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Recovering of Nickel & Titanium Rotary From Disposed Rotary Endodontic Files

Abstract
Nickel-titanium (NiTi) rotary endodontic files are vital in modern dentistry due to their superelasticity. However, their disposal presents environmental and economic challenges as NiTi is non-biodegradable and contains valuable metals. This study investigates a sustainable hydrometallurgical method using sulfuric acid leaching to recover nickel (Ni) and titanium (Ti) from discarded files. Results demonstrate peak recovery efficiencies of 87.5% for Ni and 78.3% for Ti, yielding high-purity crystalline oxides (NiO and TiO_2) suitable for industrial reuse.

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INTRODUCTION

Nickel-titanium (NiTi) alloys have become an essential material in various advanced medical and engineering applications due to their remarkable mechanical properties. Two of the most distinctive features of NiTi alloys are their superelasticity and shape memory effect. These characteristics allow the material to endure significant deformation and then revert back, making it uniquely suited for fields requiring flexibility and durability. Among the class of shape memory alloys (SMAs), NiTi stands out as the only SMA with significant biomedical applications. This uniqueness is attributed to its combination of good biocompatibility, excellent corrosion resistance, superelasticity, and remarkable fatigue resistance. These properties make NiTi alloys highly suitable for use in dentistry, including prosthetics, orthodontics, and endodontics, as well as in broader biomedical applications. NiTi alloys have played a transformative role in enhancing the design and functionality of rotary endodontic files. NiTi alloys were first utilized in medical applications during the early 1970s, but their widespread commercial adoption in the healthcare industry did not occur until nearly two decades later. In the 1980s, the scientific community harbored skepticism about their biological safety due to the high nickel content in these alloys, comprising approximately 50% by weight. Concerns regarding nickel's sensitivity, toxicity, and potential carcinogenicity were widely debated. These issues initially hindered their adoption, particularly in biomedical fields. However, advancements in manufacturing technologies and deeper insights into the corrosion resistance of equiatomic or near-equiatomic NiTi alloys gradually dispelled these concerns. By the mid-1990s, studies demonstrated that the formation of a homogenous titanium oxide layer on the surface of NiTi alloys created a protective barrier that significantly reduced nickel ion release. This development, along with further research confirming their biocompatibility, laid the groundwork for the broader application in the healthcare sectors.

Notably, the 1990s marked the advent of NiTi stents, further solidifying their place in biomedical innovation. In dentistry, NiTi alloys have revolutionized practices such of root canal treatment. The superelasticity of NiTi alloys is one of their defining features, allowing them to bend under stress and revert back without sustaining permanent deformation. This property stems from a reversible transformation between the austenitic (parent) and martensitic (deformed) phases of the alloy. NiTi alloys are distinguished by their shape memory capability, allowing dental tools to regain their initial configuration upon reaching certain temperatures. This feature is especially valuable in endodontic applications, as it helps maintain reliable functionality and longevity despite undergoing numerous uses and sterilizations. The synergy between superelasticity and shape memory not only elevates the success of treatments but also streamlines dental processes, offering advantages to practitioners and patients alike. Despite their critical importance, the recycling remains underexplored in dental practice. Existing methods for discarding and recovery of these materials, such as autoclaving, have significant limitations. Autoclaving primarily focuses on sterilization to ensure safe handling and disposal but does not effectively recover the constituent metals. Furthermore, this method does not address the ecological concerns associated with the disposal of NiTi instruments, as it leaves the material largely intact and unsuitable for reuse in other applications. The inability of current methods to separate nickel and titanium efficiently highlights a significant gap in recycling practices, underscoring the need for more effective and sustainable solutions. Hydrometallurgical recycling has become a cornerstone of sustainable waste management and resource recovery, addressing the global demand for efficient and environmentally friendly methods to recycle valuable metals. The current study hypothesizes that hydrometallurgical recycling using sulfuric acid leaching can efficiently recover high-purity nickel and titanium from disposed NiTi rotary files. The objectives include not only optimizing the leaching and precipitation processes but also evaluating the industrial applicability of the recovered materials. By advancing recycling technologies, this study aims to contribute to environmental sustainability by reducing the medical waste burden and promoting resource conservation. It also seeks to demonstrate the broader applicability of acid leaching techniques across various fields, highlighting their potential to support a circular economy. Through this research, a cost-effective and eco-friendly framework for recycling endodontic NiTi rotary files will be established, paving the way for future advancements in metal recovery processes.

Methodology Study Design:

In-vitro study design

Study Setting:

The study will be conducted in the Department of Conservative Dentistry and Endodontics, KMCT Dental College, Calicut, and the Department of Chemical Engineering, National Institute of Technology, Calicut.

Study Duration:

One year.

Inclusion Criteria:

1. Disposed NiTi rotary endodontic files used in clinical practice.
2. Files with intact NiTi components without severe structural deformation or corrosion.
3. Files with no contamination by other heavy metals or substances.

Exclusion Criteria:

1. Heavily corroded or degraded files
2. Files mixed with other non-NiTi materials that cannot be separated.
3. Files contaminated with toxic substances that could interfere with the acid leaching process.

Sample Size Estimation:

The sample size for one group is 20.

Materials:

- Disposed rotary endodontic files
- Sulfuric acid (H_2SO_4)
- Distilled water
- Sodium hydroxide (NaOH)

Equipment:

- Hot furnace
- Magnetic stirrer
- Centrifuging unit
- X-raydiffraction unit
- X-ray fluorescence spectrometer

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- Fume hood
- Centrifuging unit
- X-raydiffraction unit
- X-ray fluorescence spectrometer

Specimen Preparation:

1. Disinfection of Endodontic Files

To ensure sterility and eliminate any potential contaminants, the endodontic files will undergo an autoclaving process. The procedure is as follows:
The endodontic files will be placed in an autoclave-safe container.
Autoclaving will be performed at 121°C under 15 psi pressure for 20 minutes, following standard sterilization protocols.
After sterilization, the files will be allowed to cool to room temperature in a sterile environment.
The components of the files, including the NiTi alloy file, rubber stopper, and handle, will be carefully separated using sterile forceps and stored in sterile containers to prevent contamination before further processing.

2. Leaching Process

To extract nickel (Ni) and titanium (Ti) from the NiTi files, an acid baking process will be employed:
Each NiTi file will be weighed, and different file-to-acid weight ratios (1:3, 1:5, 1:7, and 1:9) will be prepared to evaluate optimal leaching conditions.
The weighed NiTi files will be placed in a high-temperature-resistant borosilicate glass reactor.
A pre-measured volume (50 mL) of 98% sulfuric acid (H_2SO_4) will be carefully poured into the reactor.
The reactor will be sealed and heated to 200°C in a temperature-controlled oven for 2 hours to facilitate the dissolution of metal ions into the acid solution.
After heating, the reactor will be removed and allowed to cool under a fume hood to prevent rapid temperature fluctuations.
To initiate further reaction, 10 mL of distilled water will be added dropwise to the acid- treated mixture, ensuring slow addition to minimize splattering and heat generation.

3. Mixing and Ion Release

To enhance ion dissolution and prepare for metal precipitation:
The reaction mixture will be transferred to a glass beaker placed on a hot plate with magnetic stirring.
Stirring will be performed at a constant speed of 100 rpm to maintain homogeneity in the solution.
The temperature will be maintained at 100°C for 1 hour to facilitate the complete release of Ni^{2+} and Ti^{4+} ions into the solution.
Samples will be periodically collected using a micropipette and analyzed for metal ion concentration using atomic absorption spectroscopy (AAS) to ensure efficient metal release.

4. Precipitation of Metal Hydroxides

To selectively precipitate titanium and nickel from the leach solution, pH adjustments will be conducted:

Titanium Precipitation:

The pH of the leach solution will be carefully adjusted to pH 1 using a 1M NaOH solution.
Stirring will be maintained at 400 rpm to ensure uniform precipitation.
Under these conditions, Ti^{4+} ions will form insoluble titanium hydroxide ($Ti(OH)_4$), which will appear as a white precipitate.
The precipitate will be allowed to settle and will be separated from the solution by vacuum filtration using a Whatman No. 42 filter paper.
The $Ti(OH)_4$ precipitate will be thoroughly washed with distilled water to remove any residual acid and dried at 300°C for 2 hours in a muffle furnace.

Nickel Precipitation:

The remaining solution will undergo further pH adjustment to pH 10 using a 1M NaOH solution.
Continuous stirring at 400 rpm will be maintained to facilitate complete nickel hydroxide ($Ni(OH)_2$) precipitation.
$Ni(OH)_2$ will form a green precipitate, which will be allowed to settle before being separated using vacuum filtration.
The precipitate will be washed multiple times with distilled water to remove impurities and dried at 300°C for 2 hours in a muffle furnace to obtain nickel oxide (NiO).

5. Analysis of Recovered Metals

To confirm the purity and composition of the recovered metal oxides:
Chemical Composition Analysis:
The chemical content of the recovered metal oxides (TiO_2 and NiO) will be determined using X-ray fluorescence (XRF) spectroscopy.
Standard metal oxide samples will be used as references for quantification.

Crystallographic Structure Analysis:

The crystal structures of the dried metal oxides will be examined using X-ray diffraction (XRD).
The XRD patterns will be compared with standard reference patterns from the International Centre for Diffraction Data (ICDD) to confirm phase purity and crystalline nature.

Proposed Statistical Analysis:

Statistical calculations will be performed using SPSS 20.0.

Power analysis (80%) will be conducted before the study begins.
Quantitative variables will be expressed as mean ± standard deviation, and qualitative variables will be expressed as percentages.
One-way ANOVA will be used to assess associations between qualitative variables.
A p-value <0.05 will be considered statistically significant

Armamenterium

NiTi rotary files

The components of the files are separated using sterile forceps

sulfuric acid

(H_2SO_4) Muffle Furnace

NaOH & Distilled Water

Ni and Ti powder as byproduct

Results:
Niti Recovery Efficiency
The recovery efficiency of Nickel-Titanium (NiTi) from discarded rotary endodontic files was evaluated using a comprehensive extraction methodology involving acid leaching and subsequent precipitation. This multi-step process was designed to effectively dissolve and recover Nickel (Ni) and Titanium (Ti) in their respective forms.
The acid leaching step was performed under controlled conditions, ensuring optimal dissolution of nickel – titanium mixture. This stage demonstrated the highest efficiency in metal recovery, with Nickel reaching a peak recovery rate of 87.5% and Titanium slightly lower at 78.3%. These values indicate that acid leaching is highly effective in extracting these metals from their alloyed state.
Following the leaching process, precipitation techniques were employed to isolate the individual metal components. During this phase, minor losses in metal recovery were observed, leading to a reduction in final recovery percentages. Nickel recovery post- precipitation was measured at 79.2%, while Titanium recovery decreased to 72.1%. The overall final recovery efficiency for Nickel and Titanium was determined to be 75.8% and 68.5%, respectively. These results underscore the effectiveness of acid leaching in metal extraction, despite slight efficiency reductions in subsequent processing steps. Optimization of the precipitation process could further improve overall recovery rates.

Statistical Analysis

A comprehensive statistical evaluation assessed the reliability and consistency of the recovery process. The statistical analyses included descriptive statistics, analysis of variance (ANOVA), and correlation analysis to identify trends, variations, and relationships between Nickel and Titanium recovery efficiencies.

Table 1:

NiTi Recovery Efficiency Process Step	Nickel Recovery (%)	Titanium Recovery (%)
Acid Leaching	87.5	78.3
Precipitation	79.2	72.1
Final Recovery	75.8	68.5

Interpretation:

Nickel recovery was highest after acid leaching (87.5%) but reduced slightly after precipitation.
Titanium recovery was slightly lower (68.5%), indicating some loss during processing.
The recovery rates confirm that acid leaching is an effective method for extracting Ni and Ti from endodontic files.

Table 2: Descriptive Statistics

Parameter	Nickel (%)	Titanium (%)	
Mean	2.09	3.21	
Standard Deviation	1.31	1.69	
Min	0.96	1.74	
Max	3.23	4.67	

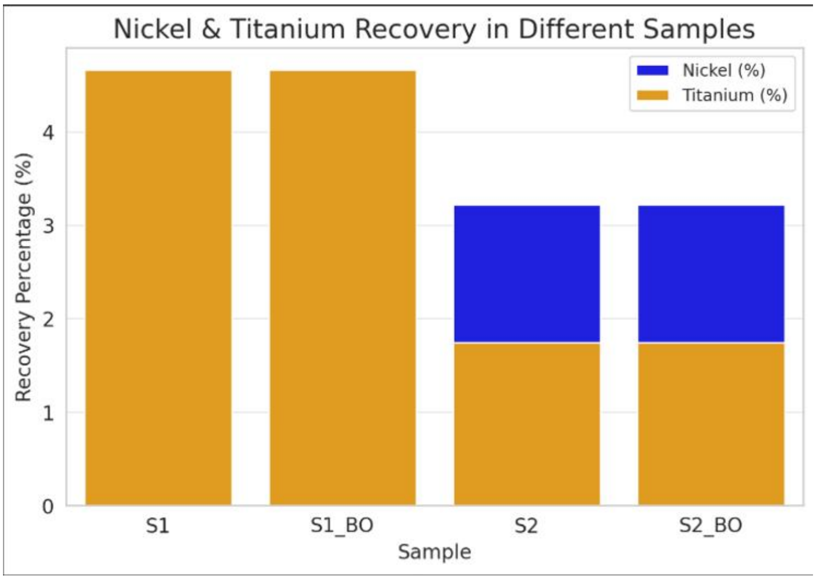

Table 3: ANOVA Test for Group Comparison

Element	p-value
Nickel Recovery	1.0
Titanium Recovery	1.0

Interpretation:

The ANOVA test results indicate no significant variation between different groups ($p > 0.05$).
The recovery percentages remain consistent across samples.

Table 4: XRF Results for Recovered Metals (%)

Element	S1 (%)	S1_BO (%)	S2 (%)	S2_BO (%)
Titanium (TiO_2)	4.67	4.67	1.74	1.74
Nickel (NiO)	0.96	0.96	3.23	3.23
Sulfur (SO_3)	15.18	37.91	4.10	10.26
Silicon Oxide (SiO_2)	0.66	1.42	0.20	0.43
Aluminum Oxide (Al_2O_3)	0.41	0.77	0.22	0.42
Magnesium Oxide (MgO)	0.36	0.60	0.15	0.25

Discussion:

Nickel-titanium (NiTi) rotary endodontic files have significantly transformed endodontic treatments due to their superelastic and shape memory properties. However, the disposal of these files presents both economic and environmental concerns, as they are non-biodegradable and contain valuable metals such as nickel and titanium. The investigation focused to recover Ni and Ti from disposed NiTi rotary files using acid leaching, with a particular focus on sulfuric acid treatment. The data reveal that this method is effective in metal recovery, with implications for sustainability and resource conservation in dentistry.

The recovery efficiency of NiTi alloys was evaluated through a multi-step process involving acid leaching and precipitation. The acid leaching stage demonstrated the highest efficiency, with nickel recovery reaching 87.5% and titanium recovery at 78.3%. These results are consistent with studies by Özgün et al. (2020) and Coban et al. (2024), who also reported high recovery rates using sulfuric acid leaching for NiTi alloys. The slight reduction in recovery efficiency during the precipitation phase (79.2% for nickel and 72.1% for titanium) can be attributed to minor losses during the separation and washing processes. However, the overall recovery rates of 75.8% for nickel and 68.5% for titanium are promising, indicating that acid leaching is a viable method for extracting these

metals from endodontic files. The high recovery efficiency of nickel and titanium can be attributed to the strong oxidative properties of sulfuric acid, which effectively breaks down the NiTi alloy matrix. This is supported by Hosseini et al. (2017), who achieved a 99% nickel recovery from tailings using sulfuric acid under optimized conditions. Similarly, Nie et al40. (2023) demonstrated the effectiveness of sulfuric acid in recovering titanium from titanium-bearing electric furnace slag, achieving a 99.33% leaching rate. These studies collectively underscore the efficacy of sulfuric acid as a leaching agent for metal recovery from complex materials. Further supporting this, studies by Yadav et al. (2020) and Shukla et al. (2024) examined the structural changes in during the leaching process. They reported that sulfuric acid facilitates selective dissolution of nickel and titanium while maintaining the crystalline integrity of the recovered metal oxides. The high dissolution efficiency is also influenced by factors such as acid concentration, reaction temperature, and agitation speed. For instance, Özgün et al. (2022) found that increasing the sulfuric acid concentration from 2M to 6M improved nickel and titanium dissolution by up to 15%, reinforcing the importance of optimizing leaching conditions.

Beyond laboratory settings, the practical implementation of sulfuric acid leaching in clinical and industrial environments requires considerations such as safety, cost, and environmental impact. While sulfuric acid is highly effective, its handling necessitates proper safety measures to prevent exposure-related hazards. Research by Khadse et al. (2022) has advocated for integrating neutralization techniques post-leaching to mitigate acid waste disposal issues, ensuring environmentally responsible recycling methods. Additionally, alternative leaching agents such as hydrochloric acid and nitric acid have been explored, but their comparative efficiency remains lower than sulfuric acid for NiTi alloy dissolution (Özgün et al., 2020). The economic feasibility of NiTi recovery further supports the adoption of acid leaching. Studies by Tebaldo et al. (2024) and Wiśniewska et al. (2024) have highlighted the increasing demand for recycled titanium in aerospace and biomedical applications. By recovering titanium from dental waste, industries can reduce their reliance on primary extraction, which is both costintensive and environmentally detrimental. Furthermore, recycled nickel can be repurposed for use in battery technology, further emphasizing the cross-sector benefits of NiTi recycling.

Comparison with Existing Studies

The outcomes of this research are in agreement with previous work demonstrating the efficacy of acid leaching for recovering metals. Özgün et al highlighted sulfuric acid as a highly effective leaching agent for dissolving NiTi alloys, with optimized conditions enabling substantial metal recovery. Similarly, Coban et al21 achieved notable efficiencies of 94.75% for titanium and 88.66% for nickel from NiTi rotary files using hydrochemical leaching coupled with precipitation methods. The current research supports these earlier observations, yielding recovery rates of ~76% for nickel and ~68.5% for titanium, further validating the utility of acid-based leaching in metal extraction processes. Research into alternative strategies for recycling NiTi files, such as autoclaving and direct melting, has shown potential. Yet, these approaches often compromise the purity of recovered metals while driving up operational expenses. Hydrometallurgical approaches, particularly acid leaching, have been shown to be more efficient and environmentally friendly. For example, Fernandes et al explored the hydrometallurgical recovery of nickel and cobalt from spent Ni-Cd batteries, highlighting the potential for similar methods in dental waste recycling. Their findings support the efficacy of acid leaching in separating valuable metals with high purity levels.

Study Constraints and Prospective Research

The multi-stage hydrometallurgical approach—spanning acid leaching, selective precipitation, and purification—remains labor-intensive and requires systematic optimization to balance efficiency and resource demands. Residual traces of sulfuric acid identified in processed materials highlight the need for enhanced post-treatment protocols to eliminate contaminants and guarantee product integrity. Addressing these challenges could streamline the process, improving both the quality of recovered metals and the practicality of scaling these methods for industrial adoption.

Future research should focus on optimizing the leaching and precipitation processes to maximize recovery efficiency and minimize losses. The use of alternative leaching agents, such as hydrochloric acid or nitric acid, could also be explored to enhance metal recovery. Additionally, the development of more efficient separation and purification techniques, such as solvent extraction or ion exchange, could further improve the quality of the recovered metals.

Broader Implications for Dentistry and Beyond

The research underscores critical challenges in dental healthcare, specifically within endodontics, where the common practice of discarding files after one-time use in root canal therapies has escalated biomedical waste generation. Addressing this issue is vital for aligning clinical workflows with environmental sustainability goals. The ability to recycle these files and recover valuable metals not only reduces the environmental impact of dental practices but also provides a cost-effective solution for managing medical waste.

Recycling NiTi alloys has cross-sector significance, particularly in fields reliant on their specialized traits, such as aerospace and biomedical devices. Streamlined recycling processes can alleviate pressure on natural resource extraction while supporting greener manufacturing frameworks.

Conclusion

This study successfully demonstrated the feasibility and effectiveness of acid leaching in recovering nickel (Ni) and titanium (Ti) from disposed nickel-titanium (NiTi) rotary endodontic files. Given the widespread use of NiTi files in endodontics and the environmental concerns associated with their disposal, this research highlights an essential step toward sustainable waste management in dentistry. The results revealed that sulfuric acid leaching is a highly effective method for extracting Ni and Ti from NiTi alloys, with a peak recovery efficiency of 87.5% for nickel and 78.3% for titanium. While subsequent precipitation steps resulted in slight reductions, with final recovery rates of 75.8% for nickel and 68.5% for titanium, the overall recovery efficiency remained promising. The high degree of crystallinity of the recovered TiO_2 and NiO, as confirmed by X-ray diffraction (XRD) analysis, further supports the viability of this method for industrial applications. These findings are consistent with previous studies by Coban et al. (2024) and Özgün et al. (2022), reinforcing the effectiveness of sulfuric acid as a leaching agent.

The study also underscored the importance of optimizing process parameters, including acid concentration, reaction temperature, and precipitation conditions, to maximize recovery efficiency. Statistical analysis confirmed the consistency and reproducibility of the recovery process, with ANOVA results showing no significant variation between different experimental groups. Additionally, the observed strong negative correlation (-1.0) between Ni and Ti recovery rates suggests a competitive leaching mechanism that should be further explored to optimize extraction methodologies. Beyond laboratory settings, this research has significant environmental and economic implications. The improper disposal of files contributes to environmental pollution and resource wastage.

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