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# Hydrometallurgical preparation of high-grade industrial manganese compound from a low-grade spessartine ore by Cyanex®272 extractant

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## ABSTRACT

This study highlights a hydrometallurgical method for the purification, extraction, and beneficiation of a multi-elemental leach liquor obtained via reductive leaching of spessartine ore, for the production of high-grade manganese compound. During these treatments, the effect of Cyanex®272 concentrations, pH, aqueous: organic (A/O) was quantified. At optimal conditions (Temperature =  $27 \pm 2^\circ\text{C}$ , 0.2 mol/L Cyanex®272, pH = 4.5, A/O = 1:1), manganese extraction efficiency was >90%. The stripping of the loaded organic phase by 0.1 mol/L sulphuric acids yielded 95.7% in a single stripping stage. The resulting aqueous solution was appreciably beneficiated to produce manganese sulphate monohydrate ( $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ : 49–315–9500, melting point =  $702.8^\circ\text{C}$ ) via precipitation and crystallisation methods. Finally, a flow sheet summarising the analytical treatment steps was provided.

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Spessartine ore; manganese; manganese sulphate; solvent extraction; Cyanex®272 extractant

## 1. Introduction

Manganese, the 12<sup>th</sup> most abundant element is the third most surplus non-ferrous transition metal found on the earth crust. The rapid increase in manganese consumption is due to some special characteristics such as hardness, corrosive resistant and high oxidising properties displayed by the metal as compared to others. These among other accounts for its essential role-played in steel manufacturing as deoxidizer and in sulphur fixation applications [1–3]. Also, purified manganese compounds such as manganese sulphate ( $\text{MnSO}_4$ ) and manganese chloride ( $\text{MnCl}_2$ ) with a wide array of applications are important raw materials for defined chemical industries [4,5]. Owing to these essentialities, the day-by-day increasing demand for purified manganese coupled with the depletion of high-grade ores has resulted in the continuous efforts channelled towards the treatment of the low-grade manganese ore with greater complexity such as spessartine ore ( $\text{Mn}_3\text{Al}_2(\text{SiO}_4)_3$ ). Spessartine, a nesosilicate ore is an admixture with several economically important elements such as iron, aluminium, tungsten, tin, calcium, etc., which must be appropriately treated to obtain high grade-purified manganese metal and relevant industrial compound if beneficiated [6]. The separation of manganese from other gangues

may be effectively achieved by various methods including cementation, ion-exchange, gaseous hydrogen reduction, electrolytic precipitation, solvent extraction among others [7,8]. However, comparing the aforementioned processes relative to the simplicity of separations, solvent extraction method characterised with a well-known mechanism of reaction, mass transfer and high product purity is an excellent technique for obtaining pure metallic specie from multi-elemental aqueous solutions with the use of organophosphorus extractants such as di(2-ethylhexyl) phosphoric acid (DEHPA), bis(2,4,4-trimethylpentyl) phosphinic acid (Cyanex®272), 2-ethylhexyl phosphonic acid-mono- 2-ethylhexyl ester (PC88A), bis(2,4,4-trimethylpentyl) monothiophosphinic acid (Cyanex®302) and tributyl phosphate (TBP) among others [7–11].

Considering the ease of complex formation with a metal ion such as manganese ion ( $\text{Mn}^{2+}$ ), various researchers have adopted the solvent extraction technique in the purification and treatment of manganese ore using one or mixture of the aforementioned extractants. For example, a hydrometallurgical route based on solvent extraction technique was investigated for selective separation of manganese by Behzad and Fereshteh [12] from Electric Arc Furnace Dust (EAFD) of ferromanganese unit which contains tar, alkalis, manganese, zinc, iron, silica, calcium, aluminium and other elements. Separation and extraction of iron and manganese with Cyanex®272/302 to D2EHPA ratio increases as extractant concentration increases with a right shift in the extraction isotherm and highest separation was achieved at 15:5%v/v. Salgado *et al.* [13] has focussed on the recovery of zinc and manganese from the spent alkaline battery using Cyanex®272 extractant. Zinc and manganese were easily separated at pH = 2 with 20% Cyanex®272 dissolved in escaid 110 diluents at a reaction temperature of 50°C.

Separation of zinc and manganese from three spent alkalis batteries (NiCd, NiMH and Li-ion) was examined in four basic steps through sorting, purification, leaching and liquid-liquid extraction using Cyanex®272 extractant by Danuza *et al* [14]. The results from the finding shows that analysis of the metal separation was best at pH ranges 4.0–4.5 for manganese, cadmium, copper and cobalt with complex formation equilibrium co-existing as  $\text{MnR}_2$  in the organic phase. Devi and Mishra [1] also investigated solvent extraction equilibrium study on Mn(II) with Cyanex®302 in kerosene. The authors reported that extraction of Mn(II) increases as extractant concentration increases up to 0.05 mol/L Cyanex®302 using O/A = 1 at pH = 7. Also, Devi *et al.* [15] investigated the extraction and separation of Mn(II) and Co(II) from sulphate solutions using sodium salt of Cyanex®272, PC 88A and D2EHPA. With the three extractants, manganese was preferentially extracted over cobalt with Na-D2EHPA regarded as the most suitable extractant at equilibrium pH of 4.45.

Therefore, this study considers the hydrometallurgical treatment of sulphuric – oxalic acid leach spessartine ore for the recovery of manganese by alkaline precipitation and solvent extraction technique by Cyanex®272 extractant in kerosene diluent. Data on the quantitative reductive leaching of the ore in acidic media have been recently reported [16].

## 2. Materials and methods

### 2.1. Materials

The processed leach solution used for this study was obtained by dissolving 10 g/L spessartine ore in 1.5 mol/L sulphuric and 1.0 mol/L oxalic acid solution at 75°C for

120 min. Under these conditions, the leach liquor was spectrophotometrically analysed and found to contain 2854.7 mg/L  $\text{Mn}^{2+}$ , 1523.2 mg/L  $\text{Fe}^{3+}$ , 205.3 mg/L  $\text{Al}^{3+}$ , 43.1 mg/L  $\text{Ca}^{2+}$  and 28.1 mg/L  $\text{Sn}^{2+}$  respectively. The sodium hydroxide and sulphuric acid (97.8%, Sigma Aldrich, Germany) used in the purification and stripping processes, bis(2,4,4-trimethylpentyl) phosphinic acid (Cyanex®272) (97%, Cytec Industries Incorporated, USA) used as the extractant for the solvent extraction process were of analytical reagent grades. The kerosene was obtained from NNPC Mega Station, Tanke Road, Kwara State, Nigeria.

## 2.2. Method

### 2.2.1. Manganese solvent extraction

Series of separation tests were carried out to determine the optimum condition for manganese extraction at room temperature ( $27 \pm 2^\circ\text{C}$ ). A 25 mL of the leach liquor and the extractant in kerosene diluent was taken into a stoppered bottle and agitated by Gallenkamp orbital shaker at a constant rate for 30 min. Thereafter, the mixture was carefully transferred into a 150 mL separating flask to allow for phase separation. After equilibrium is reached, the phases were allowed to settle and accordingly separated. The manganese, calcium and iron contents in the aqueous phase were determined directly after suitable dilutions using Buck Scientific ACCUYS 211 Atomic Absorption Spectrophotometer (AAS), while total aluminium concentration was determined with JENWAY 7300 spectrophotometer at 584 nm using ECR (Erichrome Cyanine R) as colour developing agent [17–19]. The concentrations in the organic phase were determined by mass balance and the distribution coefficient (D) calculated as the ratio of the concentration of  $\text{Mn}^{2+}$  in the organic phase to its concentration in the aqueous phase at equilibrium as summarised in Equation (1). From the D values, the percentage of manganese extracted is calculated according to Equation (2):

$$D = [\text{Mn}]_{\text{org}}^{2+} / [\text{Mn}]_{\text{aq}}^{2+} \quad (1)$$

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

Prior to solvent extraction of manganese by Cyanex®272 extractant, the iron species constituting major impurities in the leach liquor was appreciably treated. The effects of extractant concentration and solution pH on the extent of manganese extraction were examined for a better understanding of the extraction mechanisms. Iron and other dominating impurities, such as aluminium ( $\text{Al}^{3+}$ ), calcium ( $\text{Ca}^{2+}$ ) and tin ( $\text{Sn}^{2+}$ ) constituting major impurities was precipitated at pH ranges 3.0–4.3, respectively. Finally, to bring the purified extracted manganese from the loaded organic phase to the aqueous phase, the enhancement of the extracted manganese was done through stripping with 0.1 mol/L  $\text{H}_2\text{SO}_4$  solution [20,21]. The purified aqueous solution containing pure manganese was further crystallised to obtain high manganese compound comparable with industrial standards [3].

## 2.3. Solvent extraction studies

### 2.3.1. Impurity removal

Purification of the leach liquor was conducted to separate major impurities before the subsequent extraction of total manganese. Major impurities present as Fe, Al and Ca were

removed by alkaline precipitation technique through dropwise addition of freshly prepared 3 mol/L NaOH. However, with the addition of NaOH, iron in the leach liquor formed a brownish precipitate at a pH of 3.5 to form the corresponding insoluble iron hydroxide. For total aluminium treatment, the filtered liquor free of iron was further treated through pH adjustments until the colourless solution formed white precipitate at a pH of 7.3 and beneficiated [21]. Thereafter, traces of impurities including  $\text{Ca}^{2+}$  present were precipitated as  $\text{Ca}(\text{OH})_2$ . After each precipitation, the slurry was stirred for a period of 10 min and filtered. The composition of the treated purified solution used for the solvent extraction study is summarised in Table 1:

### 2.3.2. Effect of Cyanex®272 concentration

Manganese extraction efficiency was carried out varying Cyanex®272 extractant concentrations in the range of 0.01–0.25 mol/L as depicted in Figure 1. The obtained plot indicated that manganese extraction steadily increases as extractant concentration increases from 0.05 mol/L up to 0.2 mol/L, but decline slightly as concentration increases to 0.25 mol/L. At 0.2 mol/L Cyanex®272, 60.02% extraction efficiency was achieved, then declined to 43.2% as concentration increases beyond 0.2 mol/L. The extraction decrease could be as a result that at high concentration, there is a possible decrease in the fluidity of the organic phase, thereby reducing the mass transfer rate [12,22]. Hence, 0.2 mol/L Cyanex®272 was used for further optimisation studies. In this regard, the plot of log D (distribution ratio) against log [Cyanex®272] was evaluated to determine the extracting species responsible for manganese ion extraction as depicted in Figure 2. From the plot of Figure 2, the obtained slope of 0.752, indicated that a mole of extractant is exchanged with the manganese extracted specie.

**Table 1.** Results of the precipitation operation after impurity removal from the leach liquor.

| Metal ions           | Fe   | Mn     | Al   | Ca   | Sn   |
|----------------------|------|--------|------|------|------|
| Concentration (mg/L) | 0.03 | 2784.1 | 0.05 | 0.02 | 0.05 |

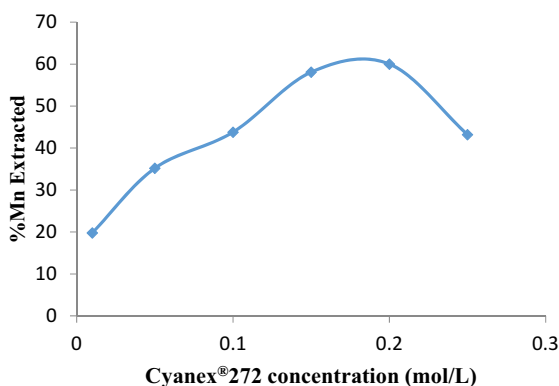


**Figure 1.** Effect of Cyanex®272 concentration on manganese extraction. *Experimental conditions:* [Cyanex®272] = 0.01–0.25 mol/L, initial manganese concentration = 2784.1 mg/L, pH = 4.0, phase ratio = 1:1, contact time = 25 min, equilibration time = 4 min.

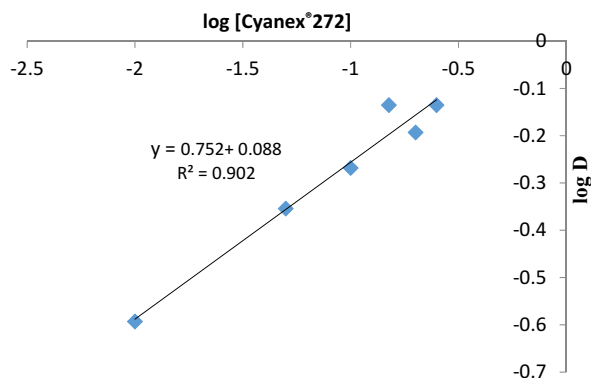


**Figure 2.** Effect of Cyanex®272 concentration on distribution ratio. *Experimental conditions:* Same as in Figure 1

### 2.3.3. Effect of pH

At an experimental set condition of 0.2 mol/L Cyanex®272, aqueous: organic (A/O) 1:1, the effect of equilibrium pH on the extraction of manganese was carried out within the pH ranges of 1.5–7.5 at room temperature ( $27 \pm 2^\circ\text{C}$ ), as summarised in Figure 3.

*Experimental Conditions:* Initial manganese concentration = 2784.1 mg/L, A/O = 1:1 pH = 1.5–7.5, contact time = 25 min, equilibration time = 4 min, Cyanex®272 concentration = 0.20 mol/L

The experimental curve of %manganese extracted versus equilibrium pH represented in Figure 3 reflected that the extraction of manganese increases with increasing pH from 1.5 to 4.5, yielding extraction efficiencies of 72.3%, 82.0%, 87.0% and 90.5% for the pH's 1.5, 2.5, 3.5 and 4.5, respectively. At pH 5.5 for example, the extraction efficiency decreases to 62% without significant changes beyond the pH of 5.5. The decrease in manganese extraction may be attributed to the notable formation of precipitates of manganese as manganese hydroxide. Thus, the distribution log D versus equilibrium pH (Figure 4) gave a linear plot with a slope of 1.043, indicating that a mole of extractant was used up during the manganese extraction process.

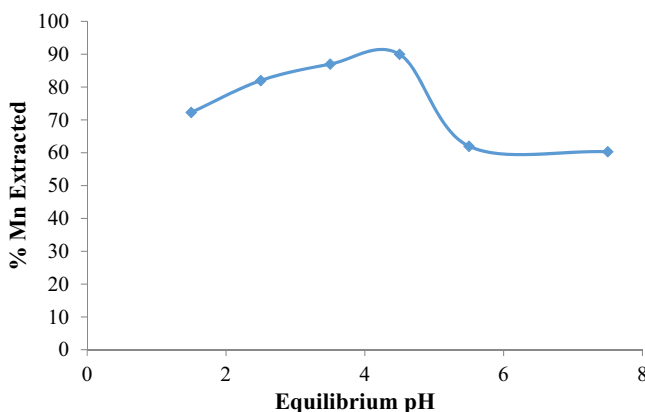


**Figure 3.** Effect of equilibrium pH on extraction of manganese using Cyanex®272 in kerosene.

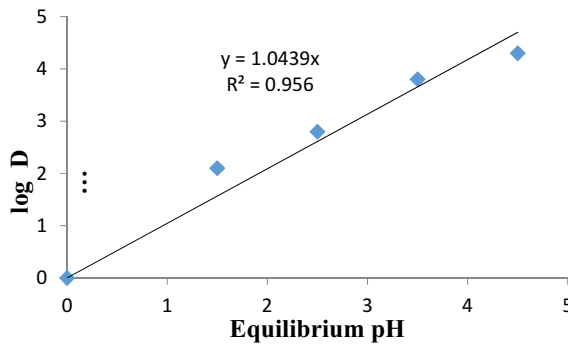
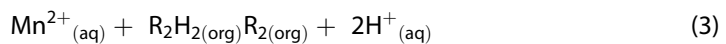


**Figure 4.** The plot of log D (distribution ratio) against equilibrium pH.

### 3. Discussion

The extraction of manganese by Cyanex®272 may appear to exist in the dimeric mixture of the form,  $R_2H_2$  and consistent with the following stoichiometry:



$R_2H_2$  represents the dimeric organo-phosphoric acid extractants (Cyanex®272),  $MnR_2$  is the probable complex in the organic phase, while (aq) and (org) denotes the aqueous and organic phases, respectively. The equilibrium constant,  $K_{ex}$  for the extraction process is:

$$K_{ex} = \frac{[MnR_2][H^+]^2}{[Mn^{2+}][RH]^2} \quad (4)$$

From Equation (4), manganese distribution ratio  $D_{Mn}$  becomes:

$$D_{mn} = \frac{[MnR_2]}{[Mn]^{2+}} \quad (5)$$

Substituting the distribution ratio (Equation 5) into  $K_{ex}$ , Equation (4) gives:

$$K_{ex} = \frac{D_{Mn}[H^+]^2}{[RH]^2} \quad (6)$$

Finally, the extraction of pure manganese by Cyanex®272 extractant in this study can be represented as:

$$\log D_{Mn} = \log K_{ex} - 2 \log [H^+] + \log [RH]_2 \quad (7)$$

#### 3.1. Stripping, purity and beneficiation studies

The organic phase at optimal extraction condition was stripped using 0.1 mol/L  $H_2SO_4$  to bring metal value to the aqueous state for purity analysis. The result of the stripping process yielded 95.7% manganese recovery at a single stage within the mixing time of 5 min and aqueous: organic (A/O) 1:1. The extractant, Mn-loaded organic and the stripped FT-IR analysis are depicted as Figure 5(a-c) respectively. The manganese solution was

further benefited by crystallisation to obtain a pure manganese sulphate monohydrate compound.

### 3.2. Extraction purity analysis by FT-IR

The analysis of the chemical bonding of the extractant, manganese organic loaded and pure stripped solutions, which enables identification of compounds present was studied using Fourier transform infrared spectroscopy (FT-IR) as summarised in Figure 5(a-c). The Cyanex®272 extractant showed a broad hydroxyl band around  $3471.1\text{ cm}^{-1}$  and a moderate intensity band around  $1423.2\text{ cm}^{-1}$  and  $1361.26\text{ cm}^{-1}$  indicating P-C and P-O, respectively, this

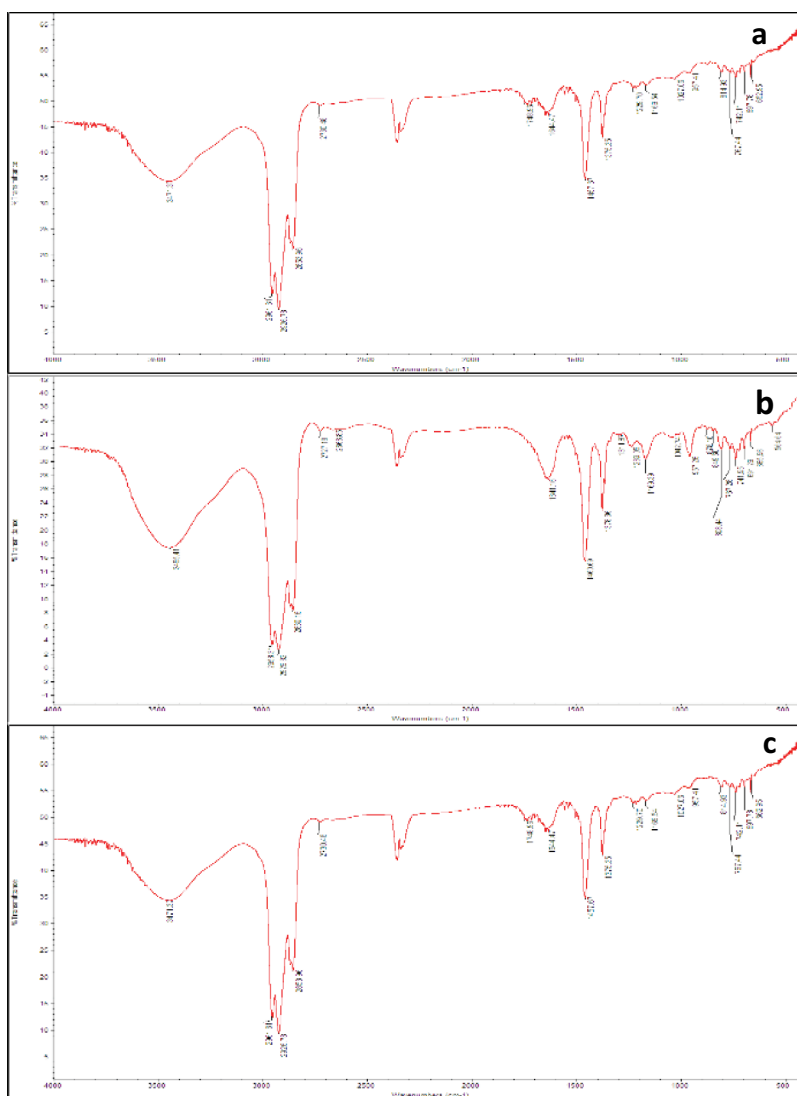


**Figure 5.** FT-IR of (a) the extractant (0.2 mol/L Cyanex®272); (b) The Manganese loaded spectrum in Cyanex®272; (c) The FT-IR spectrum after stripping with 0.1 mol/L H<sub>2</sub>SO<sub>4</sub> in Cyanex®272.

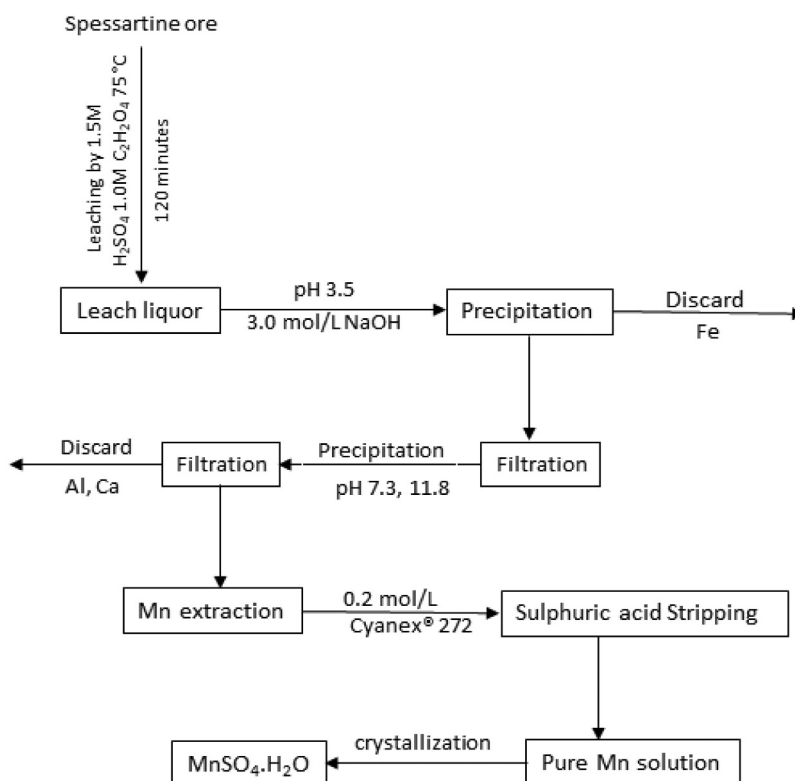


**Figure 6.** A hydrometallurgical process flow-chart for purification and recovery of manganese compound from leach liquor generated by reductive leaching of spessartine ore.

suggests the extractant to be organo-phosphorus [23–25]. From Figure 5 (i.e., manganese loaded with Cyanex®272) distinctive vibration absorption band in Figure 5(b) at 1451 cm<sup>-1</sup>, 1150 cm<sup>-1</sup>, and 957 cm<sup>-1</sup> indicate strong complexation of manganese to the P-O and P-C absorption, respectively, while the stripped loaded Cyanex®272 as represented in Figure 5(c) is similar to that of the extractant (Cyanex®272) indicating successful manganese stripping from the manganese organic loaded solution, thereby justifying the effectiveness of Cyanex®272 extractant with 0.1 mol/L H<sub>2</sub>SO<sub>4</sub> stripping for the extraction process. Finally, an hydrometallurgical flow-chart summarizing the major process of purification and recovery of manganese compound from leach liquor generated through the reductive leaching of spessartine ore is represented as Figure 6.

#### 4. Conclusions

This study affirmed the feasibility of Kaoje (Nigeria) spessartine ore treatment through a hydrometallurgical process involving purification, liquid-liquid extraction and beneficiation for the recovery of industrial manganese sulphate monohydrate (MnSO<sub>4</sub>·H<sub>2</sub>O: 49–315–9500, melting point = 702.8°C). Experimental parameters varied at room temperature (27 ± 2°C) includes extractant concentration and pH. At optimal conditions, 2519.61 mg/L was quantitatively extracted at pH of 4.5, 0.2 mol/L Cyanex®272, yielding 90.5% extraction

efficiency. A 0.1 mol/L  $\text{H}_2\text{SO}_4$  was used for the stripping of 95.7% manganese from the loaded organic phase and the stripped solution was further beneficiated via crystallisation. A hydrometallurgical flowsheet summarising the basic analytical steps for obtaining high-grade industrial manganese compound was provided. The data obtained in this study is envisaged for the country's economic diversification in substitute of the current hard-earned foreign exchange through petroleum exploration devalued by the Covid-19 pandemic.

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## Disclosure statement

No potential conflict of interest was reported by the author(s).

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