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Original Research Article

Application of SEM-EDS and XRD in metal material detection used in rubber dam clamps

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Abstract

Aim and objective: Aim is to evaluate the metal material detection of two different rubber dam clamp companies and the Objective is to compare and evaluate the metal material detection of two different rubber dam clamps using Scanning Electron Microscopy – Energy Dispersive Spectrometer and X-ray Diffraction.
Introduction: Rubber dam clamps are indispensable instruments in both endodontic and restorative dental procedures, as they ensure proper isolation of the treatment area and maintain a dry operating field. Despite their frequent use, differences in material composition, manufacturing processes, and quality control can significantly impact their structural integrity, leading to concerns about premature fracture, corrosion, and reduced longevity. Understanding the metallurgical properties of these clamps is therefore critical to improving their clinical performance. This study aims to assess and compare the elemental composition and microstructural characteristics of rubber dam clamps from two different manufacturers using Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM-EDS) and X-ray Diffraction (XRD).
Material and Methods: Sections of rubber dam clamps measuring 8 mm were prepared for analysis. To obtain a clear surface view, the samples were then etched using a solution composed of 15 ml hydrochloric acid (HCl) and 5 ml nitric acid (HNO3). After polishing, the samples were then immersed in the etching solution for 1 minute. Following etching, the samples were then divided into two groups based on their respective manufacturing companies. Each group were then examined using Scanning Electron Microscopy (SEM) combined with Energy Dispersive Spectroscopy (EDS) and X-ray Diffraction (XRD) to analyze the surface morphology and composition.
Result: Both rubber dam clamp company groups have similar content except the presence of minute quantity of nickel in group A.
Conclusion: Minute quantity of nickel in group A might be attributed to improve fracture resistance or to the electroplating done to prevent corrosion.

Keywords: Rubber dam clamps, Scanning electron microscope, Energy dispersive spectrometer, X ray diffraction, Metal material detection

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1. Introduction

Rubber dam isolation is a fundamental practice in endodontics and operative dentistry, providing effective moisture control, improved visibility, and enhanced patient safety. Central to this technique are rubber dam clamps, which secure the dam in place during clinical procedures.[1] Despite their long-standing use, there remains no internationally standardized guideline for their manufacturing quality or material composition. Clinicians frequently report fractures of certain clamps after minimal use, raising concerns about their structural integrity and clinical reliability.[2]

Most rubber dam clamps are made from various grades of stainless steel such as AISI 303, 304, 410, 416A, and 420B, or from plain carbon martensitic steel with or without electroplated coatings. Newer materials like polymer composites are also under investigation for their potential to improve performance and durability. However, inconsistencies in alloy composition and manufacturing quality can lead to early clamp failure, jeopardizing both procedural efficiency and patient safety.[3]

Advanced analytical techniques such as Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and X-ray Diffraction (XRD) offer

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valuable insights into the structural and compositional properties of dental alloys. SEM enables evaluation of surface topography and microstructure, while EDS provides elemental composition through qualitative and quantitative analysis.[4] XRD complements these methods by revealing the crystalline phases and homogeneity of metal alloys, helping to identify manufacturing defects or heat treatment inconsistencies.[5]

In our department, notable differences in the durability of rubber dam clamps from two commonly used manufacturers have been observed. This raises the need to investigate whether these differences are rooted in variations in metal composition or manufacturing processes. Understanding these factors is clinically significant, as clamp failure during treatment can compromise isolation, increase procedural time, and risk injury.

Understanding the metallurgical differences between clamps from different manufacturers can help identify the reasons behind premature failure and guide clinicians in selecting more reliable products. By identifying which alloy compositions offer better mechanical properties and corrosion resistance, the findings may lead to more informed choices in clinical practice, improved clamp design by manufacturers, and ultimately, increased safety and efficiency in dental procedures.[6]

This study aims to conduct a comparative analysis of the metal alloy composition and microstructural characteristics of rubber dam clamps from two different manufacturers using SEM-EDS and XRD. The findings will contribute to identifying the material factors influencing clamp performance and may guide clinicians in selecting more durable and reliable brands for routine clinical use.

2. Material and Methods

For this study five rubber dam clamps obtained from two different commercially available brands, referred to as Group A and Group B were taken (**Figure 1**). To facilitate uniform testing and analysis, each clamp was sectioned into smaller standardized specimens measuring approximately 8 × 8 mm using a precision cutting tool (**Figure 2**).

Figure 1: Group A AND B rubber dam clamps

Figure 2: Sectioned samples of group A & B

Following sectioning, all samples were polished to ensure a smooth and reflective surface. Polishing was performed using progressively finer abrasives to minimize surface irregularities that could interfere with imaging and analysis.

To further enhance the visibility of the microstructural features, the polished specimens were etched. The etching solution was prepared by mixing 15 ml of hydrochloric acid (HCl) with 5 ml of nitric acid (HNO_3). Each sample was immersed in this etchant for 1minute.[7]

After etching, the samples were thoroughly rinsed with distilled water, dried, and then subjected to SEM-EDS analysis (**Figure 3**). Additionally, X-ray Diffraction (XRD) analysis (**Figure 4**) was performed to determine the crystallographic phases present in the samples.

Figure 3: Scanning electron microscopy – energy dispersive spectrometry

Figure 4: X-ray diffraction

3. Results

Upon comparing the scanning electron microscopic images of the rubber dam clamps from the two companies, Group A exhibited a rough, grainy surface texture, whereas Group B displayed a comparatively smoother surface, as shown in **Figure 5Figure 6**. However, both groups demonstrated surface irregularities, including patchy areas and the presence of crevices.

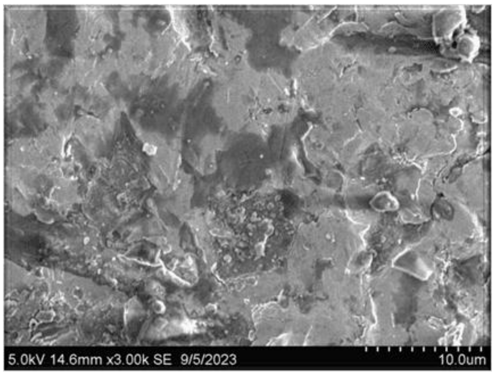


Figure 5: SEM image of group A

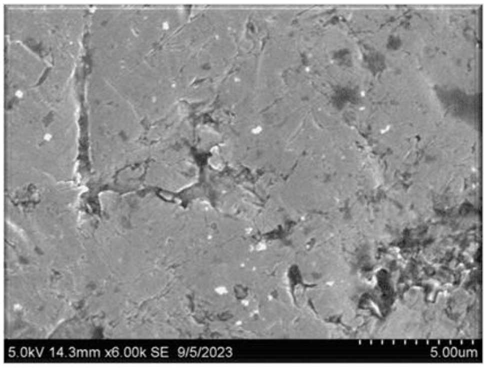


Figure 6: SEM image of group B

Subsequently, the samples were analysed using Energy Dispersive Spectroscopy (EDS) software to conduct a detailed assessment of their elemental composition. The analysis revealed that both groups shared a largely similar elemental profile; however, a trace amount of nickel was detected exclusively in the samples from Group A. This subtle compositional difference was clearly illustrated through the colour mapping (**Figure 7**,**Figure 8**), as well as in the tabulated data (**Table 1**,**Table 2**) and graphical representations (**Graph 1**,**Graph 2**).

Table 1: EDS results for Group A

Element	Weight %	Atomic %
C	6.42	19.60
O	9.18	21.03
Na	0.57	0.91
Mg	0.32	0.49
Si	4.09	5.34
Cr	10.80	7.81
Fe	68.11	44.71
Ni	0.51	0.32

Table 2: EDS results for Group B

Element	Weight %	Atomic %
C	4.35	15.66
O	4.01	10.85
Al	0.33	0.53
Si	2.09	3.22
Cr	11.24	9.35
Fe	77.97	60.38

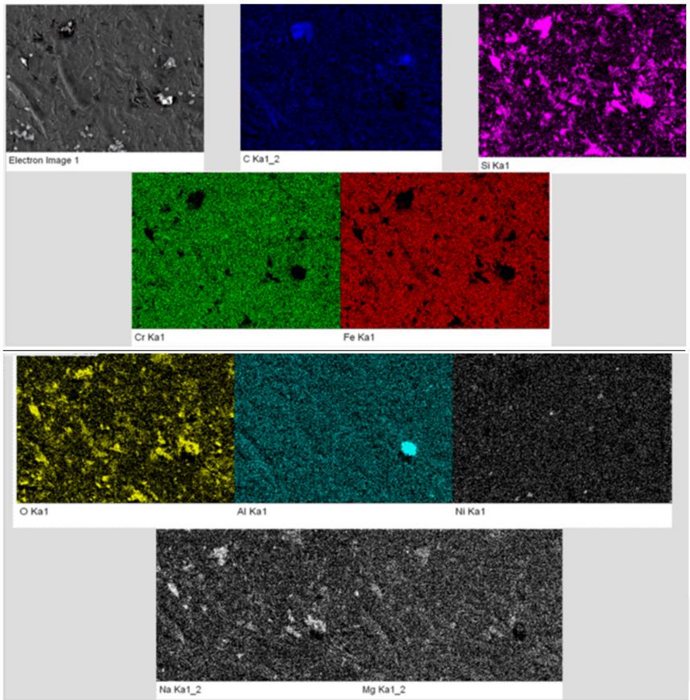


Figure 7: Colour mapping of Group A

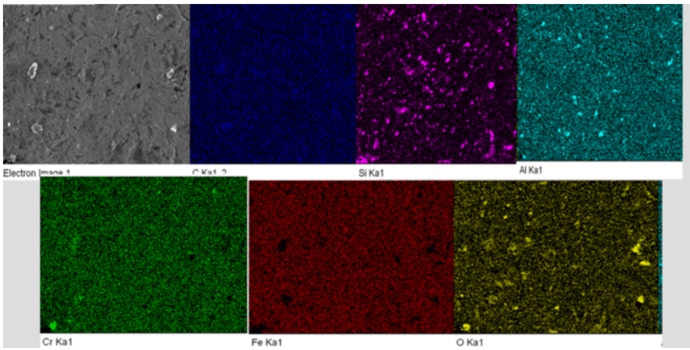
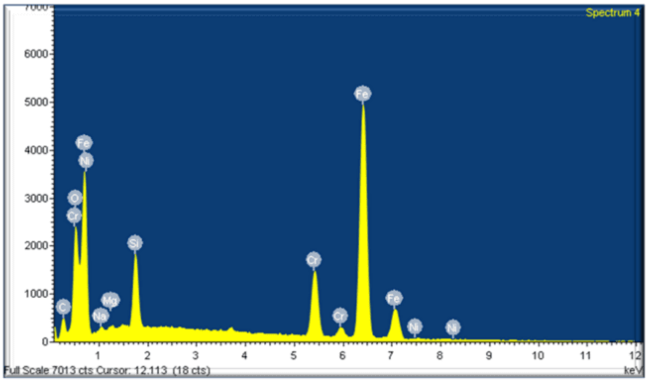
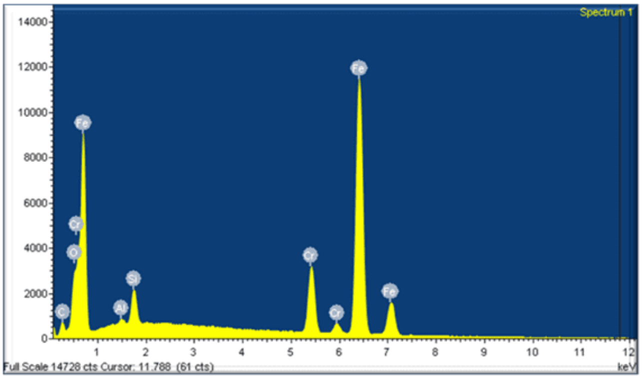


Figure 8: Colour mapping of Group B

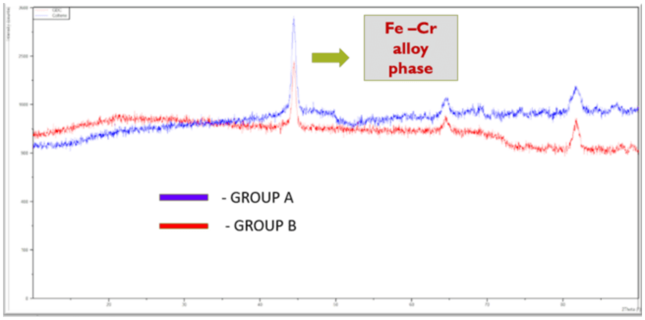
The X-ray diffraction (XRD) analysis revealed peaks at identical positions for both rubber dam clamp groups, indicating the presence of a similar Fe-Cr alloy phase in each. This finding is visually represented in **Graph 3**.



Graph 1: Graphical results of Group A



Graph 2: Graphical results of Group B



Graph 3: Graphical representation of the XRD result

4. Discussion

Rubber dam clamps have long been integral to dental procedures, particularly for achieving proper isolation and moisture control during restorative and endodontic treatments. Although widely used, their long-term durability and reliability continue to be areas of concern among dental professionals. Factors such as repeated sterilization, material fatigue, and potential for corrosion or fracture during clinical use may compromise their performance. These concerns underscore the importance of thoroughly evaluating the material composition and mechanical properties of rubber dam clamps to ensure their safety, effectiveness, and consistent performance in clinical settings.[8]

Rubber dam clamps are prone to fracture during clinical procedures, and this risk may stem from various factors such as the corrosive effects of sodium hypochlorite, mechanical fatigue due to repeated usage, and structural degradation caused by repeated autoclaving. In our department, it was observed that one brand of clamps consistently outlasted another in terms of clinical durability. This variation in performance raises concerns about the material integrity of these devices. To accurately determine the reasons behind such failures, a comprehensive understanding of the metal alloy composition and microstructural characteristics of the rubber dam clamps is essential for improving their reliability.[9]

Zinelis S et al.[6] studied the composition of the alloys used for the production of rubber dam clamps, and two types of composition was noticed. One, plain carbon martensitic steel (~0.8 wt.% carbon), electroplated with a nickel coating (600–650 Vickers hardness) and other, Martensitic stainless steel 7C27Mo (13.5 Cr1.0Mo, 0.38 C, 0.55 Mn0.40 Si wt%)(525 Vickers hardness). Alternatively, the type of 420 martensitic stainless steel is used as per US government document (Medical Procurement Item Description 1983).[10]

As in the methodology, the purpose of sectioning was to allow for consistent comparison and to fit within the sample holders of the analytical instruments. All samples were polished to ensure a smooth and reflective surface, which is critical for obtaining high-resolution images. The etching process was done to selectively corrode the surface layers, highlighting features such as grain boundaries, phase differences, and other metallurgical characteristics necessary for SEM analysis. SEM –EDS provides images of the surfaces of the metal at extremely high magnifications and helps to study metal material significantly, while XRD helps to look into the phases of the crystal lattice. Other methods to investigate the chemical composition of an alloys (or any metal), includes X-ray fluorescence spectroscopy (XRF) and optical emission spectroscopy (OES).[11]

The scanning electron microscopy (SEM) analysis did not show significant changes in the overall surface morphology of the clamps. However, it is important to consider the possible existence of microstructural imperfections that may not be visibly apparent but could still impact the mechanical performance of the clamps. Such hidden defects might affect properties like strength, durability, and resistance to fracture, highlighting the need for more in-depth structural evaluation.

EDS color mapping analysis revealed the presence of a trace amount of nickel in the clamps from Group A, whereas the clamps from Group B were found to be largely free of nickel. This subtle compositional difference may influence material properties such as corrosion resistance and mechanical strength.

According to SB Pratomo et al.,[12] increasing the nickel content from 0.3% to 1% resulted in enhanced tensile strength and hardness of the material, while the impact toughness remained largely unaffected. This suggests that even a small increase in nickel can significantly improve mechanical performance without compromising toughness.

Ahssi MA et al.[13] reported that nickel enhances the mechanical properties of metals by refining the grain structure, which increases toughness and strength. It also helps prevent the formation of scale on the material's surface. When alloyed with chromium, nickel further improves hardness, ductility, fatigue resistance, and the critical cooling rate. Moreover, due to its low diffusion coefficient, elemental nickel diffuses more slowly into iron, influencing the alloy's microstructural stability.

XRD analysis revealed the presence of Fe-Cr phases in both types of clamps, with no discernible differences in the phases between the two companies. According to Sabet H et al.[14] the absence of observable changes in the crystallographic structure indicates that the clamps have a similar composition at the macroscopic level. This suggests that, despite being manufactured by different companies, the fundamental material properties of the rubber dam clamps remain consistent, which could influence their mechanical behaviour and performance in clinical applications.

Other factors such as dimensional changes, surface corrosion, clinical handling damage, and variations in usage between operators due to repeated use and sterilization were not assessed in this study. These aspects could significantly affect the clamp's strength and durability over time. Understanding how these factors influence performance is crucial, as they may impact the clinical effectiveness and lifespan of the clamps. Therefore, these variables represent important areas for future research to provide a more comprehensive evaluation of rubber dam clamp reliability and safety in routine dental practice.[15]

5. Conclusion

Within the limitations of this study, the detection of a minute amount of nickel in Group A rubber dam clamps may be attributed either to intentional alloying aimed at enhancing fracture resistance or to surface electroplating designed to improve corrosion resistance. Nickel is known to contribute positively to mechanical properties such as toughness and strength, and its presence, even in trace amounts, could influence the performance and longevity of dental clamps. However, the scope of this investigation was limited in terms of sample size and resolution. To draw more definitive conclusions, further studies involving a larger number of samples and more advanced analytical techniques, such as nanoscale characterization, are recommended. Additionally, the current absence of standardized testing protocols for rubber dam clamps highlights the need for the development of specific guidelines to assess their material quality, mechanical durability, and clinical reliability. Such standards would help ensure consistency and safety in dental practice.

6. Source of Funding

None.

7. Conflict of Interest

None.

8. Ethical committee approval

The present study was approved by the Institutional Ethics Committee with an IEC/IRB No. KMCTDC/IEC/C/2024/05.

References

1. Ahmad IA. Rubber dam usage for endodontic treatment: a review. *Int Endod J*. 2009;42(11):963–72.
2. Bhuva B, Chong BS, Patel S. Rubber dam in clinical practice. *ENDO (Lond Engl)*.2008;2(2):131–41
3. Goodhew PJ, Humphreys J, Humphreys J. Electron microscopy and analysis. London. CRC press; 2000.
4. Garratt-Reed AJ, Bell DC. Energy dispersive X-ray analysis in the electron microscope.1st Ed. London .Taylor and Francis Group; 2003.
5. Epp J. X-ray diffraction (XRD) techniques for materials characterization. Mater. Charact. Using Nondestruct. Eval. (NDE) Methods. 2016:81–124.
6. Zinelis S, Margelos J. In vivo fracture of a new rubber-dam clamp. *Int Endod J*. 2002;35(8):720–3.
7. Yu T, Zhong F, Zhang F, Ying C, Geng G. Application of scanning electron microscopy in metal material detection.J Phys: Con Ser 2021;2002(1):012010.
8. Miao C, Yang X, Wong MC, Zou J, Zhou X, Li C, et al. Rubber dam isolation for restorative treatment in dental patients. *Cochrane Database Syst Rev*. 2021;5(5):CD009858.
9. Sutton J, Saunders WP. Effect of various irrigant and autoclaving regimes on the fracture resistance of rubber dam clamps. *Int Endod J*.1996;29(5):335–43.
10. Stainless Steel - Grade 420 (UNS S42000) [Internet].2021[23 Oct]. Available from: www.azom.com/article.aspx?ArticleID=972
11. Girão AV, Caputo G, Ferro MC. Chapter 6 - Application of Scanning Electron Microscopy–Energy Dispersive X-Ray Spectroscopy (SEM-EDS). *Compr Anal Chem*. 2017;75:153–68.
12. Pratomo SB, Oktadinata H, Widodo TW. Effect of nickel additions on microstructure evolution and mechanical properties of low-alloy Cr-Mo cast steel. *IOP Conf Ser: Mater Sci Eng*. 2019; 541: 012050.
13. Ahssi MAM, Erden MA, Acarer M, Çuğ H. The effect of nickel on the microstructure, mechanical properties and corrosion properties of niobium–vanadium microalloyed powder metallurgy steels. *Mater (Basel)*. 2020;13(18):4021.
14. Sabet H, Mirdamadi S, Kheirandish S, Goodarzi M.SEM and XRD Study of Hypoeutectic Fe-Cr-C Hardfacing Alloy Microstructure in as Weld Condition. *J Appl Chem Res*. 2010;15:70–78
15. Chhabra M, Greenwell AL. Effect of repeated sterilization on the tensile strength of rubber dam clamps. *Pediatr Dent*. 2018;40(3):220–3.

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