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Original Article

SEM and elemental analysis evaluation of the working surface of instruments from two different nickel titanium rotary file systems

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Abstract

Aim: The aim of the present study was to evaluate the surface changes in two different systems of rotary instruments before and after use by means of scanning electron microscopy (SEM) and energy dispersive analysis (EDA).

Materials and methods: We selected thirty files and divided them equally into two groups of 15 files each based on their manufacturing technology. Each file was used to prepare 5 root canals of freshly extracted human teeth. All the files were subjected to SEM and EDS before and after being used and the results compared to evaluate the surface changes. The results were then compared between the two groups using the t-test.

Results: The SEM images showed a greater mean difference in the surface changes before and after use for the system that employed only the M-wire manufacturing technology (1.46±1.68) vs. (3.07± 2.01), compared with the system that combined the M-wire and Gold manufacturing technologies (2.06±1.62) vs. (3.33±2.19). The most common observation in both systems was debris accumulation. Other notable findings included microfractures and file separation. The EDS results suggest that the more modern system has a higher loss of chemical composition after use compared to the system using only M-wire technology.

Conclusion: The present study provides evidence confirming that the use of Ni-Ti rotary file systems leads to the progression and possible creation of new surface changes. They increase the likelihood of iatrogenic errors and treatment failure in both file systems. Long-term use of files with gold manufacturing technology is not recommended, but for single use they outperform those with M-wire technology.

Keywords

endodontic instruments, fracture, root canal preparation, surface changes

Introduction

With today's modern technologies, many new endodontic instrument systems with different characteristics and unique designs are being produced. Unfortunately, the existing literature provides limited information on their surface changes before and after use. The instrument design must be taken into consideration. In addition, irregularities on the surface of instruments can cause fracture during clinical use[1] due to cracks resulting from superficial defects[2]. Most notably, separation can occur without any visible defect or previous deformation.[3] Using SEM analysis, researchers can visualize the microstructural changes that occur on the surface of these instruments after clinical use. The studies evaluating NiTi endodontic instruments indicate that the fracture of instruments inside the root canal is occasionally due to defects that are not visible.[4] Therefore, it is relevant to investigate the surface characteristics of new and used NiTi files to avoid unexpected file separation. [5] Preventive disposal of instruments before reaching the limit of their service life can prevent accidents. Therefore, in order to define the service life of endodontic instruments quantitatively and qualitatively, further studies are required.[6] Energy dispersive spectroscopy (EDS) analysis is a useful method to provide information on the chemical composition of instruments. The most common elements found are Ni and Ti. This analysis can be used to evaluate and compare the surface chemical composition of different instrument systems.

Aim

Therefore, the aim of this study was to evaluate the surface changes in rotary instruments of two different systems before and after use by means of SEM and EDS.

Materials and methods

Instruments

In this study, SEM and EDS analysis were used to compare the working surface morphology of new and used ProTaper NEXT files (Dentsply Maillefer, Ballaigues, Switzerland) and ProTaper Ultimate files (Dentsply Maillefer, Ballaigues, Switzerland). Specifically, we evaluated the changes in surface topography and surface chemical composition before and after the preparation of five root canals. A total of 30 instruments (15 from the ProTaper NEXT system and 15 from the ProTaper Ultimate system) were selected, numbered and divided into two groups: group U for the ProTaper Ultimate system and group N for the ProTaper Next system. The M-wire and Gold technology files from the ProTaper Ultimate system were compared to the M-wire technology files from the ProTaper NEXT system. It should be noted that although the alloy is similar between the Slider file of ProTaper Ultimate and the ProTaper NEXT instruments, they have different cross-sections, diameters, and taper.

Protocol of instrument usage and sterilization

Thirty freshly extracted upper molars were selected. Each instrument was used to prepare 5 root canals. After access cavity preparation, each orifice was relocated using Sx file from the ProTaper Ultimate system (Dentsply Maillefer, Ballaigues, Switzerland). This was followed by working length (WL) determination using an apex locator Propex Pixi (Dentsply Maillefer, Ballaigues, Switzerland) and Protrain (Simit Dental, Italy). The protocol for root canal shaping with the ProTaper NEXT (Dentsply Maillefer, Ballaigues, Switzerland) system consisted of manual scouting of the root canal, followed by WL determination using K-file (10/02) (Mani, Tochigi, Japan). Glide path was created manually using K-file (15/02) and (20/02) (Mani, Tochigi, Japan). This was followed by using X1, X2, and X3 instruments (Dentsply Maillefer, Ballaigues, Switzerland). The shaping protocol using ProTaper Ultimate (Dentsply Maillefer, Ballaigues, Switzerland) system consisted of simultaneous scouting, WL determination and Glide path management using the Slider instrument (Dentsply Maillefer, Ballaigues, Switzerland). In cases where the instrument did not progress in a pressureless manner, a K-file (10/02) (Mani, Tochigi, Japan) was used to complete the scouting and determine WL using Propex Pixi (Dentsply Maillefer, Ballaigues, Switzerland) and Protrain (Simit Dental, Italy). This was followed by using the Slider of Dentsply Maillefer, Ballaigues, Switzerland. The rest of the shaping procedure was completed using the Shaper, F1, F2, and F3 files (Dentsply Maillefer, Ballaigues, Switzerland). During all steps of root canal preparation, Chloraxid 2% (Cerkamed, Poland) was used as an irrigant at room temperature. After the preparation of 5 root canals, the instruments were cleaned using ultrasonic cleaners – standard (Isolab Laborgeräte GmbH, Eschau, Bayern). Following this, all instruments were autoclaved using the Universal program of the Vacuklav 23b+ (MELAG, Berlin, Germany).

Analysis of the selected instruments before and after use

Structural and morphological analysis of the rotary instruments surface before and after use was carried out using scanning electron microscopy (SEM) (Prisma E SEM, Thermo Scientific, Waltham, MA, USA). Each tool is mounted using an aluminum strip on the surface of an aluminum holder. Each of the samples was visualized at 200× and 1000× magnifications and an accelerating voltage of 20 kV using an ETD (Everhart-Thornley) detector. Each instrument was scanned once at 200× magnification and three more times at 1000× magnification. Energy dispersive (EDS) analysis was performed to determine the chemical composition of each sample at three individual points, at 200× magnification and an accelerating voltage of 20 kV, using Pathfinder™ X-ray Microanalysis Software (Thermo Scientific, Waltham, MA , USA).

The evaluation of the SEM images was performed using a customized scale adapted from the methodology proposed by Jovanović-Medojević et al.[7] This custom scale ranges from 0 to 7, with each score representing different levels of surface alterations observed on the instruments:

- 0: Represents a perfectly clean instrument surface.
- 1: Indicates the presence of small packs of debris.
- 2: Signifies the presence of large packs of debris.
- 3: Indicates a damaged cutting blade.

- 4: Represents small pitting on the surface.
- 5: Represents large pitting on the surface.
- 6: Indicates the presence of microfractures.
- 7: Indicates complete file fracture.

The SEM images were analyzed by trained observers who were blinded to the instrument groups. Each instrument was assigned a score based on the observed surface alterations according to the predefined scale. After analyzing the images of Group U and Group N, the differences were assessed between the scores of the two groups. The EDS was used to determine the relative loss in chemical composition of both group and then the results were compared.

After sterilization, all instruments were subjected to SEM and Elemental analysis using the same methodology to assess any changes in surface morphology after preparation of 5 root canals.

Statistical analysis

Python 3.10 was used to analyze the data. Python Libraries (Pandas, NumPy) were used, and Jupiter Notebook was utilized to execute the code. The T-test was used to determine the relationships between changes in the surface and the chemical composition of the instruments before and after use. The Shapiro-Wilk test presented normally distributed data (p>0.05). All statistical tests were performed at a type I error α=5% (p<0.05). The data is reported with mean values and standard deviation.

Results

The surface changes of the selected instruments from ProTaper NEXT and ProTaper Ultimate were evaluated using a scale ranging from 0 to 7, and the results are shown in **Fig. 1**. **Fig. 1A** shows the SEM results before and after using the instruments from group U, whereas **Fig. 1B** – the results after using the instruments from group N.

The data from Group U showed that the most prominent score prior to use was 1. 53.3% of all samples in the relevant group contain a visible, modest quantity of debris prior to use. The score of 3 was with a 20% frequency. The cutting blades on these instruments were damaged prior to use. **Fig. 2** shows an example of a defect in instrument 14 before use, magnified by ×1000.

After they were used, the most dominant score of the samples in group U was in the custom scale value 2 and

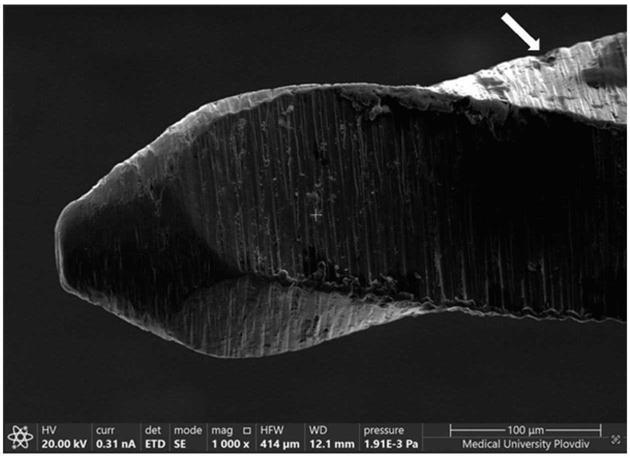

Figure 2. The white arrow points to the damaged cutting blade of tool F2 before use.

was observed in 46.7%. This indicates accumulated large amount of debris. Twenty percentages (20%) of the samples in the same group after use present with the score of 7, indicating complete file fracture. All instruments with the score 7 after use were Slider. In **Fig. 3A** and **Fig. 3B**, the SEM of instrument number 2 is displayed.

Out of all samples in group N, 40% were scored with 1 showing a residual small amount of debris before use. After use, the data shows 33.3% of the samples presented with large amounts of residual debris on their surface. **Fig. 4** shows instrument number 8 before use **(Fig. 4A)** and after use **(Fig. 4B)**.

It should be noted that we observed microfractures in 20% of all samples of group N after use. **Fig. 5** shows in-

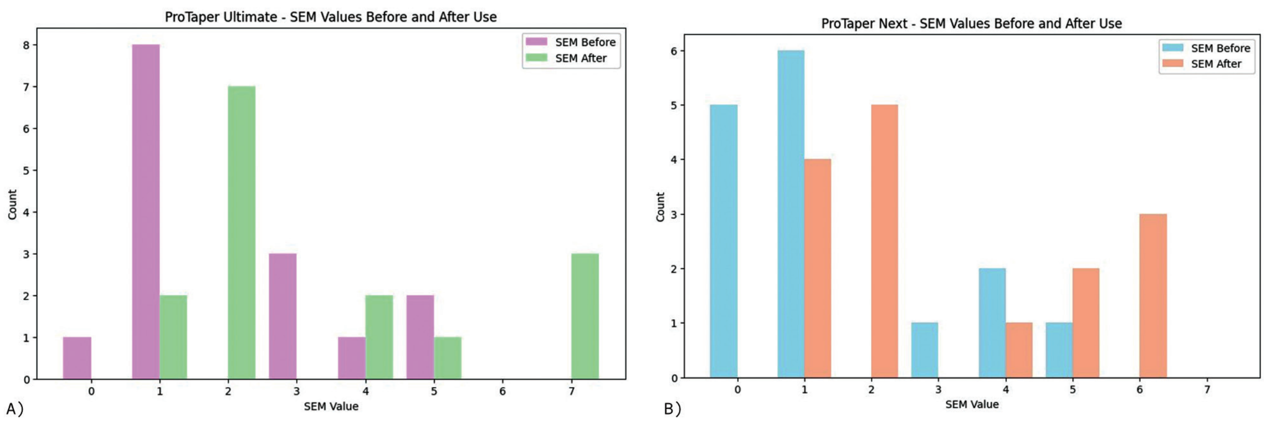

Figure 1. A) SEM results before and after using Group U instruments (purple and green); **B**) SEM results before and after using Group N instruments (blue and orange).

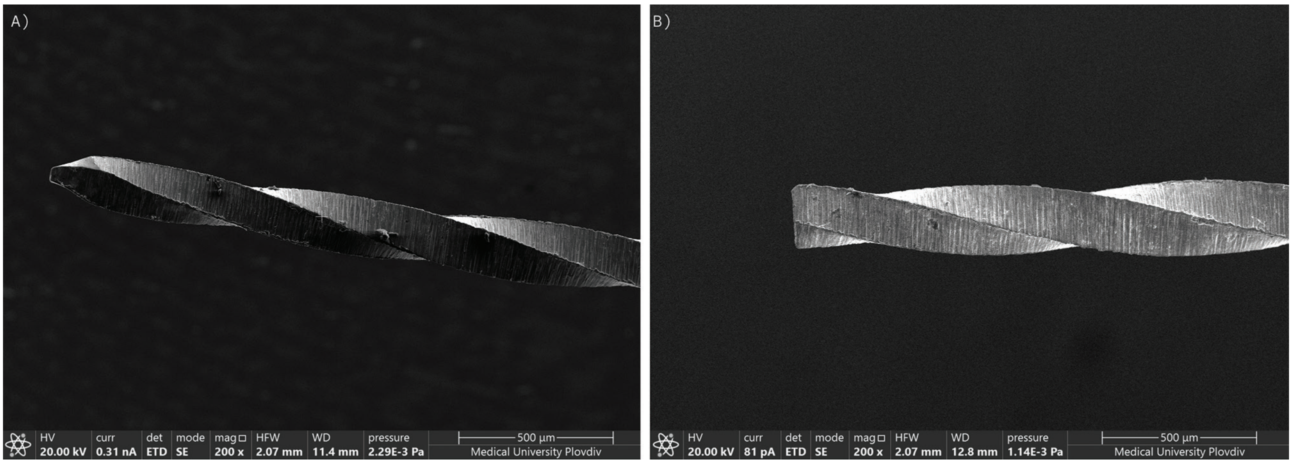

Figure 3. A) Visible small amount of debris before use of the Slider instrument; **B**) Visible complete file fracture of the Slider instrument after use.

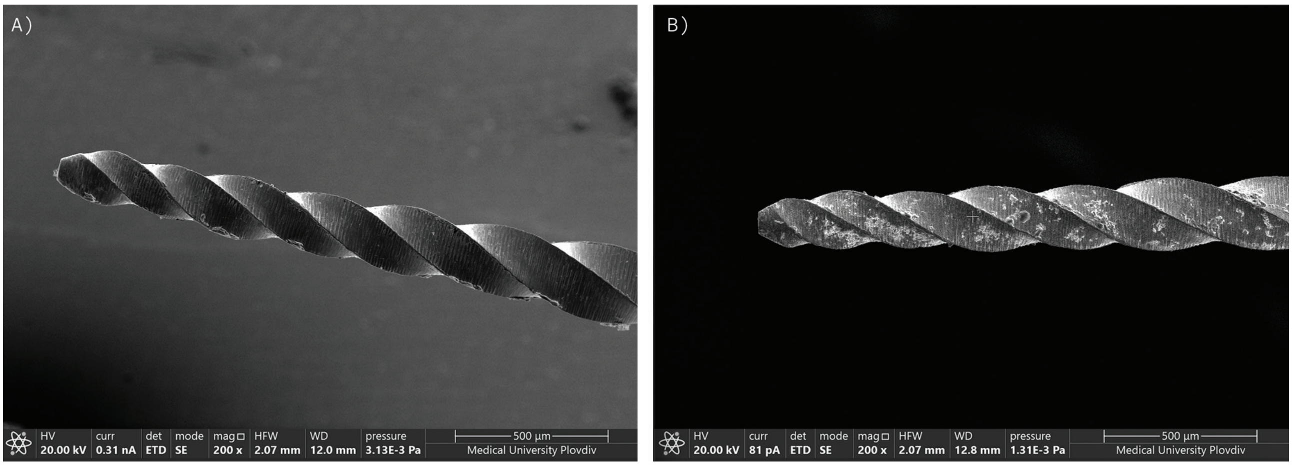

Figure 4. A) Visible small amount of debris on instrument X1 before use; **B**) Visible large amount of debris on instrument X1 after use.

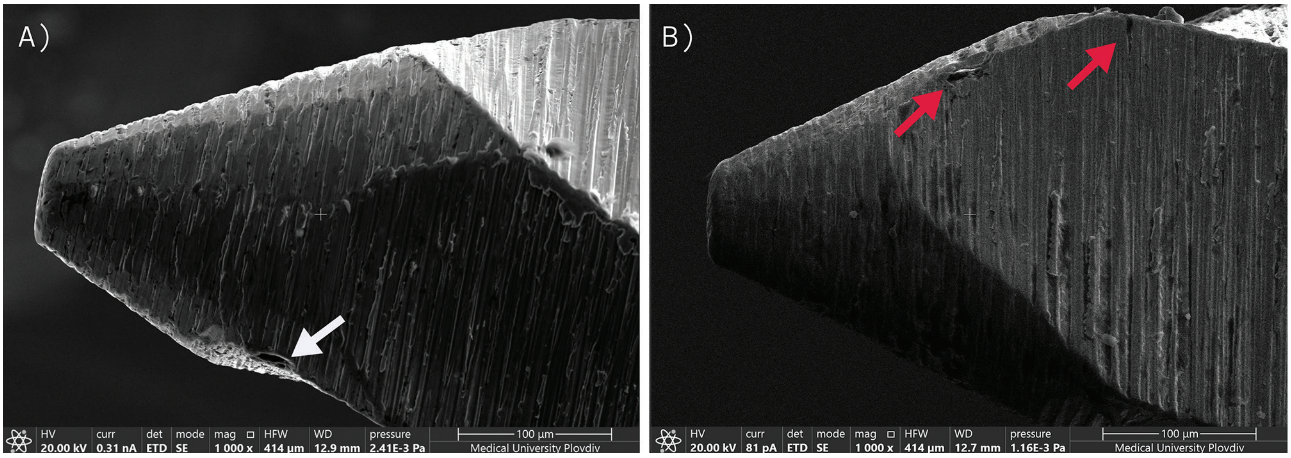

Figure 5. A) A white arrow points toward a large pitting on the X3 instrument before use; **B**) Red arrows point towards two visible microfractures on the X3 instrument after use.

strument number 10 before use **(Fig. 5A)** and after use **(Fig. 5B)** with magnification of ×1000.

The mean SEM score of all samples in group U before use was 2.067±1.62 and the mean SEM score of all samples in the same group after use was 3.333±2.19 (p=0.092). The same difference was calculated for the samples of group N. The instruments from group N before use showed a value of 1.467±1.68 and the score after use was calculated as 3.067±2.01 (p=0.020). **Table 1** displays the mean SEM scores of groups U and N.

After analyzing the results from the EDS of both groups, the mean Ni and Ti chemical composition was assessed. Then the mean difference in both elements was evaluated and displayed for group U and group N in **Table 2**. The mean difference in the chemical composition loss of Ni (p=0.256) and Ti (p=0.368) was compared between the two groups in **Figs. 6A,B**.

Discussion

The SEM and EDS analyses of ProTaper Ultimate (Dentsply Maillefer, Ballaigues, Switzerland) and ProTaper NEXT (Dentsply Maillefer, Ballaigues, Switzerland*)* before and after root canal preparation revealed significant insights into the surface morphology and integrity of these rotary endodontic instruments. During root canal preparation, rotary files are subjected to torsional and flexural fatigue.[8] Our findings show that based on SEM results, ProTaper Ultimate shows an overall better mean difference of the surface deformations before and after use compared with ProTaper NEXT. This can be explained by the fact that ProTaper NEXT is with a different manufacturing technology.[12] Our findings suggest better overall performance of ProTaper Ultimate if used in up to 5 root canals. One study conducted SEM analysis on the WaveOne (Dentsply Maillefer, Ballaigues, Switzerland) and WaveOne Gold (Dentsply Maillefer, Ballaigues, Switzerland*)* files after use observing similar surface alterations such as debris accumulation and microfractures.[9] It concluded that based on the SEM results before and after use, the samples from WaveOne showed better overall results before and after use. WaveOne is made with the M-wire technology, similar to the ProTaper NEXT (Dentsply Maillefer, Ballaigues, Switzerland). WaveOne Gold (Dentsply Maillefer, Ballaigues, Switzerland) shares the same manufacturing technology with most of the analyzed instruments in our study from ProTaper Ultimate (Dentsply Maillefer, Ballaigues, Switzerland). Our findings indicate the contrary, that

Table 1. Mean SEM values of the samples from group U and group N before and after use

Score	Before use Mean±SD	After use Mean±SD	Paired t-test *p*-value
ProTaper Ultimate	2.06±1.62	3.33±2.19	0.092
ProTaper NEXT	1.46±1.68	3.07±2.01	0.020

Table 2. Mean Ni and Ti percentages of group U and group N

Group	Mean chemical composition				Mean difference in the chemical composition before and after use	
	Before use		After use			
	Ni	Ti	Ni	Ti	Ni	Ti
Group U (Mean±SD)	52.667±2.02	43.2±1.93	47.2±5.74	38.867±4.27	5.467±5.13	4.333±4.06
Group N (Mean±SD)	55.267±0.88	45.133±2.28	51.6±3.24	42.067±2.81	3.667±3.13	3.067±3.49

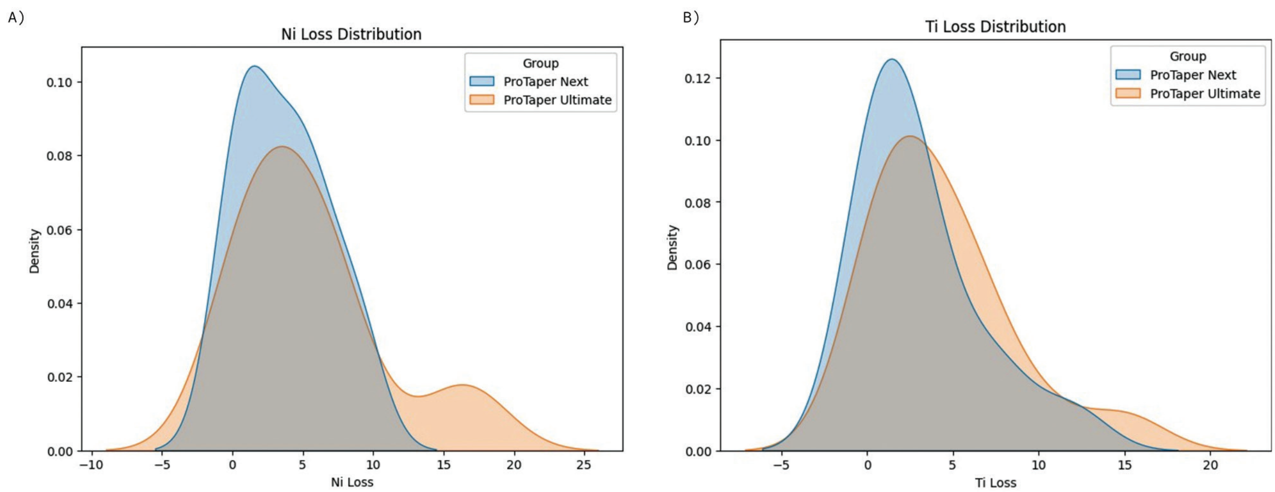

Figure 6. **A**) Ni loss difference between group U (orange) and group N (blue) (p>0.05); **B**) Ti loss difference between group U (orange) and group N (blue) (p>0.05).

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the Gold manufacturing technology showed better results when comparing the SEM values. It has been shown that defects in the manufacturing process can lead to the fracture of instruments.[10] Our study also confirm that the non-visible defects in the surface of the instruments can progress and lead to microfractures or complete file fractures. Our data noted that the Slider file from ProTaper Ultimate (Dentsply Maillefer, Ballaigues, Switzerland) has higher frequency of separation during root canal preparation. This can be associated with its designed purpose – rotary first approach[11], which is not recommended in the more difficult and narrow root canals to avoid file fracture. When discussing the EDS results, the ProTaper Ultimate (Dentsply Maillefer, Ballaigues, Switzerland) shows higher loss of Ni and Ti than ProTaper NEXT (Dentsply Maillefer, Ballaigues, Switzerland). The reason for this is most likely to be found in the production technology of the alloy.[12] During the manufacturing process, the working surface of instruments, especially their threads, might have residuals of metal strips and organic and non-organic debris which might have infective and non-specific irritating potential.[4] This accumulation of debris can potentially compromise treatment outcomes if not effectively managed.[13] Our study showed similar results with the highest rates of small amount of visible debris in both systems before use and large amounts after use.

Conclusion

By understanding the changes in the surface morphology and integrity, clinicians can make informed decisions regarding instrument selection, maintenance, and replacement to optimize treatment outcomes in root canal therapy. The frequent findings of residual debris underscore the need for regular instrument inspection, cleaning, and maintenance protocols to mitigate the risk of debris accumulation and ensure optimal instrument performance over time. Defects such as blade deformation, pitting and microcracking should encourage clinicians to limit instrument use to single use. Excessive instrument usage in more than 5 root canals, especially using the 'rotary first' approach should be avoided due to a higher risk of file separation. Further use for more than 5 root canals is not recommended for instruments with Gold heat treatment due to higher loss of chemical composition which increases the risk for procedural errors.

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