



Extraction of Iron (II) and Nickel (II) ions from Aqueous Solution using Palm Kernel Oil as a Green Extractant

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Abstract

A phase mediated, solvent extraction process was used in the removal of Fe (II) and Ni (II) ions from aqueous solutions. The organic phase was constituted with palm kernel oil (PKO) and the aqueous phase was equally modified with ethylenediaminetetraacetic acid (EDTA), as a chelating agent. Gas Chromatography Mass Spectrometry (GC-MS) and Flame Atomic Absorption Spectrophotometry (FAAS) were used to analyse the chemical composition of the PKO and the concentration of the metal ions present in the aqueous solutions, after the extraction procedures, respectively. The results from the GC-MS showed that the PKO consisted of fatty acids, mainly tridecanoic acid (32.16 %), pentadecanoic (18.02 %), dodecanoic (16.13 %), and hexadecenoic acid (12.4%).

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The concentrations of the metal ions after our extraction procedures were used to evaluate the percent extraction efficiency and the distribution ratio. High values of percent extraction efficiency, in the range of 97.37 – 99.21 percent and 97.19 – 99.78 percent, were achieved for the removal of Fe (II) and Ni (II) ions respectively. Equally, the values of the distribution coefficient were in the range of 37.03 – 124.93 as well as 34.57 – 459.79 for the removal of Fe (II) and Ni (II) ions. The percent extraction efficiency and the distribution coefficient increased with increase in initial concentration of these metal ions in the aqueous solution as well as with the volume of PKO that served as the organic phase. This was due to an equilibrium position shift in the direction that favoured the removal of the Fe (II) and Ni (II) ions from the aqueous phase. The presence of EDTA caused a further increase in the case of the extraction of Fe (II) ions but minimal effect was noted in the case of

Ni (II) ions. The results obtained revealed that PKO, with or without a chelating agent, can serve as an excellent green extractant for the removal of Fe (II) and Ni (II) metal ions from aqueous solutions.

Keywords: Solvent extraction; Iron (II) ions; Nickel (II) ions; Percent Extraction Efficiency; Distribution Coefficient; Green Extractant.

1.0 Introduction

Metals deteriorate over time due to their exposure to various environments thereby causing toxicity, even at low exposure levels (Tchounwou *et al.*, 2012; Abd Elnabi *et al.*, 2023). For instance, metals such as nickel and iron are common in industrial effluents from mining, painting, and other industries (Al-Qodah, 2006; Alam *et al.*, 2022). Thus, metals enter the environment through human activities such as manufacturing, processing, and refining (Nemerow & Agardy, 1998; Izah *et al.*, 2022). The non-biodegradability and toxicity of some metals have increased interest in viable methods for removing them from industrial waste water.

Industrial wastewater often contains Fe (II), Fe (III), and Ni (II) ions, which are released through hydro-metallurgical activities (Yang *et al.*, 2011; Mobasherpour *et al.*, 2012; Do *et al.*, 2019; Rani *et al.*, 2022). Due to the high demand for iron and nickel, it is imperative to develop efficient recovery methods for the extraction of their ions from industrial wastewater. Organic petroleum-based solvents have been used a lot for the extraction of these ions (Agrawal & Sahu, 2010; Barakat, 2011; Jisha *et al.*, 2023). Such recovered metal ions are reused or reduced to the metallic form through electrowinning processes (Komulainen *et al.*, 2009; Sureshkumar *et al.*, 2023). Industrial wastewater treatment techniques, which include electrodialysis (Barakat, 2011; Gurreri *et al.*, 2020), photocatalysis (Barakat,

2011; Bolisetty *et al.*, 2019; Gurreri *et al.*, 2020; Al-Nuaim *et al.*, 2023), chemical precipitation (Bolisetty *et al.*, 2019; Obotey Ezugbe & Rathilal, 2020), adsorption (Fu & Wang, 2011; Khulbe & Matsuura, 2018; Bolisetty *et al.*, 2019; Qasem *et al.*, 2021), as well as membrane filtration (Zhao *et al.*, 2016; Bolisetty *et al.*, 2019; Obotey Ezugbe & Rathilal, 2020) are all very expensive and require periodic maintenance procedures—with inherent downtime (Carolin *et al.*, 2017). As such, there is a need to develop efficient and cost-effective techniques in order to remove heavy metal contaminants from industrial wastewaters. It is because of this that research focus has tilted towards the development of benign and environmentally friendly extractants that can easily be used in liquid-liquid extraction of metal ions from aqueous solutions (Volesky, 2007; Carolin *et al.*, 2017; Nyamoto *et al.*, 2022).

To this end, this paper presents the results of experiments on the extraction of Fe (II) and Ni (II) ions from aqueous solutions using palm kernel oil as the organic phases in the liquid-liquid extraction processes. Palm kernel oil (PKO) contains palmitic acid, which is a typical long-chain carboxylic acid that is biocompatible and can complex with metal ions in aqueous solutions, thereby making it easy for the separation of such complexes of the metal ions (Abdel Salam *et al.*, 2011; Kalhapure & Akamanchi, 2013). Palm kernel oil is an edible plant oil obtained from the kernel of the oil palm tree (*Elaeisguineensis*), which grows very well in the southern belt of Nigeria (Poku, 2002; Gold *et al.*, 2012). PKO contains predominantly long-chain fatty acids, with Lauric acid dominating (Bhattacharya, 2003; Amri, 2011; Nainggolan & Sinaga, 2021). Our paper will pay emphasis on the effects of concentration/volume ratios of the aqueous and organic phases, the presence of the complexing agent, ethylenediaaminetetra-acetic acid

(EDTA) and the extraction time on the overall extraction efficiencies of the PKO as green extractants of Fe (II) and Ni (II) ions.

2.0 Materials and Method

2.1 Materials

The following materials were used for the various experiments that were carried out: Iron (II) sulphate (FeSO_4 ; Sigma Aldrich), Nickel (II) sulphate-hexahydrate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$; Philip Harris, $\geq 98\%$), Dichloromethane (Lobo Chemie, 98%), EDTA (Molychem), Sulphuric acid (H_2SO_4 ; Sigma Aldrich), Sodium hydroxide (NaOH ; Sigma Aldrich). These reagents were used as received without any further purification. The PKO used was produced from the palm kernel through the method presented by Poku (2002b).

In terms of equipments, we used a magnetic stirrer/hotplate, an analytical balance (Model JA 11003), a digital pH meter (LasanyL1-702), a flame atomic absorption spectrophotometer (Bulk Scientific 225/ 23x series) and an Agilent 8860 mass spectrometer with gas chromatography (GC-MS).

2.2 Method:

2.3 Preparation of aqueous and organic phases

1000 ppm stock solution of the aqueous phase containing a fixed concentration of Iron (II) (Fe (II)) and Nickel (II) (Ni (II)) ions was prepared by dissolving 1.36 g of Iron (II) sulphate (FeSO_4) and 2.24 g of Nickel (II) sulphate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$) in 500 ml deionised water respectively. Different concentrations of Fe (II) and Ni (II) solutions were obtained by diluting the stock solutions to 10 ppm, 30 ppm, and 50 ppm, respectively. 0.05 M EDTA was prepared by dissolving 9.306 g of salt in 500 ml of

deionized water. In terms of the PKO, the various volumes of the oil used for the experiments were 2 ml, 4 ml, 6 ml, 8 ml, and 10 ml.

2.4 Extraction of Iron (II) and Nickel (II) Ions from aqueous solution

The extraction of Fe (II) and Ni (II) was carried out using a solvent extraction procedure. This involved mixing 10 ml of aqueous solutions containing 10 ppm, 30 ppm, and 50 ppm of the metal ions with the different volumes of the PKO that served as the organic phase. The agitation of these two phases was done in a corked beaker by means of magnetic stirring, at 7 rms. The experimental set-up was placed on a hot plate, set at a temperature of 30°C . The stirring was done for a duration of 10 minutes, within which equilibrium was achieved. The mixture was allowed for few minutes for the partitioning and separation of the phases to occur. Finally, the aqueous and organic phases were separated by means of a separating funnel. 6 ml aliquot was taken from the aqueous phase for the determination of the metal ions concentrations through Atomic Absorption Spectrophotometry (AAS).

The percent extraction efficiency (% E) for Fe (II) and Ni (II) ions was calculated using the equation 1:

$$\% E = \frac{m_i - m_f}{m_i} \times 100 \% \quad (1)$$

where m_i and m_f are the starting and final concentrations of Ni (II) and Fe (II) ions in the aqueous phase, respectively.

The distribution coefficient (D) of Fe (II) and Ni (II) ions was calculated according to equation 2;

$$D = \frac{\text{Initial conc. of metal ion in extract} - \text{Final conc. of metal ion in aqueous phase}}{\text{Final concentration of metal ion}} \quad (2)$$

2.5 Extraction of Fe (II) and Ni (II) ions from aqueous solutions using PKO in the presence of EDTA

The extraction of Fe (II) and Ni (II) ions using PKO in the presence of EDTA was carried out using aqueous solutions containing different concentrations (10 ppm, 30 ppm, and 50 ppm) of Fe (II) and Ni (II) ions. These aqueous solutions were mixed with various volumes of PKO (2 ml, 4 ml, 6 ml, 8 ml, and 10 ml). 2 ml of EDTA was added to the mixed phases and the agitation procedure stated in 2.4 repeated.

2.6 Evaluation of the chemical compounds present in the PKO

The Agilent 8860 Gas Chromatograph linked to a 5977 °C inert mass spectrometer, with an electron-Impact source (Agilent Technologies) was used for the analysis. A HP-5 capillary column covered with 5% phenyl methyl siloxane was used as the stationary phase for the compound separation (30 m length x 0.32 mm diameter x 0.25 m film thickness). Prior to calibration, the MS was auto-tuned to perfluorotributylamine (PFTBA) using already established criteria to check the abundance of m/z 69, 219, 502 and other instrument optimal and sensitivity conditions.

The determination of the level of fatty acid and Methyl Esters Standard (FAMES) in the sample was carried out using GC-MS by operating mass selective detector (MSD) in selective ion monitoring (SIM) and scan mode to ensure low level detection of the target constituents. The samples were analysed after calibration to get the relevant fatty acids and methyl esters concentration, percentage composition, and mass spectral data. The three-point serial dilution calibration standards (5.0, 10.0, and 20.0 ppm) was prepared from the stock and used to calibrate the Gas chromatography (GC-MS). The other details of the instrument parameters and settings used in the determination are presented in our earlier

publication (Ite *et al.*, 2023) and reported in other literatures by Cao and Ito (2003), Degenhardt *et al.* (2002) and Geus *et al.* (2001) respectively.

3.0 Result and Discussion

The various results obtained in the course of this investigation are discussed here. It covers the composition of PKO, the percent extraction efficiency (% E), distribution coefficient of Fe (II) and Ni (II) ions extracted from the aqueous phase using the PKO in the organic phase.

3.1 Extraction of Fe (II) ions from the aqueous phase using PKO

PKO was used as the organic phase for the extraction of Fe (II) ions from aqueous solutions. The results in Table 1 and Figure 1 show the variation of percent extraction efficiency for removal of Fe (II) ions, at various initial concentrations in the aqueous phase, with different volumes of PKO respectively.

Table 2 and Figure 2 show the variation of the distribution coefficient of Fe (II) ions between the aqueous and the organic phases.

3.2 The extraction of Fe (II) ions from the aqueous phase using PKO in the presence of EDTA

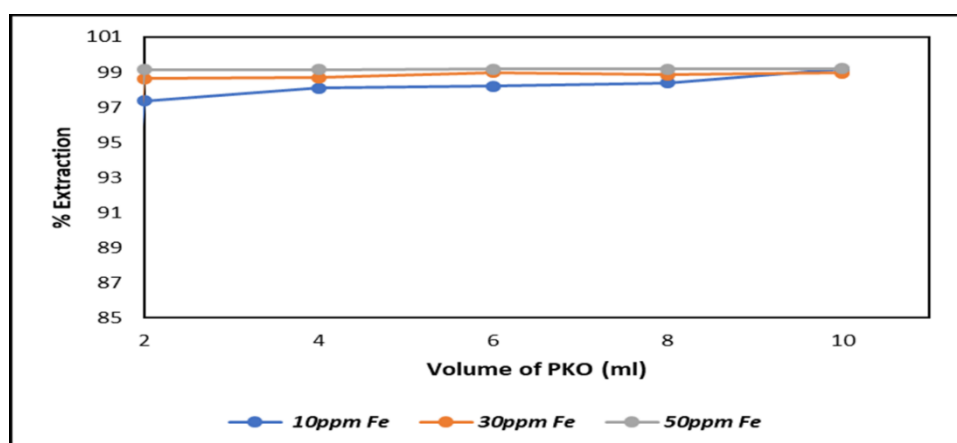
The results in Table 3 and Figure 3 show the percent extraction efficiency for Fe (II) ions in the presence of EDTA. Table 4 and the corresponding Figure 4 show the distribution coefficient of Fe (II) ions extracted in the presence of EDTA

3.3 Extraction of Ni (II) ions from the aqueous phase using PKO

Table 5 and Figure 5 show the result of the percent extraction efficiency of Ni (II) ions. Table 6 and Figure 6 show the distribution coefficient of Ni (II) ions.

Table 1: Percent extraction efficiency for Fe (II) ions using PKO. Initial concentration of Fe (II) = 10 ppm, 30 ppm, and 50 ppm

S/N	Volume of PKO (ml)	Percent extraction efficiency		
		10 ppm Fe (II)	30 ppm Fe (II)	50 ppm Fe (II)
1	2	97.37	98.63	99.14
2	4	98.10	98.69	99.14
3	6	98.21	98.97	99.16
4	8	98.38	98.85	99.18
5	10	99.17	98.95	99.21

**Figure 1:** Variation of percent extraction efficiency of Fe (II) ions with various volume of PKO**Table 2:** Distribution coefficients of Fe (II) ions, initial concentrations of Fe (II) ions = 10 ppm, 30 ppm and 50 ppm

S/N	Volume of PKO (ml)	Distribution coefficient		
		10 ppm Fe (II)	30 ppm Fe (II)	50 ppm Fe (II)
1	2	37.03	72.23	114.62
2	4	51.76	75.54	114.62
3	6	54.74	96.25	118.10
4	8	60.84	86.24	121.04
5	10	119.11	94.68	124.93

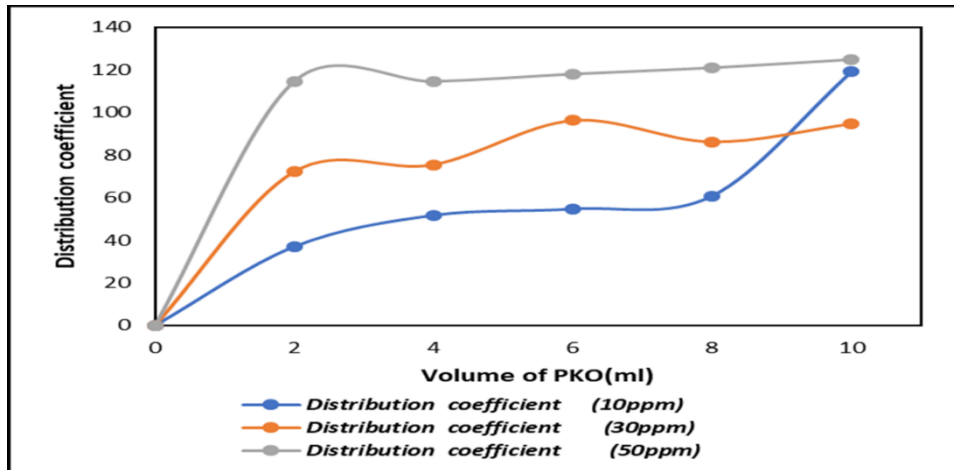


Figure 2: Variation of distribution coefficient of Fe (II) ions with various volume of PKO

Table 3: Percent extraction efficiency for Fe (II) ions using PKO in the presence of EDTA. Initial concentration of Fe (II) = 10 ppm, 30 ppm, and 50 ppm

S/N	Volume of PKO (ml)	Percent extraction efficiency		
		10 ppm Fe (II)	30 ppm Fe (II)	50 ppm Fe (II)
1	2	97.45	99.25	99.52
2	4	98.46	99.26	99.52
3	6	98.61	99.28	99.52
4	8	98.71	99.32	99.55
5	10	98.94	99.34	99.56

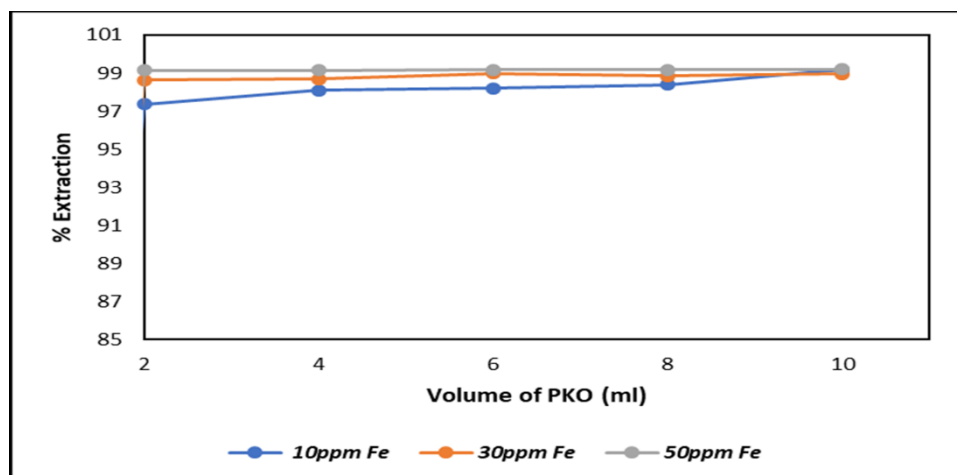
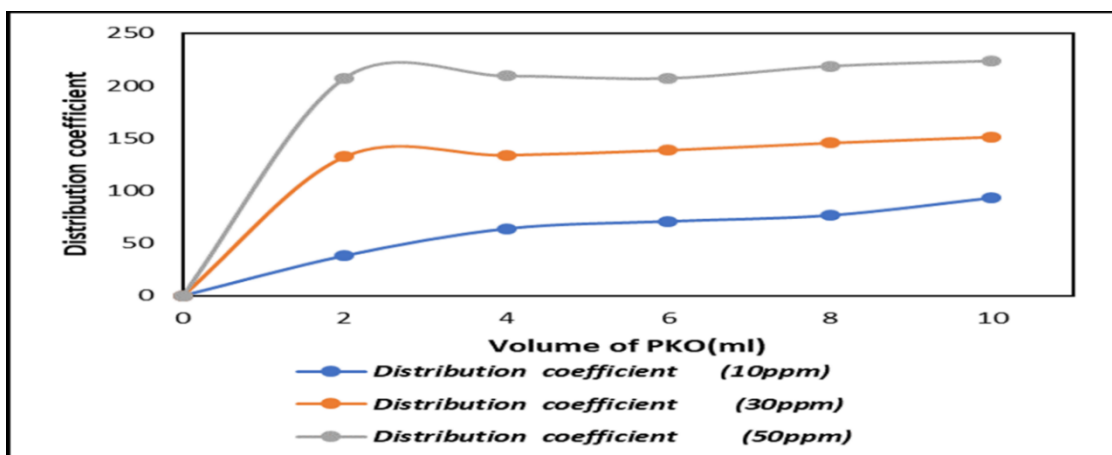


Figure 3: Variation of percent extraction efficiency of Fe (II) ions with various volumes of PKO in the presence of EDTA.

Table 4: Distribution coefficients for Fe (II) ions using PKO presence of EDTA. Initial concentration of Fe (II) ions = 10 ppm, 30 ppm and 50 ppm

S/N	Volume of PKO (ml)	Distribution coefficient		
		10 ppm Fe (II)	30 ppm Fe (II)	50 ppm Fe (II)
1	2	38.17	132.36	207.21
2	4	63.89	133.87	209.42
3	6	70.98	138.64	207.21
4	8	76.64	145.54	218.79
5	10	93.31	151.19	223.79

**Figure 4:** Variation of distribution coefficient for Fe (II) ions with the various volume of PKO in the presence of EDTA**Table 5:** Percent extraction efficiency for Ni (II) ions, initial concentration of Ni (II) ions = 10 ppm, 30 ppm, and 50 ppm;

S/N	Volume of PKO (ml)	Percentage Efficiency of Extraction		
		10 ppm Ni (II)	30 ppm Ni (II)	50 ppm Ni (II)
1	2	97.19	99.33	99.64
2	4	97.42	99.37	99.67
3	6	98.68	99.56	99.71
4	8	98.68	99.64	99.71
5	10	98.80	99.64	99.78

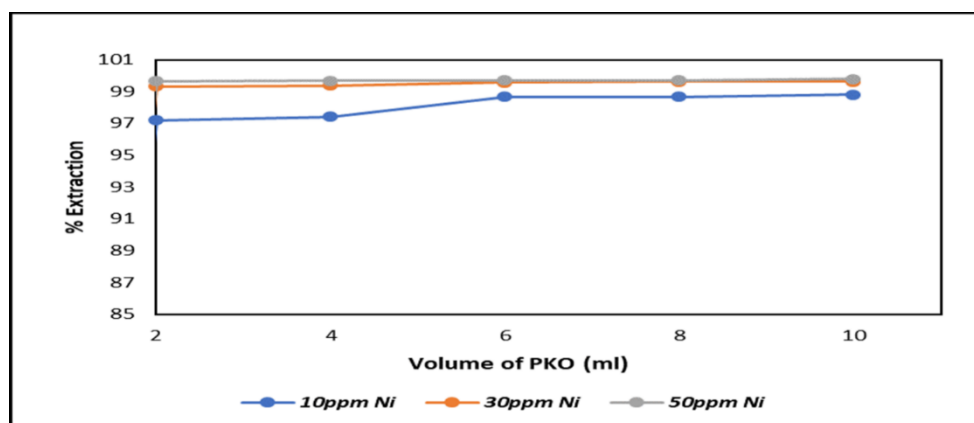


Figure 5: Variation of percent extraction efficiency of Ni (II) ions with various volume of PKO.

Table 6: Distribution coefficients for Ni (II) ions, initial concentration of Ni (II) ions = 10 ppm, 30 ppm and 50 ppm

S/N	Volume of PKO (ml)	Distribution Coefficient		
		10 ppm Ni (II)	30 ppm Ni (II)	50 ppm Ni (II)
1	2	34.57	148.57	280.60
2	4	37.74	157.67	300.11
3	6	75.03	227.09	348.57
4	8	75.03	275.48	348.57
5	10	82.32	275.48	459.79

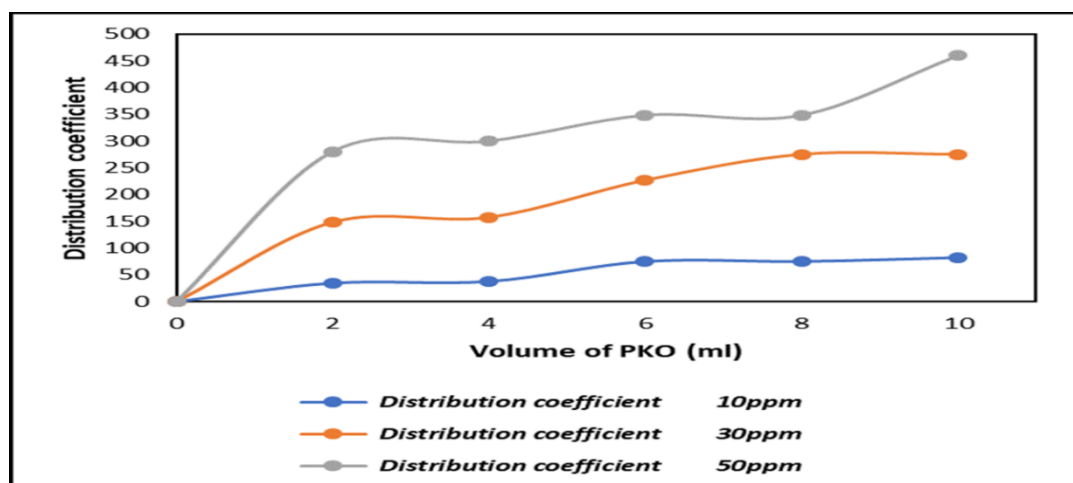


Figure 6: Variation of distribution coefficient of Ni (II) ions with various volumes of PKO

3.4 The Extraction of Ni (II) ions using PKO in the presence of EDTA

Table 7 and Figure 7 show the percent extraction efficiencies of Ni (II) ions removal from the aqueous phase using PKO and in the

presence of EDTA. Table 8 and Figure 8 show the distribution coefficient of Ni (II) ions extracted in the presence of EDTA.

Table 7: Percent extraction efficiency for Ni (II) ions using PKO in the presence of EDTA. Initial concentration of Ni (II) ions = 10 ppm, 30 ppm, and 50 ppm

S/N	Volume of PKO (ml)	Percent Efficiency of Extraction		
		10 ppm Ni (II)	30 ppm Ni (II)	50 ppm Ni (II)
1	2	96.61	99.14	99.48
2	4	96.84	99.18	99.55
3	6	97.76	99.29	99.62
4	8	97.99	99.33	99.69
5	10	97.99	99.68	99.76

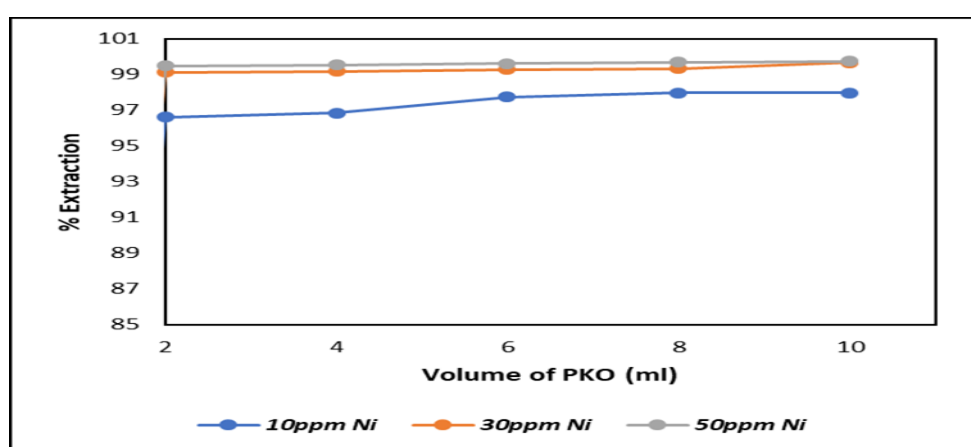


Figure 7: Variation of percent extraction efficiency of Ni (II) ions with various volumes of PKO in the presence of EDTA.

Table 8: Distribution coefficients of Ni (II) ions between aqueous and PKO with EDTA. Initial concentration of Ni (II) = 10 ppm, 30 ppm and 50 ppm

S/N	Volume of PKO (ml)	Distribution coefficient		
		10 ppm Ni (II)	30 ppm Ni (II)	50 ppm Ni (II)
1	2	28.53	115.23	192.71
2	4	30.68	120.65	222.62
3	6	43.72	140.45	263.45
4	8	48.86	148.57	322.53
5	10	48.86	308.28	415.61

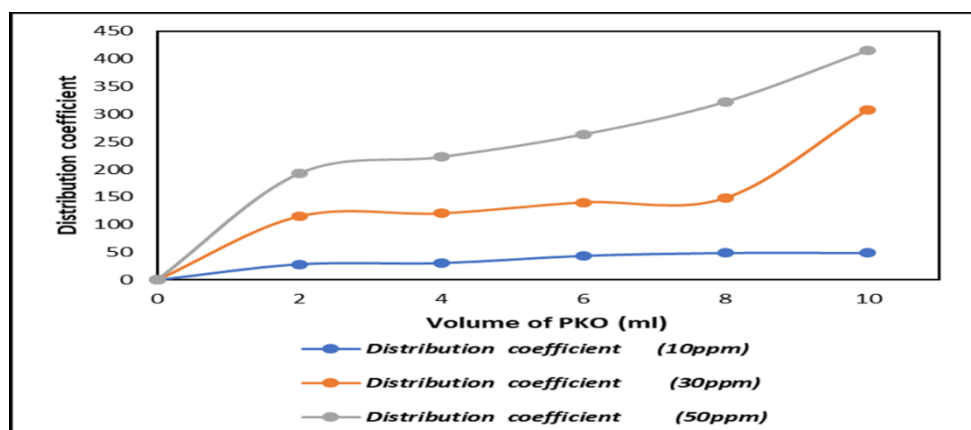


Figure 8: Variation of distribution coefficient Ni (II) ions with various volumes of PKO in the presence of EDTA

3.5 Fatty Acid Percent Composition of PKO

The composition of PKO was mostly fatty acids of various concentrations. The different fatty acids present in the PKO have been presented in our previously published article (Ite et al., 2023).

3.6 Discussion

3.6.1 The effect of the initial concentration of Fe (II) ions on the percent extraction efficiency and distribution coefficient

The initial state of the metallic solute concentration in aqueous solution often varies from one source to another, and therefore, it is necessary to evaluate the effect of the initial concentration of Fe (II) ions. The results presented in Tables 1 to 4 show that the concentration of Fe (II) ions increases from 10 ppm to 30 ppm and finally to 50 ppm, and there is a consistent rise in the percent extraction efficiencies.

At an initial Fe (II) ion concentration of 10 ppm (see Table 1), the extraction efficiencies were in the range of 97.37 % to 99.17 %. At a starting concentration of 30 ppm Fe (II) ions, the efficiencies were in the range of 98.63 % to 98.95 %, while at a 50ppm initial concentration of Fe (II), the extraction efficiencies witnessed

a further increase and were in the range of 99.14 % to 99.21 %. The rise in percent extraction efficiencies and the distribution coefficient of Fe (II) ions are attributed to the increase in the driving force for the Fe (II) ions transfer at the interface of the aqueous phase with the PKO, resulting in the equilibrium position of the extraction reaction to shift to the right.

The plots of the percent extraction efficiencies and the distribution coefficients of Fe (II) ions against the volume of PKO are presented in Figures 1 and 2, respectively. Since the rise in the initial concentration of Fe (II) ions in the aqueous phase has compelled the equilibrium position in the extraction reaction to shift in the forward direction, the percent extraction efficiencies and distribution coefficients all increased as seen in Figures 1 and 2.

3.6.2 Effect of the volume of PKO on the percent extraction efficiency and distribution coefficient for Fe (II) ions

The different volumes of PKO used for the extraction process ranged from 2 ml to 4 ml, 6 ml, 8 ml, and 10 ml, as presented in the results shown in Tables 1–4. The results obtained show that Fe (II) ions extraction from the aqueous phase increases with increase in the volume of the PKO. With the increase in the volume of PKO to 10 ml, maximum values of the percent of extraction efficiency were

obtained for all the initial concentrations of the Fe (II) ions that were in the aqueous phase. The implication is that the higher the volume of PKO, the more the free fatty acids that bind with the Fe (II) ions, thus causing more Fe (II) ions to be distributed into the organic phase. This is evident from the percent extraction efficiencies and the distribution coefficients of Fe (II) ions in the organic phase (Figures 1–4).

3.6.3 Effect of EDTA on the percent extraction efficiency and distribution coefficient for Fe (II) Ions

EDTA is a well-known complexing agent (Chang *et al.*, 2017; Zhang *et al.*, 2021; Wang & Li, 2023). When 2 ml of EDTA was added to the extraction system, considerable improvements in the extraction of Fe (II) ions were noted, as can be seen from the significantly higher percent extraction efficiencies (see Table 3 and Figure 3) and distribution coefficients (see Table 4 and Figure 4). The addition of EDTA to the aqueous solution produced a colour change immediately due to the splitting of the d-orbital of Fe (II) ions by EDTA. On exposure to the PKO-organic phase, the intense colour due to the Fe (II)-EDTA complex formation started reducing. As the volume of PKO used in the process increased, more of the carboxyl functional groups were also made available for complexation with Fe (II) ions; thus, Fe (II) ions bind with the carboxyl group and are extracted and distributed into the organic phase. From the FAAS analysis, the EDTA addition increased the extent of Fe (II) ions extracted from the aqueous phase.

3.6.4 The effect of initial concentration of Ni (II) ions on the percent extraction efficiency and the distribution coefficient.

The initial state of the metallic solute concentration in aqueous solution often varies from one source to another, and therefore, it is necessary to evaluate the impact of increasing the initial concentration of Ni (II) ions from 10

ppm to 30 ppm and finally to 50 ppm. There was a consistent rise in the percent extraction efficiencies. At an initial Ni (II) ion concentration of 10 ppm, the extraction efficiencies were in the range of 97.19% to 98.80% (See Table 5). At an initial concentration of 30 ppm Ni (II) ions, the efficiencies were in the range of 99.33% to 99.64%, while at a 50ppm initial concentration of Ni (II) ions, the extraction efficiencies witnessed a further increase and were in the range of 99.64% to 99.78%. The rise in percentage extraction efficiencies is essentially due to more sites for reaction with the metal ions, thus shifting the reaction for the extraction process to the right. The plots of the percent extraction efficiencies and the distribution coefficients against the various volumes of PKO are shown in Figures 5 and 6 respectively.

3.6.5 Effect of the Volume of PKO on the percent extraction efficiency of Ni (II) ions

The same series of volumes of PKO ranging from 2 ml to 10 ml were used for the extraction process, as shown in Tables 5 to 8. The result obtained shows that Ni (II) ions extraction from the aqueous phase increases when the volume of PKO increases. However, the maximum values of the percent extraction efficiency were obtained when the volume of PKO was 10 ml. This implies that the higher volume of PKO results in an increase in the ratio of the organic phase to the aqueous phase, resulting in more Ni (II) ions being extracted into the organic phase. This resulted in higher percentage extraction efficiencies (see Figure 5 and 7) and the distribution coefficient of Ni (II) ions in the organic phase shown in Figures 6 and 8.

3.6.6 Effect of EDTA on the percent extraction efficiency and distribution coefficient for Ni (II) ions

The effect of EDTA on the extraction of the Ni (II) ions from the aqueous phase when PKO was used as the organic phase was minimal.

The chemical constituents of PKO are mostly long chains of fatty acids. As the volume of PKO used in the process increased, more of the carboxyl functional groups should have been made available for complexation with the Ni (II) ions. However, there is competitive chelation in the sense that the EDTA might form complexes with the metal carboxylates thereby reducing the impact of EDTA in the extraction of Ni (II) ions. This explanation is responsible for the observed percent extraction efficiencies and the distribution coefficients presented in Tables 5 to 6 as compared with those in Tables 7 to 8 as well as in Figures 6, 7 and 8.

3.6.7 Fatty Acid Percent Composition of PKO

The GC-MS analysis results showed that tridecanoic acid (32.16 %), pentadecanoic (18.02 %), dodecanoic (16.13 %), and hexadecanoic acid (12.4%) were the fatty acids with the highest percentages weight composition in the PKO sample. Others such as the hexadecenoic acid palmitic acid (C16:0) and pentadecanoic acid (C15:0) were also relatively abundant in the sample. These findings of this study agrees with those of other researchers, like Edem (2002) and Gibon (2012).

4.0 Conclusion and Recommendations

The extraction of Fe (II) and Ni (II) ions from aqueous solutions using PKO as an extractant has shown that the distribution coefficient and the percentage extraction efficiency increased with a rise in the initial concentration of these ions in the aqueous phase. The presence of EDTA enhanced the extraction of Fe (II) ions when compared to cases without EDTA. However, the presence of EDTA had minimal effect on the extraction of Ni (II) ions. The study has shown that PKO is a good extractant for the removal of Fe (II) and Ni (II) ions that

are water bound. The volume of PKO used for the extraction process had a strong influence on the distribution coefficients and percentage efficiencies of Fe (II) and Ni (II) ion extraction. The findings of this work show that PKO can be used as an affordable organic extractant for the removal of metal ions that are present in water, and can serve as a preferable as green extractant due to its zero environmental impact.

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