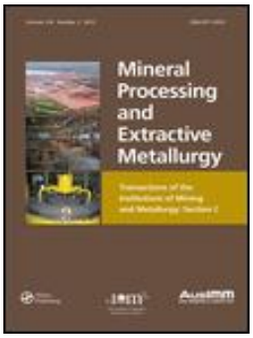

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Characterisation and treatment of spent deposited sludge of transformer oil (DSTO) by acid leaching and solvent extraction

Alafara A. Baba[a], Joshua S. Ayodele[b], Oloduowo M. Ameen[b], Abdulrasaq Jimoh[c], Uduakobong Johnson[a], Bilikisu A. Jimoh[d] and Amudat Lawal[b]

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ABSTRACT

The characterisation of spent transformer oil to determine its fitness for re-refining as well as the recovery of metals by hydrometallurgical treatment in chloride medium has been investigated. The leaching experiments on the spent material showed that its dissolution increases with leachant concentration performed at 75°C. Leaching studies showed that at optimal conditions, the dissolution reached 93% by 1.0 mol L^{-1} HCl solution within 120 min at 75°C. The solvent extraction of copper by bis(2,2,4-trimethylpentyl) phosphinic acid (CYANEX®272) in kerosene from a pregnant solution of aqueous spent deposited sludge of transformer oil containing 1243.4 mg L^{-1} Cu^{++}, 389.2 mg L^{-1} $Fe^{++/+++}$, 110.7 mg L^{-1} Ca^{++} and 235 mg L^{-1} Al^{+++} was studied. The results for the solvent extraction give an extraction efficiency of 91.77% total copper acquired via 0.2 mol L^{-1} CYANEX®272 in kerosene at 27 ± 2°C within 25 min at pH 4.0. A 0.1 mol L^{-1} HCl solution was found to be sufficient for the stripping of about 94.77% copper from the loaded organic phase. The Fourier transform–infrared spectroscopic analysis of the loaded organic phase supports the stripping experiment as there was no Cu metal ion present in the separated organic phase.

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Transformer oil; deposited sludge; leaching; solvent extraction; copper; Cyanex®272

Introduction

The degradation of transformer oil with oxygen as the reagent is known as oxidation of the oil. Thus, oxidation of transformer oil not only leads to the production of many polar compounds, such as acids, aldehydes, ketones, peroxides and alcohols, which affects the insulating properties of the oil, but also forms the sludge. For instance, the acid causes corrosion and accelerated wear, while the sludge increases the viscosity and block valves as well as other system components. These affect the heat transfer properties of the oil and often reduce the loading performance of transformers, thereby reducing their life span (Krishnamoorthy et al. 1992). Depending on the type and capacity, typical transformer oil contains 90–1485 kg weight of oil with varying degree of recoverable sludge and oil mixture after degradation (MEKSAN Transformer Ltd. 2011). Generally, transformer oil exists in two types:

(i) Paraffin-based transformer oil,
(ii) Naphtha-based transformer oil.

On comparison of both transformer oils, paraffin oil commonly used in Nigeria is less oxidised than Naphtha oil. However, spent transformer oil is often made up of materials such as copper, iron, cellulose wood, rubbers and mineral oil. Little deterioration occurs after years of service when the oil in a transformer operates under normal load conditions, adequately ventilated and free from moisture. However, if the transformer is not properly managed and is over loaded or adequately ventilated, the oil temperature will be high and oil deterioration would be accelerated (Kimball 1995).

Consequently, transformer insulating oil must withstand the rigours of thermal and electrical stress during its service life. The specification requirements for new insulating oils are that the oil shall be in accordance with the American Standard for Testing Materials (ASTM) (Allen 2009). The aging of insulating oil is associated with oxidation under the harsh environment (Shangbi 2003; Homagk et al. 2008). Thus, oil in a transformer becomes increasingly contaminated as the transformer age in service, oil quality deterioration due to the presence of oxygen and moisture at a specific temperature forming a deposited sludge (Solokov 2006). This sludge or solid wastes may contain heavy metals, such as Au, Ag, Ni, Co, Cu and Zn. These metals, which apparently may be toxic, are valuable elements if re-processed. Therefore, the development of low-cost and eco-friendly technique in the treatment of this kind of material would be a worthwhile venture

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in the area of waste-to-wealth initiatives as world global demand for metal is immeasurable. From the literature, pyrometallurgical and hydrometallurgical route or combination of the two can be employed in the metal extraction process of some secondary wastes including spent transformer oil (Leclerc et al. 2003; Turan et al. 2004). The hydrometallurgical process has been established to be cost effective and eco-friendly route for the treatment of materials with low metal contents (Leclerc et al. 2003; Somer and Nakisci Unlu 2006; Baba and Adekola 2011). Consequently, several authors have used this route as a potential means of metal extraction and beneficiation alternatives from various secondary sources (Baba et al. 2009, 2015; Manis et al. 2010; Xie and Dreisinger 2010; Ochromowicz and Chmielewski 2013). The separation of copper and zinc from a model brass pickle liquor was studied by Manis et al. (2010). The results showed that extraction of metal ions increased with pH and copper was extracted completely into the organic phase at equilibrium pH 4.9, with zinc at the pH of above 5.0. However, zinc was quantitatively extracted into the organic phase at pH 7.0.

Venkata Lakshmi and Murthy (2012) focused on corrosion phenomenon involving copper, corrosive sulphur and solid insulation of oil-immersed transformers. They used the SEM scan after aging the paper-insulated copper conductor in transformer oil with passivator at 140°C for 140 h to show the formation of thin film of passivator on the copper surface. In addition, the SEM, XRD and EDX scans after aging the paper-insulated copper conductor in transformer oil without passivator at 140°C for 140 h indicated scattered deposit of copper sulphide formation on the copper surface and on the paper insulation.

Xudong et al. (2012) studied the chemical composition variation and the influence of transformer oil aging on electrical properties, such as dielectric loss factor, and physicochemical properties, such as interfacial tension. The correlation between hydrogenated transformer oil, electrical and physicochemical properties and its composition was investigated. They are able to conclude that there is a correlation between these parameters and the chemical composition of transformer oil. Aromatic composition stays constant, alkane composition increases gradually, while cycloalkane composition decreases gradually with aging time, constant colour of the oil, acid value and viscosity increased gradually, while interfacial tension decreases as the dielectric loss factor rises slowly at the beginning of aging. In the current work, the characterisation and extraction of copper from spent deposited sludge of transformer oil was examined in chloride media by the CYANEX®272 extractant. The purified copper solution was beneficiated as copper oxide, an important compound with many diverse industrial uses such as *p*-type semi–conductor (Baba et al. 2015).

Materials and methods

Materials

The spent sludge of transformer oil used for this study was obtained from Ayetoro III 300 kVA, 11/0.415 kV distribution transformer under Baboko Business Hub, Ibadan Electricity Distribution Company, Ilorin Kwara State, Nigeria. This transformer has 245 kg oil weight capacity with recoverable 25 kg of combined sludge oil mixture. All reagents used were of analytical grade.

Deionised water and distilled kerosene was used in the preparation of all aqueous and organic solutions in this study, respectively.

Physicochemical and chemical characterisation of the material, including selected products of optimal conditions, were done using atomic absorption spectrometry (model AA320N AAS spectrometer), gas chromatography/mass spectrometry (GC-MS model: Agilent 19091S – 43HP – 5MS with column 5% phenyl methyl silox: 30 m × 250 μm × 0.25 μm), X-ray diffraction (EMPY, Shimadzu XRD model 6000) electron diffraction X-ray (energy-dispersive X-ray fluorescence (EDXRF), SEM model Leo 1450 with Lab6 Filament) based on the standard procedures available in the literature (Association of Official Analytical Chemists (AOAC) 1990; Baba et al. 2009, 2015).

Methods

Leaching procedure

Leaching experiments were carried out in 250 ml glass reactor with a mechanical stirrer and temperature control unit. For each experiment, 10 g L^{-1} of the dried deposited sludge were reacted independently with hydrochloric acid solution of different molarities (0.1, 0.2, 0.5, 1.0 and 2.0 mol L^{-1}). The reaction time was kept at 120 min in each case. The solution in the reactor after each leaching was filtered; both residue and filtrate were collected for further characterisation (Somer and Nakisci Unlu 2006). The extent of dissolution showed 28.29%, 45.22%, 74.56%, 92.83% and 90.21% reaction efficiencies. However, acid concentration above 1.0 mol L^{-1} gave decreasing dissolution rate (90.01%). The pregnant leach solution (PLS) collected at optimal leaching was used for the solvent extraction studies.

Solvent extraction

Solvent extraction experiment was carried out using the PLS from section 2.2.1 and then equilibrated with an equal volume of organic phase containing known concentration of the CYANEX®272 extractant (Figure 1) in kerosene at room temperature (27 ± 2°C) by hand shaking for a period of 25 min. Equilibrium pH was adjusted by the direct addition of concentrated

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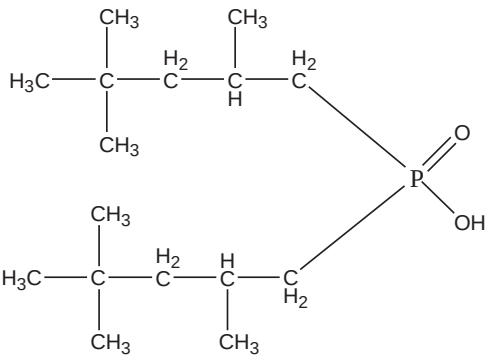

Figure 1. Structure of Cyanex®272.

HCl (0.1 mol L^{-1}) and NaOH (0.1 mol L^{-1}) solutions with HANNAH pH metre. The aqueous solutions were analysed for metal concentrations by AAS equipment. The free acid content was determined by the titration method with pH control. The concentration of copper in the organic phase was calculated from a difference between its initial concentrations in PLS and its concentration in raffinate at fixed organic/aqueous (O/A) phase ratio (Ochromowicz and Chmielewski 2013).

For every extraction experiment, the ratio of total Cu^{2+} extracted into the organic phase to its concentration in the aqueous phase is its distribution ratio (D), expressed as follows:

$$D = \frac{[Cu^{2+}]_{org}}{[Cu^{2+}]_{aq}} \quad (1)$$

where org. and aq. are organic and aqueous phases, respectively. The extraction efficiency for the process is consistent with the following relation:

$$\%\ \text{Extraction}(E) = \frac{100D}{D + V_{aq}/V_{org}} \quad (2)$$

V is the solvent volume.

Results and discussion

Characterisation studies

The results of the characterisation of the spent deposited sludge transformer oil before acid leaching are summarised in Table 1. Table 1 apparently defines the current state of deposited spent transformer oil (DSTO) compared to ASTM standard values. For example, the indication of the oil acidity expressed by its acid value, 0.8 mg KOH $gram^{-1}$. This value is below ASTM standard and as such is not good for normal operation of transformer (Association of Official Analytical Chemists (AOAC) 1990).

The GC-MS results (Figure 2, Table 2) of the DSTO indicate the presence of compounds such as Tridecane, Heptadecane, Nonadecane, 2-Bromodecane, 2-Buten-1-one, Octadecane, 4H-1,3-Benzochoxin-4-one, Pentadecane and Butylated hydroxytoluene. It is evident from Figure 2 that heptadecane having the highest yield is the most abundant compound present in the used oil.

The elemental composition of the DSTO by EDXRF gave SiO_2 (17.07%), TiO_2 (0.37%), P_2O_5 (4.1%), Fe_2O_3 (54.53%), CaO (6.75%), MgO (0.04%), Na_2O (0.3%), K_2O (0.23), V_2O_5 (0.03%), Cr_2O_3 (0.055%), CuO (13.14%), ZnO (0.77%) and Eu_2O_3 (0.6%). The structural morphology of spent sludge showing detailed images examined by the SEM before and after leaching is depicted in Figure 3.

Before leaching (Figure 3(a)), the image shows pyramidal particles in different sizes with thick surface body, whereas after optimal leaching (Figure 3 (b)), the image gave a deviation indicating the influence of the acid resulting in the formation of a thick cluster surface evident of metal extraction as compared to the initial raw surface particles. The Energy Diffraction Spectrum (EDS) data for the elemental composition of the spent deposited sludge before and after leaching with 1.0 mol L^{-1} HCl solution at 75°C for 120 min are presented in Table 3.

The composition of the PLS examined by atomic absorption spectrophotometer gave 1243.4 mg L^{-1} Cu^{++}, 789.2 mg L^{-1} $Fe^{++/+++}$, 110.7 mg L^{-1} Ca^{++} and 23.5 mg L^{-1}, Al^{+++}. Before solvent extraction investigations, the iron constituting major impurities in the leach liquor was eliminated by adjusting the pH of the solution from 1.2 to 3.6 with ammoniacal solution (Baba et al. 2009) to obtain the following results: 1239.6 mg L^{-1} Cu^{++}, 0.6 mg L^{-1} $Fe^{++/+++}$, 2.1 mg L^{-1} Ca^{++}, and 1.2 mg L^{-1} Al^{+++}.

Table 1. The physicochemical properties of DSTO as compared with ASTM D1816.

Property	Experimental value	ASTM D1816 STANDARD
Density	0.82 g cm^{-3}	0.55–0.89 g cm^{-3}
Viscosity	9.7 cSt	3.0–12 cSt (max)
Flash point	140°C	145°C (max)
pH	4.27	5.5–8.2 (min–max)
Specific gravity	0.86	0.91 (max)
Cloud point	9°C	7–15°C
Water content	1.32×10^5 mg L^{-1}	30 mg L^{-1}
Free fatty acid	0.04 mg KOH g^{-1} oil	0.01–0.08 mg KOH g^{-1} oil (min–max)
Acid number	0.8 mg KOH g^{-1} oil	0.01–0.03 KOH g^{-1} oil (min–max)
Pour point	−7°C	−8°C −6°C (min–max)
Boiling point	220°C	120–230°C (min–max)

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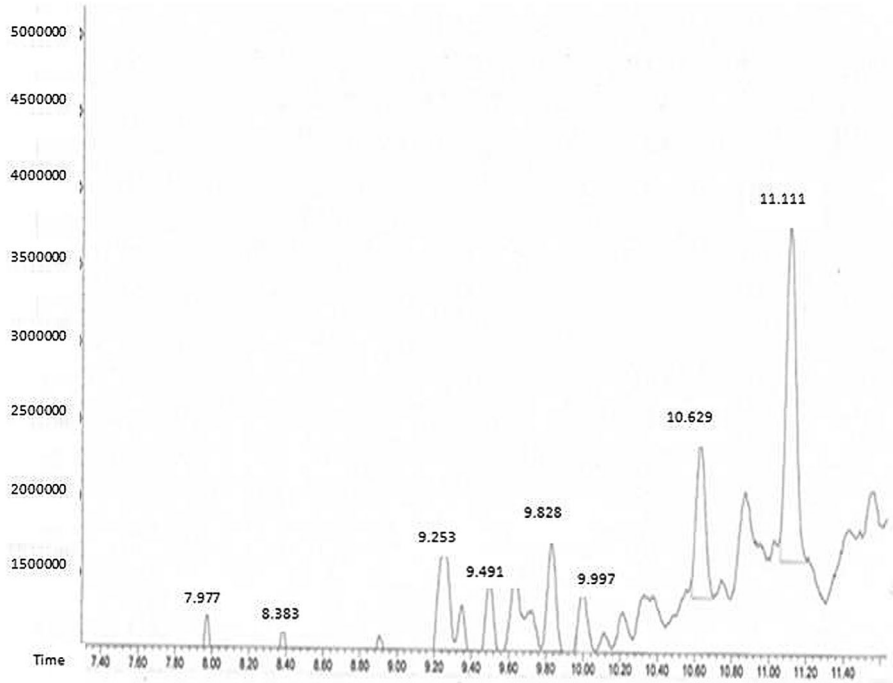

Figure 2. GC spectrum of spent DSTO.

Solvent extraction studies

Effect of equilibrium pH

To investigate the effect of pH on the extraction of Cu from its aqueous solution by 0.2 mol L^{-1} Cyanex in kerosene, experiments were carried out in the equilibrium pH ranges of 1–5 at 27 ± 2°C for 25 min. Figure 4 presents a summary of the percentage of total Cu extracted at different equilibrium pH. Figure 4 shows that the percentage extraction of copper varies from 34.6 to 91.66 with increasing pH from 1 to 4. On further increase of pH above pH 4, the percentage extraction decreased to 91.50% at pH 5. Hence, pH 4 was used for further optimisation of other parameters. The possible reason for this observation as reported by Hosseini et al. (2011) was, at high pH where high metal extraction is envisaged, the distribution coefficient considerably increases, while extraction percentage decreases or increases only slightly.

To assess the number of mole of hydrogen ion required to extract a mole of copper metal ion at defined extractant concentration, the plot of log D vs log [Cyanex] was made (Figure 5). This plot gave a straight line with a slope of 0.47. This slope 0.47 ~ 0.5 ($R = 0.955$) indicates that 0.5 or ½ mole of hydrogen ion is required to extract 1 mole of Cu^{2+} by Cyanex extractant.

Effect of Cyanex concentration

To confirm the effect of Cyanex®272 on copper extraction in the leachate at optimal leaching, the concentration of Cyanex®272 in kerosene was varied in the range of 0.01–0.25 mol L^{-1} at pH 4.0. The result showed the extraction of copper increased with increasing Cyanex®272 concentrations, as presented in Figure 6.

Experimental conditions: [Cyanex] 0.05–0.25 mol L^{-1}, temperature: 27 ± 2°C, contact time: 25 min.

Figure 6 shows that the percentage extraction of Copper increases from 43.7 to 91.7% with increasing Cyanex concentration from 0.05to 0.2 mol L^{-1}. However, a further increase above 0.2 mol L^{-1} gave drastic decrease in extraction to 90.1% using 0.05 mol L^{-1} Cyanex. Hence, 0.2 mol L^{-1}, Cyanex concentration giving the highest extraction yield was kept as optimal concentration and was used for further studies. For better understanding of the number of moles of extractant required to extract a mole of Cu^{2+} ion, the plot of log D vs log [Cyanex] revealed a straight line with a slope of 1.72, approximately 2 (Figure 7). This indicates the association of two moles of extractant per mole of metal ion during the extraction process.

Discussion

Based on the aforementioned results, the extraction equilibrium of copper (M^{2+}) with Cyanex®272 (HA) in kerosene is consistent with the following stoichiometry:

$$M^{2+}_{aq} + 2HA_{org} \leftrightarrow [MA_2]_{org} + 2H^{+}_{aq} \quad (3)$$

The equilibrium constant, K_{ex}, derived from Equation (3) gave the following:

$$K_{ex} = \frac{[MA_2][H^+]^2}{[M^{2+}][HA]^2} \quad (4)$$

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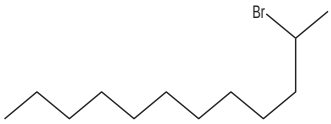
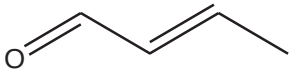
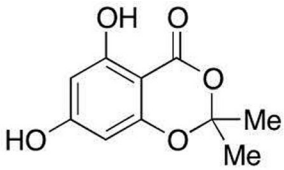
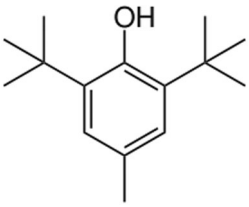
Table 2. Compounds present in the spent transformer oil.

Compound	RT (min)	Yield	Structure
Tridecane ($C_{13}H_{28}$)	5.606	6.30	
Heptdecane ($C_{17}H_{36}$)	6.751	10.24	
Nonadecane ($C_{19}H_{40}$)	6.820	3.17	
2-Bromododecane ($C_{12}H_{25}Br$)	7.239	9.14	
2-Buten-1-one ($C_{13}H_{20}O$)	8.383	4.78	
Tritetracontane ($C_{43}H_{88}$)	9.253	11.90	
Octadecane ($C_{18}H_{38}$)	9.491	4.48	
Tetradecane ($C_{14}H_{30}$)	7.977	13.38	
4H-1,3-Benzochoxin-4-one ($C_{10}H_{10}O_5$)	9.997	3.66	
Pentadecane ($C_{15}H_{32}$)	10.629	9.94	
Butylated hydroxytoluene ($C_{15}H_{24}O$)	11.111	23.01	
Total		100	

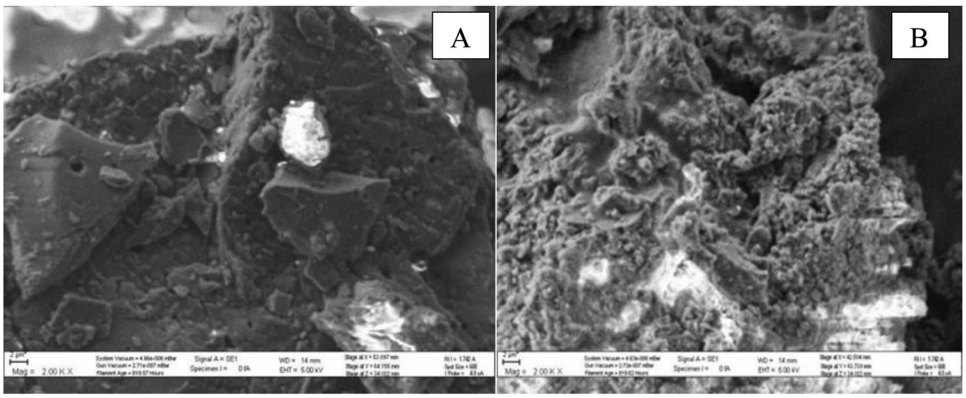


Figure 3. The SEM images of the raw sample before leaching (a) and after optimal leaching (b).

Table 3. The EDS data of the DSTO before and after leaching.

Elements (%)	O	C	Fe	Al	Ca	Cl	Si	S	Cu
Before leaching	50.47	31.50	4.24	1.10	0.53	–	0.73	0.64	10.79
After leaching	47.93	46.12	0.07	0.51	0.09	2.78	0.92	0.37	0.02
Efficiency of extraction	–	–	98.3	53.6	–	–	–	–	99.8

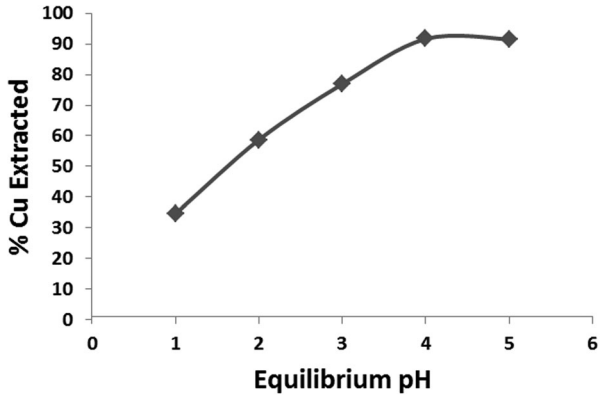


Figure 4. Effect of equilibrium pH on the per cent of Cu extracted by 0.2 mol L^{-1} Cyanex®272 in kerosene.

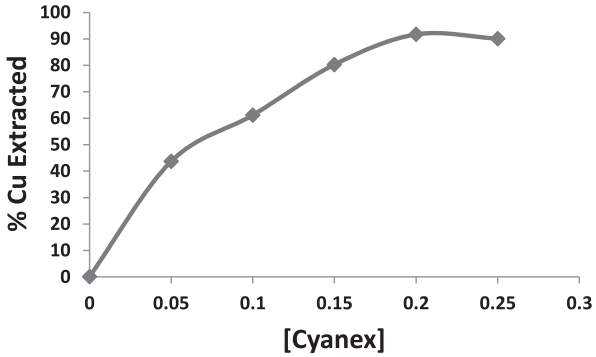


Figure 6. % Copper extracted vs. Cyanex concentration.

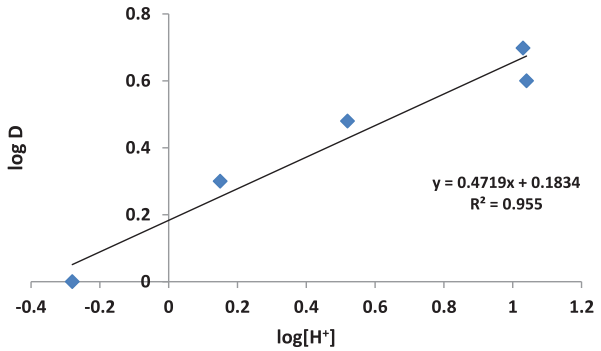


Figure 5. log D vs. log [H^+]. *Experimental conditions:* Same as Figure 3.

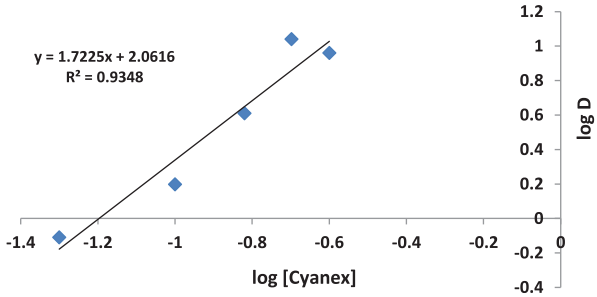


Figure 7. Plot of log D vs log [Cyanex] *Experimental conditions*. Same as in Figure 6.

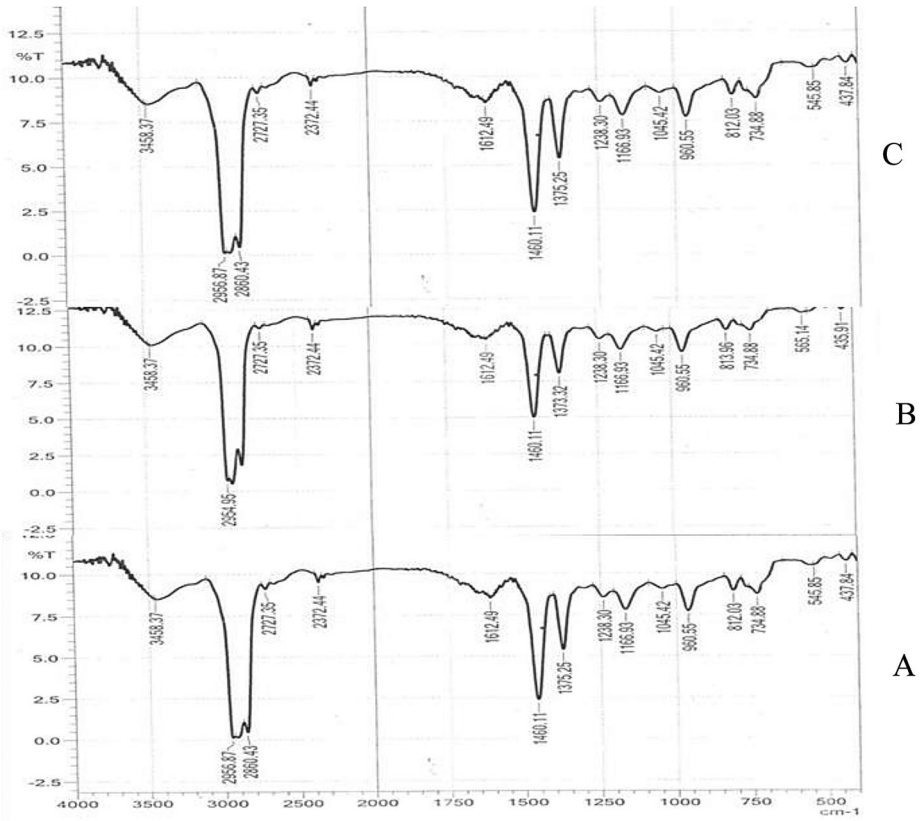


Figure 8. FT-IR spectral pattern of (a) the extractant (0.2 mol L^{-1} Cyanex in kerosene); (b) Copper-loaded organic phase at optimal condition; (c) organic phase after metal stripping by 0.1mol L^{-1} HCl solution.

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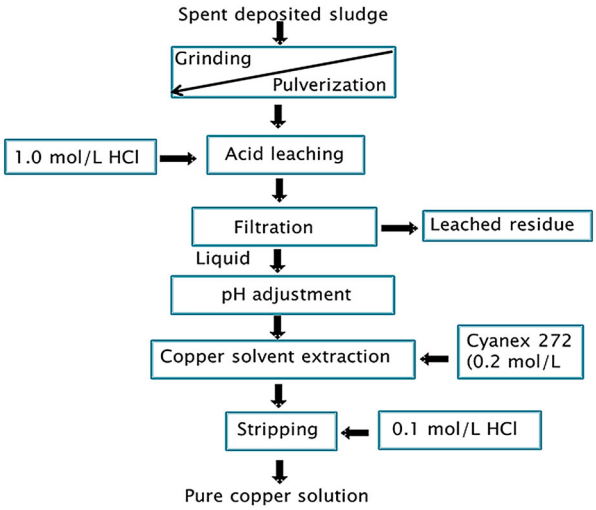

Figure 9. A proposed hydrometallurgical route for copper extraction from spent DSTO.

Rearrangement of Equation (4) implies:

$$\log D = \log k_{ex} + 2\log[HA] + 2\,pH \quad (5)$$

where distribution ratio, D is expressed as

$$D = \frac{[MA_2]}{[M^{2+}]} \quad (6)$$

Consequently, the product purity during Cu extraction by 0.2 mol L^{-1} Cyanex®272 in kerosene during 25 min contact at pH 4, where the extent of extraction process (91.77%) was examined by the Shimadzu H400F Fourier transform–infrared spectrometer (FT-IR) in the spectral range 400–4000 cm^{-1}. The bonding and surface properties of the loaded organic phase solutions and the compounds formed were obtained. The FT-IR spectrum presented in Figure 8(a–c) shows that the extraction mechanism occurs via metal–organic complexation. Comparing the Figure 8(b) before and after complexation with Cyanex, absence of the peak at 2860.43 cm^{-1} indicates metal extractant bonding, but exists in Figure 8(a,c). This showed that there is complete stripping of the copper ion, and the extractant regenerated. Also, the FT-IR spectra of the copper-loaded organic phase showed a peak at 435.91 cm^{-1}, indicating an interaction between the oxygen atom of the extractant and copper. The peak observed at 1375.25 cm^{-1} for the extractant was shifted to 1373.32 cm^{-1} in the copper-loaded cyanex extractant. This was assigned for the v(P=O) and the shift is attributed to the interaction of copper with the oxygen atom of the alkanol group in the extractant.

Copper recovery flow chart

A proposed hydrometallurgical scheme summarising the analytical procedures for the treatment of spent deposited sludge for the copper recovery is shown in Figure 9.

Conclusions

Conclusively, the results of this study provide the following valuable information:

(i) The characterisation results give a verdict in the variation of the transformer oil potency with respect to the ASTM D1819 regulations implies that the transformer oil is fit for re-refining.
(ii) The leachability of heavy metals especially, copper from spent deposited sludge of transformer oil by acid leaching and solvent extraction is possible. The mineralogical analysis of spent deposited sludge of transformer oil by EDXRF showed that the major chemical components of spent deposited sludge of transformer oil under investigation include: CuO, ZnO, Fe_2O_3 and SiO_2 with compounds such as Cr_2O_3, V_2O_5 and MgO as minor components. The EDXRF complements the results revealed by the XRD and the presence of other compounds such as SiO_2 (silicate) and other insoluble compounds are the residual products after optimal leaching.
(iii) At optimal leaching conditions, about 93% of initial 10 g L^{-1} DSTO sample was dissolved within 120 min. Extraction of copper by Cyanex®272 increased with increasing extractant concentration and equilibrium pH. A 0.1 mol L^{-1} HCl solution was found to be efficient for the stripping of about 94.77% from organic loaded spent deposited sludge which was complemented by the chemical bond examination obtained from the FT-IR analysis. The purified copper solution could further be processed to obtain copper metal of industrial value through electrowining process (Ochromowicz and Chmielewski 2013; Baba et al. 2015).
(iv) Beneficiation of the organic products from DSTO obtained in this study shall be reported in due course.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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