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RESEARCH ARTICLE

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Investigation of four nickel titanium endodontic instruments, with cyclic fatigue resistance, scanning electron microscopy, and energy dispersive x-ray spectroscopy

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Abstract

The aim of this study was to compare of four different nickel-titanium (Ni-Ti) endodontic files and evaluate in terms of cyclic fatigue resistance and metallurgical properties. Four different type Ni-Ti root canal files Protaper Next X2 (PTN) (Dentsply Maillefer, Ballaigues, Switzerland), One Curve (OC) #25.06 (Micro Mega, Besancon, France), EndoPlus Flex Plus Gold X2 (EPG) (Turkuaz Dental, Denizli, Turkey), and EndoPlus Flex Plus Blue #25.06 (EPB) (Turkuaz Dental, Denizli, Turkey) files were tested for cyclic fatigue resistance ($n = 20$). During experiments artificial zirconia block canal was used. The artificial canal designed with curvature 60° and 5-mm radius. The number of cyclic to fracture (NCF) was noted. Fractured length (FL) parts of Ni-Ti files were recorded to assessment of fracture volumetric point. All fractured surfaces of Ni-Ti files were assessed by scanning electron microscope (SEM) to confirm the type of fractures. Descriptive evaluation become accomplished for the fundamental composition of units with the aid of using energy-dispersive x-ray spectroscopy (EDX). NCF data were evaluated via Bonferroni test with post hoc multiple comparison method. OC showed the highest NCF values ($p < .05$). The standardization of the study was confirmed as the FL of files was statistically similar in length ($p > .05$). SEM analysis confirmed that all scanned samples were fractured due to cyclic fatigue. EDX analysis confirmed that EPB established the poorest Ni content file.

Research Highlights

- The cyclic fatigue-related failure of One Curve was significantly more resistant than Protaper Next and EndoPlus files.
- Scanning electron microscopy images showed that One Curve and Protaper Next have round tips
- Energy dispersive x-ray spectroscopy showed that all four endodontic instruments mainly have Nickel and Titanium elements

KEYWORDS
cyclic fatigue, EDX, EndoPlus flex plus, one curve, Protaper next

1 | INTRODUCTION

Today, it is impossible to imagine modern endodontics without nickel-titanium files (Ni-Ti). Ni-Ti files cause less complications such as perforations, zips, and ledges compared with stainless steel files (Cheung, 2007; Peters, 2004). In addition, Ni-Ti files are more flexible than stainless steel files, so it is more resistant to breakage in root canals with sharp curvatures (Alapati et al., 2005; Iqbal et al., 2006). As clinicians search for more durable Ni-Ti files, many companies are search to increase the strength of Ni-Ti files (Parashos et al., 2004). Various technologies have been developed to increase the durability of Ni-Ti files against both cyclic and torsional fatigue (Plotino et al., 2009). One of the important technologies is surface electropolishing processes to prevent Ni-Ti file fractures caused by micro cracks on the surface of the Ni-Ti files (Peters & Paqué, 2010). The fact that the cross-sections of the Ni-Ti files are designed in different ways is one of the elements that increase their strength (Martins et al., 2022). In addition, nonactive rounded tip of the files contribute to reducing the risk of perforation during root canal preparation (Morales et al., 2021). However, increasing the durability of Ni-Ti files by heat treatment has shown a dramatic improvement in the resistance of the files (Martins et al., 2023). Manufacturer companies increase the temperature of Ni-Ti files to a certain temperature, and then quickly cool them down to a certain temperature. Depending on the differences in this heating process, martensitic and austenitic phase balances of Ni-Ti files are tried to be adjusted. The more martensitic phase of the Ni-Ti files, the more durable and softer it is. However, since the dentin penetration of martensitic phase Ni-Ti files is low, a balance must be established between the martensitic phase and the austenitic phase (Hou et al., 2021). There are many studies in the literature investigating the cyclic fatigue of Ni-Ti files. Although most of these studies were done on heat-treated files, it was understood that the files had different resistances (Hou et al., 2021; Peters & Paqué, 2010; Plotino et al., 2009). Therefore, the strengths of heat-treated files differ. In this study, four different file systems, which were heat treated, were subjected to cyclic fatigue testing. In addition, the tips of the files were examined by scanning electron microscope (SEM) analysis and files metallurgical properties by energy dispersive x-ray analysis (EDX). These files are;

ProTaper Next X2 (PTN) (Dentsply Maillefer, Ballaigues, Switzerland) is a Ni-Ti file system has M-Wire heat treatment technology. It is also designed off-centered rectangular cross-section and has regular taper (#25.06) (Koçak et al., 2021).

One Curve (OC) (Micro Mega, Besancon, France) Ni-Ti file is produced for instrumentation of the root canal with a single file and it has regular taper (#25.06). OC Ni-Ti file is produce with C-wire heat treatment technology. Also the manufacturer's declared that the OC file has control memory and it can be prebent to preserve the shape of the root canal. OC has two different cross-section region, the apical 3 mm region has variable three cutting-edges design and the remainder coronal region has two cutting-edges design (Ertuğrul, 2019).

EndoPlus Flex Plus Gold (EPG) and EndoPlus Flex Plus Blue (EPB) (#25.06) (Turkuaz Dental, Denizli, Turkey) are newest produced Ni-Ti files and they have different colors because of different heat treatment process. Also they have triangle cross-section design along the file (EndoPlus Endodontic System, 2023).

The aim of this study was to compare of four different nickel-titanium (Ni-Ti) endodontic files and evaluate in terms of cyclic fatigue resistance and metallurgical properties. The null hypothesis of the study was the newest EPG and EPB files have been higher cyclic fatigue resistance than other files. Also EPG and EPB files have safety non-active round tips.

2 | MATERIALS AND METHODS

Based on data from previous studies (Ertuğrul & Orhan, 2019; Grande et al., 2006), a power calculation was performed using G*Power 3.1 (Heinrich Heine University, Dusseldorf, Germany) software. The calculation indicated that the sample size for each group should be a minimum of 18 files. Four different type of Ni-Ti rotary files were tested in this study. Size #25.06 files were examined with stereomicroscope (SMZ1500, Nikon Corp., Tokyo, Japan) at 20× magnification, which has been detected any defect and deformity Ni-Ti file was replaced with a fit file.

2.1 | Cyclic fatigue tests

For cyclic fatigue tests total of 80 Ni-Ti files (PTN #20.05, OC #20.05, EPG #25.06, and EPB #25.06) were randomly selected in four groups for each Ni-Ti file system ($n = 80$). The static artificial canal was made of zirconia block (Upcera Technologies Inc., Shenzhen, PRC) described in previous studies (Ertuğrul & Orhan, 2019). The artificial canal curvature was setup with 60° angle and 5-mm radius of curvature. Synthetic oil (WD Company, Milton Keynes, UK) was used in the artificial canal in order to minimize the torsional forces acting on the Ni-Ti files during the experiments. All Ni-Ti files were rotated with endodontic motor (Reciproc Silver, VDW GmbH, Munich, Germany) at 300 rpm in the artificial root canal according to the manufacturer's instructions. The Ni-Ti files were rotated up to 19 mm in the artificial zirconia canal Figure 1. Each file was rotated in the artificial root canal until fractured. A digital camera was used for confirm the fracture time. All cyclic fatigue tests were occurred under 25°C. The time to file breakage was measured with a 1/100 s precision chronometer and recorded for each Ni-Ti file. Then, how many cycles each file made until fracture occurred was calculated with the following formula:

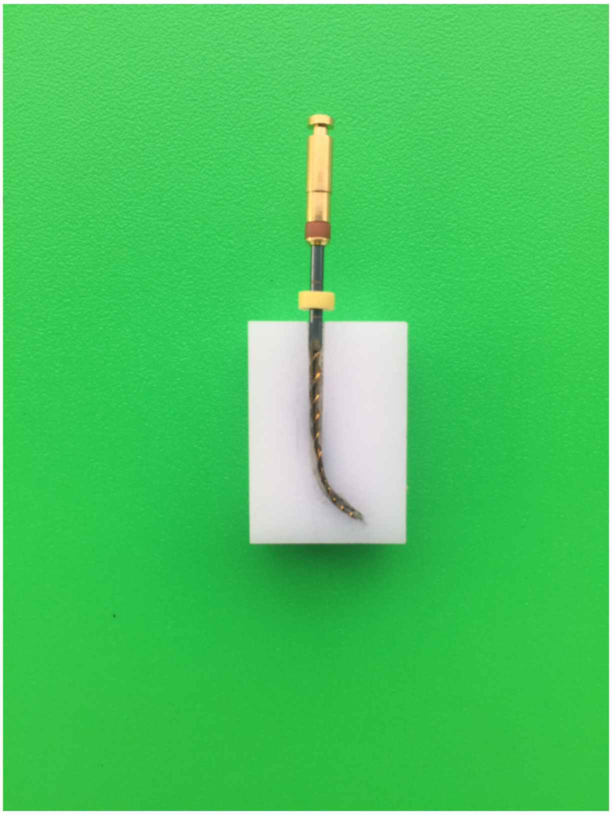

FIGURE 1 Experimental cyclic fatigue test model set-up photo.

The number of cycles to failure (NCF) = Revolutions per min (rpm)/60 × Time to fracture (s).

The fragment length (FL) of Ni-Ti files were measured with a digital caliper (Absolute Digimatic; Mitutoyo Corp, Kawasaki, Japan) and then recorded for each fragment. FL was statistically evaluated to confirm whether the files were positioned correctly in artificial root canal.

2.2 | SEM and EDX spectroscopy examination

All fractured parts were cleaned in alcohol with ultrasonically before fractured surface observation as described by Inan and Aydin (2012). Fractured Ni-Ti files surfaces were evaluated under SEM (Zeiss Supra 40VP, Carl Zeiss SMT Inc., Oberkochen, Germany) for detecting signs of cyclic fatigue failure. SEM used for two different magnifications (450× & 3000×) under 15.00 kV accelerating voltage. In addition, the form of the tip parts of the files was also observed with SEM under 500× magnification. EDX analysis was used to determined metallurgical properties.

2.3 | Statistical analysis

Quantitative data in the study were presented as mean–standard deviation marked with "±." Analysis of variance (ANOVA) for repeated measures was adopted to analyze NCF differences among the four groups. Bonferroni test was applied as a post hoc multiple comparison method. "$p < .05$" was considered as statistically significant level. The SPSS version 20.0 software 0 (SPSS, Inc., Chicago, IL) was used for the statistical analysis.

TABLE 1 Mean ± SD of the number of cycles to failure (NCF) and fragment length (FL) of Ni-Ti files.

	NCF* Mean ± SD	FL** Mean ± SD
Protaper Next	585.75 ± 73,15[a]	4,61 ± 0,09
One Curve	1073.75 ± 156,3[b]	4,42 ± 0,1
EndoPlus Flex Plus Gold	545.25 ± 129,65[a]	4,37 ± 0,15
EndoPlus Flex Plus Blue	695 ± 131,77[c]	4,49 ± 0,09

*Means with the same superscript lowercase letter are not significantly different ($p > .05$).
**There was no significant difference in the length of fracture fragment between files ($p > .05$).

3 | RESULTS

The NCF of the OC file was found to be statistically significant higher than PTN, EPG, and EPB files ($p < .05$). The NCF of PTN and EPB files no statistically significant difference found ($p > .05$) however NCF of PTN and EPB files is statistically significant higher than the EPG files ($p < .05$) Table 1. There is no statistically significant difference was found between the FL of all Ni-Ti files. Thus, it was understood that the file areas exposed to compression and tensile forces distribution were the close points. SEM images of the fractured planes showed typical signs of cyclic fatigue in all files Figure 2.

With the 500× SEM observation, OC and PTN files showed non-cutting rounded safety tips. EPG and EPB files showed unrounded tips. Additionally, from the SEM images, it was seen that the surface of the OC files was quite smooth than other files Figure 3. EDX analysis showed that all files were composed primarily of nickel and titanium, with slightly different percentages depending on the files tested Table 2.

4 | DISCUSSION

Present study is the first cyclic fatigue study with Ni-Ti EndoPlus files. OC and PTN files, which have been tested in many studies in the literature, were compared with EndoPlus files (Bürklein et al., 2013; Pérez-Higueras et al., 2014; Uygun et al., 2016; You et al., 2010). As a result of the study, the null hypothesis was rejected.

One of the reasons for early breakage of files is micro cracks on file surface (Gutmann & Gao, 2012). Files were examined under a microscope at 20× magnification to prevent premature fractures caused by microcracks and deformities.

Experimental work by Pruett et al. (1997) described the angular forms of ideal setup for cyclic fatigue tests. Although the test setups

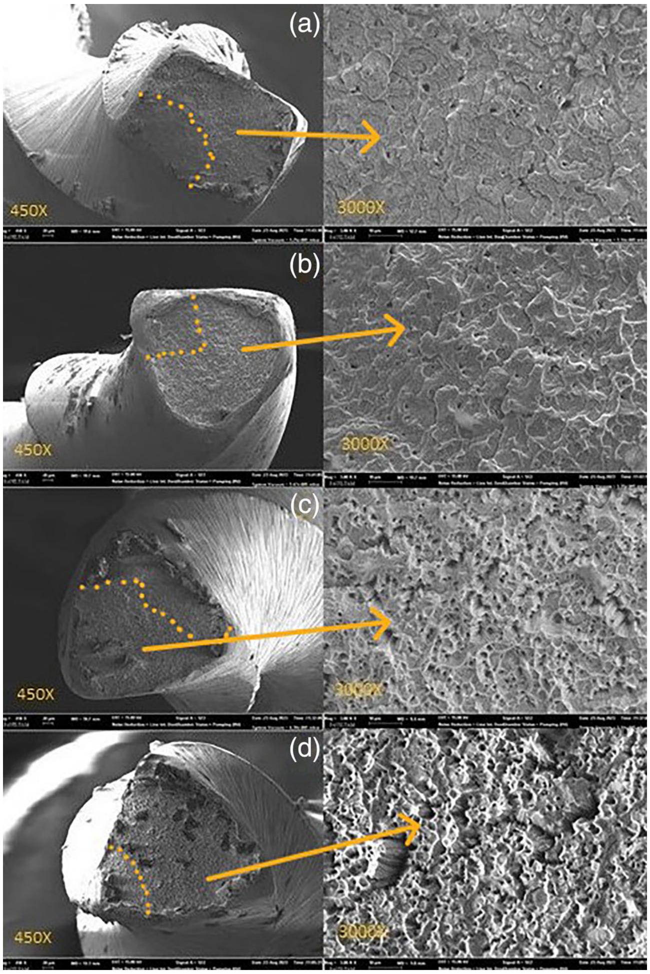

FIGURE 2 Scanning electron microscopic images of the fractured surfaces view of “A” PTN, “B” OC, “C” EPG, and “D” EPB with low (450×) and high (3000×) magnification view instruments. The crack initiation signs and the surface form with pits and bulges (circled area) are observed in the fracture surfaces.

of some studies were manufactured according to the Schneider approach, after the study of Pruett et al., cyclic fatigue tests in the literature were generally performed according to this angle system (Plotino et al., 2009). In present study, dental zirconia block material was used as an 60° angle and 5 mm radius of curvature artificial canal. Zirconium is a very suitable material for computer-aided manufacturing with micron precision. In addition, zirconium is resistant to abrasions (Thandalam et al., 2015). As in many previous cyclic fatigue studies, a lubricating synthetic oil was used in the artificial canal during the experiment to reduce the friction (Zhang et al., 2008). Present study, the torsional forces accumulated on the file were tried to be reduced with synthetic oil. In previous studies, both static and dynamic test mechanisms were used as cyclic fatigue tests. There is no study has been found showing the superiority of these test mechanisms over each other (Plotino et al., 2009). The reason why we preferred the static mechanism in our study is that the standard pecking motion used in dynamic tests is incompatible with clinical practice. In the artificial root canal, the files were rotated at a standard 19 mm depth. This method allowed compressive and tensile forces to act on the same point of the file (Inan & Aydin, 2012; Plotino et al., 2009). Many studies have used SEM analysis to confirm whether files are broken by cyclic forces and/or torsional forces. If there are spiral-shaped scars on the surface of the fractured file, this is known as torsional fatigue fracture (Griffiths et al., 2000; Plotino et al., 2009; Pruett et al., 1997). If there are traces in the form of rupture-separation, this is known as cyclic fatigue fracture (Alapati et al., 2005). In present study, SEM analysis showed that cyclic fatigue tests were successful. Because of Areas of crack initiation and growth displayed small nucleation regions and slow crack propagation, known as smooth regions, located around the cross-section's periphery. Crack propagation was characterized by striations, each indicating the crack's advancement due to tensile stress during the instrument's rotation. The manufacturer declared that the OC file has C-wire technology that is heat treated (Micoogullari Kurt et al., 2020). The PTN is a super-elastic Ni-Ti file (Elnaghy & Elsaka, 2014). Newly produced gold-colored and blue-colored EndoPlus Ni-Ti files were used in the study. It is known that the colors of NiTi instruments change after heat treatment. These colors are usually gold and blue. Also in the literature that the blue colored files are more flexible and durable by more martensitic phase depending on the heat treatment (Peters & Paqué, 2010). The present study also confirms this result. However, as the martensitic properties of the files increase, their penetration into the dentin decreases. The brochure of EndoPlus files does not contain information about the cross-section of the file (EndoPlus Endodontic System, 2023). When the SEM analyzes are examined, it is seen that the file has a triangular cross-section Figure 2. While the cross-section of EndoPlus and OC files is triangular, the cross-section of PTN files is rectangular. OC files have an off-centered cross-section at apical 2 mm part. PTN files have off-centered cross-section at all along (Ha et al., 2017).

One of the study shows that triangular files are more flexible than rectangular files (Versluis et al., 2012). However, it has been stated in finite element analysis study that files with triangular cross-sections are more resistant to cyclic fatigue than files with rectangular cross-sections (Cheung et al., 2011). In this study, it was understood that the triangular cross-section OC files were more resistant to cyclic fatigue resistance than the rectangular cross-section PTN files. However, there was no statistically significant difference between the triangular cross-section EndoPlus files and the rectangular cross-section PTN files against cyclic fatigue resistance.

In all cyclic fatigue tests, the zone where the fracture occurs is generally at the point of maximum stress and compression. The metal volume at the critical cyclic zone where the maximum stress point is located is one of the factors affecting the cyclic fatigue resistance of the file. If the metal volume is low at critical zone, the file will be more resistant, and if it is more, the file will be less resistant to cyclic fatigue (Grande et al., 2006). Therefore, files with the same taper were used for standardization of the study. In addition, the standardization of the study was confirmed by comparing the lengths of the broken file pieces with each other Table 1.

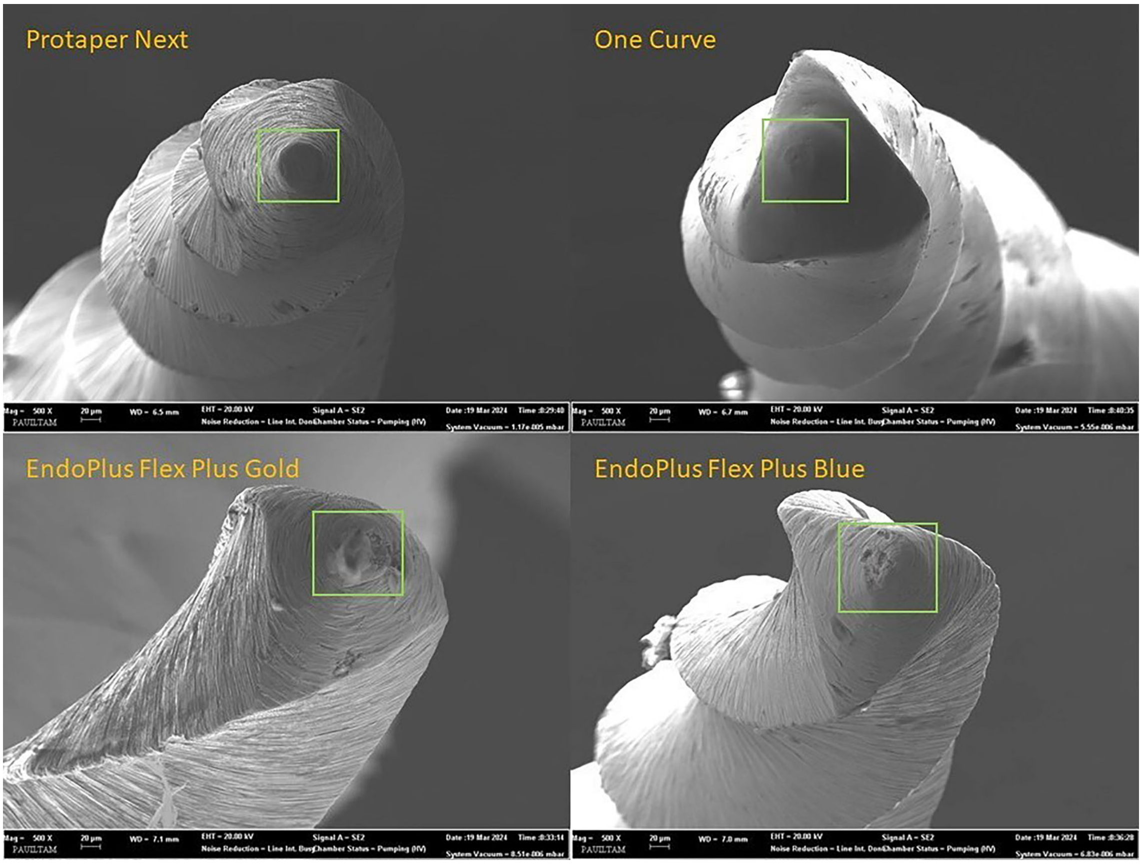

FIGURE 3 Scanning electron microscope analysis showing the surfaces and tips of the files.

TABLE 2 Energy-dispersive x-ray analysis presents the mean wt% ± standard deviations of each element of the four files.

	C	Si	Ti	Ni
Protaper Next	4.77 ± 0.12		42.94 ± 0.62	52.29 ± 0.71
One Curve	4.48 ± 0.18	0.48 ± 0.21	42.44 ± 0.58	51.81 ± 0.67
EndoPlus Flex Plus Gold	18.15 ± 0.45		38.35 ± 0.4	43.5 ± 0.58
EndoPlus Flex Plus Blue	4.48 ± 0.24	0.48 ± 018	42.44 ± 0.48	51.81 ± 0.62

Abbreviations: C, carbon; Si, silicon; Ti, titanium; Ni, nickel.

One of the previous study, OC and PTN files have been compared and it has been found that OC files are more resistant to cyclic fatigue as in present study (Ertuğrul, 2019). Although EndoPlus files have just been introduced to the market, they have been shown to be statistically less resistant to cyclic fatigue than OC files. In many studies in the literature, it has been reported that heat-treated files have higher cyclic fatigue resistance than non-heat-treated files (Keskin et al., 2018; Koçak et al., 2021; Plotino et al., 2009). However, when the cyclic fatigue resistance of EndoPlus files is compared with PTN files, there is no significant difference between them ($p > .05$).

Tip design plays a crucial role in the effectiveness and safety of root canal instrumentation. It guides the file through the canal and influences the outcome of shaping the root canal system. Tip designs can generally be classified into different types. Cutting tips are very aggressive in cutting dentin and have the ability to cut at the tip. They are particularly useful for navigating through narrow and calcified canals, as they facilitate easier access to the root apex. However, they require cautious use because their aggressive cutting action can lead to perforation of the root canal if not managed properly. Noncutting tips are designed solely to guide the instrument through the root canal without actively cutting dentin. They are less prone to torsional fracture compared to cutting tips. Noncutting tips are beneficial for cases where a more delicate approach is needed, such as in fragile or curved canals. Partially cutting tips combine characteristics of both cutting and noncutting tips. They have a partially active tip that provides some cutting action while also guiding the instrument through the canal. Partially cutting tips, like those found in the ProTaper Universal System, offer a balance between cutting efficiency and safety, reducing the risk of torsional fracture while still facilitating effective canal shaping (Bergmans et al., 2003; Griffiths et al., 2000).

Choosing the appropriate tip design depends on various factors such as the anatomy of the canal, degree of calcification, and the clinician's skill level. It is essential to understand the advantages and limitations of each tip design to achieve successful outcomes in root canal procedures while minimizing the risk of complications. Many previous studies have reported that files with rounded tips are safety during root canal preparation (Al-Saffar & Al-Gharrawi, 2023; Arias & Peters, 2022; Atique et al., 2022; Dos Reis et al., 2023). In the presented study, OC and PTN files showed rounded tips at 500× SEM magnification.

AUTHOR CONTRIBUTIONS

İhsan Furkan Ertuğrul: Conceptualization; methodology; data curation; supervision; investigation; writing – original draft; visualization. **Havva Kübra Arslan:** Methodology; conceptualization; writing – original draft.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are openly available in [repository name] at [URL], reference number [reference number].

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