Determination and Removal of Phosphate and Nitrate from the Ground and Surface Water at the Three Counties of Guyana

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Authors’ contributions
Present work was carried out in collaboration between both authors. Author JAC done the experimental work of research project, write first draft of manuscript and part of revision work. Author BBT as supervisor of research project finalized the revise manuscript to the standard of publications. Both authors read and approved the final manuscript.

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ABSTRACT
Standard analytical procedures that are based on colourimetry facilitated the determination of PO₄³⁻ and NO₃⁻ in the ground and surface waters from the three (3) counties of Guyana. Phosphates and nitrates beyond their permissible levels in natural waters can pose significant threats to the aquatic environment, its inhabitants, and the health of humans and terrestrial animals. For this reason, this study aims to investigate the use of metal (nickel, cadmium, cobalt) ferrocyanides (octahedral complexes that facilitate adsorption of anions primarily on its two divalent transitional metals) as adsorbents to facilitate the removal of the aforementioned anions. Results revealed that nickel ferrocyanide is the most effective adsorbent for treating ground and surface waters that contain high levels of PO₄³⁻, while it was also the most effective adsorbent for treating surface waters that contained NO₃⁻. Fixed volumes of water samples were treated with 50 mg, 100 mg, and 150 mg of the adsorbent. The correlation coefficient computations revealed that they were instances of both positive and negative correlations, which implied that increasing adsorbent dosage resulted in increased adsorption and other instances decreased adsorption, respectively.

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This occurrence went against literature, possible reasons for this are explained in detail within the discussion portion of the manuscript. Adsorption isotherms and Langmuir plots were used to facilitate adsorption studies, in various instances, the generic appearance of these isotherms was not observed, possible reasons for this occurrence are also explained in more detail within the article. Based on observations within the article, the use of cobalt ferrocyanide for treating groundwater and removing $\text{PO}_4^{3-}$ is not recommended, while cadmium ferrocyanide is not recommended to be used to facilitate the removal of $\text{NO}_3^-$, due to the possible formation of $\text{Co}_3(\text{PO}_4)_2$ and $\text{Cd}(\text{NO}_3)_2$, respectively, which consequently affected the results. Technologies like those studied in this research project should be explored and studied more in-depth (particularly with regards to the stability of the adsorbents) with regards to the treating of ground and surface water supplies, to facilitate the removal of $\text{PO}_4^{3-}$ and $\text{NO}_3^-$, since the results observed, though on a small scale, can be seen to move us closer towards a green and sustainable environment, inclusive of safeguarding the health of the World’s people and expanding the scope of cost-effective and simple methods that aid in the removal of anions from natural waters.

Keywords: Phosphate; nitrate; determination; removal; adsorption; metal ferrocyanides.

1. INTRODUCTION

“Water is critical for sustainable development, including environmental integrity and the alleviation of poverty and hunger, and is indispensable for human health and well-being” [1].

Ground Water is referred to as “all the water which is below the surface of the ground in the saturation zone and is in direct contact with the ground or subsoil” [2], such water is contained within geological formations referred to as aquifers (porous and permeable underground layer of water-bearing rocks). Whereas Surface Water is referred to as that water found above that of groundwater which originates from precipitation inclusive of moisture from clouds, rain, and snow [3]. This form of water is usually found in a river, lake, or other surface cavities. In Guyana most (approximately 60%) of the domestic water supply (i.e. potable water) comes from groundwater resources, while most of the water supply for agriculture (sugarcane and rice) and industry comes from surface water [4]. As a consequence, knowledge of the status and integrity of natural waters both ground and surface water found within Guyana is of vital importance. This research focuses primarily on the levels of phosphates and nitrates in the water supplies since each of them if present or consumed in large quantities can affect the aquatic environment and its inhabitants and also negatively impact one’s health, respectively. Phosphate stimulates the growth of plankton and aquatic plants (food source for fish), where the continuous supply of food would increase the fish population, and subsequently improve water quality [5]. However, the natural levels (NL) of phosphate within aquatic systems usually range from 0.005 to 0.05 mg/L. If these levels are not maintained there is a high possibility of long-term eutrophication occurring, as discussed in more detail in the discussion portion of the paper. Nonetheless, this can be prevented if the total phosphorus levels are kept below 0.5 ppm and 0.05 ppm, respectively [6].

Nitrate similar to phosphate is an important plant nutrient. It is, however, one of the most frequent groundwater pollutants, where it enters groundwater through fertilizer runoff, urban sewage effluents, and animal waste. Levels of nitrate above the permissible limit in drinking water sources can lead to Methaemoglobininaemia or what is commonly referred to as “blue baby syndrome” [7]. This disease entails the conversion of nitrate to nitrite in the digestive system, where nitrite oxidizes the iron in the hemoglobin of the red blood cells to form methemoglobin, which lacks the oxygen-carrying ability of hemoglobin. This occurrence provides adequate conditions for a baby under 6 months to develop the aforementioned disease, where their skin and veins appear blue [7].

The concept of adsorption has been one of the many methods commonly used to aid in the removal of heavy metal ions from water and wastewater, to improve the integrity of water bodies [8]. This phenomenon is defined as the adhesion of a chemical species onto the surface of particles [9]. Metal ferrocyanides (Ni, Co, Cd) were the adsorbent materials utilized during the study – in varying quantities, with aim of determining which adsorbent material was most effective while considering the anion, water form, and quantity of adsorbent. Knowledge of metal
ferrocyanides is paramount, particularly since they have been widely applied as adsorbents and ion exchangers [10]. Research has shown that it has been used in the adsorption of amino acids, dyes (methylene blue), and heavy metal ions (Ti$^{4+}$, Hg$^{2+}$, Mg$^{2+}$, Fe$^{3+}$, etc.) [11]. However, after research, it is evident that literature about the interaction of metal ferrocyanides with anions, in particular, is limited. The metal ferrocyanides being studied are octahedral complexes (i.e. six ligands), where Fe$^{3+}$ is the central metal ion, surrounded by six nitrile functional groups, inclusive of two atoms of the respective divalent transitional metal. It is the surface of the divalent metal, upon which much of the adsorption of the anion is said to occur. The transitional property of the metals is what allows the adsorbent to possess the property of it being insoluble. Premised on the fact that much literature is not available concerning the use of metal ferrocyanides to aid in the removal of phosphates and nitrates from the water and wastewater, attempts have been made to ascertain how effective cobalt, cadmium, and nickel ferrocyanides are for the removal of the aforementioned anions. The effectiveness and feasibility of each adsorbent material were determined by varying the quantity of the adsorbent materials.

The potential use of biochar to remove phosphorus from wastewater was studied by Nobaharan et al. [12]. Rajaniemi et al. [13] have described the removal of phosphate and ammonia from water through electrochemical and chemical precipitation of struvite. The simultaneous nitrate and phosphate adsorption capacity of solid waste residue generated from Awash Melkassa aluminum sulfate and sulfuric acid are reported by Berkessa et al. [14]. The bioremediation of nitrogen and phosphorus of beverage effluent was investigated by Bhammad et al. [15] using the potential strains of Bacillus sp. (BK1) and Aspergillus sp. (BK2). Fetene and Addis [16] have demonstrated the removal of phosphate from wastewater using Ethiopian rift pumice. A comprehensive study on phosphate removal from water using phragmites australis biochar loaded with aluminum, calcium, iron, lanthanum, and magnesium oxides was studied by Wang et al. [17]. Bunce et al. [18] have presented a review of phosphorus removal technologies and their applicability to small-scale domestic wastewater treatment systems. Phosphate removal from contaminated water using activated carbon-supported nanoscale zero-valent iron particles was studied by Bezzi and Chirwa [19]. The present study showed that silica-coated zero-valent iron particles could be promising adsorbents for the removal of phosphate from polluted waters. Biftu et al. [20] have studied the adsorption of phosphate, nitrate, and chromate through adsorption on activated carbon derived from the stem of the Carissa Carandas plant. Phosphate removal from aqueous solution was studied by Mekonnen et al. [21] using low-cost volcanic rocks such as pumice and scoria obtained from the Ethiopian Great Rift Valley. Phosphate removal and recovery from wastewater using layered zinc hydroxide as an adsorbent were investigated by Almasri et al. [22]. Zhang et al. [23] has described the adsorption of phosphorus from wastewater on zeolite synthesized from fly ash.

2. METHODOLOGY

2.1 Chemicals

2.1.1 Determining the presence of PO$_4^{3-}$

Ammonium persulfate, Sodium Hydroxide (1 N), Phenolphthalein indicator solution, Potassium dihydrogen phosphate (pre-dried), Ammonium molybdate solution: 20 g of ammonium molybdate was dissolved in 500 ml distilled water, and stored in a glass stoppered volumetric flask. Antimony potassium tartrate: 1.4 g of antimony potassium tartrate was dissolved in 400 mL water in a 500 mL volumetric flask and diluted to volume, and stored in a paper-covered glass volumetric flask. Ascorbic acid solution (0.1M): 1.76 g of ascorbic acid was dissolved in 100 mL distilled water. The solution was stored in a fridge (~4°C) when not in use. Sulfuric acid (5 N): 70 mL of concentrated H$_2$SO$_4$ was diluted to 500 ml with distilled water.

2.1.2 Determining the presence of NO$_3^-$

Sodium salicylate (0.5 % w/v): 0.5 g of sodium salicylate was dissolved in 100 mL of distilled water. Sodium hydroxide (25% w/v): 50g of sodium hydroxide was dissolved in 140 mL of distilled water, where it was allowed to cool in an ice bath. After which, the solution was made up to 200 mL. Concentrated sulphuric acid (instrumental grade), Stock nitrate solution (186 mg/L NO$_3^-$): 0.255 g NaN$O_3$ (pre-dried for 2 hrs.) dissolved in 1L of distilled H$_2$O. Working nitrate solution (50 mg/L NO$_3^-$): 135 mL of the stock solution made up to 500 mL.
2.1.3 Preparation of adsorbent materials

Potassium ferrocyanide, Nickel chloride, Cobalt chloride, Cadmium chloride.

2.1.4 Water samples

Groundwater samples were collected from water wells/reservoirs, while the surface water samples were collected from flowing lakes and canals.

2.2 Synthesis and Preparation of Adsorbent Materials

The synthesis of nickel, cobalt, and cadmium ferrocyanides followed a method similar to that reported by Kourim et al. [24]. 500 ml of the nickel or cadmium chloride (0.1M) was added to 167 ml potassium ferrocyanide solution (0.1M) with constant stirring. The reaction mixture was heated on a water bath for 3 hours and kept at room temperature. The resulting precipitate was vacuum filtered, washed several times with distilled water, and dried in an air oven at 60°C. The dried product was ground and sieved to 125 µm particle size, and stored in tightly sealed containers. With regards to cobalt ferrocyanide, the use of one volume of 0.5M potassium ferrocyanide and 2.4 volume of 0.3M cobalt chloride [25] the resulting solution was gently stirred for 30 minutes at room temperature. The resulting slurry of cobalt ferrocyanide was vacuum filtered, washed several times with distilled water, and dried. The granules produced were dried and powdered to 125 µm particle size, and stored in tightly sealed containers. The neutral and basic aluminum oxides obtained from the laboratory stores were ground and sieved to 125 µm particle size.

2.3 Characterization of Adsorbent Materials

After the synthesis of CdFc, CoFc, and NiFc, the colours observed were dark brown, off-white (with a tinge of green), and olive green, respectively. Elemental analysis was performed via an Agilent Technologies 200 Series Atomic Absorption Spectrophotometer and GTA 120 Graphite Tube Automizer, which was used to analyze for the respective percentage metal concentrations, the results of this analysis are highlighted in Table 1. Whereas, spectral studies were conducted by recording the infrared spectra (4000 cm⁻¹ – 1000 cm⁻¹) of the metal ferrocyanides in KBr (1:10) on Buck Scientific Infrared Spectrophotometer M630. The results of this analysis are highlighted in Table 2.

2.4 Collection of Water Samples

Water samples were collected in sterilized plastic containers - plastic containers were used since they do not have active sites that are capable of binding with the inorganic ions [26]. The samples were preserved by storing them in an ice chest/ridge at a temperature of ~4°C. The containers were also labeled after collection. The maps for surface and ground water sampling locations from the three counties in Guyana, viz. Berbice, Demerara, and Essequibo are shown in Figs. 1 – 6, respectively.

2.5 Determination of Phosphate in Water Samples

The method used was primarily based on that which was reported by Murphy and Riley [27] in their article titled "A modified single solution method for the determination of phosphate in natural waters". An aliquot (50 mL) of water sample was pipetted into a conical flask, to which one (1) drop of phenolphthalein indicator solution was added. In the instances, where a red colour developed, the hydrolyzing solution was added dropwise to discharge the colour. 1 mL of hydrolyzing solution and 0.4 g of ammonium persulfate was added to the reaction mixture. The reaction mixture was allowed to boil on a hot plate until the solution volume met 10 mL. The solution was left to cool, then diluted to 30 mL with distilled water, after which a drop of phenolphthalein indicator was added, where the reaction mixture was neutralized to a faint pink colour with 1N NaOH, 8 mL of combined reagent was to the reaction medium and thoroughly mixed. The reaction medium was made up to 100 mL, after which it was allowed to stand for at least 10 minutes but no more than 30 minutes, to facilitate maximum colour development.

The absorbance of the samples was measured at 880 nm, where a reagent blank was used as the reference solution. With the use of the plotted standard curve, the concentration of phosphate in the water samples was determined. Potassium dihydrogen phosphate was used as the source of phosphate for the standards, with concentrations of 0.01, 0.025, 0.05, 0.10, 0.25, 0.50, 1.00, 1.50, and 2.00 mg/L PO₄³⁻, 8 mL of the combined reagent was also added.
Fig. 1 Berbice surface water sampling location
Location and coordinates: Adventure Channel/Atlantic Ocean - 6°12'45.7"N 57°16'51.3"W
Fig. 2. Berbice ground water sampling location
Location and coordinates: Adelphi Pump Station - 6°13'43.2"N 57°28'57.0"W
Fig. 3. Demerara surface water sampling location
Location and coordinates: University of Guyana Caiman Pond - 6°48'43.1"N 58°07'03.6"W
Fig. 4. Demerara ground water sampling location
Location and coordinates: Guyana Water Incorporation Better Hope Water Treatment Plant - 6°49'21.1"N 58°05'19.6"W
Fig. 5. Essequibo surface water sampling location
Location and coordinates: Tuschen Canal - 6°52'26.9"N 58°21'31.2"W
Fig. 6. Essequibo ground water sampling location

Location and coordinates: Guyana Water Incorporation Vergenoegen Water Treatment Plant - 6°52'37.4"N 58°21'21.7"W
2.5 Determination of Nitrate in Water Samples

The method used was primarily based on that which was reported by Yang, et al. [28], in their article titled “A Simple Spectrophotometric Determination of Nitrate in Water, Resin, and Soil Extracts”. An aliquot (50 mL) of water sample was pipetted into a conical flask. To which 2.0 mL of sodium salicylate solution was added. This solution was mixed well and evaporated to dryness on a hot plate. After evaporation to dryness and cooling, with the use of a pipette 1.0 mL of concentrated sulphuric acid was added, mixed, and allowed to stand for ~10 minutes. The solution was diluted to 50 m L. After which, with the use of a pipette 10 mL of sodium hydroxide solution was added and mixed thoroughly. When the reaction medium was cooled, it was made up to 100 m L. The absorbance of the samples was measured at 410 nm, where a reagent blank was used as the reference solution. With the use of the plotted standard curve, the concentration of nitrate in the water samples was determined.

2.6 Removal of PO$_4^{3-}$ and NO$_3^-$ Using Adsorbent Materials

50 mg of adsorbent was added to 100 mL of the water samples retrieved from the various water bodies. The reaction mediums were shaken manually, after which they were left in a fridge for ~2 days to facilitate settling and the supernatant was re-analyzed for the specific anion via the aforementioned analytical procedure. All data was recorded, inclusive of adsorption isotherms, standard deviation, and correlation coefficient values to highlight the efficiency of the adsorption process. The aforementioned steps were repeated with 100 mg and 50 mg for each adsorbent material, with the respective anion.

2.7 Statistical Treatment

The ANOVA statistical test was used to conclude whether there is a significant difference in the final concentration of the anion (PO$_4^{3-}$/ NO$_3^-$) in the H$_2$O samples when increasing adsorbent dosages are used to treat the individual water samples.

The Correlation Coefficient was used to determine whether what is expected based on literature, actually occurred experimentally, with regards to an increase in the adsorbent dosage, leads to increasing percentage adsorption, since increased availability of active sites with increasing amounts of adsorbent was expected, and hence increased adsorption.

3. RESULTS

Concerning the statistical analysis conducted under the ANOVA test, it was observed that the p-value is extremely lower than 0.05 (varying from $1.0 \times 10^{-22} - 1.0 \times 10^{-05}$), thus indicating that there is strong evidence against the null hypothesis ($H_0$), and it is therefore rejected, while the alternative hypothesis ($H_A$) has been accepted. This meant that increasing adsorbent dosages to treat the water samples, may have resulted in decreased concentration of the respective anion in some instances, but the extent of its effectiveness was not significant, this is shown in Tables 4 and 5. Similarly, the same conclusions were made with regards to the $F$ value and $F$ critical values, wherein in all instances also, the $F$ value was extremely greater than the $F$ critical value.

3.1 Effect of Mass Concentration of Metal Ferrocyanides on the Adsorption of Phosphate and Nitrate

It was observed that percentage adsorption concerning increasing dosages of the respective adsorbents yielded a strong positive correlation since the values are very close to +1. However, they were instances where strong negative correlations were observed (i.e. values are very close to -1), particularly when analyzing for nitrates after treatment with adsorbent materials. Despite this observation, when NiFc adsorbents were used its negative correlations were lower or there was no negative correlation but rather a weak positive correlation, thus indicating that its activity agreed with the literature. This comes as no surprise since referring to Figs. 7 and 8, NiFc was the most effective adsorbent.

The Correlation Coefficient of Determination ($R^2$) values on the adsorption isotherms were above 0.6 in practically all instances, thus indicating a significant relationship existing between the adsorption of the anion and the respective adsorbent. Similarly, this was observed for the Langmuir plots (in not all, but most instances), thus indicating that the plotted data fitted the Langmuir Isotherm Model.
Table 1. Elemental analysis of nickel, cadmium, cobalt ferrocyanides

| Metal Ferrocyanides | Metal Concentration (mg/L) |
|---------------------|----------------------------|
| NiFc                | 0.50 ± 0.049               |
| CdFc                | 12.16 ± 0.084              |
| CoFc                | 60.80 ± 4.897              |

*NiFc, nickel ferrocyanide; CdFc, cadmium ferrocyanide; CoFc, cobalt ferrocyanide

Table 2. Infrared spectral data of nickel, cadmium, cobalt ferrocyanides

| Adsorbent | Adsorption Frequency (cm⁻¹) |
|-----------|-----------------------------|
|           | O-H/ H₂O Molecule Bond | H-O-H Bending | C≡N Bond |
| NiFC      | 3728.8 ± 0.00             | 1600.00 ± 0.00 | 2078.65 ± 7.15 |
| CoFC      | 3728.8 ± 0.00             | 1600.00 ± 0.00 | 2078.65 ± 7.15 |
| CdFC      | 3721.65 ± 7.15            | 1600.00 ± 0.00 | 2057.2 ± 0.00  |

*NiFc, nickel ferrocyanide; CdFc, cadmium ferrocyanide; CoFc, cobalt ferrocyanide

Table 3. Concentration values for the untreated water samples

| SAMPLE | Phosphate | Nitrate |
|--------|-----------|---------|
|        | Concentration (mg/L) | Natural Level (NL) exceeded | NO₃⁻ Concentration (mg/L) | Maximum Contaminant Level (MCL) exceeded |
| BS     | 1.30 ± 0.00506 | + | 0.62 ± 0.0009 | - |
| BG     | 0.23 ± 0.0009 | + | 0.45 ± 0.0021 | - |
| DS     | 0.22 ± 0.00074 | + | 0.08 ± 0.0005 | - |
| DG     | 0.62 ± 0.00036 | + | 0.17 ± 0.009 | - |
| ES     | 0.69 ± 0.00071 | + | 0.34 ± 0.004 | - |
| EG     | 0.35 ± 0.00021 | + | 0.58 ± 0.0026 | - |

*(+) – natural level exceeded; (-) – maximum contaminant level not exceeded; B - Berbice; D- Demerara; E- Essequibo; S- Surface water; G- Groundwater

Fig. 7. The overall effectiveness of adsorbents when used to treat surface water samples
Table 4. Adsorption values after treatment with metal ferrocyanides at varying mass concentrations for the removal of nitrates

| Flask ID | Mass of adsorbent (g) | Volume of sample (mL) | NiFc Conc. after NiFc (mg PO₄³⁻/L)- Qeq | Amount of solute adsorbed (mg/L) | CdFc Conc. after CdFc (mg PO₄³⁻/L)- Qeq | Amount of solute adsorbed (mg/L) | CoFc Conc. after CoF (mg PO₄³⁻/L)- Qeq | Amount of solute adsorbed (mg/L) |
|----------|-----------------------|-----------------------|----------------------------------------|-----------------------------------|----------------------------------------|------------------------------------|----------------------------------------|-----------------------------------|
| BS       | 0.15                  | 50                    | 0.54                                   | 0.004                             | 0.03                                   | 4.71                               | -0.205                                | -1.36                             |
|          | 0.10                  | 50                    | 1.01                                   | -0.20                             | -0.20                                  | 0.47                               | 0.008                                 | 0.08                              |
|          | 0.05                  | 50                    | 0.55                                   | 0.004                             | 0.07                                   | 2.35                               | -0.087                                | -1.73                             |
|          | 0.00                  | 50                    | 0.62                                   | 0.000                             | 0.00                                   | 0.62                               | 0.000                                 | -                   |
| BG       | 0.15                  | 50                    | -                                      | -                                 | -                                      | -                                  | -                                      | -                   |
|          | 0.10                  | 50                    | 0.12                                   | 0.017                             | 0.17                                   | 4.41                               | -0.198                                | -1.98                             |
|          | 0.05                  | 50                    | 0.13                                   | 0.016                             | 0.32                                   | 1.06                               | -0.031                                | -0.61                             |
|          | 0.00                  | 50                    | 0.45                                   | 0.000                             | 0.00                                   | 0.45                               | 0.000                                 | -                   |
| DS       | 0.15                  | 50                    | 0.29                                   | -0.011                            | -0.07                                  | 5.9                                | -0.291                                | -1.94                             |
|          | 0.10                  | 50                    | 0.21                                   | -0.007                            | -0.07                                  | 4.23                               | -0.198                                | -2.08                             |
|          | 0.05                  | 50                    | -0.04                                  | 0.000                             | 0.12                                   | 1.67                               | -0.080                                | -1.59                             |
|          | 0.00                  | 50                    | 0.08                                   | 0.000                             | 0.00                                   | 0.08                               | 0.000                                 | -                   |
| DG       | 0.15                  | 50                    | 0.09                                   | 0.004                             | 0.03                                   | 6.82                               | -0.333                                | -2.22                             |
|          | 0.10                  | 50                    | 0.12                                   | 0.003                             | 0.03                                   | 2.93                               | -0.133                                | -1.38                             |
|          | 0.05                  | 50                    | 0.25                                   | -0.004                            | -0.08                                  | 2.06                               | -0.095                                | -1.89                             |
|          | 0.00                  | 50                    | 0.17                                   | 0.000                             | 0.00                                   | 0.17                               | 0.000                                 | -                   |
| ES       | 0.15                  | 50                    | -0.05                                  | 0.020                             | 0.13                                   | 3.71                               | -0.169                                | -1.12                             |
|          | 0.10                  | 50                    | -0.09                                  | 0.022                             | 0.22                                   | 4.32                               | -0.199                                | -1.99                             |
|          | 0.05                  | 50                    | 0.06                                   | 0.014                             | 0.28                                   | 1.58                               | -0.062                                | -1.24                             |
|          | 0.00                  | 50                    | 0.34                                   | 0.000                             | 0.00                                   | 0.34                               | 0.000                                 | -                   |
| EG       | 0.15                  | 50                    | 0.03                                   | 0.028                             | 0.18                                   | 4.58                               | -0.200                                | -1.33                             |
|          | 0.10                  | 50                    | 0.036                                  | 0.027                             | 0.27                                   | 3.53                               | -0.148                                | -1.48                             |
|          | 0.05                  | 50                    | 0.1                                    | 0.024                             | 0.48                                   | 1.48                               | -0.045                                | -0.90                             |
|          | 0.00                  | 50                    | 0.58                                   | 0.000                             | 0.00                                   | 0.58                               | 0.000                                 | -                   |

(Temperature= 25±1°C, NO₃ =max= 410nm, particle size= 125µm)
Table 5. Adsorption values after treatment with metal ferrocyanides at varying mass concentrations for the removal of phosphates

| Flask ID | Mass of adsorbent (g) | Volume of sample (mL) | Conc. after NiFc (mg PO₄³⁻/L)-Ceq | Amount of solute adsorbed (mg/L) | Conc. after CdFc (mg PO₄³⁻/L)-Ceq | Amount of solute adsorbed (mg/L) | Conc. after CoFc (mg PO₄³⁻/L)-Ceq | Amount of solute adsorbed (mg/L) | x/m (mg/g) | Qeq |
|----------|-----------------------|-----------------------|-----------------------------------|---------------------------------|-----------------------------------|---------------------------------|---------------------------------|---------------------------------|-----------|------|
| BS       | 0.15                  | 50                    | 0.06                              | 0.062                           | 0.41                              | 0.48                            | 0.041                           | 0.27                            | 1.00      | 0.015|
|          | 0.10                  | 50                    | 0.13                              | 0.059                           | 0.59                              | 0.64                            | 0.033                           | 0.33                            | 0.66      | 0.032|
|          | 0.05                  | 50                    | 0.19                              | 0.056                           | 1.11                              | 0.76                            | 0.027                           | 0.54                            | 1.49      | -0.01|
|          | 0.00                  | 50                    | 1.30                              | 0.000                           | 0.00                              | 1.30                            | 0.000                           | -0.01                           | 1.30      | 0.000|
| BG       | 0.15                  | 50                    | 0.06                              | 0.009                           | 0.06                              | 0.00                            | 0.012                           | 0.08                            | 0.45      | 0.043|
|          | 0.10                  | 50                    | 0.04                              | 0.010                           | 0.10                              | -0.01                           | 0.012                           | 0.12                            | 1.25      | 0.003|
|          | 0.05                  | 50                    | 0.20                              | 0.002                           | 0.03                              | 0.15                            | 0.004                           | 0.08                            | 0.74      | 0.028|
|          | 0.00                  | 50                    | 0.23                              | 0.000                           | 0.00                              | 0.23                            | 0.000                           | -0.01                           | 0.23      | 0.054|
| DS       | 0.15                  | 50                    | -0.01                             | 0.012                           | 0.08                              | 0.05                            | 0.009                           | 0.06                            | 0.05      | 0.009|
|          | 0.10                  | 50                    | 0.02                              | 0.010                           | 0.10                              | 0.06                            | 0.008                           | 0.08                            | -0.01     | 0.012|
|          | 0.05                  | 50                    | 0.01                              | 0.011                           | 0.21                              | 0.10                            | 0.006                           | 0.12                            | 0.18      | 0.002|
|          | 0.00                  | 50                    | 0.22                              | 0.000                           | 0.00                              | 0.22                            | 0.000                           | -0.01                           | 0.22      | 0.000|
| DG       | 0.15                  | 50                    | 0.29                              | 0.017                           | 0.11                              | 0.57                            | 0.003                           | 0.02                            | 0.16      | 0.023|
|          | 0.10                  | 50                    | 0.41                              | 0.011                           | 0.11                              | 0.59                            | 0.002                           | 0.02                            | 0.09      | 0.027|
|          | 0.05                  | 50                    | 0.63                              | -0.001                          | -0.01                             | 0.59                            | 0.002                           | 0.03                            | 1.25      | -0.032|
|          | 0.00                  | 50                    | 0.62                              | 0.000                           | 0.00                              | 0.62                            | 0.000                           | -0.01                           | 0.62      | 0.000|
| ES       | 0.15                  | 50                    | 0.00                              | 0.035                           | 0.23                              | 0.05                            | 0.032                           | 0.21                            | -0.01     | 0.035|
|          | 0.10                  | 50                    | 0.03                              | 0.033                           | 0.33                              | 0.07                            | 0.031                           | 0.31                            | -0.01     | 0.035|
|          | 0.05                  | 50                    | 0.03                              | 0.033                           | 0.66                              | 0.06                            | 0.032                           | 0.63                            | 0.06      | 0.032|
|          | 0.00                  | 50                    | 0.69                              | 0.000                           | 0.00                              | 0.69                            | 0.000                           | -0.01                           | 0.69      | 0.000|
| EG       | 0.15                  | 50                    | 0.08                              | 0.014                           | 0.09                              | 0.17                            | 0.009                           | 0.06                            | 0.09      | 0.013|
|          | 0.10                  | 50                    | 0.06                              | 0.015                           | 0.15                              | 0.16                            | 0.010                           | 0.10                            | 0.13      | 0.011|
|          | 0.05                  | 50                    | 0.03                              | 0.016                           | 0.32                              | 0.17                            | 0.009                           | 0.18                            | 0.08      | 0.014|
|          | 0.00                  | 50                    | 0.35                              | 0.000                           | 0.00                              | 0.35                            | 0.000                           | -0.01                           | 0.35      | 0.000|

(Temperature = 25±1°C, PO₄⁻₃ max= 880nm, particle size = 125µm)
Fig. 8. The overall effectiveness of adsorbents when used to treat groundwater samples

4. DISCUSSION

4.1 Effect of Metal Ferrocyanides on Water Samples

When nickel ferrocyanide was added to samples, within 3 minutes clearing up of the samples was observed, this was particularly observed in the case of the surface waters. After the settling period, white particles were observed on the surface of the adsorbent. Upon addition of cadmium ferrocyanide to samples, qualitatively it is observed to be more effective than that of nickel ferrocyanide. However, during re-analysis of the sample for NO$_3^-$ after treatment with cadmium ferrocyanide, in all instances, based on quantitative analysis, the sample seems to have more NO$_3^-$ than there initially was in the sample. The cobalt ferrocyanide worked similarly to that of cadmium ferrocyanide upon addition to samples. However, this only applies to surface waters, in the case of groundwater, the innate characteristic of these adsorbents being insoluble did not apply to cadmium ferrocyanide. Rather, the water samples were recolored to brown (i.e. the colour of the adsorbent). Also, when the samples were re-analyzed for the respective anion, the solution recoloured to blue and produced precipitate in all instances, samples, therefore, had to be filtered before analysis. Further, based on the results obtained, the adsorbents appear to be consistently more effective when used to remove PO$_4^{3-}$ as compared NO$_3^-$.

4.2 Phosphate Concentration after Treatment with Metal Ferrocyanide

Waters treated with NiFc and CdFc produced concentrations quite similar to their initial. However, CoFc treated waters, upon application of heat, the solutions recoloured to blue and produced excessive amounts of a precipitate. I have not been able to confirm this claim; however, I am suggesting that this occurrence may have occurred due to Co$^{2+}$ having a greater affinity for the PO$_4^{3-}$ ion than that of the ferrocyanide complex, resulting in the formation of an inorganic compound known as cobalt phosphate, Co$_3$(PO$_4$)$_2$, which is purple-blue. Nonetheless, its influence on the absorbance of the samples was minimized, via filtering of the sample, which revealed some drastic changes,
as the sample was coloured due to the precipitate which evolved during heating, while CoFc seemed to be a good adsorbent based on qualitative analysis, however quantitatively it was not, due to low percentage adsorption values.

With regards to the analysis for PO$_4^{3-}$ in the samples before treatment, the resulting concentration of PO$_4^{3-}$ for all water samples collected exceeded the natural level of (i.e. 0.05 mg/L – 0.005 mg/L), as shown in Table 3. The observed exceeded natural concentration of phosphate in the waterways may have occurred, due to contamination by detergent, agricultural and urban runoff, or industrial and municipal wastewater treatment plant effluent, algal bloom [5]. This, therefore, indicates the possibility of long-term eutrophication (excessive growth of aquatic plants) occurring in the water bodies [6]. Which can subsequently lead to low dissolved oxygen levels within these water bodies, and thus the death of the aquatic organisms [5]. With regards to the groundwaters, it was unexpected to find such high levels of PO$_4^{3-}$, this may be as a result of contamination of our groundwaters and also improper maintenance of our reservoirs (suggested due to observation). The levels of PO$_4^{3-}$ are not to the extreme high, thus it poses no potential health threat to animals or humans, but the levels should be controlled, cleaning and proper maintenance of our reservoirs and proper disposal of our waste materials should be made a practice four our own health and safety, else we may be faced with health complications - digestive problems [29]. This should be taken seriously since simple water treatment systems do not facilitate the removal of PO$_4^{3-}$.

Despite this, after the treatment of the PO$_4^{3-}$ contaminated water samples, it was observed that NiFc was not only deemed the most effective adsorbent, but it also removed enough PO$_4^{3-}$ so that the water samples (in most instances) are within the prescribed NL of PO$_4^{3-}$ in water. Long-term eutrophication, therefore, avoided as highlighted on Table 3, and depicted on Figures 7 and 8. Optimum removal of PO$_4^{3-}$ from surface waters was achieved when 150 mg of NiFc was used, while for ground waters NiFc was also most effective, however when 100 mg of it was used instead.

4.3 Nitrate Concentration after Treatment with Metal Ferrocyanides

Waters treated with NiFc and CoFc, was quite similar to when the initial water samples were analyzed, besides the fact that all of them had to be filtered before analysis. However, CdFc treated waters, during pre-treatment the intensity of the resulting yellow, qualitatively it was such that more NO$_3^-$ was being added to the test sample. Unlike CoFc and the analysis for PO$_4^{3-}$, no precipitate caused the intensified colouration, and in the instances where there was precipitate, despite filtration the sample kept the same yellow. I have not been able to confirm this claim, however, I am suggesting that this occurrence may have occurred due to Cd$^{2+}$ having a greater affinity for the NO$_3^-$ ion than that of the ferrocyanide complex, resulting in the formation of an inorganic compound known as cadmium nitrate Cd(NO$_3$)$_2$, which is yellow. As a result of this occurrence, extremely high absorbance values were produced (higher than the initial solution), thus subsequently obtaining negative percentage adsorption values. Notwithstanding, optimum results with regards to the removal of NO$_3^-$ from surface and ground waters was achieved when 50 mg of NiFc was used.

![Image 2. Nitrate](image)

With regards to the analysis for NO$_3^-$ in the samples before treatment, the resulting concentration of NO$_3^-$ for all water samples collected did not exceed the MCL (i.e. 10 mg/L), as shown in Table 3. This is great, particularly considering the ground waters because if they had contained levels of NO$_3^-$ beyond the MCL, the water treatment measures set in place could not have removed it. The ground waters from the collection areas are therefore deemed safe for consumption with respect to nitrates (i.e. after water treatment/purification), and the health and well-being of infants and pregnant women within these areas, need not be of concern with regards to high levels of nitrate within their domestic water supply – low probability of Methaemoglobinaemia (blue baby syndrome) and its associated health effects.
4.4 Atomic Absorption Spectroscopy (AAS) Analysis

Referring to Table 2, the concentration of the respective metal in 1g of NiFc and CdFc yielded somewhat reproducible results (i.e. referring to the standard deviation), however in the case of CoFc only two of the three concentration values were used since they were the closest, this however still resulted in a high standard deviation. It is believed, that something is wrong with this adsorbent, particularly considering its behavior during the analysis for PO$_4^{3-}$.

4.5 Infrared Spectral Analysis

With regards to the metal ferrocyanides, a peak characteristic for the nitrile group was observed, and O-H/ H$_2$O molecule peak and H-O-H bending. While only the two latter were observed in the case of the aluminum oxides – these observations correlated with the results highlighted [30,31]. Each of the IR Spectrums had peaks at the same absorbance frequency: 771.20 ± 0.00 cm$^{-1}$, 871.30 ± 0.00 cm$^{-1}$, 1986.20 ± 0.00 cm$^{-1}$, 2343.20 ± 0.00 cm$^{-1}$ and 3814.60 ± 0.00 cm$^{-1}$.

4.6 Adsorption Isotherms

The purpose of the adsorption isotherms is to show the mg of adsorbate present on a gram of the adsorbent as a function of its concentration. Concerning this project, the equilibrium concentration (C$_e$) which is present on the X axis– is the concentration of the contaminant after treating the water samples with varying masses of adsorbents; from the left of the adsorption isotherms.

Studying the adsorption isotherms, and considering the literature, many of the adsorption isotherms do not resemble that of a usual adsorption isotherm. This is so because, in some instances, the expectation of increasing adsorbent dosage leads to increased adsorption (decreased absorbance values) did not occur, but rather increased adsorbent dosage lead to decreased adsorption. Regarding the Q$_e$ (adsorption capacity) and C$_e$ (equilibrium concentration) values on the Y and X-axis respectively, according to literature, lower adsorbent dosages should yield maximum adsorption capacity values, as a result of not many active sites being present (adsorption capacity is met faster), thus resulting in a leveling off of the curve as concentration increases, while larger adsorbent dosages will yield lower adsorption capacities, as a result of their being more active sites (adsorption capacity is met slower) - this is observed on a few of the adsorbent isotherms. In other instances, higher adsorbent dosages yielded higher adsorption capacities as compared to when smaller adsorbent dosages were used, thus resulting in somewhat of an upturned adsorption isotherm.

The non-characteristic appearance of the adsorption isotherms was expected, since the absorbance values did not agree with the literature, with regards to increasing adsorbent dosage. I believe that the fact that the test samples were natural, and their full composition was unknown, that there may have been some contributing factors with regards to their composition (interferences), experimenters errors, and to some extent the method used for determination, particularly with regards to NO$_3^-$ - that would have contributed to the deviation from literature.

4.7 Langmuir Plots and Constants

A note was made of the non-characteristic appearance of the adsorption isotherms, and the possible reasons behind it. The same was expected with regards to the Langmuir plots, particularly since the values that were used to plot the adsorption isotherms, were part of the calculation for the respective Langmuir plots. Characteristically Langmuir plots should show an increase from left to right, however, this was not observed in all instances. The R$^2$ values for the Langmuir plots were in most instances more than 0.6, thus indicating that the plotted data fitted well to the Langmuir isotherm model. In addition to the Langmuir plots, the Langmuir constants were computed, according to research, the negative values for the Langmuir constants can be as a result of the adsorbates experiencing difficulty adsorbing to the adsorbents. Moreover, with regards to the negative intercepts obtained, research has suggested that this may have been as a result of too few data points, outliers, or large error bars.

Adsorption of anions (phosphate and nitrate) on metal ferrocyanides may have occurred due to the presence of divalent cations (viz. Ni$^{2+}$, Co$^{2+}$, Cd$^{2+}$) present at the outer coordination sphere of metal ferrocyanides. It is unlikely for the phosphate and nitrate anion to enter into the coordination sphere of metal ferrocyanides by replacing strong ligand like cyanide. The high
uptake of phosphate and nitrate on the surface of nickel ferrocyanide may be due to the high porosity of nickel ferrocyanide in comparison to cobalt and cadmium ferrocyanides. The high adsorption of phosphate in comparison to nitrate in each case may be due to its high molecular weight and anionic charges. The present method has significant advantages over existing methods for the determination and removal of phosphate and nitrate due to its low cost and also the mere fact that this method explores the possibility of using metal ferrocyanides for the removal of anions from ground and surface water.

Parallel studies in the determination and removal of anions through adsorption on various adsorbents have been reported in the chemical literature. A potential and cost-effective treatment method utilizing thermally activated bentonite was evaluated for the treatment of highly loaded real petroleum processing waste water was reported by El-Shamy et al. [32]. El-Shamy et al. [33] evaluated and compared the capability of polyacrylamide or ferric chloride or aluminum sulfate individually and mixed in treatment of the petroleum wastewater. They have also investigated corrosion behavior for untreated and treated wastewater. The effect of Cu²⁺ and Ni²⁺ ions in treated wastewater on the corrosion behavior of steel pipelines was demonstrated by Shehata et al. [34]. The effect of Cu²⁺ and Ni²⁺ on the corrosion behavior of steel pipelines revealed that the corrosion resistance of steel for treated and untreated water is almost the same. The theoretical approach of calculating intermolecular interaction energy of H-bonded systems of water clusters under the effect of a magnetic field is performed using DFT (Density Function Theory) level with B3LYP function on Gaussian 09 program by El–Kashef et al. [35]. The use of fungal biomass in batch and continuous flow systems for chromium (VI) recovery was studied by Saad et al. [36]. El-Shamy et al. [37] have described experimental work focused on the effect of magnetic treatment on certain water parameters such as temperature, electrical conductivity, total dissolved salts, and pH by exposing water to a permanent magnetic field with magnetic flux density (β = 1.45 T ±0.05).

5. CONCLUDING REMARKS

1. Nickel ferrocyanide is the most effective adsorbent to remove phosphate from both ground and surface waters. It is also the most effective adsorbent to remove nitrate from surface water. However, to further support this claim additional attributes should be varied, for instance temperature.

2. The adsorbent dosages used (in increasing increments) did not have a large effect on the removal of the anions. This was concluded for all the samples via an ANOVA test, there is no significant difference when increasing increments of the respective adsorbent are used to treat the H₂O samples, thus rejecting the null hypothesis (H₀).

3. Based on results and observations, the use of CoFc and CdFc to treat the water samples containing PO₄³⁻ and NO₃⁻ respectively, is not recommended. However, further studies can be conducted to ascertain whether the aforementioned compounds are being formed during the treatment of water samples with CoFc and CdFc. Subsequently, if possible, measures can be put into place to counteract the interference.

4. Within Guyana and the world as a whole, technologies like those studied in this research project should be explored and studied more in-depth with regards to treating of ground and surface water supply to facilitate the removal of primarily PO₄³⁻ but also NO₃⁻. Despite the results not being perfect, nonetheless a start, I believe that, that which was observed (if much more study is done particularly concerning the stability of these adsorbents in the presence of natural samples) can be applied to water treatment systems.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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