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DEVELOPMENT OF AN ENVIRONMENTALLY FRIENDLY AND HIGHLY EFFICIENT METHOD FOR THE SEPARATION OF Co(II) AND Ni(II) IONS FROM Cd–Co–Ni CAKE INTERMEDIATES

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ABSTRACT

Selective recovery of cobalt from Cd–Co–Ni cake intermediates is problematic due to the chemical similarities among accompanying ions and the environmental impacts of traditional separation techniques. Most existing techniques have low selectivity for Co(II) and are reagent-hungry, with secondary waste generation, especially in real industrial by-products. Here, an environmentally friendly hybrid pyrometallurgical-hydrometallurgical method is proposed for the selective separation and recovery of Co(II) from industrial Cd–Co–Ni cakes. The method consists of a sequence of steps, namely controlled oxidative-sulfation pretreatment, pH-regulated separation of interfering ions, and selective complexation of Co(II) with 2-nitroso-1-naphthol-3.6-disulphonic acid (NNDA). The effective conceptual separation of interference suppression and cobalt complexation is the method's novelty, enabling the selective recovery of Co(II) from complex multicomponent matrices. The formation and stability of the Co-NNDA complex were confirmed by UV–Vis spectrophotometry, X-ray diffraction, IR spectroscopy, and analysis of particle size. In the pH 5.5–6.0 and 25–30 °C ranges, selective retention and recovery of cobalt were achieved with high phase purity and reproducibility (RSD < 5%). The results indicate the potential of the developed method to recover cobalt from metallurgical waste streams, from economic and environmental perspectives.

Keywords: Sustainable Metallurgy, Selective Separation, NNDA Complexation, Cobalt Recovery, Cd–Co–Ni Cake.
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INTRODUCTION

The continuous growth of the metallurgical and chemical industries has generated significant volumes of industrial by-products containing highly toxic,[1-4] strategically essential metals.[5-7] One of the most complex and environmentally hazardous waste streams is the Cd–Co–Ni cake intermediates generated during cadmium production.[8-10] This is due to their multicomponent nature and their ability to persist in the environment for long periods.[11] The indiscriminate dumping of such residues not only facilitates the contamination of the soil and water with highly toxic metals but also represents a significant economic loss due to the available metals in the waste.[12-14] Cobalt presents an exceptional paradox: it is both a toxic heavy metal and a vital resource for rechargeable batteries,[15-17] superalloys, catalysts, pigments, and advanced technologies in aerospace and medicine.[18-20] As global demand for cobalt rises, interest in mining it from secondary sources and industrial waste is growing.[21-,24] Cobalt in Cd–Co–Ni cakes is customarily found with other economically valuable metals and metalloids,[25-27] including nickel, zinc,

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cadmium, copper, iron, and antimony, making the selective separation of cobalt from the alloy a complex challenge.[28-30] There has been extensive research and literature reporting industrial and research methods for cobalt separation,[31] including in situ solvent extraction,[32] ion exchange, adsorption, and selective precipitation.[33] The majority of these methods apply to simple or ideal systems, lack sufficient separation of Co(II) and Ni(II)[34], require large quantities of organic solvents,[35] and generate secondary pollution in real industrial matrices.[36] Additionally, many researchers focus on analytical measurements,[37] with little focus on the practical recovery of cobalt from complex metallurgical wastes.[38] This work has bridged the first gap in the literature by demonstrating a selective, environmentally friendly,[39,40] and industrially scalable method for isolating Co(II) from real Cd–Co–Ni cake intermediates, using low amounts of reagents.[41] The novelty of this work is in the combination of (i) selective precipitation of non-cobalt ions at specific pH values in the presence of interfering ions and (ii) ligand-selective stabilisation of cobalt by 2-nitroso-1-naphthol-3.6-disulphonic acid (NNDA). Separating the removal of interference and the fixation of cobalt as separate procedures improves selectivity and reproducibility.[42,43] This study aims to provide the first proof of a validated and environmentally sustainable method for the efficient recovery of Co(II) from Cd–Co–Ni cake intermediates and will be supported by data from spectrophotometry,[44] infrared spectroscopy, crystallography, and other physicochemical analyses.[45]

EXPERIMENITAL

All reagents and chemicals were used from the experimental laboratory, and deionised water was used for the whole experimental procedure. For pH adjustment, sulfation, and selective precipitation, 98% H_2SO_4 (sulfuric acid, 37% HCl (hydrochloric acid, and sodium hydroxide (NaOH) were used in the experiment in a precise concentration. The used organic complexing reagent 2-nitroso-1-naphthol-3.6-disulphonic acid (NNDA) serves as a selective ligand for cobalt. 0.1% (w/v) solutions were made by dissolving 0.01 g of reagent into 10 mL of 96% ethanol. Ethanol was used due to NNDA's high solubility and stability. The NNDA solution was placed in an amber glass bottle to prevent photodegradation. Buffer solutions of universal type, with a pH range of 1-12, were made with a combination of the following: H_3BO_3- boric acid, H_3PO_4- phosphoric acid, and CH_3COOH- acetic acid. The pH was adjusted to 0.2 N NaOH.

Preparation of Standard Cobalt Solutions

A stock solution of cobalt (II) at 1 mg/mL was prepared by weighing 1.000 g of $CoCl_2 \cdot 6H_2O$, dissolving it in deionised water, and bringing the solution to the 1-litre mark in a volumetric flask. To avoid precipitation and hydrolysis of Co (II) ions, a few drops of HCl were added. Standard working solutions were prepared in the range of 0.5-25 mg/L and diluted daily from the stock solution.

Characterisation of Cd–Co–Ni Cake

The Cd–Co–Ni cake utilised in this study was sourced from an industrial cadmium production process. Before analysis, the sample was dried (105°C for 4 hrs) and homogenised. The elemental composition was analysed by X-ray fluorescence spectroscopy and helped in the selective separation strategy of primary and secondary metal constituents in the sample.

Oxidative–Sulfation Pretreatment

The sample was oxidatively roasted, and then sulfated to change the metallic sulfides of the cake to water-soluble sulfates. The sulphide phases were oxidised in the following way:

$$MeS + O_2 \rightarrow MeO + SO_2\uparrow \quad (1)$$

The oxides were then subjected to sulfuric acid to obtain water-soluble metal sulfates:

$$MeO + H_2SO_4 \rightarrow MeSO_4 + H_2O \quad (2)$$

The metal ions Co^{2+}, Ni^{2+}, Zn^{2+}, Cd^{2+}, Cu^{2+}, etc. that were in the sulfate solution were selectively separable.

Stepwise pH-Controlled Selective Precipitation

The interfering metal ions were removed selectively by the addition of NaOH solution under constant stirring. At a pH of 3-4, $Fe(OH)_3$ was precipitated. When the pH was 5-6, Zinc(II) and Antimony(III) were eliminated and then Copper(II), then Cadmium(II). The precipitates were filtered after every precipitation, and the filtrate that contained increased Co^{2+} ions was stored to be processed further.

Formation of the Co(II)–NNDA Complex

In order to obtain the selective complexation of cobalt, the correct quantity of NNDA solution was introduced to the cobalt-enriched filtrate. The pH of 5.5-6.0 was measured, and the reaction was left to run for 10 minutes at room temperature (25- 30 °C). These conditions formed the Co-NNDA complex that has a stable and intensive color.

Spectrophotometric Measurements

Using a double-beam spectrophotometer, the UV-Visible spectrophotometric measurements were done for the Co–NNDA complex at 530 nm, and the measurements were compared to a reagent blank. Calibration curves were obtained using a standard cobalt solution, and to check the reproducibility of the method, repeated measurements (n = 6) were done.

Infrared and X-Ray Diffraction Analysis

FTIR spectra for the Co–NNDA complex were recorded after isolating and drying the complex. The spectra were evaluated between 4000 and 400 cm^{-1} to identify possible coordination sites. The divided cobalt species and the retrieved metallic cobalt were characterised for phase composition and crystallinity using X-ray Diffraction (XRD) analysis.

Stability and Particle Size Analysis

The zeta potential and particle size distribution of cobalt species in solution were measured using a DLS analyser. It was employed to determine the dispersion stability and reproducibility of the cobalt complex under the determined optimal conditions.

RESULTS AND DISCUSSION

Composition of Cd–Co–Ni Cake and Process Implications

X-ray fluorescence (XRF) analysis was utilised to identify the industrial Cd-Co-Ni cake sample. In the XRF spectrum of the sample shown in Figure-1, many constituents are observed. Zinc, which makes up a fair portion of the sample (36-45%), dominates the spectrum. Other elements include: cobalt (3.5-6.5%), copper (3.5-10%), cadmium (2.5-8%), nickel (2.5-4.6%), and minor constituents of antimony and iron. Very likely, the phenomenon of spectral signal attenuation superposition is just a reflection of the complexity of the sample matrix, which explains the poor performance of conventional selective separation methods in complex matrices. It is also worth noting that the Co concentration is within the economically recoverable range, thereby confirming the selective extraction approach as economically justifiable.

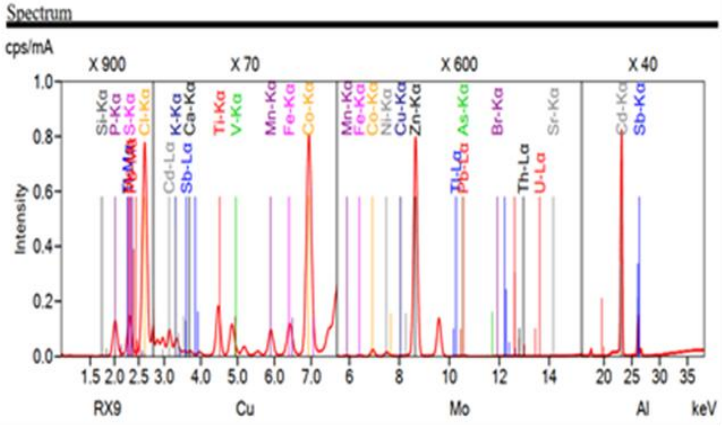


Fig.-1: X-ray fluorescence spectrum of the Cd–Co–Ni cake intermediate

Mechanism of Selective Separation

The two-stage mechanism accounts for the selectivity of the proposed method. In the first stage, oxidative sulfation treatment converts metal sulfides to soluble sulfates, enabling controlled solution chemistry. In the second stage, interfering metal ions are removed sequentially using regulated-pH hydroxide precipitation. Iron (III) ions are removed at pH 3-4, followed by zinc and antimony ions at pH 5-6, and then copper ions at pH 7-8. Cadmium ions are removed at pH 8-10. This step-wise mechanism removes the competing ions while keeping cobalt ions mostly in solution. The formation of complexes with NNDA selectively stabilises the remaining cobalt ions. In the mildly acidic to near-neutral ranges, Co(II) has a higher coordination affinity to NNDA than Ni(II) does, which explains the selectivity.

Spectrophotometric Evidence of Co(II)–NNDA Complex Formation

Spectrophotometry in the UV-Vis range was used to study the interactions between Co (II) and NNDA. A complex formed, resulting in a pronounced colour change and an absorption peak at 530 nm. Figure-2 demonstrates the most intricate parameters as the best example of building complexes. Good reproducibility (RSD less than 4.5, n = 6) and linearity were achieved in the set range of 0.5 -25 mg L-1 Co(II). These parameters prove the appropriateness of the method for applied and industrially relevant concentrations of cobalt.

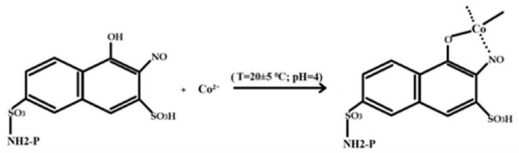


Fig.-2: Reaction Equation and Optimal Conditions for the Formation of the Co(II)–NNDA Complex

Infrared Spectroscopic Characterisation

Infrared spectroscopy proved the coordination mechanism of cobalt with NNDA. Figure-3 demonstrates the IR spectrum of the Co-NNDA complex. The loss or displacement of the -OH stretching band and the change in the typical -N=O and -C=S vibrations are evidence of coordination with oxygen and sulfur donor atoms. This spectral evidences affirm that NNDA is a multidentate ligand, which generates a stable chelate with Co(II).

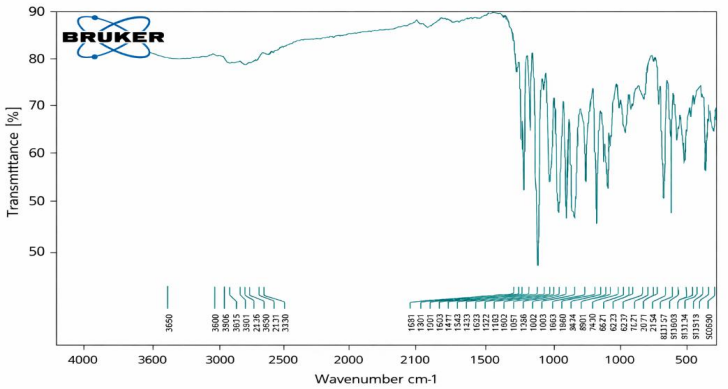


Fig.-3: IR Spectrum of the Cobalt(II)–NNDA Complex

Crystallographic Confirmation of Cobalt Species

The phase composition of the separated products containing cobalt was proved by X-ray diffraction (XRD) analysis. Figure-4 is the XRD pattern of the cobalt species after selective separation, and clearly, there are diffraction peaks of cobalt phases and no dominant peaks of zinc, copper or cadmium. This confirms the effectiveness of the interference elimination strategy.

Cobalt has been obtained not just through the processed form; cobalt was also obtained as the metallic form after extra processing. Figure-5 indicates that the reclaimed cobalt metal is highly crystalline in nature with high purity in terms of phases. This kind of crystal purity and phase purity indicates that the versatility is a result of recovery and separation of the metal.

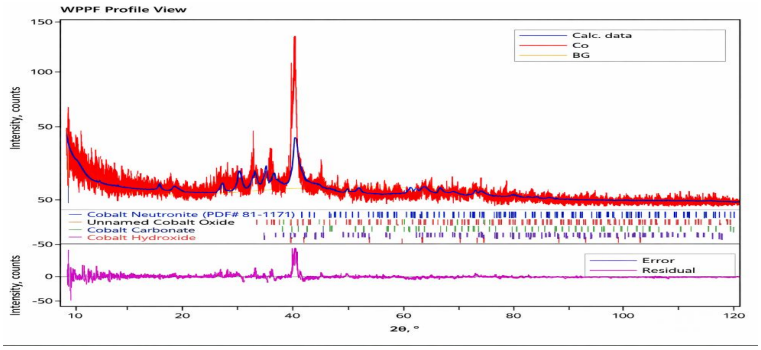


Fig.-4: XRD Pattern of the Selectively Separated Co(II) Species

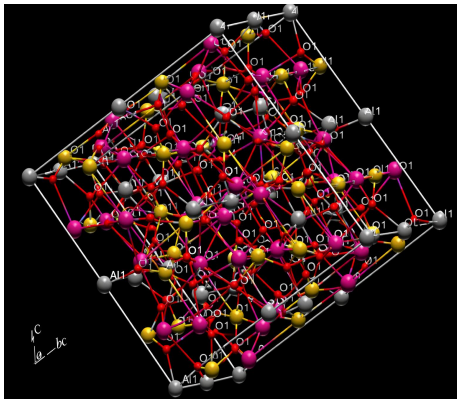


Fig.-5: XRD and Crystal Structure Analysis of Recovered Metallic Cobalt

Particle Size and Stability Analysis

The dispersion of Co species was measured through the particle size and the zeta potential of the dispersed particles. Particles with co-containing such as Figure-6 seem to have a homogeneous distribution in the nanometer range; thus, it can be concluded that the NNDA is highly stabilised and aggregated to a minimum. It is also observed that the electrostatic stabilisation evidences the reproducibility and reliability of the spectrophotometric study.

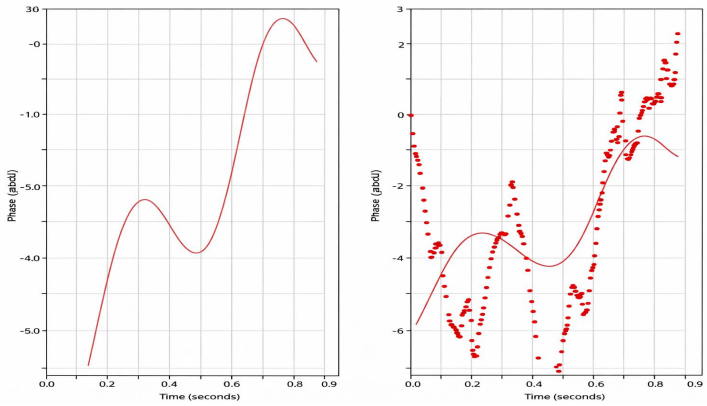


Fig.-6: Particle Size Distribution of Co (II) Species in Solution

Method Performance and Limitations

The developed procedure works best at pH 5.5-6.0 and moderate temperatures (25-30 degree C). Very large amounts of nickel might necessitate more washing or more precipitation. However, the use of organic solvents, reagents, and secondary wastes is considerably minimised, so the proposed method is in line with the green and sustainable metallurgy principles, compared to solvent extraction or ion-exchange methods.

CONCLUSION

A process for the selective recovery and separation of Co(II) in Cd-Co-Ni cake intermediates in an environmentally safe, structurally integrated manner is provided. The key findings of the scientific work of this study include the following. A discriminatory process of phase-controlled suppression the interfering ions with the help of pH, as well as the ligand-specific stabilisation of cobalt, has been outlined. The NNDA reagent was able to specifically and systematically form a complex with Co (II) in real multicomponent industrial matrices. The selectivity, stability and phase purity of the recovered cobalt was confirmed by all the spectrophotometric, IR and XRD analyses as well as particle size analysis. The method has proved to be highly reproducible, to be used with very few chemicals and shows great potential for industry. Finally, it is an addition to the significant lack of recovery technologies for the extraction of cobalt and will enable a sustainable recycling of metallurgical residues to transform the hazardous Cd-Co-Ni residues into valuable cobalt.

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CONFLICT OF INTERESTS

The authors state that there is no conflict of interest in this research.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-12: Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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