

# Parametric Investigation of Fluidized-Bed Homogeneous Granulation for Simultaneous Recovery of Copper and Nickel From Wastewater: Flow Rate Effects on Granule Formation and Applications in Human Health and Water Safety

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## Abstract

Environmental pollution, particularly due to heavy metals, is a growing global issue, with industries like electronics making a significant contribution. The uncontrolled and intensive processing of electronic waste (e-waste) has resulted in the widespread release of heavy metals into the environment, leading to elevated concentrations in nearby air, water, soils, sediments, and agriculture. Among these pollutants, metals such as copper and nickel, commonly discharged from printed circuit board (PCB) manufacturing, pose significant environmental and health threats due to their high toxicity. This investigation explores the simultaneous removal and recovery of copper (Cu<sup>2+</sup>) and nickel (Ni<sup>2+</sup>) ions from synthetic printed circuit board (PCB) wastewater through the application of a fluidized-bed homogeneous granulation technique. The research specifically examines how key operational variables—namely, the flow rate of the carbonate precipitant, pH, and carbonate ion concentration, influence both metal removal efficiency and granule formation. The synthetic effluent used in the trials contained copper and nickel ions at initial concentrations of 6.3 mM and 3.4 mM, respectively. Experimental conditions were maintained at a pH of 6.5, with a total carbonate concentration of 17 mM. Findings indicated that a precipitant flow rate of 15 mL/min was optimal for effective copper removal and granulation, whereas nickel demonstrated superior removal performance at a higher flow rate of 25 mL/min. Elevated flow rates, however, were observed to reduce granulation efficiency and promote sludge accumulation, thereby hindering the overall performance of the system. Morphological analysis via scanning electron microscopy (SEM) revealed granules with irregular surfaces and notable porosity, likely due to the concurrent precipitation of multiple metal species. The results underscore the viability of fluidized-bed granulation as a promising, sustainable, and cost-effective approach for treating heavy metal-laden industrial wastewater. Nonetheless, further refinement of process parameters is recommended to enhance metal recovery yields and granule quality.

**Keywords:** carbonate; copper; fluidized-bed; nickel; granulation

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## 1. Introduction

The world is currently grappling with significant environmental concerns like food shortages, the unsustainable use of energy resources, and pollution issues, with heavy metal contamination being one of the most pressing challenges (Arazo et al., 2016; Li et al., 2024; Mitra et al., 2022). While many heavy metals are crucial in various human activities, they are known carcinogens that do not biodegrade, are persistent in the environment, highly water-soluble, and challenging to eliminate from wastewater (Fu & Wang, 2014; Moukadiri et al., 2024; Quimada et al., 2022). Moreover, other waterborne contaminants present in natural and engineered systems exhibit similar persistence, high solubility, and resistance to conventional treatment processes, further complicating effective water purification and environmental protection efforts (Alfar et al., 2025). Heavy metal pollutants in wastewater can

originate from industrial discharges, agricultural runoff, mining activities, construction and demolition sites, household wastewater, landfills, wastewater treatment plants, and electronics production (Song & Li, 2014).

The rapid progress in information technology has resulted in a substantial rise in the production of electrical and electronic equipment (EEE). Printed circuit boards (PCBs) are essential components in virtually all EEs. However, the continuous technological progress in EEE has resulted in the obsolescence of electronic devices, consequently generating large quantities of PCB (Jadhav & Hocheng, 2015; Quimada et al., 2020, 2022, 2024). The PCB industry employs nickel and copper ions for electroplating due to their favorable corrosion resistance and thermal stability properties. Recognizing the importance of addressing this matter, there is a growing emphasis on devising and implementing effective

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strategies to eliminate heavy metals from wastewater. Industrial wastewater containing heavy metals is often treated using various cost-effective methods. Several physico-chemical technologies have been explored for removing these persistent and non-biodegradable contaminants from polluted water (Daniel et al., 2015). Commonly employed techniques to address the issue include ion exchange adsorption, biosorption, electrodialysis, membrane separation, and precipitation. While these techniques possess certain limitations, including suboptimal removal efficiency, high energy consumption, substantial maintenance requirements, generation of waste by-products, significant sludge volumes, and additional costs associated with dewatering, a more favorable method for treating wastewater containing heavy metals has emerged that utilizes granulation in a fluidized-bed reactor (FBR). The increasing need to address environmental challenges has made sustainability a focal area in various fields, including management (Javed et al., 2024; Quimada et al., 2023; Quimada, N. E. & Ogdiman, R. A., 2025). The recovery and treatment of heavy metals in fluidized-bed reactors rely on controlled granulation or crystallization processes, which have gained notable attention for their ability to achieve high removal efficiencies with minimal sludge generation. This approach supports efficient mixing of reactants, enhances the uniformity and purity of the resulting precipitates, and minimizes chemical consumption—making it both economically and environmentally favorable (C. Chen et al., 2015; J. P. Chen & Yu, 2000; Salcedo et al., 2015). Fluidized-bed systems have been widely applied to treat wastewaters contaminated with various inorganic pollutants such as nickel, copper, arsenic, zinc, fluoride, and acids including boric and phosphoric. Since their development in the 1980s, fluidized-bed crystallization technologies have been effectively employed for metal removal via carbonate precipitation, especially from effluent sources such as electroplating industries, refineries, and acid mine drainage (Lertratwattana et al., 2017; Quimada et al., 2022).

In this context, the current work evaluates the fluidized-bed homogeneous granulation method for the simultaneous treatment and recovery of copper ( $\text{Cu}^{2+}$ ) and nickel ( $\text{Ni}^{2+}$ ) ions from simulated printed circuit board (PCB) wastewater. A parametric investigation was performed to identify the optimal precipitant flow rate while maintaining constant pH and carbonate concentrations, aiming to determine its influence on metal removal efficiency. In addition, the granules formed during the process were subjected to morphological and compositional assessment using scanning electron microscopy (SEM).

## 2. Materials and Methods

In this research, synthetic wastewater simulating printed circuit board (PCB) effluent was prepared using copper chloride ( $\text{CuCl}_2$ ) and nickel sulfate ( $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ), both procured from Shikmayu's Pure Chemicals with a 99% purity. Sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), supplied by

PanReac with a 98% purity, was used as the precipitant for the recovery of metal ions. To regulate the pH in the reaction zone, small amounts of sodium hydroxide ( $\text{NaOH}$ , 97%, from Sigma-Aldrich) or nitric acid ( $\text{HNO}_3$ , 90%, from Sigma-Aldrich) were added. All chemicals were of reagent grade and used as received, without additional purification. Deionized (DI) water, produced using a Millipore Milli-Q system with a resistance greater than 18.  $\text{M}\Omega$ , was employed for preparing all solutions.

The experimental setup, shown in Figure 1, consisted of a cylindrical laboratory-grade Pyrex glass reactor with a 450 mL capacity. The reactor was divided into two regions: a reaction zone with a 2 cm diameter and 80 cm height, and an effluent zone with a 4 cm diameter and 20 cm height. The abrupt expansion between these regions was designed to reduce hydraulic loading and prevent small particles from draining into the effluent area. Three peristaltic pumps were used to control the upward flow of synthetic PCB wastewater, the carbonate precipitant solution, and the recirculating fluid into the fluidized bed reactor (FBR), allowing for precise regulation of the recirculation flow rate in the lower section of the reactor. To support the granulation bed and ensure uniform flow distribution, a layer of glass beads (0.5 cm in diameter) was packed to a height of 4 cm at the reactor's base. A pH/ORP meter (Suntex), with an accuracy of  $\pm 0.05$ , was used to monitor the pH.

The initial concentrations of copper and nickel ions in the synthetic wastewater were set at 6.3 mM and 3.4 mM, respectively, which are typical concentrations found in PCB wastewater. The pH of the carbonate precipitant solution was adjusted to 6.5 using  $\text{NaOH}$  or  $\text{HNO}_3$  before injection into the FBR. The experiment was carried out at ambient room temperature ( $25^\circ\text{C}$ ) for 14 days. The total carbonate loading was maintained at a constant value of 17 mM, and the flow rates of both solutions were varied (10, 15, 20, 25, and 30  $\text{mL}\cdot\text{min}^{-1}$ ) to determine the optimal flow rate. The initial recirculation flow rate was set at 20  $\text{mL}\cdot\text{min}^{-1}$  and later adjusted up to 100  $\text{mL}\cdot\text{min}^{-1}$  as needed. The inflow rates were manipulated to achieve the desired hydraulic retention time (HRT), thereby increasing the residence time of copper and nickel carbonates within the reactor. During the reaction, small nuclei formed in a supersaturated state and aggregated to form larger granules through collisions and bonding. The continuous interaction between copper and nickel ions with the carbonate precipitant resulted in sufficient nucleus clustering, leading to granulation. Fine granules began to form within 2–3 days, and they became fluidized due to continuous collisions at the bottom of the reactor. The effluent pH was recorded at the end of each experimental run. The granules formed were extracted from the reactor via an outlet fitted with glass beads, rinsed with deionized (DI) water, air-dried, stored at  $4^\circ\text{C}$ , and later characterized for further analysis. Additionally, the competition between copper and nickel carbonates during granule formation was studied, with effluent samples taken at regular intervals throughout the experiment. All experiments were performed in

triplicate to ensure the accuracy and reliability of the results.

The initial concentration of copper ions in the synthetic wastewater was established at 6.3 mM, while nickel ions were set at 3.4 mM, reflecting actual concentrations in PCB wastewater. The pH of the carbonate precipitant solution was adjusted via the addition of NaOH or HNO<sub>3</sub> prior to injection into the FBR and maintained at 6.5 pH. The experiment was conducted at ambient room temperature (25°C) over a total duration of 14 days. The total carbonate loading was maintained at a constant value of 17 mM, while the precipitant flow rates for both solutions were varied to 10, 15, 20, 25, and 30 mL·min<sup>-1</sup> to determine the optimal flow rate. The initial recirculation flow rate was set at 20 mL·min<sup>-1</sup> and later modulated up to 100 mL·min<sup>-1</sup> as necessary. The inflow rates were adjusted to achieve the desired hydraulic retention time (HRT), enhancing the residence time of copper and nickel carbonates within the reactor.

During the reaction, small nuclei formed in a supersaturated state and aggregated to create larger granules as they collided and bonded. The continuous reaction of copper and nickel ions with the carbonate precipitant facilitated sufficient nucleus clustering, resulting in granule formation. Within 2–3 days, the emergence of fine granules was noticeable, and the granules became fluidized through continuous collisions at the reactor's bottom. The effluent pH was recorded at the conclusion of each experimental run. The formed granules were subsequently extracted from the reactor via the outlet equipped with glass beads, rinsed with DI water, air-dried, stored at 4°C, and characterized for further analysis. Additionally, the competition between copper and nickel carbonates in granule formation was assessed, with effluent samples taken at predetermined intervals throughout the study. All experiments were conducted in triplicate to ensure accuracy and reproducibility of results.

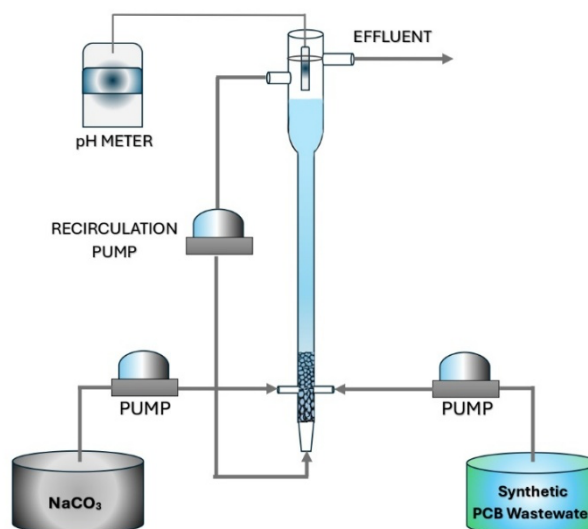


Figure 1. Fluidized-bed Reactor Set-up

### 3. Results and Discussion

#### Effects of precipitant flow rate

The recovery of Cu<sup>2+</sup> and Ni<sup>2+</sup> was achieved through a fluidized-bed homogeneous granulation process. The study investigated the effects of varying the precipitant flow rate while maintaining consistent carbonate loading and pH values to identify the optimal conditions for the precipitant flow rate. The majority of the reaction occurred in the lower section of the fluidized-bed reactor, where the reactant feed was situated. The introduction points for the reactants were positioned on opposite sides of the reactor, facilitating mixing through the adjustment of the recirculation flow rate and the

regulation of the peristaltic pump's speed within the recirculation stream.

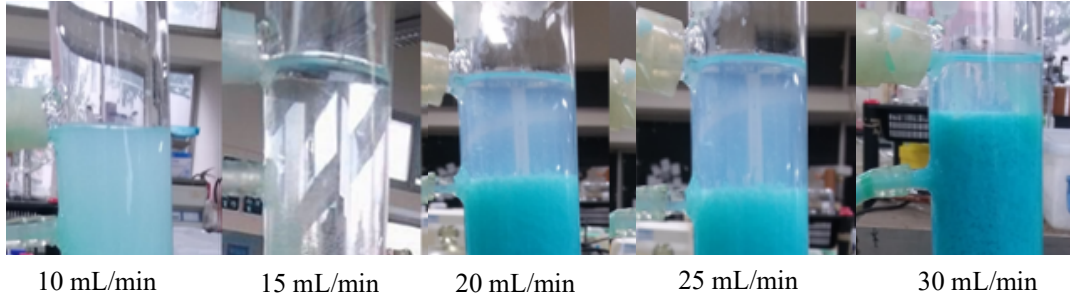
In this study, experiments were conducted to examine the effect of precipitant flow rate by varying the flow rates (Q<sub>CO<sub>3</sub></sub>) to 10, 15, 20, 25, and 30 mL/min. The copper concentration was kept constant at 6.3 mM, nickel concentration at 3.4 mM, carbonate concentration at 17 mM, and pH was maintained at 6.5. For each experimental run, the flow rate of the wastewater entering the reactor was kept the same as the precipitant flow rate. A summary of the experimental conditions is provided in Table 1.

Table 1. Summary of the influent flow rate and operational conditions.

Q <sub>CO<sub>3</sub></sub> (ml/min)	Q <sub>Cu</sub> (ml/min)	Initial pH	[Cu <sup>2+</sup> ] (mM)	[Ni <sup>2+</sup> ] (mM)	[CO <sub>3</sub> <sup>2-</sup> ] (mM)
10	10	6.5±0.2	6.3	3.4	17

15	15
20	20
25	25
30	30

At elevated flow rates, the hydraulic retention time decreased, resulting in an increase in the cross-sectional loading of metal ions. The results indicated that using a high precipitant flow rate was not suitable for copper recovery. As demonstrated in Figure 2 below, a high influent flow rate generated a significant amount of sludge in the upper section of the reactor.



**Figure 2.** The condition of the reactor during the treatment process with varying flow rates

Figure 3 illustrates the impact of the precipitant flow rate on copper removal and granulation. The results demonstrate that both copper removal and granulation are more favorable at lower precipitant flow rates compared to higher influent flow rates. When a higher carbonate flow rate (30 mL/min) was applied, copper removal decreased to 97.98%, and copper granulation dropped to 94.15%, representing reductions of 8.34% and 18.70%, respectively.

As depicted in Fig. 4, varying the carbonate flow rate from 10 mL/min to 30 mL/min significantly influenced the removal and granulation efficiencies of copper, respectively. At carbonate flow rates of 10 mL/min and 15 mL/min, the copper granulation efficiencies were 94.15% and 93.88%, respectively, indicating only a 0.28% difference in granulation efficiency. However, when the flow rate was increased to 20 mL/min, the granulation efficiency declined to 8.13%.

The experimental results indicate that operating at high influent flow rates leads to the production of a substantial volume of sludge in the upper section of the reactor, which significantly reduces copper granulation efficiency [19]. Despite the decrease in granulation, copper removal remained high since the sludge

produced during filtration was unable to pass through the syringe filter, resulting in less than a 5% difference across each precipitant flow rate condition.

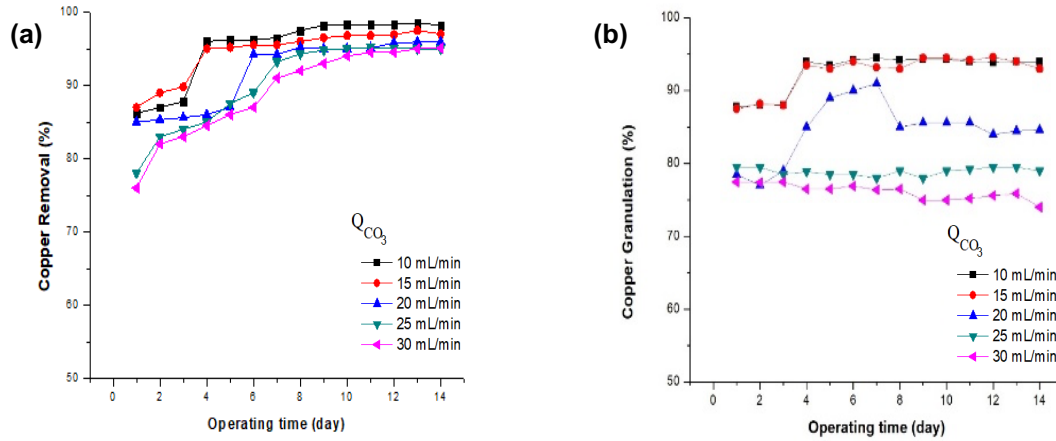
As mentioned earlier, increasing the influent flow rate leads to a reduction in hydraulic retention time (HRT), which was calculated using the formula provided in Equation 1 below. The results of these calculations are summarized in Table 2. A shorter HRT limits the collision and aggregation of small nuclei, which are essential for the nucleation and growth of granules. As a result, sludge forms and accumulates in the upper section of the fluidized-bed reactor (FBR). The reduced HRT also negatively impacts copper granulation efficiency, as many fine particles are carried away in the effluent before they have the chance to coalesce into larger granules.

$$HRT = \frac{V}{Q_T}$$

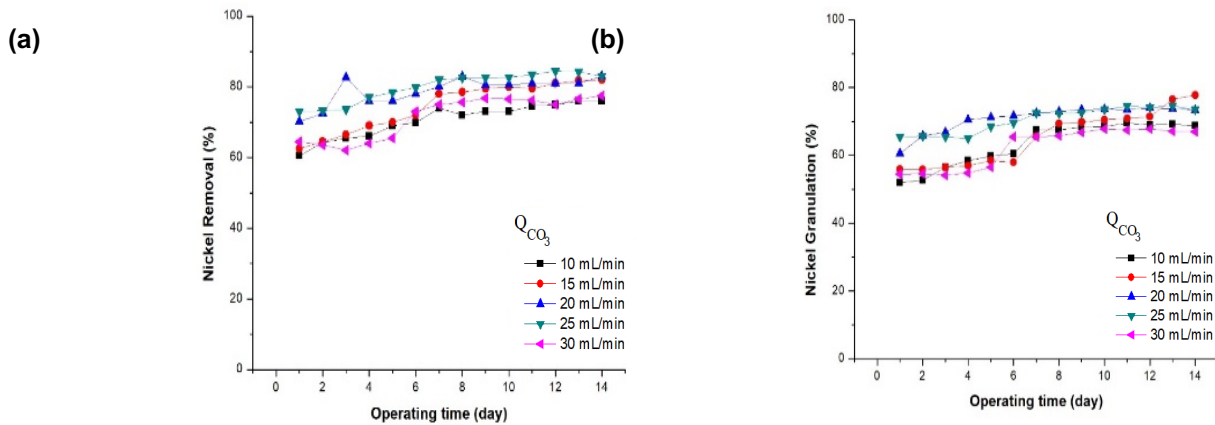
Where; HRT is a hydraulic retention time, min  
 V is a volume of reactor, = 450 ml  
 Q<sub>T</sub> is a total influent flow rate, ml/min

**Table 2.** Computation of Hydraulic Retention time at Every Influent Flow Rate

Q <sub>CO<sub>3</sub></sub> (ml/min)	Q <sub>Cu+Ni</sub> (ml/min)	HRT (min)
10	10	22.5
15	15	15
20	20	11.25
25	25	9
30	30	7.5



**Figure 3.** Efficiency over operating time at varying precipitant flow rate on (a) copper removal and (b) copper granulation.



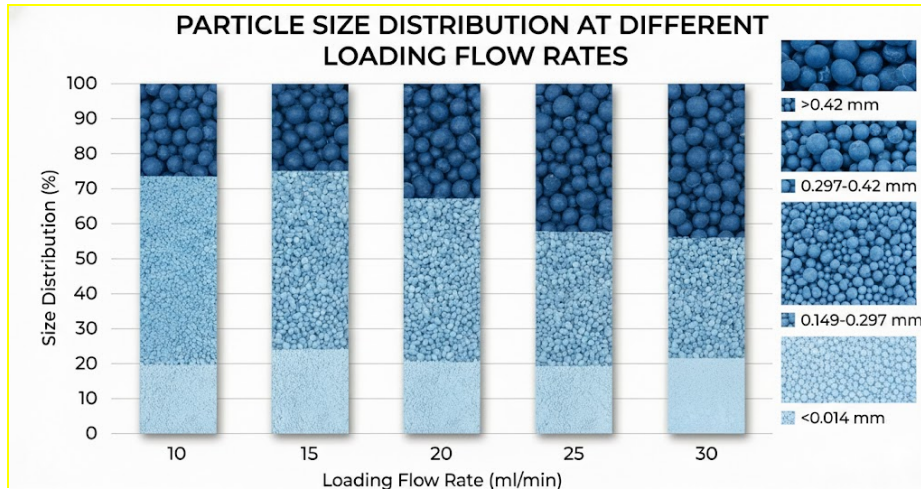
**Figure 4.** Efficiency over operating time at varying precipitant flow rate on (a) nickel removal and (b) nickel granulation.

In contrast to the observations for copper ions, the removal and granulation efficiencies of nickel ions were highest at a precipitant flow rate of 25 mL/min. As depicted in Fig. 4, both parameters showed an increasing trend from 10 to 25 mL/min before declining at 30 mL/min. These findings highlight the significant influence of the influent flow rate ( $Q_i$ ) on the removal and granulation efficiencies of both nickel and copper ions. Notably, the optimal precipitant flow rates for achieving maximum efficiency vary between these ions.

For nickel, the highest recorded efficiencies were 83.10% for removal and 73.46% for granulation, both occurring at a flow rate of 25 mL/min.

#### *Effect of the Precipitant Flow Rate on Size Distribution*

Figure 5 shows that increasing influent flow rate produces finer granules and the lowering of the recovery of larger granules. As mentioned, the reactions hindered nucleation and growth because of the short HRT.



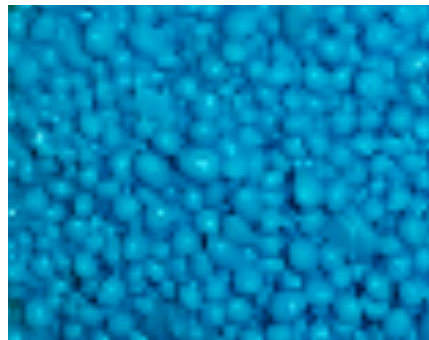
**Figure 5.** Effect of the Precipitant Flow Rate on Size Distribution

Research on copper removal in a fluidized-bed reactor has shown that as hydraulic loading increases, copper removal efficiency declines. This reduction is primarily due to granule erosion within the fluidized bed and increased porosity, which facilitates the formation of micro-particles within the water phase among fluidized grains (Lee, 2004). Likewise, a study by Chen and Yu (J. P. Chen & Yu, 2000) on lead removal using a fluidized bed with sand crystal seeding found that higher recirculation ratios led to the washout of precipitates initially attached to the grain surfaces. This was attributed to heightened turbulence, which subsequently increased effluent concentrations (J. P. Chen & Yu, 2000). These observations align with the behavior of copper ion removal and granulation. However, for nickel

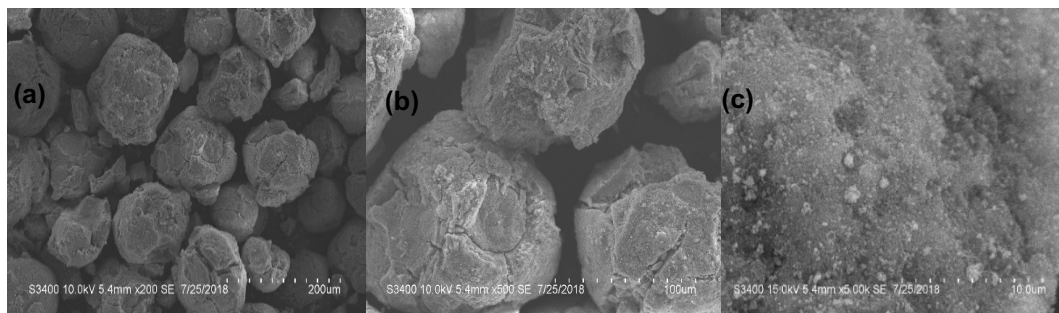
ion removal and granulation, a higher influent flow rate appears to be more effective. To achieve optimal efficiencies, a flow rate of 25 mL/min is required (Quimada et al., 2022; Salcedo et al., 2015).

**Characterization of the Formed Granules**

After the operation time of 14 days, the produced granules in the condition of pH - 6.5,  $\text{Cu}^{2+}$  - 6.3 mM,  $\text{Ni}^{2+}$  - 3.4,  $\text{CO}_3^{-2}$  - 17 mM, and precipitant flow rate of 15 mL/min were analyzed to determine their morphology. As illustrated in Fig. 5, the granules exhibited varying particle sizes, which is further evident in the actual image of the collected granules presented in Fig. 6. The granules having the largest particle size were taken and subjected to physical characterization.



**Figure 6.** Granules produced after applying the optimal removal flow rate from wastewater containing copper and nickel ions



**Figure 7.** SEM images showing the morphology of the granules produced in FBR at different magnification (a) 200x Magnification (b) 500x Magnification (c) 5,000x Magnification

Figure 7 displays SEM images of granules formed under specific conditions, including an initial pH of 6.5, a total carbonate concentration of 17 mM, a carbonate flow rate of 15 mL/min, and initial concentrations of 6.3 mM for copper and 3.4 mM for nickel. Initially, these granules formed as small particles, which grew progressively through collisions with other particles. Over time, these smaller particles continued to cluster together, leading to further growth and granulation. As illustrated in Figure 7, the granule surfaces were not smooth, likely due to the presence of multiple metal ions in the synthetic wastewater, which contributed to surface roughness (Shimizu & Hirasawa, 2013). Additionally, after air-drying, some granules exhibited cracks and a hollow structure, which may have resulted from friction and attrition between particles as evident in Fig. 7(b). Under higher magnification, the granules appeared structurally weakened, mainly due to their porosity. This occurrence can be theoretically linked to the diffusion of carbonate ions from within the particles (Aldaco et al., 2006).

This study investigated the removal of copper ( $\text{Cu}^{2+}$ ) and nickel ( $\text{Ni}^{2+}$ ) ions from synthetic wastewater representative of printed circuit board (PCB) effluents through a fluidized-bed homogeneous granulation system. Experimental findings emphasized the significance of operational variables, particularly the flow rate of the carbonate precipitant, in determining both metal recovery performance and granule development. The optimum conditions for copper granulation were observed at a flow rate of 15 mL/min, while nickel removal reached its maximum efficiency at 25 mL/min. Exceeding these flow rates resulted in diminished granulation effectiveness and increased sludge generation, highlighting the need to regulate hydraulic retention time (HRT) to maintain process stability.

The results reveal that at higher precipitant flow rates, the HRT decreases, limiting the collision and aggregation of small nuclei essential for nucleation and granule growth. This led to sludge formation in the upper section of the reactor, reducing granulation efficiency. Although copper removal remained high across all flow rates (less than 5% variation), granulation efficiency significantly declined due to shortened HRT and particle washout. These findings are consistent with previous research showing that increasing hydraulic loading in fluidized-bed reactors reduces copper removal efficiency due to granule erosion and higher bed porosity, which enhances micro-particle formation (Lee, 2004). Similarly, Chen and Yu observed that higher recirculation ratios in lead removal systems led to the washout of precipitates because of intensified turbulence, which increased effluent concentrations.

For nickel ions, the opposite trend was observed—higher influent flow rates enhanced both removal and granulation up to the optimal rate of 25 mL/min. This behavior can be attributed to nickel's distinct precipitation kinetics and its interaction with carbonate ions under fluidized conditions (Quimada, et. al, 2022; Salcedo et al., 2015). However, beyond this point, excessive turbulence disrupted the stability of forming

granules, leading to decreased efficiency. These results underscore the critical role of influent flow rate and HRT in governing metal ion behavior within the reactor.

Morphological characterization through SEM analysis revealed that granules formed under optimal conditions (15 mL/min for  $\text{Cu}^{2+}$  and 25 mL/min for  $\text{Ni}^{2+}$ ) displayed rough and porous surfaces. The surface roughness likely resulted from the coexistence of multiple metal ions in solution, while porosity was associated with limited carbonate diffusion and particle abrasion during collisions (Shimizu & Hirasawa, 2013; Aldaco et al., 2006). Cracks and hollow structures observed in dried samples further suggest internal stresses and attrition among particles during operation. Such structural inconsistencies may influence granule integrity and mechanical stability, which are critical for sustained reactor performance.

Overall, the results affirm the potential of fluidized-bed granulation as an effective strategy for the simultaneous removal and recovery of copper and nickel from complex industrial wastewater streams. The process offers advantages such as low chemical consumption, reduced sludge production, and the formation of reusable granules. Nevertheless, careful optimization of operational parameters—including flow dynamics, hydraulic retention time, and recirculation ratio—is essential to enhance granule integrity, minimize sludge formation, and improve overall treatment efficiency.

#### 4. Conclusions

The experimental data indicates that precipitant flow rate acts as a primary determinant for removal efficiency, granulation behavior, and final granule morphology within a fluidized-bed homogeneous granulation reactor. When treating synthetic PCB wastewater containing  $\text{Cu}^{2+}$  and  $\text{Ni}^{2+}$  ions, it was observed that the hydraulic environment—specifically hydraulic retention time (HRT)—directly dictates the interplay between nucleation kinetics and particle aggregation.

Copper ions demonstrated a distinct preference for lower precipitant flow rates; 15 mL/min emerged as the optimal threshold for maximizing both removal and granulation. Advancing the flow rate beyond this point triggered a decline in granulation performance, likely a consequence of shortened HRT and heightened turbulence. These conditions facilitate the washout of fine nuclei, subsequently increasing sludge buildup in the reactor's upper zones. Interestingly, while granule formation was sensitive to these hydraulic changes, copper removal percentages remained robust, suggesting that the chemical precipitation of copper is less vulnerable to hydraulic loading than the physical aggregation process.

Nickel removal followed a contrasting trajectory, where higher flow rates actually enhanced performance, peaking at 25 mL/min. This divergence underscores the unique precipitation kinetics of nickel-carbonate species under fluidized conditions. Nevertheless, a "tipping point" was observed where excessive turbulence

eventually destabilized granule formation, regardless of the metal species involved.

Analyses of particle size distributions verified that increased influent flow rates correlate with a shift toward finer particle populations, effectively capping the growth of larger, mechanically resilient granules. Morphological assessment via SEM supported these findings; granules produced under peak conditions displayed rough, porous surfaces. This texture is likely a byproduct of multi-metal precipitation and carbonate diffusion constraints, coupled with mechanical abrasion during inter-particle collisions. The observation of internal voids and surface fissuring in dried samples points toward significant internal stresses and attrition, factors that could impact the structural longevity of the granules in industrial settings.

Ultimately, these results confirm the utility of fluidized-bed systems for dual-metal recovery. However, the study makes it clear that system stability hinges on a precise equilibrium between flow velocity and retention time. To scale this technology for industrial wastewater applications, operators must prioritize hydraulic control to balance high recovery rates with granule durability and minimal sludge production.

## 5. Acknowledgements

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## 6. Author's Contributions

Nathaniel E. Quimada conducted the investigation, interpreted the results, and was responsible for validation, as well as writing and editing the manuscript.

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## 7. Conflicts of Interest

The authors declare that they have no competing interests.

## 8. AI declaration (if any)

Artificial intelligence (AI) was used solely as a rephrasing and language-editing tool to refine wording and improve English grammar and sentence construction. The intellectual content, analysis, and conclusions of the work remain entirely the authors' own.

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