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Purification of Mn-Electrolyte Solution from Leaching of Zn-MnO_2 Spent Batteries Using Cementation Technique

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Abstract

This paper investigates the purification of a manganese electrolyte solution, produced by leaching spent Zn-MnO_2 primary batteries, using cementation with metallic manganese powder. The battery paste, composed mainly of 59.98% Mn and 37.23% Zn, was roasted at 900 °C for 30 minutes to remove carbon and convert MnO_2 to Mn_3O_4. After roasting, the composition changed to 63.65% Mn and 31.73% Zn. Ascorbic acid and sulfuric acid were used as reducing and leaching agents, respectively, at 70°C for 3 hours with a solid-to-liquid ratio of 1/20 g/mL and a stirring rate of 200 rpm. The resulting leachate solution contained Mn and Zn at concentrations of 16.5 and 9.2 g/L, respectively. Cementation with manganese powder was then used to remove zinc from the leachate solution. The effects of reaction temperature, time, stirring rate, and amount of manganese powder on zinc removal were investigated. The optimal conditions for zinc removal (98.4%) were found to be 60 °C, 600 rpm, and 20 minutes. Analysis of the cemented powder revealed a composition of 82% Zn and 17% Mn. Further investigation determined that using the ideal amount of manganese powder achieved a purification efficiency of 99.67%, reducing the zinc concentration in solution to 0.03 g/L.

Keywords*: Spent Zn-MnO2 battery; Cementation; Manganese; Zinc.*

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1. Introduction

Improper disposal of Zn-MnO_2 batteries creates significant environmental problems due to their heavy metal content. Recycling batteries offers a solution to minimize waste. It is the most effective way to manage spent batteries, driven by environmental concerns, resource conservation, and potentially even for financial gain [1]. Improper disposal of Zn-MnO_2 batteries creates significant environmental problems due to their heavy metal content. Recycling batteries offers a solution to minimize waste. It is the most effective way to manage spent batteries, driven by environmental concerns, resource conservation, and potentially even for financial gain.

Zn-MnO_2 batteries are single-use, non-rechargeable cells that generate electricity through a reduction reaction at the manganese cathode and an oxidation reaction at the zinc anode. Their ease of manufacture, safe handling, and widespread use in household devices like radios, toys, and various gadgets contribute to their popularity over other battery types [2]. In Zn-MnO_2 batteries, manganese dioxide (MnO_2) serves as the positive electrode (cathode), while zinc acts as

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the negative electrode (anode). These components are housed in a stainless-steel casing along with an electrolyte solution and additional conductive materials such as a carbon rod. Ammonium chloride and zinc chloride solutions, along with starch, may also be included in the electrolyte depending on the specific battery design [3].
The collection and processing of these batteries differ depending on the chosen method, pyrometallurgical or hydrometallurgical. Unfortunately, significant amounts of electrode powder, rich in manganese and zinc, are often incinerated or landfilled instead of being recycled [4]. These metals have diverse properties, making them valuable in numerous applications, from automobiles and construction to batteries and alloys [5].

There are two significant recycling processes for treating Zn-MnO2 batteries: pyrometallurgy and hydrometallurgy. The majority of pyrometallurgy processes in the EU, including those of Batrec in Switzerland, Snam-Savan in France, and Inmetco in Pennsylvania [6,7] .In the pyrometallurgical process, materials are conflagrating, incinerated, and smelted in a plasma arc furnace as well as sintering, and melting at high temperatures. It illustrated the need for a gas purification system and the high energy required, which was a drawback of the pyrometallurgy process [8].On the other hand, hydrometallurgy is thought to be an economical and ecologically sustainable way of battery treatment. This process begins with crushing followed by magnetic separation, separation from paper, iron scraps, and plastic. Spent batteries are found to contain 2.25% plastics, 6.12% paper, 28.94 % steel cases, and 55.3% battery powder [9]. The next stage involves converting the metals from powder to aqueous liquor by leaching them using acidic solutions. Finally, metals are extracted by many methods, such as ion exchange, precipitation by controlling pH, and cementation.

The hydrometallurgical ores processing industry has long employed the cementation method to remove unwanted metals from the leach solution or to obtain the desired metal species [10]. Moreover, it is the process by which a less noble metal precipitates out of a more noble metal's salt solution. The electrochemical potentials of the reacted species in solution determine the general reaction for a cementation technique based on their redox reaction [11,12]. Numerous research has been conducted on the cementation and cementation kinetics of metals have been done, as an illustration of this kind of kinetic study is the cementation of copper from updated solutions onto a rotating aluminum and it was found that the reactants' diffusion through the fluid layer which had a 14.1 kJ/mol of activation energy [13]. Utilizing a rotating disc geometry, the kinetics of copper cementation were investigated on pure iron substrates [14]. Research on the cementation method of copper recovery also showed that scrap iron could be effectively used to remove copper ions from waste solutions from sulphate-type copper electroplating [15].The cementation process has been thoroughly examined in numerous studies, such as cementation of copper by iron [16], additionally research on gold onto zinc using rotating disc electrode system [17], also cementation cobalt, nickel and cadmium by zinc metal [18], in addition ,research on cobalt from an industrial zinc electrolyte in the presence of Cu, Cd, Pb, Sb and Sn additives and less than 10% of the zinc dust dissolves [19],research on cementation copper from cyanide liquors using zinc and study different conditions of temperature ,free cyanide concentrations and solution PH and concluded at higher temperature and lower PHs the cementation rate increases [20],silver ions onto copper from acidic sulphate solutions were investigated using a rotating cylinder system and study the effect cylinder rotation speed, initial concentration of silver ions, concentration of sulphuric acid, temperature and oxygen in the system [21], a hydrometallurgical method for lead acid battery and extract lead from ammonium sulphate solution by nickel powder using cementation technique was studied [22], copper from complex sulfide leach liquor was studied [23], analysis of the cementation of silver ions after the cementation is the mechanical activation and obtained that it impacts the mechanical properties of cementator [24], analysis was done on copper recovery using metallic aluminum and found that the rate of reaction rises with increasing copper concentration ,stirring speed ,temperature and decreasing PH [25],optimization study on copper cementation from reefing waste water [26], examine the extraction of copper from monometallic solution by cementation on zinc was efficiently done in a simple agitated reactor [27], a thorough analysis was conducted on the recovery ruthenium using zinc, examining the interplay between reaction temperature, reaction time , amount of zinc ,shaking and sodium chloride addition [28], an investigation onto the nonferrous metals' cementation process using brine leaching solution using different cementation agents such as zinc and iron [29], using metallic aluminum particles to apply to the response surface method to copper cementation [10], efficient parameters analysis on the use of zinc powder in the cementation reaction of cadmium from the point of diffusion [30], modeling and optimization of oxide copper cementation kinetics and achieved a cementation recovery of roughly 90% [31], aluminum is used to recover concentrates and metallic lead from zinc plant residue [32], studying cementation copper from industrial waste solutions [33],analyze the temperature and PH of the solution as well as the purification process of the zinc sulphate solution from cadmium and copper using the cementation technique and concluded that the cementation process is more effective at changing temperatures[34]. However, there is no study on extracting zinc using manganese powder by the cementation process.

The purpose of this work is to purify the Mn electrolyte solution that is made by leaching spent Zn-MnO2 primary batteries from zinc ions by cementation technique using metallic manganese powder and to obtain the optimum conditions utilized to separate cemented zinc powder from the solution. The parameters of cementation were solution temperature, stirring rate, the time of the cementation process and the amount of manganese powder added to the solution for precipitation of cemented zinc powder.

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2. EXPERIMENTAL WORKS

2.1. Material

For this study, the Zn-MnO_2 batteries were dismantled to extract the internal paste, which was then crushed into a powder to ensure a uniform consistency. This roasted battery powder was leached using ascorbic acid ($C_6H_8O_6$) as a reducing agent in a sulfuric acid (H_2SO_4) solution. Following the leaching step, cementation for selective removal of zinc ions was performed using manganese powder (Mn). The steps of this experimental method are illustrated in Figure 1.

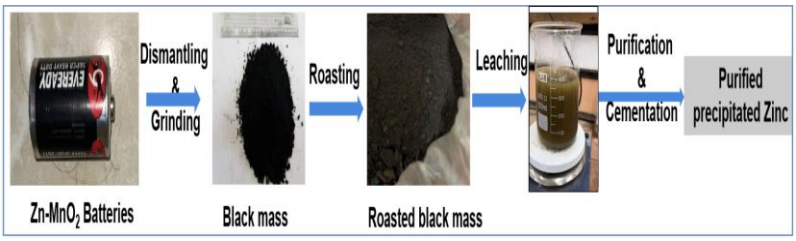

Figure 1: Illustration of the experimental workflow for zinc extraction from Zn- MnO2 spent batteries.

2.2 Characterization of Black Powder and Roasted Powder

Prior to experimentation, the composition of the black powder from spent Zn-MnO_2 batteries was analyzed using X-ray fluorescence (XRF) for Mn, Zn, Al, Fe, Si, Ca, Ag, and Ni. X-ray diffraction (XRD) using Cu Kα radiation (λ = 1.54 Å) and scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDX) were also employed to characterize the phases and morphology of the powder. Roasting the black powder in a furnace facilitated its dissolution by removing carbon and converting manganese oxide phases. Following this step, XRF and XRD were again used to analyze the roasted powder. Finally, an S4AA system (China) measured the concentrations of leached zinc and manganese ions.

3. RESULTS AND DISCUSSION

3.1. Characterization of The Spent Zn-MnO2 Primary Batteries

The black powder of batteries was thoroughly mixed to guarantee uniformity prior to being ground to the necessary 90 µm particle size. The chemical elements of the black powder of the Zn-MnO_2 spent batteries are illustrated in Table 1. The major elements of the obtained black powder from the dismantled Zn-MnO_2 spent batteries were 59.98% Mn and 37.23% Zn. XRF results were obtained after roasting the samples at 900 °C, so no carbon content of graphite appeared while it was determined by EDX analysis.

TABLE 1. XRF analysis of black powder of the spent Zn-MnO2 batteries.

Components	Wt.%
Mn	59.98
Zn	37.23
Al	1.12
Fe	0.429
Si	0.579
Ca	0.246
Ag	0.059
Ni	0.019

According to the XRD analysis, manganese dioxide (MnO2) and Simonkolleite (Zn5(OH)8Cl2.H2O) were the two main phases of the black powder as shown in Figure 2 and Figure 3 illustrates a black powder's SEM picture. The irregularly shaped and rough surfaced particles range in diameter from 10 µm to 50 µm, and many of the smaller particles stick to the larger ones. Table 2 shows the percentage of carbon in black powder by EDX analysis and find that before roasting, the EDX analysis of the black mass sample showed a high carbon content of 44.97%.

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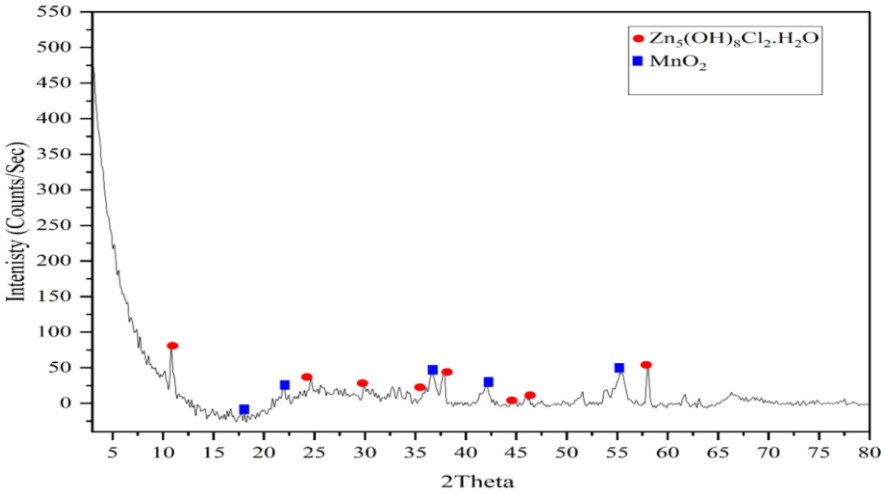

Figure 2: XRD analysis of paste of Zn-MnO_2 battery.

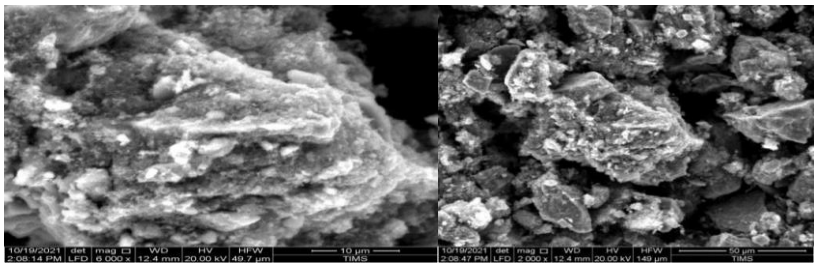

Figure 3: Microstructure of paste of Zn-MnO_2 spent battery.

Table 2. EDX analysis of paste of Zn-MnO_2 spent battery.

Element	Wt.%
C	44.97
O	13.03
Na	1.07
Al	2.25
Cl	9.50
Mn	23.94
Zn	5.24

3.2. **Roasting Process**

The process of roasting involves thermal gas- solid reactions, such as pyro hydrolysis, oxidation, reduction, chlorination, and sulfation. This process involves heating the ores to a high temperature below their melting point while removing excess air from them to release their oxides, which are then used to extract the metals.

Due to the large percentage of carbon in the black powder and it is hindering the MnO_2 leaching, the black powder was roasted for 30 min at 900° C in air to remove carbon and to obtain Mn_3O_4 instead of MnO_2 [35], as shown in Figure 4. Manganese percentage was 63.65% and Zinc percentage was 31.73 % in the roasted powder, and it is also clear that the percentage of manganese increased in the powder.

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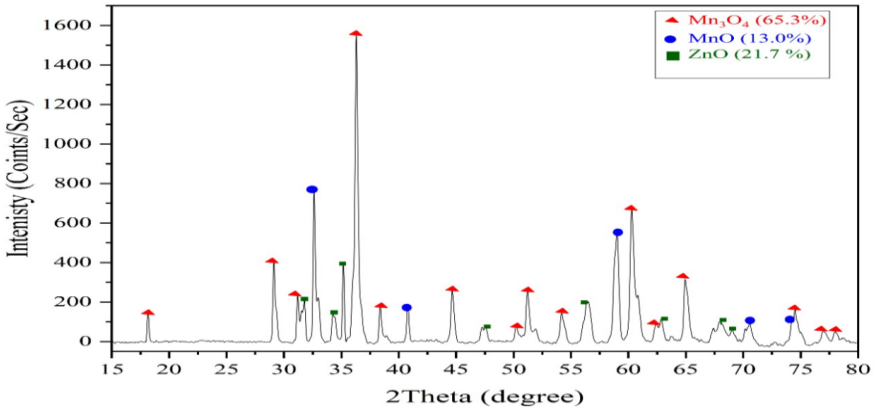

Figure 4: XRD analysis of roasted powder.

3.3. **Leaching Process**

In this research, the roasted black powder of the Zn-MnO_2 spent batteries was leached with sulfuric acid as a leaching agent using ascorbic acid as a reducing agent. Leaching conditions were set, as previously reported [9], at 70° C for 3 h using 0.5 M of sulfuric acid concentration with the addition of 13 g/dm³ ascorbic acid, and 1/20 g/cm³ in the ratio of solid /liquid while stirring rate set at 200 rpm. Table 3 shows the Mn and Zn concentrations results after the black powder leaching process before and after roasting and roasted powder. Before and after roasting, two samples of the black powder of the spent Zn-MnO_2 were leached to compare the effective leaching on both samples. The roasted samples showed a 3.9 times improvement in Mn recovery compared to the black paste without roasting, while Zn recovery was improved by 2.9 times higher than the sample before roasting. These results confirm the effective leaching of Mn and Zn metals from the roasted black powder of the Zn-MnO_2 spent batteries.

Table 3. Results of leaching of black mass and roasted mass.

Conditions of Sample	Mn (g/L)	Zn (g/L)
Before Roasting	**4.24**	**3.22**
After Roasting	**16.5**	**9.2**

3.4. **Precipitation and Purification Processes**

The cementation process carries out precipitation by adding manganese powder to the solution, which contains manganese and zinc ions. The reaction describes the cementation of zinc from aqueous solution:

$$Mn^{o}\,(\text{manganese powder}) + Zn^{2+} \longrightarrow Zn^{o}\,(\text{cemented zinc}) + Mn^{2+} \quad (1)$$

The extraction of zinc has been done by adding 8.6 gm for each litter of manganese powder by the stoichiometric ratio ,8.6 g of manganese powder was added to the solution to precipitate zinc. The experiment was carried out in 20 mL of solution. Experimental parameters used in this study are solution temperature (25, 40, and 60 °C), stirring rate (200, 400, and 600 rpm), time (20, 40, and 60 min) of the cementation process of Zn metal and amount of manganese powder added to solution (X,1.25X,1.5X,1.75X and 2X), where (X) is the amount of manganese powder added to solution by stoichiometric ratio

The following equation calculates the amounts of zinc precipitated:

$$\text{Precipitated Zinc (\%)} = \frac{C_o - C_f}{C_o} \times 100 \quad (2)$$

Where:

(C_o) the initial concentration of zinc in solution before cementation

(C_f) the final concentration of zinc in the same solution after cementation.

3.4.1 **Effect of Stirring Rate and Time on Zinc Cementation at Room Temperature**

Both Figure 5 and Figure 6 reveal the percentage of purification of zinc from manganese solution and the zinc concentrations in solution after cementation at room temperature with (200, 400, 600) rpm stirring rate and (20, 40, 60) min as time of cementation process. It's clear that the precipitation percentage of zinc metal increases with increased stirring rate at room temperature. As the stirring increases the rate of chemical reaction increases the speed of dissolution of the manganese powder added to the solution, thus making it easier to get rid of zinc ions. It is evident that as the

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cementation process duration increases with increasing the stirring rate to 400 and 600 rpm the percentage of purification increases.

Evidently, the cementation of zinc increased at 400 rpm, 60 minutes, and room temperature, the zinc concentration reached 0.199 g/L and the purification ratio of the solution from zinc was 97.83%.

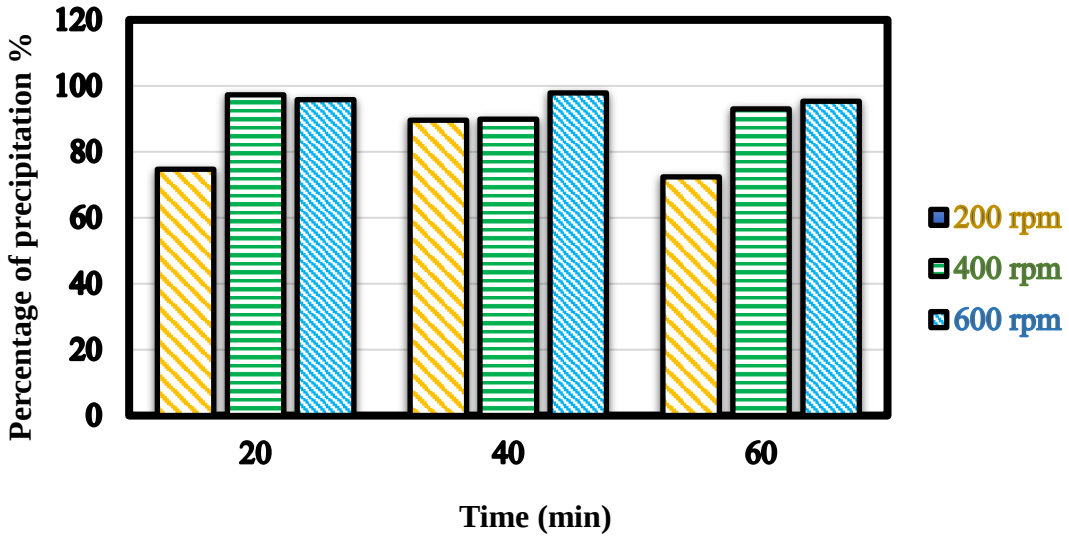

Figure 5: Results of precipitation percentage of zinc by cementation at room temperature.

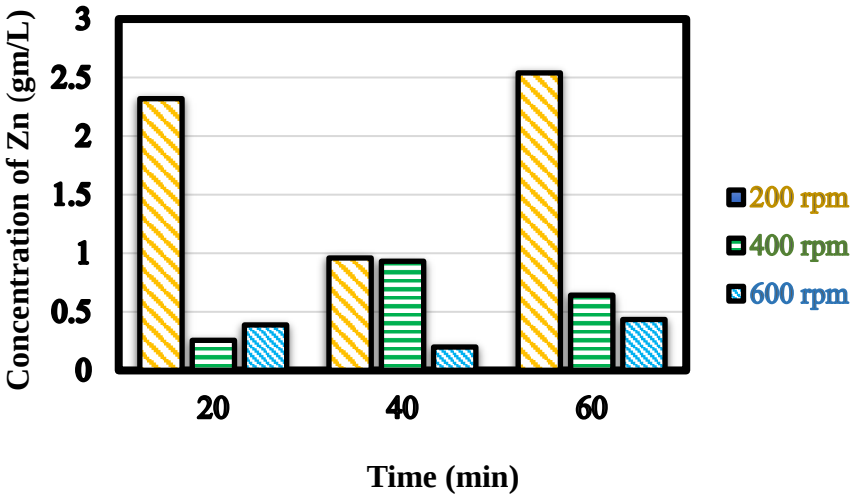

Figure 6: Results of concentration of zinc after cementation at room temperature.

3.4.2 **Effect of Stirring Rate and Time on Zinc Cementation at 40° C**

As shown in Figure 7, the percentage of zinc precipitation increases with a higher stirring rate. The concentration of zinc in solution after cementation is shown in Figure 8. Increasing the temperature of the system agitates the particles more. This higher energy allows for a faster cementation reaction rate, leading to higher zinc purification percentages at 600 rpm (regardless of whether the reaction time is 20, 40, or 60 minutes). Conversely, increasing the process temperature to 600 rpm reduces reaction time compared to room temperature. This results in a lower final zinc concentration of 0.155 g/L and a higher purification percentage of 98.3% after 40 minutes at 600 rpm.

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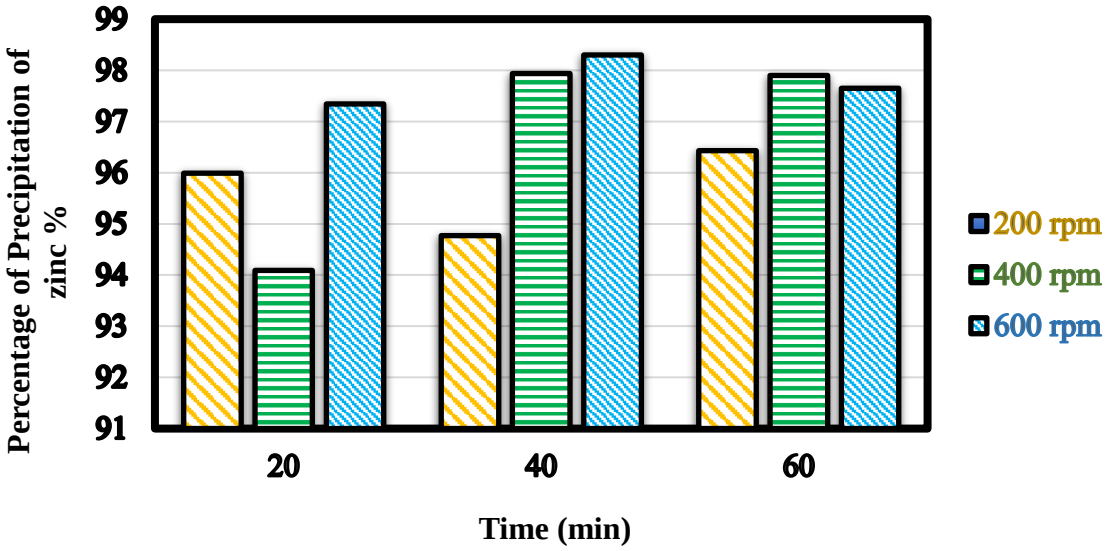

Figure 7: Results of precipitation percentage of zinc by cementation at 40 ℃.

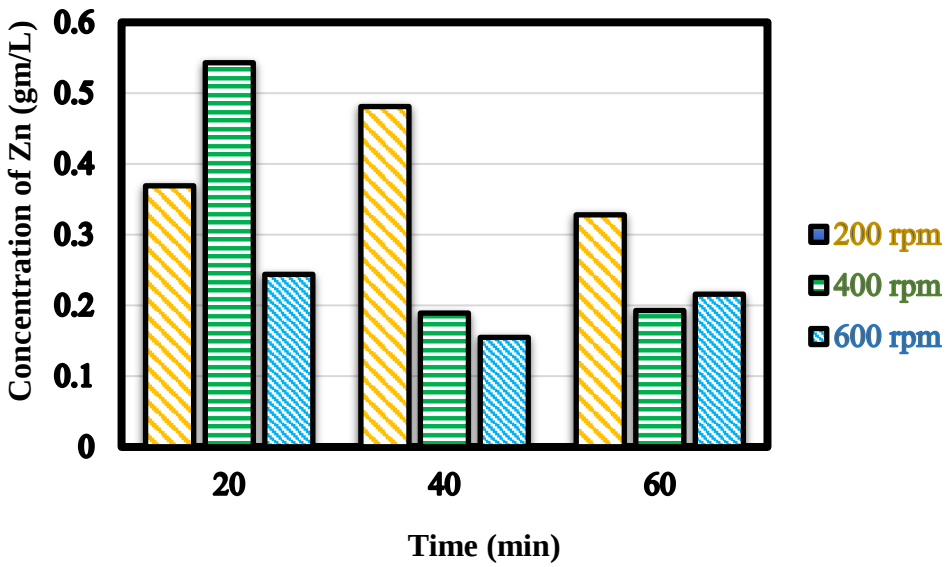

Figure 8: Results of concentration of zinc after cementation at 40° C.

3.4.3. **Effect of Stirring Rate and Time on Zinc Cementation at 60° C**

It was obvious that raising the temperature facilitates the cementation process. As the temperature rises, the cementation process reacts more quickly and improve the rate of reaction. There is an inverse relationship between time and temperature. When increasing the temperature, the time spent decrease to purify the solution from zinc ions. Figure 9 shows that the high percentage of purification of solution was 98.4% and the zinc concentration becomes 0.148 g/L in solution at 600 rpm and 20 min as shown in Figure 10, and the manganese concentration in the solution becomes 29.94 g/L.

Finally, the obtained conditions are 60℃ as cementation temperature, 600 rpm stirring rate, and 20 min time cementation. The obtained powder from the cementation process at this condition was 82% and %17 % for Zn and Mn by using XRF analysis. 98.4 % of zinc was extracted from the solution by cementation, while the percentage precipitation of zinc by pH change process by adding NaOH was 95.35 % [9]. The XRD analysis of the obtained powder as cemented zinc from optimum conditions is shown in Figure 11. The XRD analysis showed characteristic peaks at 2θ (degree) 36.38, 38.95, 43.2, 54.36, and 70.19. The crystallite showed a hexagonal shape of the precipitated zinc after purification.

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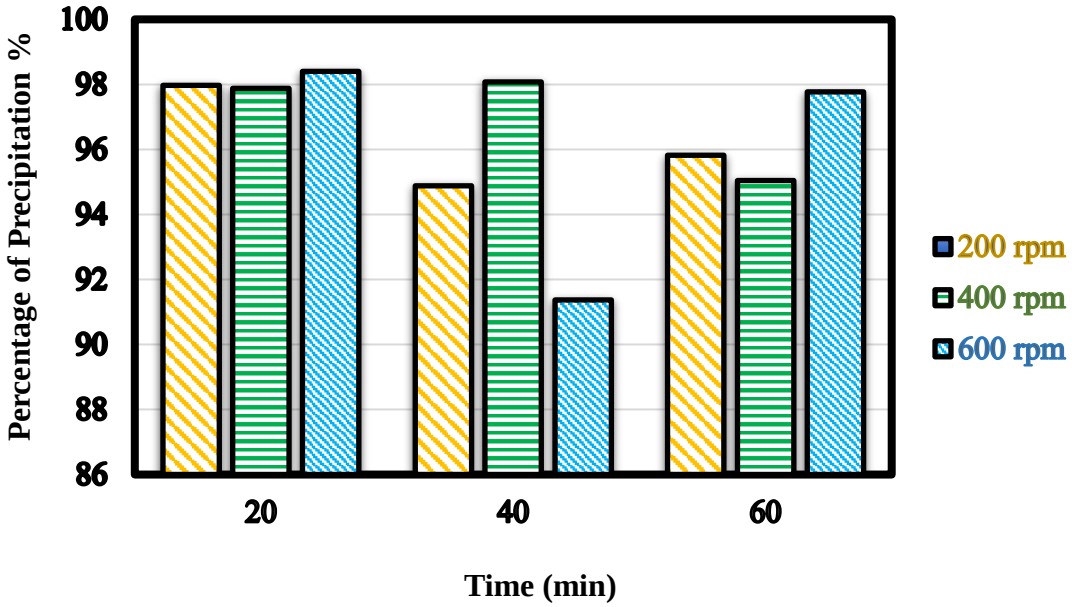

Figure 9: Results of precipitation percentage of zinc by cementation at 60℃.

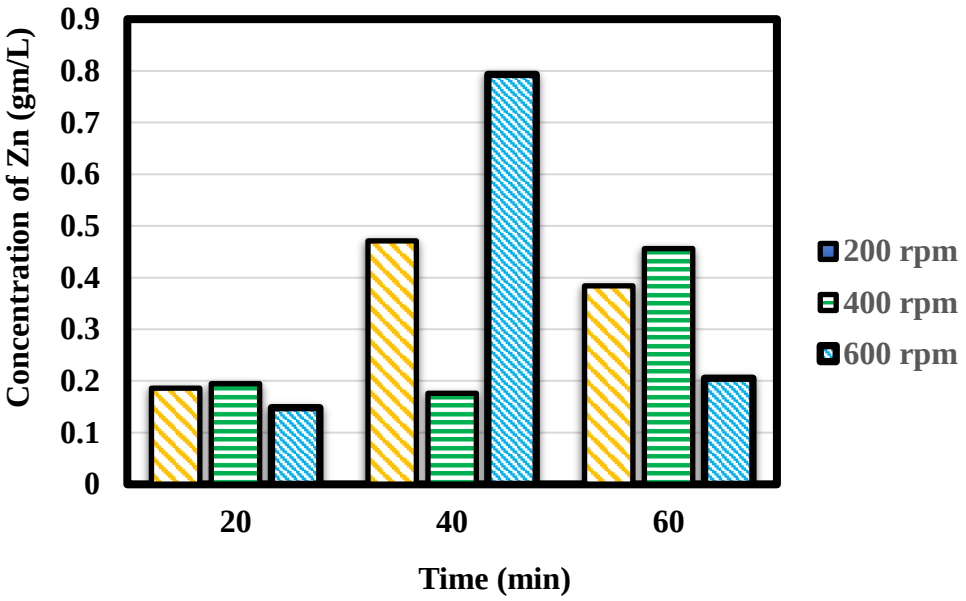

Figure 10: Results of zinc concentration after cementation at 60° C.

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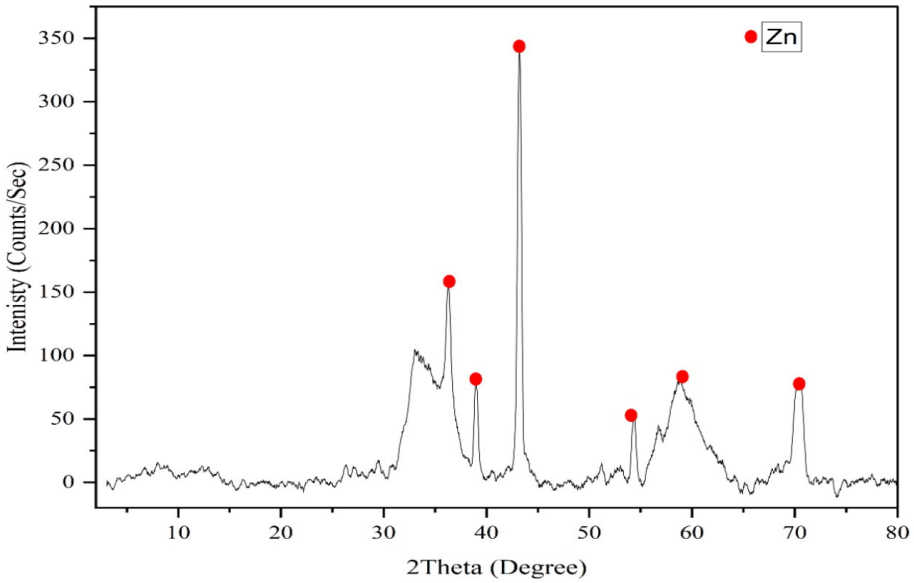

Figure 11: XRD analysis of cemented zinc powder.

3.4.4. **Effect of Amount of Manganese Powder**

Having identified the optimal cementation conditions (60°C temperature, 600 rpm stirring rate, and 20 minutes), we investigated the effect of manganese content on zinc cementation. Experiments were conducted using Mn dosages of 1X, 1.25X, 1.5X, 1.75X, and 2X the stoichiometric amount (X refers to the amount needed for complete zinc removal). Zinc removal efficiency increased from 98.4% at 1X Mn stoichiometry to over 99% at 1.5X and 1.75X Mn stoichiometry Figure 12. However, adding a 2X Mn dosage resulted in a decrease in zinc precipitation, as shown in Figure 13. Like when removing copper by aluminum plate from refining wastewater, the percentage removal of copper ion about 99% at aluminum stoichiometry 1X,1.5X.2X [26].

The most logical and straightforward way to precipitate zinc appears to be cementation with using manganese powder to create metallic zinc sediments suitable for metallurgical processing. The component of manganese has been selected to be special so that no other element is added to the solution and will be an obstacle in the deposition of manganese by electrowinning later. The possible form of the cementation reaction appears below due to different standard reduction potentials [36].

$$Zn^{2+}+2e=Zn \quad (E°=-0.7618V) \tag{3}$$

$$Mn^{2+}+2e=Mn \ (E°= -1.185\ V) \tag{4}$$

This process is redacted, oxidized on the anode, and resuspended in the metal cathode through electrochemical means. The difference of potential of the general reaction is shown in following equation.

$$Mn°\ (s)+ Zn^{2+}\ (aq) \longrightarrow Zn°(s)+Mn^{2+}\ (aq) \quad (\ E°=0.432V) \tag{5}$$

To complete the precipitation, it must be E° for zinc bigger than E° for manganese [37] and if it is less, the metal will not precipitate.

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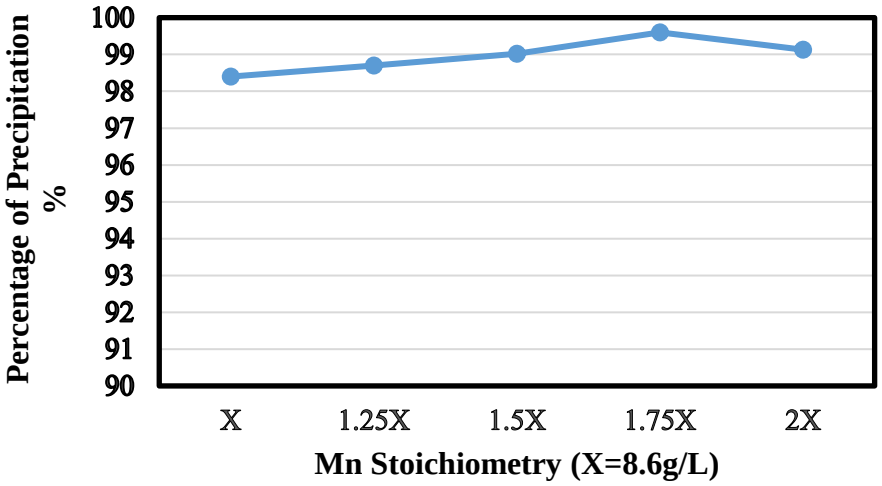

Figure 12: Effect of amount of manganese powder on zinc cementation at 60 ℃, 600 rpm and 20 min.

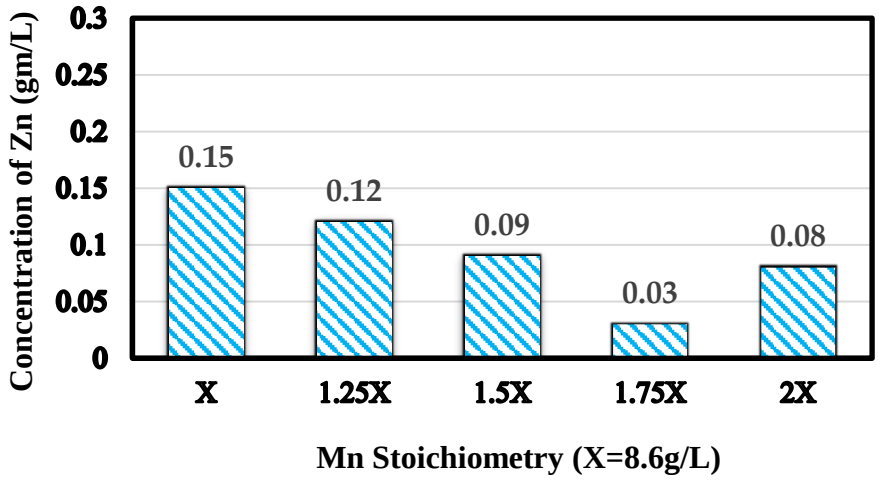

Figure 13: The zinc concentrations in solution after cementation at 60 ℃, 600 rpm and 20 min.

4. CONCLUSIONS

This study investigated how to purify the Mn electrolyte solution that was produced by leaching paste of Zn-MnO_2 spent batteries from zinc ions using cementation process by adding metallic manganese powder. Based on the findings, it is possible to draw the following conclusions:

1. The cementation experiments' findings demonstrated that the three parameters of temperature, stirring rate and time had the most influence on the zinc cementation process. Finally, cementation time of 20 minutes, stirring rate of 600 rpm and 60℃ were selected as the optimum cementation conditions, resulting in 98.4 % of zinc recovery.
2. The obtained powder as cemented zinc was 82% Zn and 17% Mn, and the obtained concentrations in solutions were 0.148 g/L and 29.94 g/L for Zn and Mn respectively.
3. After obtaining the optimum conditions for the cementation process, experiments were made to study the impact of the added amount of manganese powder to the solution on the concentration of the retained zinc ions after the cementation process, and it was concluded that the optimum amount of manganese powder was 1.75X (=15.05 g/L) where the percentage of purification was increased to 99.67% and concentration of zinc ions in solution became only 0.03 gm/L.

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