# Recovery of Bismuth from Anode Sludge in Sn-Bi Electroplating Processes from Industry

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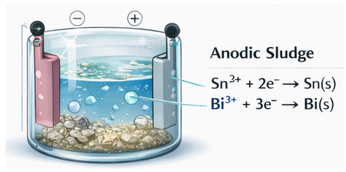
## Abstract

This study investigates the recovery of bismuth from anodic sludge generated during industrial tin–bismuth (Sn–Bi) electroplating. The sludge, considered industrial waste, contains valuable metals such as bismuth and tin. Chemical and phase analysis using ICP and XRD revealed that the sludge contains 74.34 wt.% bismuth, mainly in the form of $Bi_2O_3$. Selective leaching using methanesulfonic acid ($CH_3SO_3H$) was applied to separate bismuth from other components. The effects of acid concentration, temperature, leaching time, agitation speed, and solid-to-liquid ratio were investigated. The optimal conditions include temperature 20 °C, 160 min, stirring speed of 530 rpm, 400 g/L Methanesulfonic and a liquid-to-solid ratio of 4.8/1 , resulting in a leaching efficiency leaching of 63.70 %. The recovered bismuth showed suitable purity for reuse in Sn-Bi Electroplating process. This approach demonstrates a promising method for recovering valuable metals from electroplating waste while reducing environmental impact and supporting resource sustainability.

## Introduction

### 1 Sn-Bi Electroplating

In tin–bismuth (Sn–Bi) electroplating, $Sn^{2+}$ and $Bi^{3+}$ ions are electrochemically reduced and deposited onto the cathode surface, while insoluble reaction by-products gradually accumulate and form anodic sludge during the plating process.




### 2 Anodic Sludge Formation

Anodic sludge forms at the anode as a result of metal ion oxidation and the accumulation of oxide residues and impurities during Sn–Bi electroplating.




### 3 Hydrometallurgical leaching

Methanesulfonic acid (MSA) is employed as a leaching agent to selectively dissolve bismuth oxide ($Bi_2O_3$) from anodic sludge. In this process, bismuth oxide reacts with methanesulfonic acid to form soluble bismuth methanesulfonic according to the following reaction:

$$Bi_2O_3 + 6CH_3SO_3H \rightarrow 2Bi(CH_3SO_3)_3 + 3H_2O$$

This reaction facilitates the dissolution of bismuth from the solid sludge into the solution, enabling its subsequent separation and recovery.

### 4 Selective Leaching Process




Selective leaching is a hydrometallurgical process used to selectively dissolve the target metal from solid materials while minimizing the dissolution of other components. The efficiency of this process is influenced by parameters such as acid concentration, temperature, leaching time, agitation speed, and solid-to-liquid ratio.

### 5 Inductively Coupled Plasma (ICP)

Analyzes the chemical composition in samples.

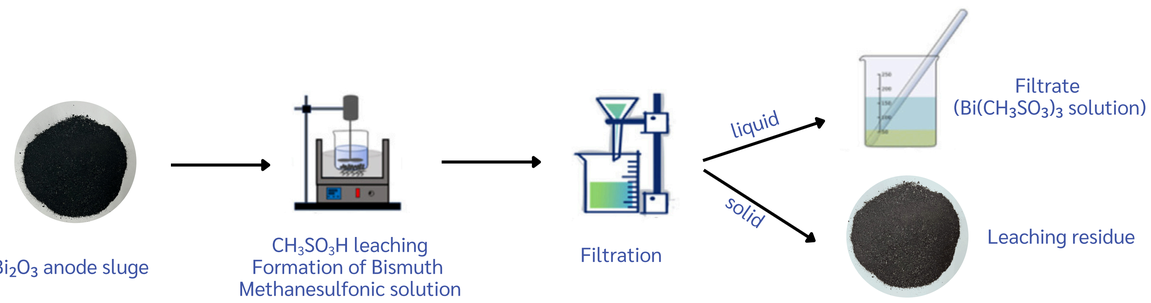



### 6 X-Ray Diffraction

Examines the crystalline structure and phase of compounds.



## Experiment




Experimental Conditions

| | |
|---|---|
| Leaching temperature (°C) | 20 , 50 , 70 , 90 |
| Leaching time (min) | 5 , 10 , 20 , 40 , 80 , 160 , 320 , 640 , 1280 |
| Stirring speed (rpm) | 0 , 530 , 800 |
| $CH_3SO_3H$ concentration (g/L) | 200 , 400 , 600 , 800 , 1000 , 1277 |
| liquid-to-solid ratio | 3.0:1 , 3.4:1 , 4.0:1 , 4.8:1 , 6.0:1 |

## Conclusion

Anodic sludge generated from industrial Sn–Bi electroplating contains a high bismuth content of 74.34 wt.%, primarily in the form of bismuth oxide ($Bi_2O_3$).

Selective leaching using methanesulfonic acid (MSA) was applied to extract bismuth from the anodic sludge.

The optimal leaching conditions were temperature 20 °C, 160 min, stirring speed of 530 rpm, were 400 g/L Methanesulfonic and a liquid-to-solid ratio of 4.8/1

Under these conditions, a bismuth leaching efficiency of 63.70 % was achieved, demonstrating the potential for effective recovery of bismuth from industrial waste.

## Objective

To recovery of bismuth from anodic sludge produced during industrial Sn–Bi electroplating and to improve the efficiency of bismuth recovery from industrial waste materials.

## Results & Discussion

### 1. X-ray Diffraction (XRD) Analysis

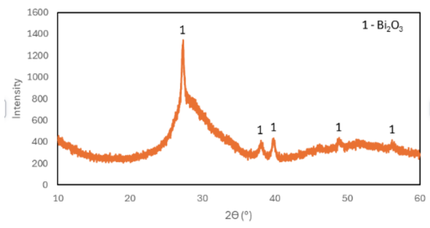



Fig. 1. XRD pattern of $Bi_2O_3$ anodic sludge sample.

### 2.Inductively Coupled Plasma (ICP) Analysis

| Composition | Bi | Other |
|---|---|---|
| wt. % | 74.34 | 25.66 |

Table. 1 ICP analysis of $Bi_2O_3$ anodic sludge from the Sn–Bi electroplating process.

### 3.Effect of leaching conditions

3.1 Leaching rates of Bi and decreased concentration of $CH_3SO_3H$ at different temperatures.

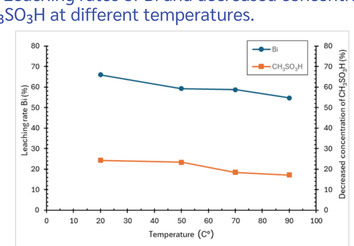



Fig.3 (a)

3.2 Leaching rates of Bi and decreased concentration of $CH_3SO_3H$ at different times.

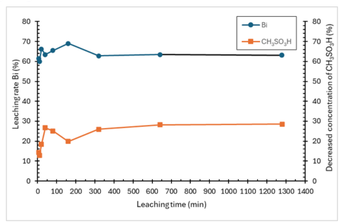



Fig.3 (b)

3.3 Leaching rates of Bi and decreased concentration of $CH_3SO_3H$ at different stirring speed.

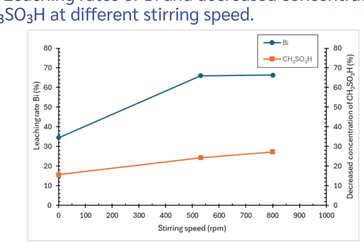



Fig.3 (C)

3.4 Leaching rates of Bi and decreased concentration of $CH_3SO_3H$ at different $CH_3SO_3H$ concentration.

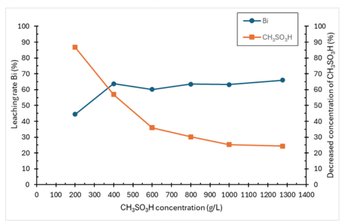



Fig.3 (d)

3.5 Leaching rates of Bi and decreased concentration of $CH_3SO_3H$ at different liquid to solid ratio.

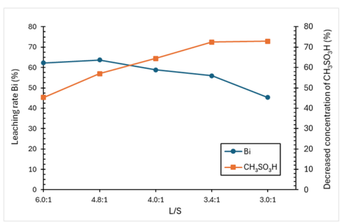



Fig.3 (e)

### 4.Scanning Electron Microscopy (SEM) Analysis

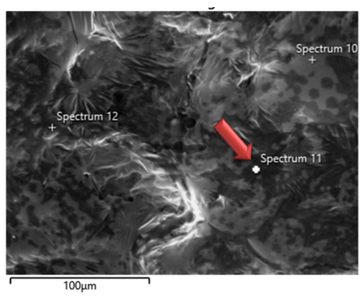
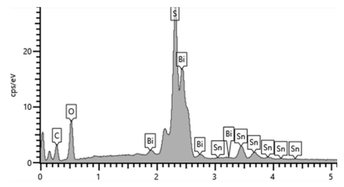



Fig.4 SEM micrograph of $Bi_2O_3$ sludge after leaching with $CH_3SO_3H$




**Spectrum 11**

| Element | Signal Type | Line | Wt% | Wt% Sigma | Atomic % |
|---|---|---|---|---|---|
| O | EDS | K series | 17.44 | 0.19 | 51.13 |
| S | EDS | K series | 23.29 | 0.18 | 34.07 |
| Sn | EDS | L series | 12.75 | 0.19 | 5.04 |
| Bi | EDS | M series | 46.51 | 0.36 | 10.44 |
| Total | | | 100 | | 100 |

Fig.5 spectrum 11 (Fig.4) shows the elemental composition of $Bi_2O_3$ sludge after leaching with methanesulfonic acid (MSA).

## References

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Direct oxidative pressure leaching of bismuth sulfide concentrate in methanesulfonic acid medium.
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