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Development of Technology for the Recovery of Residual Lithium from Its Processing Waste

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Development of Technology for the Recovery of Residual Lithium from Its Processing Waste

ABSTRACT

Objective - To develop a sustainable and efficient technology for the recovery of residual lithium from waste generated during its processing, contributing to the mitigation of environmental impacts and the optimization of the utilization of this critical resource.

Methodology - The research involved laboratory experiments based on flotation, chemical reaction, and washing, using different reagents, including vinegar, acetone, hydrochloric acid, sodium carbonate, kerosene, 99.5% alcohol, liquid soap, eucalyptus oil, and surfactant-containing remover. The samples were analyzed by X-ray fluorescence spectrometry, mass spectrometry, and X-ray diffraction, and the data were subjected to statistical analyses, including variation coefficients and Pearson correlation.

Originality/relevance - The study fills a gap in lithium recovery from industrial waste, considering the technical and environmental feasibility of different processes. The research contributes to the circular economy and the reduction of dependence on primary sources, a topic of growing interest in the global energy transition.

Results - The results indicated that acid leaching presented the highest efficiency in lithium concentration, achieving a 105% increase in metal concentration. A positive correlation was observed between lithium and aluminum (r = 0.956) and a negative correlation with calcium (r = -0.987), providing guidelines for process optimization. The chemical reaction method demonstrated greater reproducibility (CV = 28.48%) compared to flotation (CV = 97.74%) and washing (CV = 79.90%).

Theoretical/methodological contributions - The research establishes parameters for optimizing lithium recovery processes, providing insights for future investigations aimed at improving efficiency and industrial scalability of these techniques.

Social and environmental contributions - Recovering lithium from waste reduces the need for primary extraction, minimizing environmental impacts, water consumption, and emissions associated with conventional mining. The findings can guide public policies and industrial strategies focused on the sustainable management of mineral resources.

KEYWORDS: Residual lithium. Lithium recovery. Sustainability. Industrial waste. Chemical processes.

Desenvolvimento de tecnologia para a recuperação de lítio residual a partir dos resíduos de seu processamento

RESUMO

Objetivo - Desenvolver uma tecnologia sustentável e eficiente para a recuperação de lítio residual a partir dos resíduos gerados durante seu processamento, contribuindo para a mitigação dos impactos ambientais e a otimização do aproveitamento desse recurso crítico.

Metodologia - A pesquisa envolveu experimentos laboratoriais baseados em flotação, reação química e lavagem, utilizando diferentes reagentes, incluindo vinagre, acetona, ácido clorídrico, carbonato de sódio, querosene, álcool 99,5%, sabão líquido, óleo de eucalipto e removedor contendo surfactantes. As amostras foram analisadas por espectrometria de fluorescência de raios X, espectrometria de massa e difração de raios X, e os dados foram submetidos a análises estatísticas, incluindo coeficientes de variação e correlação de Pearson.

Originalidade/relevância - O estudo preenche uma lacuna na recuperação de lítio a partir de resíduos industriais, considerando a viabilidade técnica e ambiental de diferentes processos. A pesquisa contribui para a economia circular e para a redução da dependência de fontes primárias, tema de crescente interesse na transição energética global.

Resultados - Os resultados indicaram que a lixiviação ácida apresentou a maior eficiência na concentração de lítio, atingindo um aumento de 105% na concentração do metal. Houve uma correlação positiva entre lítio e alumínio (r = 0,956) e negativa com cálcio (r = -0,987), fornecendo diretrizes para otimização do processo. O método de reação química demonstrou maior reprodutibilidade (CV = 28,48%) em relação à flotação (CV = 97,74%) e à lavagem (CV = 79,90%).

Contribuições teóricas/metodológicas - A pesquisa estabelece parâmetros para a otimização dos processos de recuperação de lítio, fornecendo subsídios para futuras investigações sobre a melhoria da eficiência e escalabilidade industrial dessas técnicas.
Contribuições sociais e ambientais - A recuperação de lítio a partir de resíduos reduz a necessidade de extração primária, minimizando impactos ambientais, consumo de água e emissões associadas à mineração convencional. Os achados podem orientar políticas públicas e estratégias industriais voltadas à gestão sustentável de recursos minerais.

PALAVRAS-CHAVE: Lítio residual. Recuperação de lítio. Sustentabilidade. Resíduos industriais. Processos químicos.

Desarrollo de tecnología para la recuperación de litio residual a partir de los residuos de su procesamiento

RESUMEN
Objetivo - Desarrollar una tecnología sostenible y eficiente para la recuperación de litio residual a partir de los residuos generados durante su procesamiento, contribuyendo a la mitigación de los impactos ambientales y a la optimización del aprovechamiento de este recurso crítico.
Metodología - La investigación involucró experimentos de laboratorio basados en flotación, reacción química y lavado, utilizando diferentes reactivos, incluyendo vinagre, acetona, ácido clorhídrico, carbonato de sodio, queroseno, alcohol 99,5%, jabón líquido, aceite de eucalipto y removedor con surfactantes. Las muestras fueron analizadas mediante espectrometría de fluorescencia de rayos X, espectrometría de masas y difracción de rayos X, y los datos fueron sometidos a análisis estadísticos, incluyendo coeficientes de variación y correlación de Pearson.
Originalidad/relevancia - El estudio llena un vacío en la recuperación de litio a partir de residuos industriales, considerando la viabilidad técnica y ambiental de diferentes procesos. La investigación contribuye a la economía circular y a la reducción de la dependencia de fuentes primarias, un tema de creciente interés en la transición energética global.
Resultados - Los resultados indicaron que la lixiviación ácida presentó la mayor eficiencia en la concentración de litio, logrando un aumento del 105% en la concentración del metal. Se observó una correlación positiva entre el litio y el aluminio (r = 0,956) y una correlación negativa con el calcio (r = -0,987), proporcionando directrices para la optimización del proceso. El método de reacción química demostró una mayor reproducibilidad (CV = 28,48%) en comparación con la flotación (CV = 97,74%) y el lavado (CV = 79,90%).
Contribuciones teóricas/metodológicas - La investigación establece parámetros para la optimización de los procesos de recuperación de litio, proporcionando insumos para futuras investigaciones sobre la mejora de la eficiencia y la escalabilidad industrial de estas técnicas.
Contribuciones sociales y ambientales - La recuperación de litio a partir de residuos reduce la necesidad de extracción primaria, minimizando los impactos ambientales, el consumo de agua y las emisiones asociadas a la minería convencional. Los hallazgos pueden orientar políticas públicas y estrategias industriales enfocadas en la gestión sostenible de los recursos minerales.

PALABRAS CLAVE: Litio residual. Recuperación de litio. Sostenibilidad. Residuos industriales. Procesos químicos.

INTRODUCTION

Lithium has emerged as a strategic element in the global energy transition, driving sustainable technologies and high-performance batteries. Its growing demand, especially for electric vehicles and renewable energy storage systems, has intensified the exploration of primary sources, such as salt flats and pegmatite deposits (Frota, Lima, 2023). However, intensive extraction presents significant environmental and economic challenges, including high water and energy consumption, as well as the generation of waste harmful to ecosystems.

The long-term security and stability of lithium supply are growing concerns due to the geographic concentration of reserves in few countries (Peiró, Méndez, Ayres, 2013). As an alternative, the recovery of residual lithium emerges as a promising solution, aligning with the principles of circular economy. Garcia *et al.* (2023) emphasize that recovery strategies are fundamental to ensuring sustainability in the lithium production chain, reducing dependence on primary sources and mitigating environmental impacts associated with mining.

For Brazil, a country with significant reserves of this metal, mastering advanced recovery technologies represents a strategic opportunity. The development of these technologies can strengthen the country's position in the global lithium value chain, fostering innovation, generating qualified jobs, and promoting environmental sustainability in the mineral sector. According to Braga and França (2013), the improvement of these technologies can confer a competitive advantage to Brazil in the international scenario.

Despite recent advances, there is a gap in the development of technologies that integrate high recovery efficiency, low environmental impact, and economic viability. The recovery of residual lithium presents significant technical challenges, mainly due to the complexity of the residue matrix and the chemical interactions involved in the process (Liu et *al.,* 2023). Given this scenario, this study seeks to fill this gap by developing and evaluating innovative methods for recovering residual lithium.

This study aims to develop sustainable and efficient technologies for the recovery of lithium from waste generated during its processing. The specific objectives include: (i) identifying the most effective chemical route for the recovery of lithium present in the waste; (ii) evaluating the technical, economic, and environmental viability of the recovery process; (iii) investigating the potential of different chemical substances to improve process efficiency; and (iv) comparing the efficiency of the proposed technology with other existing routes for lithium recovery. Preliminary results indicate that acid leaching is the most promising method, achieving a lithium concentration rate of 105% in the processed waste.

Thus, this research can contribute to the development of sustainable lithium recovery processes, optimizing the use of mineral resources and reducing environmental impacts. Additionally, it can provide technical insights for industry and public policies aimed at the sustainable management of strategic metals.

METHODOLOGY

This study is characterized as experimental and was conducted in the Soils Laboratory of the Federal University of Jequitinhonha and Mucuri Valleys (UFVJM), Mucuri campus, in

Teófilo Otoni, Minas Gerais. The experimental material consisted of residual aluminum silicate, generated during the processing of spodumene for lithium extraction, provided by the Brazilian Lithium Company (CBL). The average chemical composition of the experimental material is presented in Table 1.

Table 1: Composition of the silicate used in the experiments

RESULTS OF ANALYSES MATERIAL: LANDFILL SILICATE											
Composition	Li_2O (%)	Na_2O (%)	K_2O (%)	P_2O_5 (%)	CaO (%)	Al_2O_3 (%)	Fe_2O_3 (%)	Mn_2O_3 (%)	MgO (%)	SiO_2 (%)	SO_4^{2-} (%)
Mean	0.869	1.403	0.391	0.149	4.490	21.061	0.719	0.108	0.189	59.877	2.796
SD	0.092	0.123	0.036	0.031	1.657	1.731	0.088	0.010	0.036	1.001	0.402

Source: Brazilian Lithium Company (CBL) (2023).

Experimental Procedures

Thirteen distinct experiments were conducted, defined based on specific combinations of reagents and techniques, as described below. The experiments were organized into three main categories: flotation, chemical reaction, and washing. All trials were performed in triplicate, and control samples (untreated silicate) were used for direct comparison.

Flotation: Six experiments were carried out, using the following reagents as collecting agents: vinegar, acetone, liquid soap, eucalyptus oil, 99.5% alcohol, and kerosene. In each test, 100 g of residual aluminum silicate in aqueous solution were used, with flotation time varying between 10 and 12 hours. The aeration system was adapted, using an air pumping mechanism similar to that employed in aquariums.

Chemical Reaction: Experiments were conducted using hydrochloric acid (HCl $\cong$ 2%) and a combination of HCl with sodium carbonate (Na_2CO_3). Each test used 50 g of residual aluminum silicate, with a reaction time of 20 hours.

Washing: The washing process was performed with distilled water, including sieving through a 1.18 mm mesh for particle size separation.

Analyses and Characterization

The resulting samples were analyzed by SGS Geosol Laboratórios LTDA, using the following chemical and structural characterization techniques:

- X-Ray Fluorescence Spectrometry (XRF).
- Inductively Coupled Plasma Mass Spectrometry (ICP-MS).
- X-Ray Diffraction (XRD).

Statistical Analysis

The data were analyzed using IBM SPSS Statistics software (version 26), applying the following statistical methods:

- Analysis of Variance (ANOVA): to compare the efficiency of different treatments.

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- Tukey's test: for multiple comparisons between experimental groups.
- Pearson's correlation: to evaluate relationships between the analyzed variables.
- Significance criterion: a significance level of 5% ($p < 0.05$) was adopted for all analyses.
- Standard error calculation: to evaluate the precision of measurements.

The efficacy of the different methods was evaluated by comparing the lithium concentration in the treated samples relative to the control sample, also considering reagent consumption and processing time.

RESULTS

The following are the results obtained in the flotation, chemical reaction, and washing experiments, highlighting the lithium concentration in the treated residues. It is important to emphasize that the tested processes did not promote the total recovery of lithium, but resulted in an increase in the metal concentration in the processed samples, along with other compounds present in the residues. The experimental material presented an initial composition of 0.869% Li_2O (4,032 ppm Li), 21.061% Al_2O_3, 0.719% Fe_2O_3, 0.108% Mn_2O_3, 59.877% SiO_2, 4.490% CaO, and 0.189% MgO.

Flotation Experiments

The six flotation experiments showed variations in lithium concentration in the processed residues. Flotation with vinegar obtained the highest relative concentration, reaching 2,720 ppm Li (67.3% ±2.5%). Acetone resulted in 1,841 ppm (45.6% ±3.0%), while liquid soap and eucalyptus oil presented lower values, with 491 ppm (12.2% ±1.7%) and 274 ppm (6.8% ±2.1%), respectively.

Treatments with 99.5% alcohol and kerosene did not show significant variation, with final concentrations of 4,460 ppm and 3,960 ppm, values close to the initial sample. These results indicate that flotation, despite having promoted variations in lithium concentration in the processed residues, was not effective in the selective separation of the metal.

Chemical Reaction Processes

Treatment with 2% HCl presented the highest concentration of lithium in the treated residues, reaching 4,240 ppm (105% ±2.0%). The combination of HCl with Na_2CO_3, applied for 30 minutes, resulted in 2,828 ppm (70% ±3.5%), while extending the reaction time to 60 minutes led to 2,472 ppm (61.2% ±4.2%).

These results suggest that the chemical reaction promotes modifications in the composition of the residues, increasing the concentration of lithium along with other elements present in the treated material, without achieving selective recovery of the metal.

Washing Experiments

Simple washing with distilled water resulted in a reduction in lithium concentration in

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the residues, reaching 264 ppm (6.5% ±1.9%). However, the separation of the supernatant in water washing presented a concentration of 4,462 ppm (110.4% ±2.2%).

Particle size fractionation also influenced the distribution of lithium in the residues, resulting in concentrations of 4,279 ppm (105.9% ±2.4%) for the fine fraction and 4,879 ppm (120.8% ±1.7%) for the coarse fraction. These data indicate that granulometric segregation may have favored the relative enrichment of lithium, without promoting its efficient separation.

Statistical Correlations

Pearson's Correlation analysis revealed that the lithium concentration in the treated residues has a significant positive association with aluminum ($r = 0.956$, $p < 0.001$) and a negative association with calcium ($r = -0.987$, $p < 0.001$). These results suggest that aluminum may be related to the stabilization of lithium in the residues, while calcium may act as a limiting factor in the process. Additionally, moderate correlations with iron ($r = 0.714$, $p = 0.014$) and weak correlations with manganese ($r = 0.238$, $p = 0.480$) were observed.

The evaluation of the reproducibility of the methods revealed that the chemical reaction presented the lowest coefficient of variation (CV = 28.48%), indicating greater stability in the tests. The washing obtained a CV of 79.90%, while flotation presented the highest variability (CV = 97.74%), which demonstrates low reproducibility of this method in the experiments conducted.

The global average concentration of lithium in the processed residues was 2,613.64 ppm (±1,736.13 ppm), reinforcing that the tested methods altered the distribution of lithium in the treated material, but did not promote its selective and complete recovery.

DISCUSSION

The obtained results demonstrate that the tested processes did not promote the selective recovery of lithium, but resulted in an increase in its concentration in the treated residues, indicating that the applied methods influenced the redistribution of chemical elements in the processed material. This observation is aligned with previous studies suggesting that lithium recovery from industrial waste is a challenging process, as its selective extraction is frequently limited by the presence of other compounds in the mineral matrix (WANG *et al.,* 2023).

Flotation, frequently used in the selective separation of minerals, presented varied results, with better performance for the treatment with vinegar (67.3%) and acetone (45.6%). However, the coefficient of variation values (CV = 97.74%) indicate low reproducibility, suggesting that factors such as surface adsorption and the interaction of lithium with other elements may have negatively influenced the efficiency of the process. Recent studies point out that lithium flotation may be limited by the interference of silicates and aluminosilicates, which reduce the selectivity of the process (Mendes *et al.,* 2018).

In the chemical reaction experiments, acid leaching with HCl was the process that presented the highest relative concentration of lithium in the treated residues (105%), while the HCl/Na_2CO_3 combination presented a lower concentration (70% in 30 minutes and 61.2% in 60 minutes). These results suggest that HCl had a more effective role in the partial dissolution of the residue matrix, promoting a relative enrichment of lithium along with other components, but without achieving a selective separation. Previous studies indicate that acid leaching is efficient in removing impurities and releasing lithium in solution, but its selectivity depends on variables such as pH, temperature, and reaction time (Salakjani, Singh, Nikoloski, 2016; Dessemond *et al.,* 2018).

The washing of residues, in turn, presented variation in lithium concentration according to the applied technique. Simple washing with distilled water resulted in a decrease in the metal concentration (6.5%), indicating that this isolated process is not sufficient to significantly modify the distribution of lithium in the residues. However, the separation of the supernatant after washing presented a lithium concentration of 110.4%, while the granulometric fractionation demonstrated a tendency for higher concentration in the coarse fraction (120.8%) compared to the fine fraction (105.9%). These data reinforce the hypothesis that granulometry and physical segregation may have a relevant role in the differential concentration of lithium in the processed material, as defended by Wills and Napier-Munn (2006).

Pearson's correlation analyses indicated a positive and significant relationship between lithium and aluminum ($r = 0.956$, $p < 0.001$) and a negative association with calcium ($r = -0.987$, $p < 0.001$). The positive correlation with aluminum suggests that lithium may be associated with aluminosilicate phases, corroborating studies that indicate that lithium in industrial tailings may be linked to minerals such as spodumene and lepidolite (Ibsaine *et al.,* 2023). On the other hand, the negative correlation with calcium may indicate that the presence of this element reduces the efficiency of the employed methods, possibly due to the formation of less soluble compounds or competitive adsorption in the mineral matrix.

From the perspective of method reproducibility, the chemical reaction presented the lowest coefficient of variation (CV = 28.48%), indicating greater stability in the conducted tests. Flotation, on the other hand, presented the highest variability (CV = 97.74%), which suggests the need for optimization of operational parameters to improve the efficiency of this method. Washing obtained an intermediate CV (79.90%), reinforcing that its efficacy depends on factors such as contact time and granulometric segregation.

The obtained results reinforce that the total recovery of lithium was not achieved in the tested processes, but the data demonstrate the significant influence of different methods on the redistribution and concentration of the metal in the treated residues. Recent studies point out that the combination of methods can be a more effective strategy, with chemical leaching associated with physical techniques, such as selective flotation and membrane separation, having demonstrated greater efficiency in lithium recovery from industrial tailings (Hasan, Hossain, Sahajwalla, 2023).

Beyond the direct recovery of lithium, residues from the processing of this metal, such as aluminum silicate and calciner fine particles, have potential for sustainable applications in other production chains. Research demonstrates that lithium processing residue, composed mainly of aluminum silicate and calciner fine particles, can be used as an alternative fertilizer in castor bean cultivation (Ricinus communis L.). The results indicated that doses of 8 and 10 t/ha of the residue, associated with 50% of conventional fertilization, promoted greater plant development and productivity. In the analysis of total fresh and dry mass, these treatments presented the highest yields, while the oil content analysis showed that the same dosages resulted in values similar to those found in the literature (35-55%), proving the viability of using these residues as a conditioner-fertilizer (Costa *et al.,* 2024). This approach not only reduces the environmental impacts of inadequate disposal but also offers a viable solution for agriculture in nutrient-poor soils.

The integration of lithium recovery strategies with solid waste management public policies is essential to maximize their environmental and economic benefits. As evidenced by Aguiar *et al.* (2024), the effectiveness of these policies depends on collaboration between social actors (waste collectors, companies, and government) and the adoption of models such as the circular economy, which prioritize the reuse of materials and social inclusion. The typology proposed by these authors highlights emerging themes, such as municipal waste management, and driving forces, such as the environment, reinforcing the need for coordinated actions to reduce environmental impacts and promote sustainability in the lithium production chain.

Given these observations, it is recommended that future investigations evaluate combined strategies, optimizing variables such as reaction time, type of reagent, and temperature, as well as the implementation of pre-treatment techniques to improve the selectivity of the processes. Additionally, the environmental impact of the tested methods should be analyzed, considering the industrial viability of lithium recovery from tailings.

CONCLUSION

This study evaluated different methods for the recovery of residual lithium from industrial waste generated during spodumene processing. The results demonstrated that, although none of the processes promoted the selective recovery of lithium, there was an increase in its concentration in the treated residues, evidencing the influence of the applied methods on the redistribution of the metal in the mineral matrix.

Acid leaching with HCl presented the best results, reaching a concentration of 4,240 ppm of Li (105%), suggesting that the partial dissolution of the residue matrix contributed to a relative enrichment of lithium, without, however, providing its selective separation. Flotation, in turn, showed variable results and low reproducibility (CV = 97.74%), indicating that the interaction of lithium with other components of the residues may have limited the efficiency of the process. Simple washing was not effective, but granulometric fractionation indicated a higher concentration of lithium in the coarse fraction (120.8%), suggesting that physical segregation may play a relevant role in the differential enrichment of the metal in the residues.

Statistical analyses revealed a significant positive correlation between lithium and aluminum ($r = 0.956$, $p < 0.001$) and a negative association with calcium ($r = -0.987$, $p < 0.001$), indicating that the presence of these elements may influence the efficiency of the employed methods. Additionally, the chemical reaction presented the lowest variability (CV = 28.48%), demonstrating greater stability in the experiments, while flotation presented the highest data dispersion, suggesting the need for optimization of operational parameters.

The findings of this research reinforce the need to develop more effective strategies for the selective recovery of lithium, combining different physicochemical approaches. Future studies should evaluate hybrid techniques, such as the association between chemical leaching and physical-mechanical processes, as well as the optimization of operational parameters to

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improve the efficiency and reproducibility of the methods.

From an environmental perspective, the recovery of lithium from industrial waste can reduce dependence on primary extraction, mitigating environmental impacts associated with traditional mining. However, for these methods to be viable on an industrial scale, it is essential to consider economic aspects, reagent consumption, and the generation of secondary waste.

Therefore, this study has the potential to contribute to the advancement of research aimed at the valorization of industrial tailings containing lithium, providing relevant data for the improvement of metal recovery processes. The development of more selective and sustainable technologies can favor the circular economy in the mineral sector, reducing environmental impacts and increasing the efficiency in the utilization of strategic resources.

REFERENCES

DECLARATIONS

CONTRIBUTION OF EACH AUTHOR

Alexandre Sylvio Vieira da Costa and Elton Santos Franco
Study Conception and Design, Data Curation, Investigation, Methodology, Final Review and Editing, Supervision.

Alexssander Ferreira de Sousa, Nubia Aparecida de Aguilar, and Tales Ceniro Campos
Data Curation, Formal Analysis, Investigation, Writing - Initial Draft, Writing - Critical Review.

DECLARATION OF CONFLICTS OF INTEREST

We, **Alexssander Ferreira de Sousa, Alexandre Sylvio Vieira da Costa, Elton Santos Franco, Nubia Aparecida de Aguilar, and Tales Ceniro Campos,** declare that the manuscript entitled **"Development of Technology for the Recovery of Residual Lithium from Its Processing Waste":**

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