# SOLVENT EXTRACTION OF ZINC FROM BURNT TYRE ASH USING TRI BUTYL PHOSPHATE


## Abstract

**The article reports the solvent extraction of zinc (Zn) from burnt tyre ash using tributylphosphate (TBP) as an extractant. The increasing interest in environmental protection and resource recovery has prompted research into extracting valuable metals from waste materials, such as Zn from tyre ash, a significant secondary source. The optimal conditions for zinc leaching and extraction were determined through a series of hydrometallurgical processes. Zn was successfully extracted under specific HCl concentration, temperature, and pH conditions, with TBP showing effective performance as an extractant.** We found that increasing the initial concentration of TBP in kerosene improves the extraction efficiency of Zn from burnt tyre ash. The highest extraction rate achieved was 48.68% using 0.30 mol/L TBP at pH 4.5. The relationship between log D and log [TBP] was done to determine how many moles of TBP were involved in the extraction system. A linear plot with a slope of ~ 1.00 was obtained and indicated the liberation of one mole of [H+] during the extraction of one mole of $Zn^{2+}$ from the burnt tyre ash. **The study highlights the potential for resource recovery from waste materials, contributing to both environmental sustainability and the supply of industrial metals.**



*Keywords*: ***Zn, Burnt tyre ash, leashing, Tributylphosphate (TBP), extraction.***

## 1.0 INTRODUCTION

With the growing interest in environmental protection in recent years, among the concerns under discussion are emissions of hazardous chemical species during waste incineration, spontaneous combustion of the waste in storage, and secondary pollution by heavy metals highly concentrated in the combustion residue.

Automobile tyres consist of rubber, carbon black, and various organic and inorganic additives (including plasticizers, anti-aging agents, sulfur and ZnO, etc) (Niezgoda et al., 2020; Malinova, 2022). Heavy metals are concentrated in combustion ashes during the incineration process, and the residues are disposed of as industrial waste under strict regulation. However, whole tyre wastes annually generated an estimated value of 7000 tonnes for Zn, drawing attention to the recovery of valuable metals from waste scrap and dumpsite ashes (Xu et al., 2020).

Zn is one of the minerals that has been neglected previously but is recently gaining importance as a base metal for various applications in the metallurgical and chemical industry (Matinde et al., 2018); it is widely extracted from its primary ore (sphalerite) and can also be recovered from secondary sources (Šajn et al., 2022; Akitoye et al., 2024), such as Zn ash, Zn dross, flu dust of electric arc and automobile shredder scrap. The chemical nature of the secondary source is such that they are classified as hazardous waste, and their toxicity is mainly due to the presence of different metals such as lead, cadmium, cobalt, arsenic, and chromium (Khatun et al., 2022). Therefore, with the depletion of the primary sources of metal, researchers have begun to search for alternatives, which has resulted in metals like Zn now being extracted from secondary sources like galvanizing plant dross and ash, spent primary batteries, Zn scrap, leach residue flue dust generated from iron making and steel making plant however spent galvanizing (Khatun et al., 2022).

Much work has been reported in the literature regarding the use of extractants of diverse nature and in various media, such as dithizone and cyclohexanones, which have limited solubility in organic solvents and so are not recommended when Zn concentration is greater than $10^{-3}$ mol/L. Carboxylic acids, amines, organophosphorus compounds, and chelex-100 have been cited as good candidates for the extraction of Zn (Khatun et al., 2022).

TBP (Tributylphosphate) has been cited to be a good candidate for the extraction of Zn (II) in acid media (Sathityatiwat, 2019). There is practically little or no information on the ability of triphenylphosphine (TPP) to extract Pb or Zn, except a report on its use for the extraction of palladium (Krishnan et al., 2021).

Zn ash is an important secondary source of Zn recovery as it constitutes a source from which a high amount of Zn can be recovered. Pyrometallurgical and hydrometallurgical processes are employed in the treatment of secondary sources, and they are more environmentally friendly and cost-effective in treating Zn-containing materials through leaching and selective precipitation by solvent extraction (Loy et al., 2018).

The hydrometallurgical process usually involves the dissolution of the secondary sources (sometimes after ashing) in an acidic media, sometimes with prior water treatment, and recovering the Zn from the solution. Ammonium carbonate has been used for Zn recovery from steel dust, automobile scrap, and incinerator fly ash (Kaya et al., 2020) in the latter case, with a 400 C roasting step included. Combination treatments have also been used, such as sulphuric acid and chloride leaching with electro-galvanizing dust, waste acid and alkali for fly ash, sulphuric acid and sodium bisulphide/carbonate for flue dust, and a combination of acid/oxygen/ H20 & S20, with blast furnace dust. Sulphuric acid leaching has been used for steel dust, and it is the most commonly used approach for the solubilization of solid Zn waste solids (Binnemans et al., 2020). About 30-40% of Zn consumed comes from reclaimed Zn sources (Kania *et al.*, 2023; Kaya et al., 2020). Pyrometallurgical processes generally involve heating at temperature (usually in the furnace), the secondary sources of Zn to remove the non-metallic content of the sample, and then leaching the Zn-rich ash in suitable acid. It is employed in the processing of most metals and finds application in the extraction of base metals like Zn, tin, etc. Researchers have used extractants such as TBP to exploit the recovery of Zn from spent batteries (Tanong et al., 2017).

### 1.1 Extractant

Tributylphosphate is an organophosphorus compound with the chemical formula (CH3CH2CH2CH2O)3PO. It is a colorless and odorless liquid, and its application is found in its use as an extractant and a plasticizer (Jangid *et al.*, 2023). It is an ester of phosphonic acid with n-butanol. It is used as a solvent for extracting and purifying rare earth metal from the ores. An extractant aims to provide high selectivity and efficiency for metal chelation. It is also referred to as a solvating extractant.

### 1.2 Leaching

Leaching involves the removal of contaminants (organic or inorganic) from a solid state into a liquid phase when a substance is subjected to different processes, such as mineral dissolution, complexation, and desorption (Isak *et al.*, 2023). Leaching is one of the important processes involved in the field of metallurgy (Šajn *et al.*, 2022).

## 2.0 Significance of the Study

*Zn* is an important industrial metal used in various applications, including galvanizing steel, manufacturing brass and other alloys, and producing batteries, paints, and fertilizers widely used in industries such as construction, automotive, electrical, and electronics (Jones et al., 2014; Mielcarz-Skalska & Smolińska, 2017; Sabnavis et al., 2018).

Zn has also been used as an anode in many historical galvanic elements. These include, among others, the Voltasche column, the Daniell element, and the Bunsen element. To a lesser extent, Zn is also used for negative electrodes in accumulators (rechargeable batteries); Zn is mixed with small amounts of other metals. These include lead, bismuth, indium, aluminum, and calcium. Zn die casting is the common name for die-cast parts made of refined Zn casting alloys. These alloys provide far better casting values than are possible when casting pure Zn. Zn was used in times of need, most recently in the two world wars, in the form of zinc alloys for coinage. The global Zn market is influenced by supply and demand dynamics, global economic conditions, technological advancements, and factors (Sverdrup et al., 2019), hence the drive for the demand for zinc ores and products.

## 3.0 METHODS

### 3.1 Sample Collection

Burnt tyre ashes (1 kg) were collected from a waste dumpsite in the Dogarawa area of Zaria, Kaduna state, Nigeria, in November 2022. Tyres were burned after a regulatory agency confiscated them.

### 3.2 Methods

Ash (2.5 g) was dissolved in aqua regia (250 ml) at an ambient temperature of 25 °C for less than 2 hours (1:3 of HCl: HNO3), and the total concentration of zinc in the ash was measured in the resulting solution by atomic absorption Spectrophotmetry after filtration and dilution.

### 3.3 Determination of Total Zinc Content in the Dumpsite Ash and Optimum Leaching Condition for Zn

The optimum leaching conditions of HCl concentration, temperature, leaching time, and agitation speed were determined and are 2.968 mol/L of HCl, 68.85, 97 minutes, at an agitation rate of 400 rpm. The amount of Zn leached was 32009.205 mg/L.

### 3.4 Leaching of Zn from the Dumpsite Ash

2.5 g of burnt tyre ash was weighed and introduced into a 250 cm3 prepared solution of HCl in a 500 cm3 beaker and was introduced on a heating magnetic stirrer at 68.85 °C. A magnetic stirrer bar was inserted, and the solution was covered with a glass lid. The burnt tyre was left to be in contact with the lixiviant for 97:703 minutes, at the end of the contact time, the mixture obtained was then filtered and the insoluble residue left on a filter paper was washed thoroughly with a little of the hot filtrate. The filtrate was then carefully transferred into a 250 ml standard volumetric flask. The solution was made to mark with distilled water; the filtrate in the standard volumetric flask was then adjusted to pH 3.5 with a pH meter before analysis, using Atomic Absorption Spectroscopy (AAS) as presented in plate 1.

*Plate.1 : Preparation of leach liquor*

### 3.5 Preparation of TBP solution

Tributylphosphate $(CH_3CH_2CH_2CH_2O)_3PO$ has an essay % Purity = 99%, Specific gravity = 0.98 g/cm$^3$, Molar mass = 266 g/moL

Calculation of molarity of the stock TBP

$$M = \frac{\%P \times Spg \times 10}{Mm}$$

$$M = \frac{99 \times 0.98 \times 10}{266}$$

M = 3.65 M (concentration of the stock TBP)

Using dilution formula, volume of TBP needed to prepare a standard solution can be calculated;

$C_1V_1=C_2V_2$

To prepare 0.30M TBP

$$V_1 = \frac{0.30 \times 50}{3.65}$$

$V_1$ = 4.10 cm$^3$

About 4.10 cm$^3$ of TBP was measured and slowly added to 25 cm$^3$ kerosene in a 50 cm$^3$ volumetric flask and made up to the 50 cm$^3$ volume with kerosene.

The same process was carried out for the preparation of other concentration of TBP which were 0.05 M, 0.10 M, 0.15 M, 0.20 M, 0.25 M and 0.30 M.

### 3.6 Procedure for the solvent extraction of Zn from leach liquor

The experiment was performed using the leachate obtained by the optimal conditions of the temperature leaching, concentration, and time of Zn in the leached liquor in section 3.4. AAS carried out analysis at room temperature 25±2 °C. Equal volumes of 10 cm3 of the predetermined concentration of the extractant (TBP), which were 0.05 M to 0.30 M TBP with 10 cm3 of leachate from section 3.4 were mixed in a 250 cm3 conical flask and shaken using a mechanical shaker for 25 minutes at room temperature. The pH of the pregnant leach solution in each case was ensured to be pH 4.5. Plate 2 presents the preparation of the aqueous and organic phases.

*PLATE 2 : Preparation of aqueous phase and organic phase*

After the equilibration phase disengagement was carried out for each experimental group in 250 $cm^3$ separating funnel, it was allowed to form two separation layers. The upper one contained Zn loaded organic phase while the lower part had a clear solution, after the formation of two distinct layers the clear solution (aqueous phase) was drained out by opening the tap. The concentration of Zn in each experimental group of TBP concentration was calculated using AAS analysis, to determine the Zn composition left in the aqueous phase.

*Plate 3: Separating funnel setup*

The concentration of the metal in an organic phase was calculated from the differences between its concentration in the aqueous phase before and after the extraction (Yamada *et al.*, 2023). Then % Zn extracted against TBP concentration was plotted.

**3.7 Effect of pH on the Solvent Extraction of Zn**

The percentage of extracted Zn against concentration of TBP that gave the highest percentage of co-extracted was used for optimization of other parameter which included equilibrium pH of the aqueous media (Mohammadzadeh *et al.*, 2022). The pH of the leach liquor in section 3.4 was adjusted to a pH 2.5, 4.5, 5.5 and the % of zinc extracted using solvent extraction, was repeated for each case as in section 3.6. The optimum pH was then determined. A plot of % zinc extracted against pH was plotted.

$$\% E = \frac{[M]org}{[M]aqu.initial}$$

$$D = \frac{[M]org}{[M]aqu}$$

Equation of chemical reaction

$$Zn^{2+}_{(aq)} + HCl + 2TBP_{(o)} \longrightarrow Zn(Cl)_2\ TBP)_2 + H^+$$

### 3.8 Stripping of zinc

Zn was stripped from the loaded organic phase by contacting the experimental organic phase with a 0.05 mol/L HCl solution and shaking for 25 min thereafter; the mixture was allowed to reach equilibrium and then separated with a separating funnel. The aqueous phase contains the metals (Zn), and the organic phase will be TBP. The Zn ion concentration in the stripping aqueous was spectrophotometrically measured using AAS.

## 4.0 RESULT AND DISCUSSION

*Table .1: Effect of TBP concentration on the amount of Zn unextracted from the leached liquor*

| [TBP] /pH | [Zn] (ppm) | SD | Mean Abs |
|---|---|---|---|
| [0.05 M] at pH 2.5 | 2.2353 | 0.0033 | 0.3635 |
| [0.10 M] at pH 2.5 | 1.7878 | 0.0036 | 0.2907 |
| [0.15 M] at pH 2.5 | 1.7074 | 0.0053 | 0.2777 |
| [0.20 M] at pH 2.5 | 1.6981 | 0.0019 | 0.2761 |
| [0.25 M] at pH 2.5 | 1.6511 | 0.0028 | 0.2685 |
| [0.30 M] at pH 2.5 | 1.6107 | 0.0027 | 0.2619 |
| [0.05 M] at pH 3.5 | 2.5260 | 0.0105 | 0.4108 |
| [0.10 M] at pH 3.5 | 2.4873 | 0.0024 | 0.4045 |
| [0.15 M] at pH 3.5 | 2.4571 | 0.0045 | 0.3996 |
| [0.20 M] at pH 3.5 | 2.4092 | 0.0063 | 0.3918 |
| [0.25 M] at pH 3.5 | 2.3516 | 0.0060 | 0.3824 |
| [0.30 M] at pH 3.5 | 2.2809 | 0.0085 | 0.3709 |
| [0.05 M] at pH 4.5 | 1.7656 | 0.0061 | 0.2871 |
| [0.10 M] at pH 4.5 | 1.6340 | 0.0059 | 0.2657 |
| [0.15 M] at pH 4.5 | 1.6292 | 0.0043 | 0.2650 |
| [0.20 M] at pH 4.5 | 1.5012 | 0.0036 | 0.2441 |
| [0.25 M] at pH 4.5 | 1.4420 | 0.0055 | 0.2345 |
| [0.30 M] at pH 4.5 | 1.3042 | 0.0055 | 0.2121 |
| [0.05 M] at pH 5.5 | 1.9604 | 0.0092 | 0.3188 |
| [0.10 M] at pH 5.5 | 1.8855 | 0.0109 | 0.3066 |
| [0.15 M] at pH 5.5 | 1.5793 | 0.0014 | 0.2568 |
| [0.20 M] at pH 5.5 | 1.4508 | 0.0012 | 0.2359 |
| [0.25 M] at pH 5.5 | 1.4348 | 0.0002 | 0.2333 |
| [0.30 M] at pH 5.5 | 1.4247 | 0.0027 | 0.2317 |

**CALCULATION**

Formula for leaching

Actual concentration = Instrument reading x Dilution factor

For aqueous initial

Instrument reading = 2.5477 ppm,

Dilution factor =100

Actual concentration – 254.77 mg/L or 254.77 ppm

The initial concentration of Zn in the leached gotten was 254.77 mg/L

***Table 2 Effect of concentration of TBP against % Zn Extracted into organic phase at pH 2.5***

| TBP Concentration | Instrument reading (ppm) | Actual Conc.(mg/L) | [M] org | % Extraction | Distribution ratio (D) |
|---|---|---|---|---|---|
| 0.05 M | 2.2353 | 223.53 | 31.24 | 12.26% | 0.139 |
| 0.10 M | 1.7876 | 178.76 | 76.01 | 29.83% | 0.423 |
| 0.15 M | 1.7074 | 170.74 | 84.03 | 32.98% | 0.492 |
| 0.20 M | 1.6981 | 169.81 | 84.96 | 33.35% | 0.500 |
| 0.25 M | 1.6511 | 165.11 | 89.66 | 35.19% | 0.543 |
| 0.30 M | 1.6107 | 161.07 | 93.70 | 36.79% | 0.582 |

*Table 3: Effect of concentration of TBP against % Zn Extracted into organic phase at pH 3.5*

| TBP concentration | Instrument reading (ppm) | Actual conc. (mg/L) | [M] org | % Extraction | Distribution ratio |
|---|---|---|---|---|---|
| 0.05 M | 2.526 | 252.6 | 2.17 | 0.88% | 0.089 |
| 0.10 M | 2.4873 | 248.73 | 6.04 | 2.37% | 0.024 |
| 0.15 M | 2.4571 | 245.71 | 9.06 | 3.55% | 0.037 |
| 0.20 M | 2.4092 | 240.92 | 13.85 | 5.20% | 0.057 |
| 0.25 M | 2.3516 | 235.16 | 19.61 | 7.69% | 0.083 |
| 0.30 M | 2.2809 | 228.09 | 26.68 | 10.47% | 0.117 |

*Table 4: Percentage of Zn extracted into the organic phase with TBP concentration at pH 4.5*

| Concentration TBP | Instrument reading (ppm) | Actual conc. (mg/L) | [M]org | % Extraction | Distribution ratio (D) |
|---|---|---|---|---|---|
| 0.05 M | 1.7656 | 176.56 | 78.21 | 30.69% | 0.443 |
| 0.10 M | 1.634 | 163.4 | 91.37 | 35.86% | 0.559 |
| 0.15 M | 1.6292 | 162.92 | 91.85 | 36.05% | 0.564 |
| 0.20 M | 1.5012 | 150.12 | 104.65 | 41.08% | 0.697 |
| 0.25 M | 1.4421 | 144.21 | 110.57 | 43.39% | 0.767 |
| 0.30 M | 1.3042 | 130.42 | 124.03 | 48.68% | 0.947 |

*Table .5: Effect of concentration of TBP against % Zn extracted into organic phase at pH 5.5*

| TBP concentration | Instrumental reading(ppm) | Actual concentration | [M]org | % Extraction | Distribution ratio |
|---|---|---|---|---|---|
| 0.05 M | 1.9604 | 196.04 | 58.72 | 23.05% | 0.299 |
| 0.10 M | 1.8855 | 188.55 | 66.22 | 25.99% | 0.351 |
| 0.15 M | 1.5793 | 157.93 | 96.84 | 38.01% | 0.613 |
| 0.20 M | 1.4508 | 145.08 | 109.65 | 43.04% | 0.756 |
| 0.25 M | 1.4348 | 143.48 | 111.29 | 43.68% | 0.776 |
| 0.30 M | 1.4247 | 142.47 | 112.3 | 44.08% | 0.762 |

*Table .6: Summary of the effect of concentration of TBP against pH of the extraction of Zn*

| Concentration of TBP | pH 2.5 | pH 3.5 | pH 4.5 | pH 5.5 |
|---|---|---|---|---|
| 0.05 M | 0.139 | 0.024 | 0.443 | 0.299 |
| 0.10 M | 0.423 | 0.037 | 0.559 | 0.351 |
| 0.15 M | 0.492 | 0.057 | 0.564 | 0.613 |
| 0.20 M | 0.500 | 0.087 | 0.694 | 0.756 |
| 0.25 M | 0.543 | 0.089 | 0.767 | 0.762 |
| 0.30 M | 0.582 | 0.117 | 0.942 | 0.776 |

*Table 7: Effect of concentration of TBP against Distribution ratio*

| Concentration of TBP | log x | Distribution ratio | log D |
|---|---|---|---|
| 0.05 M | -1.30103 | 0.206 | -0.68613 |
| 0.10 M | -1 | 0.343 | -0.46471 |
| 0.15 M | -0.82391 | 0.436 | -0.36051 |
| 0.20 M | -0.69897 | 0.508 | -0.29414 |
| 0.25 M | -0.60206 | 0.539 | -0.26841 |
| 0.30 M | -0.52288 | 0.604 | -0.21896 |

Where x = [TBP]

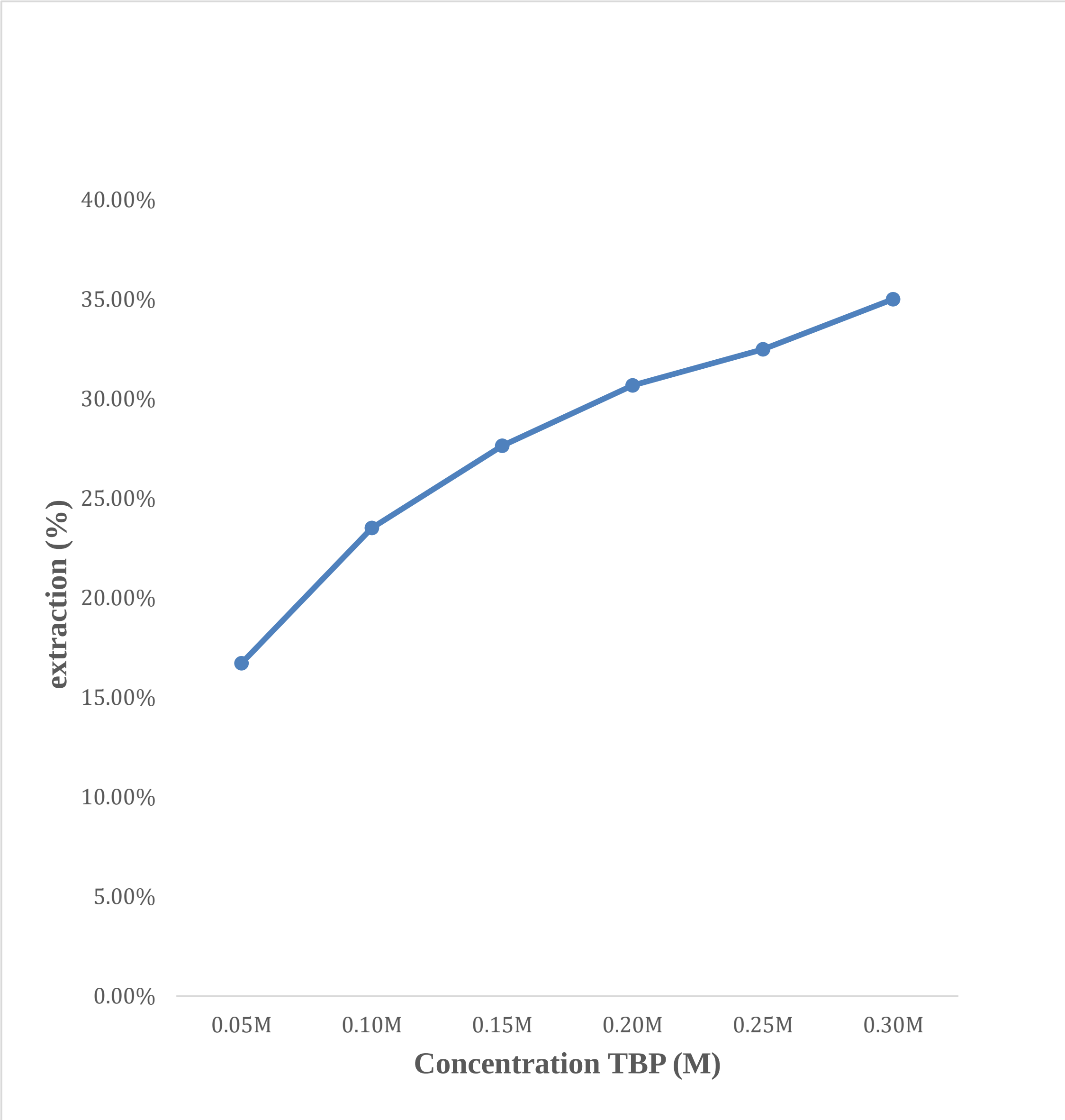


***Figure 6: Plot of % of Zn extracted against concentration of TBP***

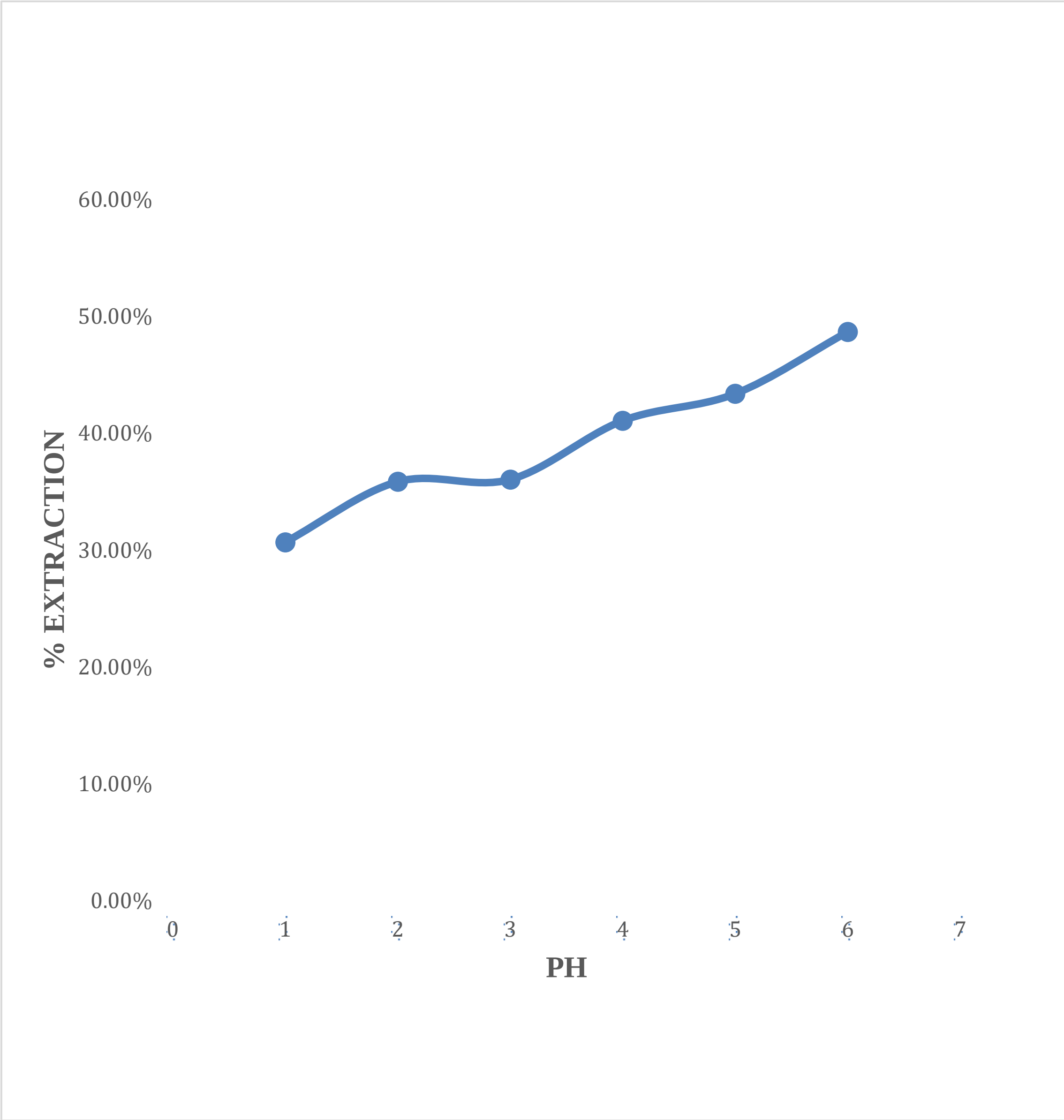


*Figure 7: Effect of pH % of Zn extracted*

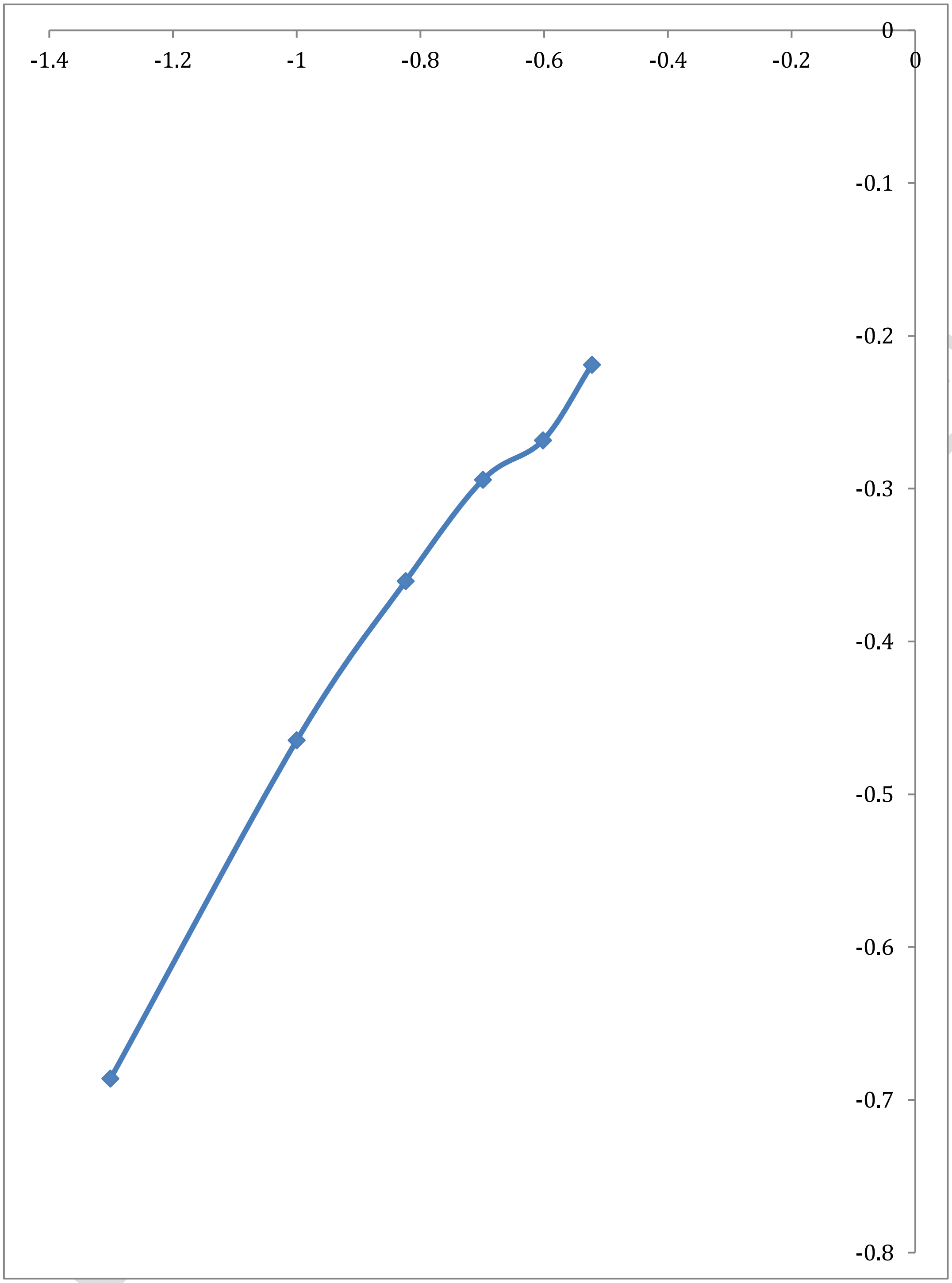


***Figure 8: plot of log D against log [TBP]***

### 4.1 Effect of TBP concentration on the extraction of Zn from burnt tyre ash

The initial concentration of Zn in the leached liquor was 254.77 mg/L. The effect of the extractant concentration on the extraction of Zn from the leached liquor was carried out over the TBP concentration range of 0.05-0.30 mol/L at a pH of 4.5. The results obtained in Table 1 showed that the percentage of Zn extracted increased from 30.69% to 48.68% with an increase in TBP concentration. Thus, a plot of the percentage of Zn extraction against the TBP concentration was obtained to get the optimal TBP concentration. The highest percentage of Zn extracted was 48.68% using 0.30 M TBP at pH 4.5.

### 4.2 Effect of TBP concentration on distribution ratio

The effect of TBP concentration on the distribution ratio D of Zn was also studied, and the result in Table 7 showed an increase from 0.206 to 0.604. The distribution ratio increased with an increasing extractant concentration. The slope of the plot of log D against log TBP in Table 7 gave ~1.00 and indicated that 1 mole of the extractant was involved in the extraction process. One mole of [H+] from the extractant, TBP, was exchanged for one mole of Zn2+ at each process of the extraction of Zn from the burnt tyre ash obtained from Dogarawa, Zaria, Nigeria.

### 4.3 Effect of pH on the extraction process

The effect of pH on the extraction efficiency of zinc using TBP was systematically examined across an equilibrium pH range from 2.5 to 5.5, as detailed in Table 6. The extraction efficiency, expressed as the percentage of zinc extracted, was plotted against pH. The data indicated a positive correlation between the pH of the aqueous phase and the extent of zinc extraction, with extraction efficiency increasing progressively as the pH increases. Notably, at pH 4.5, the extraction efficiency reached its peak, with approximately 48.68% of zinc being successfully extracted from the burnt tire ash. This observation suggests that pH 4.5 represents the optimal condition for zinc extraction using TBP, under which the maximum yield was achieved.

### 4.4 Effect of TBP concentration

The plot of log D against log [TBP], as shown in Figure 8, demonstrates that the distribution ratio (D) increases with increasing [TBP] concentration. The linearity of the plot, with a slope of approximately 1, indicates that one mole of [$H^+$] is liberated during the extraction of one mole of $Zn^{2+}$ from the burnt tire ash. This linear relationship observed in Figure 8 suggests a straightforward stoichiometric interaction between TBP and $Zn^{2+}$ during extraction.

### 4.5 Stripping studies

The stripping study of Zn from the loaded TBP was carried out with dilute HCl solution. The result of the stripping investigation showed that 0.05 mol/L HCl stripped Zn from the loaded TBP at a 1: 1 phase ratio.

## 5.0 CONCLUSION

- The result obtained in the solvent extraction of Zn by TBP in kerosene, indicated that an increase in the initial extractant concentration (organic phase) enhanced the extracted percentage of Zn from burnt tyre ash with the highest percentage of Zn being 48.68% by 0.30 mol/L TBP at pH 4.5.
- The Liquid-Liquid Extraction of $Zn^{2+}$ using Tributylphosphate in kerosene was found to have the potential of extracting Zn from burnt tyre ash leach liquor at optimum condition with one mole of [$H^+$] being liberated during extraction of one mole of $Zn^{2+}$ from the burnt tyre ash.
- The study showed that a phase ratio of 1:1 for the organic phase ; aqueous phase, extracted 48.68% of the total Zn into the organic phase at a pH of 4.5.



## ETHICAL APPROVAL

As per international standards or university standard written ethical approval has been collected and preserved by the author(s).

**DISCLAIMER (ARTIFICIAL INTELLIGENCE)**

Author(s) hereby declare that no generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc) and text-to-image generators have been used during writing or editing of manuscripts.

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