PRECIPITATION OF NICKEL FROM SYNTHETIC WASTEWATER USING MODIFIED WASTE GLASS

ZAMEN KARM1,*, SINAN S. HAMDI2, ZAINAB MOHAMMED SELLAB3

¹Department of Production Engineering and Metallurgy,
University of Technology, Baghdad, Iraq

²Nanotechnology and Advanced Materials Research Center,
University of Technology, Baghdad, Iraq

³Department of Materials Engineering, University of Technology, Baghdad, Iraq

*Corresponding Author: Zamen.K.mekhelf@uotechnology.edu.iq

Abstract

A feasible new technique has been developed to extract nickel ions from synthetic industrial wastewater. This study successfully modified waste glass containing silica, sodium, magnesium, and calcium oxides using an acidification approach to develop and evaluate it as an effective precipitating agent for heavy metal ion recovery. The waste glass was activated using 1 M hydrochloric acid at different times of 5, 10, 15, and 20 h. The morphology and physiochemical characterization of modified waste glass (MWG) were ascertained by X-ray diffraction, Fourier transform infrared and scanning electron microscopy. Atomic absorption spectroscopy was used to estimate the precipitation efficiency of metal nickel ions as a function of MWG particle size with varying immersion times. The laboratory findings revealed that the prepared participating agent effectively removed metal nickel ions from wastewater through a precipitation technique at alkaline conditions. The highest precipitation efficiency of metal nickel ions was observed with a fine MWG particle size of 87 µm by about (99 %), compared to other particle sizes. These results indicate that MWG has promising performance for precipitating heavy metal ions and maintains the alkalinity of aquatic environments.

Keywords: Chemical leaching, Chemical precipitation, Nickel (ll), Wastewater, Waste glass.

1. Introduction

The depletion of non-renewable resources and raw materials has become a major global concern in recent years because of their strong correlation with detrimental environmental effects and primary energy consumption. Moreover, there has been an upsurge in concerns regarding the waste disposal and recycling shortage of massive quantities of solid and liquid material debris. In this respect, many researchers have been encouraged to find an effective way to economically use waste materials and reduce environmental pollution.

Waste glass forms over 10.9 thousand tons of solid waste, of which 39.7 tons are derived from municipal solid waste [1, 2]. However, silica is composed of two naturally occurring elements, and their major constituents, oxygen and silicon, form over 90% of the Earth's crust. The wide range of applications of silica and products containing silica demonstrate the ubiquity of this material in daily life, and silica can be upgraded to improve its applications and make it more environmentally friendly [3].

The recycled waste glass was used as a low-cost [4, 5], naturally available adsorbent material to reduce the use of synthetic adsorbents that cause pollution during the synthesis process to adsorb Cd (II), Cu (II), and Pb (II) from contaminated mediums [6, 7]. Acid pretreatment is essential for synthesizing amorphous silica with high activity and specific surface area [4]. Fundamentally, several techniques have been employed to remove Ni ions from wastewater, including solvent extraction [8-10], electrolysis [11], ultrafiltration [12], codeposition [13-15], and electrooxidation flocculants [16].

Despite the beneficial performance of these processes in waste treatment, their technical, environmental, and economic feasibility has restricted their use at the field scale. This is mainly due to waste sludge, which is enriched with heavy metals and is deemed highly toxic and unsafe to the environment [17, 18]. Additional expenses are incurred during the disposal of these materials [19, 20]. Recently, precipitation and flocculation have been introduced as a low-cost and environmentally friendly technique for removing Ni ions from wastewater [2-23].

Many studies have reported conventional precipitation approaches that utilize chemicals to facilitate the removal of heavy metals. However, handling sludge from precipitation products still suffers from restrictions, similar to the previously mentioned conventional methods [2, 5]. To address these challenges, a few researchers have attempted to develop eco-friendly precipitating agents and improve the removal efficiency of heavy metals. Kostrzewa et al. [24] reported that the precipitation efficiency of heavy metal ions was enhanced by augmenting the pH of the real medium up to pH = 5 through a hydrometallurgical process. They also claimed that the best removal of heavy-metal Ni ions was approximately 56% with 30% NaOH chemical additives.

Wang et al. [25] introduced the chemical participant of developed silica xerogel for recovering the heavy metal in wastewater. The synthesized participant agent exhibited promising removal efficiency of Ni metal (99.65%) beyond 72 h in an alkaline pH range (10-11). The main objective of the present study is to synthesize a new and economical precipitant agent through a simple and direct method that can effectively recover heavy metal Ni ions from wastewater while maintaining a consistently high pH level.

2. Materials and Methods

2.1. Sample collecting

Glass bottle waste was collected from household discards. Initially, the glass bottles were washed multiple times with water to remove soluble impurities. The bottles were then crushed and ground into a powder, and sieved to obtain a mesh-sized powder ($\pm 200 \, \mu m$). Subsequently, the waste glass powder was washed and exposed to an air drier in a dry oven at $\pm 100 \, ^{\circ} C$ for 12 h and stored for later use. The preparation process of modified waste glass powder (MWG) was used to formulate a precipitation agent from waste glass, as presented in Fig. 1.

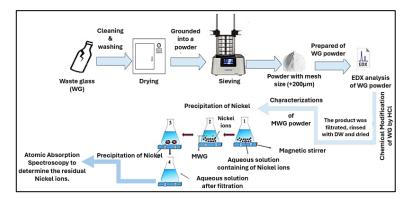


Fig. 1. Schematic diagram of preparation precipitant agent from waste glass.

The elemental composition of the harvested waste glass (WG) was analyzed using energy-dispersive X-ray spectroscopy (EDX), as shown in Fig. 2.

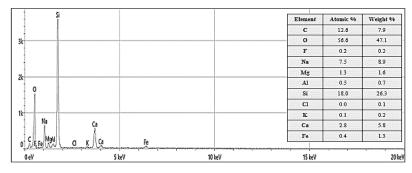


Fig. 2. EDX analysis of waste glass.

2.2. Chemical modification of the waste glass

The modification process of waste glass was modified by an effortless and direct method through immersion with 3 g of prepared waste glass powder in 600 ml of 1.5 M hydrochloric acid (HCl). The immersed samples were stirred at 160 rpm for 5, 10, 15, or 20 h. The glass waste containers with acidic solutions were filtered and rinsed with deionized water until the pH of the water became (pH = 7) to remove the modified WGP from the solution. The final samples were filtered and dried in an oven at 100 °C for 1 h to dehydrate and acquire modified waste glass

powder. The developed samples were defined based on their immersion time in an acidic solution, as illustrated in Table 1.

Table 1. Samples of waste glass were used in this study.

No.	Time of immersion in 1.5 M HCl (hours)	
Sample 1	5	
Sample 2	10	
Sample 3	15	
Sample 4	20	

2.3. Characterization of modified waste glass (MWG)

The MWG samples were analyzed using morphological, physical, and chemical techniques. The glass samples were characterized by X-ray diffraction (XRD) using (XRD 6000 Shimadzu (Japan), and the XRD patterns were obtained using an angle diffractometer 2θ in the range ($10\text{-}80^\circ$) at room temperature with Cu K α radiation. The chemical moieties on the glass surface were analyzed using Fourier transform infrared spectroscopy (Bruker ALPHA FTIR, Germany) in the wavenumber range ($4000\text{-}400\text{ cm}^{-1}$).

The surface morphologies of the MWG samples were investigated using scanning electron microscopy (SEM), type TESCAN, Czech Republic. The research's methodological process has used an image processing program (ImageJ) to determine the average particle diameter of the modified glass powder after the modification process.

2.4. Precipitation of nickel from synthetic industrial wastewater

Nickel sulfate (NiSO₄· $6H_2O$) in this research work has been dissolved in distilled water to produce synthetic industrial wastewater. The Ni ions concentration in the synthetic wastewater was 100 mg/L. After this process, different particle sizes of 3 g of activated MWG were incorporated into the solution. Further, the resulting mixture was agitated for over 50 h at pH 7.5 and room temperature. An atomic absorption spectroscopy has been used to periodically determine the residual nickel concentration in the solution, and the following formula (1) was used for computing the precipitation efficiency (PE) [26]:

$$PE = C_0 + Cf \tag{1}$$

3. Results and Discussions

The study has identified XRD as a method for examining the crystal structures of various materials. Also, X-ray diffraction is used to determine the atomic arrangement of the sample and the diffraction pattern. This method is also applied in other fields of science, such as chemistry and geology, to identify and characterize crystalline materials. Each MWG sample was investigated by XRD analysis at the Nanotechnology and Advanced Materials Research Center, Iraq.

The presence of SiO_2 was indicated by a distinct peak at $2\theta = 22^\circ$. In addition, the wide range of peaks between $20\text{-}30^\circ$ indicates silicates, sodium silicate, calcium, and other impurities, and these materials are the original components of waste glass in the XRD measurements of all the specimens, as illustrated in Fig. 3.

Moreover, the convexity of the peak in the same range implies that the powder is amorphous, without any characteristic peaks related to the crystalline structure [27]. Interestingly, the sharpness of this peak decreased with increasing acid immersion time for the waste glass powder. These observations confirmed the successful modification of the glass surface with chemical ions during the immersion process in the HCl solution.

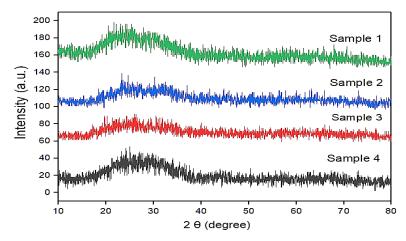


Fig. 3. XRD of MWG samples with different immersed times in 1.5 M HCl.

The FTIR spectra of MWG powder via acid-leached at 5, 10, 15, and 20 hours, as depicted in Fig. 4. The peaks observed at 471.21,471.03,469.73, and 439.16 cm⁻¹ are related to Si-O-Si bending vibrations. The stretching vibrations of Si-O-Al is represented by the peaks at 660.30 cm⁻¹ [5]. The peaks ascribed to Si-O-Si asymmetric stretching at 1,027.37 cm⁻¹ and 1,001.58 cm⁻¹. The hydroxyl group (-OH) from the silanol (Si-OH) or adsorbed water molecule trapped within the silicate structure of the waste glass particles appeared at the peaks 3,725.22, 3,565.88, 3,820.43, and 3,718.27 cm⁻¹, respectively [25].

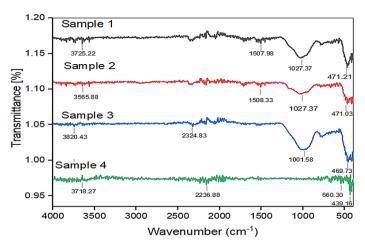


Fig. 4. FTIR of MWG samples in an acid solution of HCl as a function of immersion time.

The presence of silicates, carbon, metal oxides, and metal halides in the waste glass particles was generally confirmed by the different peaks in the spectra [5]. It is worth noting that the intensity of peaks at 1,027.37 and 1,001.58 cm⁻¹ decreased with increasing the immersion time in the HCl solution. These functional groups might be attributed to the silanol groups and adsorbed water molecules through precipitation, electrostatic interaction, and other processes. Also, all the details of the peaks are mentioned in Table 2.

		-	-
No.	Wavenumber (cm ⁻¹)	Appearance	The assignment of the functional group
1	439.16	medium	Si-O-Si bending
2	469.73	medium	Si-O-Si bending
3	471.03	medium	Si-O-Si bending
4	471.21	medium	Si-O-Si bending
5	660.30	strong	Si-O-Al stretching
6	1001.58	strong	S=O stretching
7	1027.37	strong	S=O stretching
8	1507.98	strong	N-O stretching
9	1508.33	strong	N-O stretching
10	2236.88	weak	C-H bending
11	2324.38	strong	O=C=O stretching
12	3565.88	strong	O-H stretching
13	3718.27	medium	O-H stretching
14	3725.22	medium	O-H stretching
15	3820.43	medium	O-H stretching

Table 2. FTIR analysis of MWG samples.

Figures 5 and 6 show the SEM images and particle size distribution of the glass powder after different activation times using a chemical solution of HCl. The morphology of the developed glass particles was somewhat similar to that of flakes or shards, which were reduced with extended immersion times in the acidic solution; these results agree with [28]. The mean MWG particle diameter was determined from the microstructure images of the SEM analysis, which revealed the average diameter value of all the glass powder particles in the range of 180-81 μm , as presented by the particle size distribution in Fig. 6.

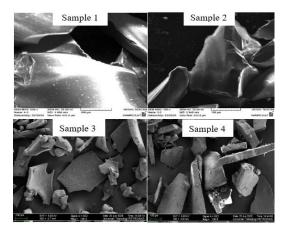


Fig. 5. SEM of prepared MWG samples.

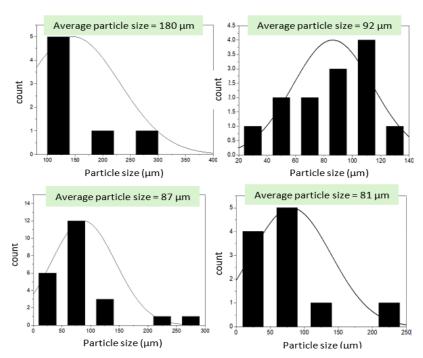


Fig. 6. Particle size distribution of prepared MWG samples.

The results of the precipitation efficiency of the heavy metal Ni and the final pH as a function of the precipitation time at different particle sizes of the precipitant agent, using 3 g of the synthesized activated waste glass samples in a nickel-containing solution at a concentration of 100 mg/L. However, the nickel removal efficiency increased dramatically in almost all MWG additive samples versus the variation in the precipitation time, as shown in Figs 7-10.

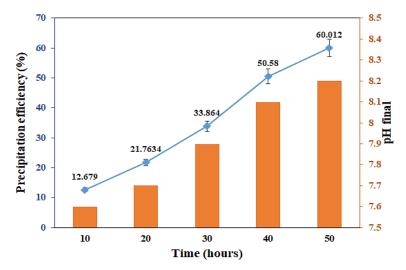


Fig. 7. The precipitation efficiency of MWG and final pH with particle size 180 μm as a function of time.

Journal of Engineering Science and Technology

June 2025, Vol. 20(3)

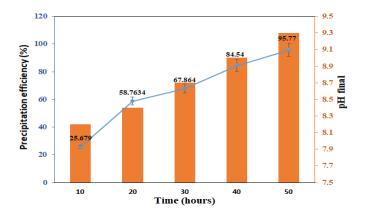


Fig. 8. The precipitation efficiency of MWG and final pH with particle size 92 μ m as a function of time.

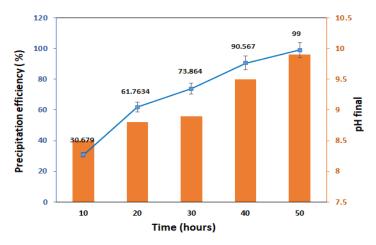


Fig. 9. The precipitation efficiency of MWG and final pH with particle size 87 μ m as a function of time.

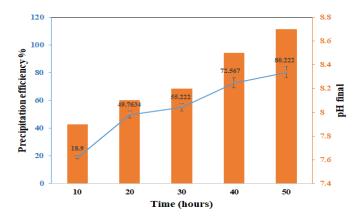


Fig. 10. The precipitation efficiency of MWG and final pH with particle size 81 μm as a function of time.

Journal of Engineering Science and Technology

June 2025, Vol. 20(3)

Figures 7-10 show that the efficiency of nickel removal was progressively enhanced by 18.9, 25.6%, 30.679, and 12.7% with MWG precipitant particle sizes of 180 μ m, 92 μ m, 87 μ m, and 81 μ m, respectively, after 10 h under ambient conditions. The results of chemical precipitation indicated that the precipitation efficiency of heavy metal Ni ions increased with a decrease in the particle size of the developed precipitants, which is related to the high surface area of the fine particles. However, the highest precipitation efficiency (99 %) was obtained using a particle size of 87 μ m of MWS.

In this study, nickel ions were deposited under conditions at a pH of 7.5. We noticed that after adding the concentration of the precipitated material from the modified glass waste, the pH increased after 10 h to 8.2, 9.3, 9.9, and 8.7 at particle sizes of 180 μm , 92 μm , 87 μm , and 81 μm at 50 h under ambient conditions, respectively; these results are in agreement with [29]. Meanwhile, the modified glass waste under alkaline pH and the abundance of hydroxyl groups (-OH) led to ionization of the glass surface.

These groups act as heavy metal binding agents and enhance precipitation efficiency. In comparison, the efficiency of heavy metals' chemical precipitation declined with a finer particle size of the developed precipitant at 81 μm . This behaviour is ascribed to the low absorption capacity, even with the fine particles, due to the loss of active functional groups, such as silanol groups, during the acidic treatment. This finding corresponded with FTIR analysis results and affirmed that functional groups on the surface-modified glass play a significant role in enhancing precipitation efficiency.

4. Conclusions

In this study, low-cost precipitant agents were synthesized from glass waste to find a suitable green environmental solution focusing on sustainable aspects and green chemistry. Waste glass has been successfully modified through an eco-friendly process (acidification process). We can summarize the results as follows:

- XRD analysis of MWG samples indicates the existence of silicates, sodium silicate, and calcium, which are the main components of waste glass.
- The XRD analysis findings are confirmed the functional groups that detected through the examination of FTIR analyses.
- The particle size of the glass powder decreased with increased immersion time using a chemical solution of HCl.
- The MWG with 87 μm particle size revealed high precipitation efficiency of heavy metal Ni ions under alkaline conditions and room temperature after 50 hours.
- The precipitation of nickel increased with increased immersion time.
- The optimum conditions of precipitation findings exhibited that the ultimate precipitate of metal Ni ions was 99% after 50 hours by the inclusion of MWG with a particle size of 87 μm.
- In all particle sizes of precipitant agents, we noticed that the pH increased with time after adding the concentration of the precipitated material from the modified glass waste. Consequently, it can be deduced that the modified waste glass with functional groups has considerable potential as a low-cost precipitant agent for wastewater treatments.

Nomenclatures

 C_{o} Initial concentration before precipitation, mg/L. C_{f} Final concentration after precipitation, mg/L.

PE Precipitation efficiency.

Abbreviations

FTIR Fourier Transform Infrared

HCl Hydrochloric acid MWG Modified Waste Glass

SEM Scanning Electron Microscopy

XRD X-Ray Diffraction

References

1. Wang, H.; and Lei, Z. (2023). Energy supply from oil and gas, mineral depletion, and total natural resource rents: Impact of oil equivalent energy use CO₂ intensity. *Resources Policy*, 86(Part B), 104172.

- 2. Dhaneswara, D.; Fatriansyah, J.F.; Situmorang, F.W.; and Haqoh A.N. (2020). Synthesis of amorphous silica from rice husk ash: Comparing HCl and CH₃COOH acidification methods and various alkaline concentrations. *International Journal of Technology*, 11(1), 200-208.
- 3. Mariaková, D.; Mocová, K.A.; Fořtová, K.; Pavlů, T.; and Hájek, P. (2021). Waste glass powder reusability in high-performance concrete: Leaching behavior and ecotoxicity. *Materials*, 14(16), 4476.
- Suprapto, S.; Augista Nur Azizah, P.; and Lailun Ni'mah, Y. (2023). Silica gel from chemical glass bottle waste as adsorbent for methylene blue: Optimization using BBD. *Journal of Renewable Materials*, 11(12), 4007-4023.
- Ghani, U.; Hussain, S.; Ali, A.; Tirth, V.; Algahtani, A.; Zaman, A.; Mushtaq, M.; Althubeiti, K.; and Aljohani, M. (2022). Hydrothermal extraction of amorphous silica from locally available slate. ACS Omega, 7(7), 6113-6120.
- 6. Ibrahim, H.S.; Ammar, N.S.; Abdel Ghafar, H.H.; and Farahat, M. (2012). Adsorption of Cd (II), Cu (II) and Pb (II) using recycled waste glass: Equilibrium and kinetic studies. *Desalination and Water Treatment*, 48(1-3), 320-328.
- Mekhelf, Z.K.; Hamied, R.S.; and Subhi, A.D. (2022). Separation of copper metal from produced water. *Proceedings of the 2nd International Conference* on *Petroleum Technology and Petrochemicals (ICPTP* 2021), Baghdad, Iraq, 2443(1), 030034.
- 8. Qin, B.; Luo, H.; Liu, G.; Zhang, R.; Chen, S.; Hou, Y.; and Luo, Y. (2012). Nickel ion removal from wastewater using the microbial electrolysis cell. *Bioresource Technology*, 121, 458-461.
- 9. Qasem, N.A.A.; Mohammed, R.H.; and Lawal, D.U. (2021). Removal of heavy metal ions from wastewater: A comprehensive and critical review. *npj Clean Water*, 4(1), 36.
- 10. Karimova, L.; Kairalapov, Y.; Tussupbekova, T.; Oleinikova, T.; Makasheva, G. (2024). Hydrometallurgical processing of molybdenum middlings from

- shatyrkul-zhaysan cluster ore. *Journal of Mining and Metallurgy*, *Section B*: *Metallurgy*, 60(1), 71-83.
- 11. Li, M.; Liu, C.; Ding, A.; and Xiao, C. (2023). A review on the extraction and recovery of critical metals using molten salt electrolysis. *Journal of Environmental Chemical Engineering*, 11(3), 109746.
- 12. Yang, Q.; Xie, Y.; Zhu, B.; Zeng, Y.; Zhou, H.; Ai, P.; and Chen, G. (2022). Positively charged PVC ultrafiltration membrane via micellar enhanced ultrafiltration for removing trace heavy metal cations. *Journal of Water Process Engineering*, 46,102552.
- 13. Porto, M.B.; Alvim, L.B.; and de Almeida Neto, A.F. (2017). Nickel removal from wastewater by induced co-deposition using tungsten to formation of metallic alloys. *Journal of Cleaner Production*, 142(Part 4), 3293-3299.
- 14. Rashed, M.N.; Gad, A.A.; and AbdEldaiem, A.M. (2018). Preparation of low-cost adsorbent from waste glass for the removal of heavy metals from polluted water. *Journal of Industrial and Environmental Chemistry*, 2(2), 7-18.
- 15. Zhao, Y.; Wang, Y.-H.; Wu, J.-J.; and Ma, W.-H. (2024). Study on the behavior of impurity removal from lithium-iron-phosphate slag using the ultrasonic-assisted sulphuric acid leaching. *Journal of Mining and Metallurgy*, *Section B: Metallurgy*, 60(1), 59-70.
- 16. Guan, W.; Tian, S.; Cao, D.; Chen, Y.; and Zhao, X. (2017). Electrooxidation of nickel-ammonia complexes and simultaneous electrodeposition recovery of nickel from practical nickel-electroplating rinse wastewater. *Electrochimica Acta*, 246, 1230-1236.
- 17. Silva, T.S.; Meili, L.; Carvalho, S.H.V.; Soletti, J.I.; Dotto, G.L.; and Fonseca, E.J.S. (2017). Kinetics, isotherm, and thermodynamic studies of methylene blue adsorption from water by Mytella falcata waste. *Environmental Science and Pollution Research*, 24(24), 19927-19937.
- 18. Hussain, Z.; Sultan, N.; Ali, M.; Naz, M.Y.; AbdEl-Salam, N.M.; and Ibrahim, K.A. (2020). Thermochemical conversion of waste glass and mollusk shells into an absorbent material for separation of direct blue 15 azo dye from industrial wastewater. *ACS Omega*, 5(29), 18114-18122.
- 19. Lago, D.; Kraxner, J.; Galusek, D.; and Bernardo, E. (2023). Novel glass-based membranes for Cu adsorption: From alkali activation to sintering. *Heliyon*, 9(8), e18221.
- Song, B.; Zeng, G.; Gong, J.; Liang, J.; Xu, P.; Liu, Z.; Zhang, Y; Zhang, C.; Cheng, M.; Liu, Y.; Ye, S.; Yi, H.; and Ren, X. (2017). Evaluation methods for assessing effectiveness of in situ remediation of soil and sediment contaminated with organic pollutants and heavy metals. *Environment International*, 105, 43-55.
- 21. Pang, F.M.; Teng, S.P.; Teng, T.T.; and Omar A.K.M. (2009). Heavy metals removal by hydroxide precipitation and coagulation-flocculation methods from aqueous solutions. *Water Quality Research Journal*, 44(2), 174-182.
- 22. Baltpurvins, K.A.; Burns, R.C.; Lawrance, G,A.; and Stuart, A.D. (1996). Use of the solubility domain approach for the modeling of the hydroxide precipitation of heavy metals from wastewater. *Environmental Science & Technology*, 30(5), 1493-1499.

- 23. Coman, V.; Robotin, B.; and Ilea, P. (2013). Nickel recovery/removal from industrial wastes: A review. *Resources, Conservation and Recycling*, 73, 229-238.
- Kostrzewa, M.; Staszak, K.; Ginter-Kramarczyk, D.; Kruszelnicka, I.; Góra, W.; Baraniak, M.; Lota, G.; and Regel-Rosocka, M. (2022). Chromium(III) removal from nickel(ii)-containing waste solutions as a pretreatment step in a hydrometallurgical process. *Materials*, 15(18), 6217.
- 25. Wang, R.; Ng, D.H.L.; and Liu, S. (2019). Recovery of nickel ions from wastewater by precipitation approach using silica xerogel. *Journal of Hazardous Materials*, 380, 120826.
- Junuzović, H.; Begic, S.; Selimovic, A.; Dozic, A.; Cvrk, R.; and Ahmetovic, M. (2019). Efficiency of carbonate precipitation and removal of copper and nickel ions from their monocomponent and two-component aqueous solutions. *International Journal for Research in Applied Sciences and Biotechnology*, 6 (6), 11-15.
- 27. Torres-Carrasco, M.; Palomo, J.G.; and Puertasa, F. (2014). Sodium silicate solutions from dissolution of glasswastes. Statistical analysis. *Materiales de Construcción*, 64 (314),e014.
- 28. Azat, S.; Sartova, Z.; Bekseitova, K.; and Askaruly, K. (2019). Extraction of high-purity silica from rice husk via hydrochloric acid leaching treatment. *Turkish Journal of Chemistry*, 43(5), 1258-1269.
- 29. Chen, Q.; Luo, Z.; Hills, C.; Xue, G.; and Tyrer, M. (2009). Precipitation of heavy metals from wastewater using simulated flue gas: Sequent additions of fly ash, lime and carbon dioxide. *Water Research*, 43(10), 2605-2614.