	11.50	15.13	1.69	17.37
ZrFc	34.66	10.30	12.97	2.90	15.41

^aSbFc, antimony hexacyanoferrate (II); CdFc, cadmium hexacyanoferrate (II); ZrFc, zirconium hexacyanoferrate (II)

Table 2. Infrared spectral data of antimony, cadmium, and zirconium hexacyanoferrates (II)

Metal Hexacyanoferrates (II) ^a	Adsorption frequency (cm ⁻¹)				
	H ₂ O molecules/OH groups	HOH bending	ν C \equiv N stretching	ν Fe-C	Metal-N*
SbFc	3600	1600	2000	580	490
CdFc	3600	1620	2000	600	490
ZrFc	3600	1600	2000	580	500

^aSbFc, antimony hexacyanoferrate (II); CdFc, cadmium hexacyanoferrate (II); ZrFc, zirconium hexacyanoferrate (II), * Metal - N bond shows degree of polymerization

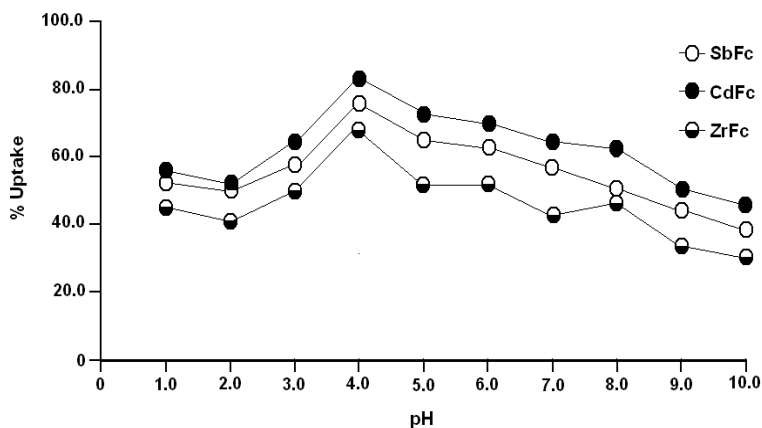


Fig. 1. Effect on pH on adsorption of 2, 4-DNP on metal hexacyanoferrates (II). (Temperature is $30 \pm 1^\circ\text{C}$; concentration is 10^{-4} M; amount of metal hexacyanoferrate (II) is 25 mg)

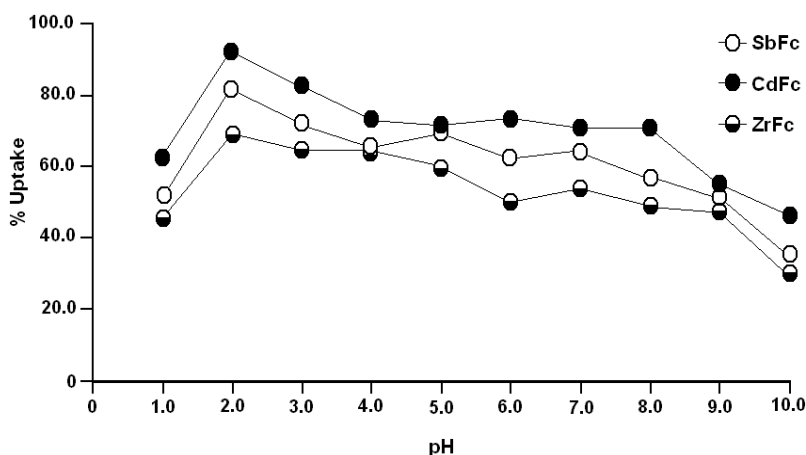


Fig. 2. Effect on pH on adsorption of 2, 4, 6-TNP on metal hexacyanoferrates (II). (Temperature is $30 \pm 1^\circ\text{C}$; concentration is 10^{-4} M; amount of metal hexacyanoferrate (II) is 25 mg)

Table 3. Percentage adsorption of 2, 4-dinitrophenol on metal hexacyanoferrates (II) (Room temperature: $30 \pm 1^\circ\text{C}$; λ_{max} 2, 4-DNP = 360 nm; 2, 4-DNP pKa = 3.96)

pH	Percentage (%) adsorption		
	SbFc	CdFc	ZrFc
1.0	49.8	52.7	44.6
2.0	46.7	47.3	40.9
3.0	58.7	62.4	50.3
4.0	76.4	80.5	68.2
5.0	52.6	71.2	55.4
6.0	62.0	66.4	55.4
7.0	57.6	61.0	42.8
8.0	50.3	60.2	47.5
9.0	42.6	48.6	36.2
10.0	38.9	44.7	32.6

2, 4-DNP, 2,4-dinitrophenol

Present trend of adsorption is justified by specific surface area (SSA) of metal hexacyanoferrates (II) (CdFc = 101.67; SbFc = 74.30; ZrFc = 47.30) (m^2g^{-1}) were calculated by basic methylene blue dye adsorption. The Langmuir plots for adsorption 2, 4-DNP and 2, 4, 6-TNP on antimony, cadmium and zirconium hexacyanoferrate (II) complexes are shown in Figs. 5 & 6, respectively. The Langmuir constants (b and Q_0) values are given in Table 6. Sorption data have been correlated with Langmuir adsorption model given by equation [24,25].

$$\frac{1}{Q_{eq}} = \frac{1}{Q_0} + \frac{1}{bQ_0 \cdot C_{eq}}$$

where C_{eq} is equilibrium concentration of phenol, b is the constant related to the equilibrium

constant or binding energy ($b \propto e^{-\Delta H/RT}$ the parameter b reflects the steepness of the approach to saturation, more precisely to the b values is the reciprocal of concentration at which half saturation of the adsorbent is attained). Q_{eq} is amount (mg) of adsorbate adsorbed per gram of adsorbent and Q_0 is the adsorption maxima, i.e. mg of phenol required per gram of metal hexacyanoferrate (II) for forming a complete monolayer on the surface.

Table 4. Percentage adsorption of 2, 4, 6-trinitrophenol on metal hexacyanoferrates (II) (Room temperature: $30 \pm 1^\circ\text{C}$; λ_{max} 2, 4, 6-TNP = 382 nm; 2, 4, 6-TNP pKa = 0.38)

pH	Percentage (%) adsorption		
	SbFc	CdFc	ZrFc
1.0	50.7	60.6	46.3
2.0	81.7	87.6	72.2
3.0	72.0	80.2	64.0
4.0	64.3	71.9	64.0
5.0	66.3	68.5	60.9
6.0	60.2	70.6	50.7
7.0	62.7	66.9	56.0
8.0	57.9	70.2	52.3
9.0	52.3	54.8	50.2
10.0	36.0	45.3	34.9

2,4,6-TPN, 2,4,6-trinitrophenol

The linear nature of Langmuir plots confirm the formation of monolayer of 2, 4, 6-TNP molecules on metal hexacyanoferrates (II). The values of Langmuir constants b and Q_0 for all the systems were obtained from the slope and intercept of the plots, respectively. The values of Q_0 have been

interpreted as a measure of the accessibility of the adsorption sites and the b may be regarded as a measure of the affinity of the metal hexacyanoferrate (II) for the adsorbed substituted phenols [26]. The values of Langmuir constants indicate favourable

conditions for adsorption. The Q_0 values are found to be maximum for CdFc – TNP system while it is a minimum for the ZrFc – DNP system. The b value is found to be a maximum for ZrFc – DNP system and lowest for CdFc-TNP system.

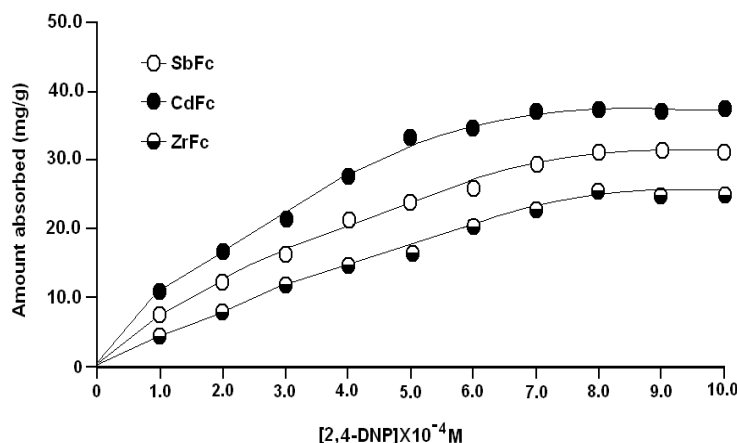


Fig. 3. Adsorption isotherms of 2, 4-DNP on metal hexacyanoferrates (II). (Temperature is $30 \pm 1^\circ\text{C}$; pH is 7.0 ± 0.01 , amount of metal hexacyanoferrate (II) is 25 mg)

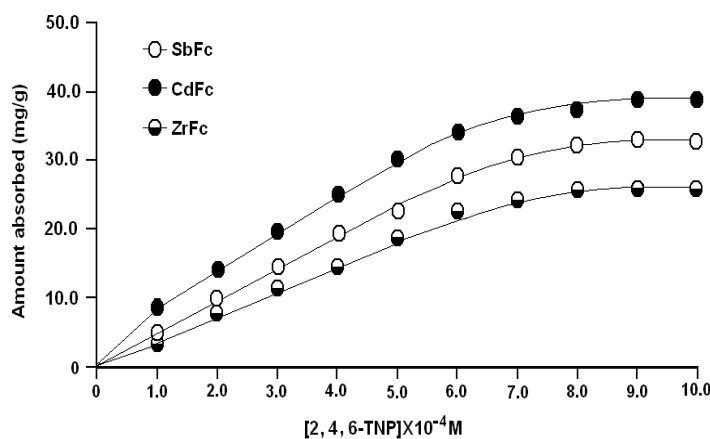


Fig. 4. Adsorption isotherms of 2, 4, 6-TNP on metal hexacyanoferrates (II). (Temperature is $30 \pm 1^\circ\text{C}$; pH is 7.0 ± 0.01 , amount of metal hexacyanoferrate (II) is 25 mg)

Table 5. Maximum uptake of 2, 4-dinitrophenol and 2, 4, 6-trinitrophenol on metal hexacyanoferrates (II) (Room temperature = $30 \pm 1^\circ\text{C}$; pH = 7.0 ± 0.01)

Substituted phenols	Metal hexacyanoferrates(II)	Particle size(μm)	Maximum uptake(mg/g)
2, 4 – DNP ($pka = 3.96$) $\lambda_{\text{max}} = 360 \text{ nm}$	SbFc	125	30.7
	CdFc	125	37.0
	ZrFc	125	25.0
2, 4, 6 – TNP ($pka = 0.38$) $\lambda_{\text{max}} = 382 \text{ nm}$	SbFc	125	33.3
	CdFc	125	39.0
	ZrFc	125	26.2

2, 4-DNP, 2, 4- dinitrophenol, 2,4,6-TNP, 2,4,6- trinitrophenol

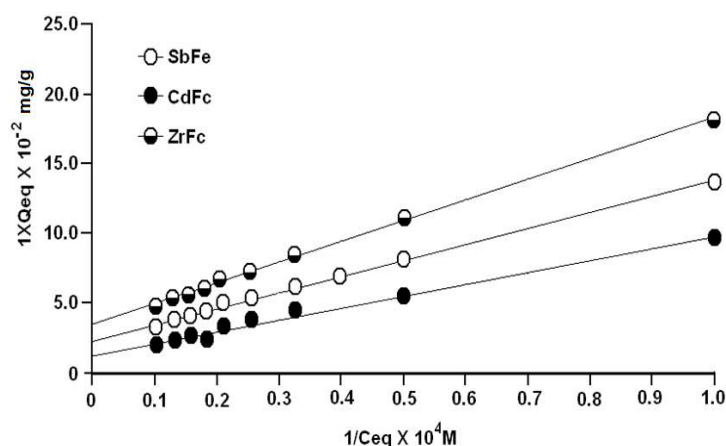


Fig. 5. Langmuir plots of 2, 4-DNP on metal hexacyanoferrates (II). (Temperature is $30 \pm 1^\circ\text{C}$; pH is 7.0 ± 0.01 , amount of metal hexacyanoferrate (II) is 25 mg)

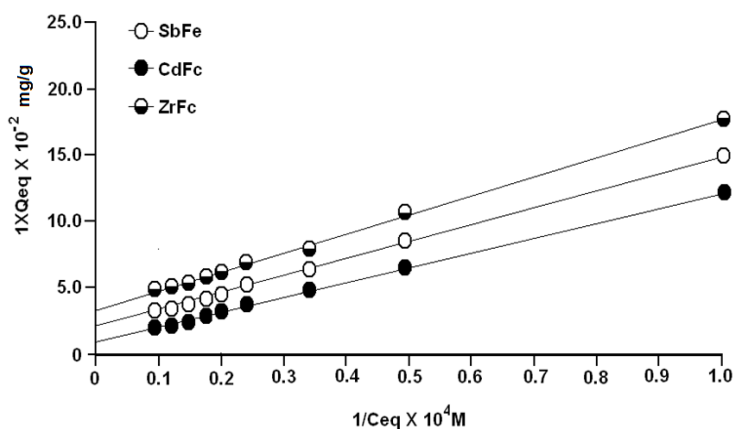


Fig. 6. Langmuir plots of 2, 4, 6-TNP on metal hexacyanoferrates (II). (Temperature is $30 \pm 1^\circ\text{C}$; pH is 7.0 ± 0.01 , amount of metal hexacyanoferrate (II) is 25 mg)

Table 6. Langmuir constants for adsorption of 2, 4-dinitrophenol and 2, 4, 6-trinitrophenol on metal hexacyanoferrates (II) (Room temperature = $30 \pm 1^\circ\text{C}$; pH = 7.0 ± 0.01)

Substituted phenols	Metal hexacyanoferrates (II)	Particle size (μm)	Langmuir constants	
			$b \times 10^2 (\text{l mol}^{-1})$	$Q_0 (\text{mg g}^{-1})$
2, 4 – DNP ($pK_a = 3.96$) $\lambda_{\text{max}} = 360 \text{ nm}$	SbFc	125	16.69	45.45
	CdFc	125	11.39	100.00
	ZrFc	125	20.02	32.26
2, 4, 6 – TNP ($pK_a = 0.38$) $\lambda_{\text{max}} = 382 \text{ nm}$	SbFc	125	12.46	58.82
	CdFc	125	4.80	166.67
	ZrFc	125	17.91	38.46

2, 4-DNP, 2, 4- dinitrophenol, 2,4,6-TNP, 2,4,6- trinitrophenol

Species $[\text{Fe}(\text{CN})_6]^{4-}$ in metal hexacyanoferrates (II) exist with an octahedral geometry, where six cyanide ligands surround the central iron atom [27]. Due to strong field of the CN⁻ ligands, all six electrons become paired to give the electronic

configuration t_{2g}^6 . Although the cyanide ligands bond with iron via σ -donation, there is sufficient back bonding from iron d_{π} orbitals to CN⁻ ligands anti bonding P_{π} orbitals. Transition metal hexacyanoferrates (II) usually have a polymeric

lattice structure with $[\text{Fe}(\text{CN})_6]^{4-}$ anions, where the outer transition-metal ions may be coordinated through the nitrogen end of cyanide ligand. As proposed earlier concerning on the adsorption of several biomolecules on different metal hexacyanoferrates (II), the adsorption of substituted phenols on hexacyanoferrates (II) is probably due to a phenolate anion interaction with replaceable metal ions present outside of the coordination sphere of the hexacyanoferrates (II).

4. CONCLUDING REMARKS

1. Antimony, cadmium and zirconium hexacyanoferrates (II) were synthesized, characterized and utilized for the removal of 2, 4- DNP and 2, 4, 6- TNP from the aqueous solution.
2. The most effective pH for the removal of 2, 4-DNP and 2, 4, 6-TNP were found to be 4.0 and 2.0, respectively.
3. Cadmium and zirconium hexacyanoferrates (II) were found to have maximum and minimum adsorption, respectively for both adsorbates.
4. Adsorbents cheaper than metal hexacyanoferrates (II) are available for the removal of phenols from the environment but present study indicates feasibility of using metal hexacyanoferrates (II) for the treatment of phenol bearing wastewater.
5. 2, 4, 6-TNP found to be more adsorbed in comparison to 2, 4-DNP on all three metal hexacyanoferrates (II) studied.
6. Results in the present study suggest the potential feasibility for the use of metal hexacyanoferrates (II) in the removal of 2, 4-DNP and 2, 4, 6-TNP from industrial waste, potable water, environment and agricultural soil.

COMPETING INTERESTS

Author has declared that no competing interests exist.

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