UNIVERSIDADE MANTIDA PELA COMPANHIA
NILZA CORDEIRO HERDY DE EDUCAÇÃO E CULTURA
CNPJ 29.403.763/0001- 65

PROPEP
Pró-Reitoria de
Pós-Graduação e Pesquisa

UNIVERSIDADE DO GRANDE RIO
PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA

EXPLORANDO A PERFORMANCE E A FUNCIONALIDADE DE INSTRUMENTOS ENDODÔNTICOS A PARTIR DE UMA ABORDAGEM MULTIMÉTODO

TESE DE DOUTORADO

NATASHA CÂMARA AJUZ DEMIER

2023

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Orientador(a):
Prof. Dr. Emmanuel João Nogueira Leal da Silva

2023

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Aprovada em 09 de agosto de 2023

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DEDICATÓRIA

Dedico esta tese primeiramente a Deus e a minha família e meus filhos que são a minha fonte diária de inspiração.

AGRADECIMENTOS

Agradeço primeiramente a Deus.

Agradeço a minha família que me apoiou em todos os momentos que precisei, muitas vezes preocupados, mas nunca duvidaram que eu conseguiria.

Agradeço aos meus filhos Joaquim e Valentina, meus amores maiores e minha motivação, que são uma fonte de energia na minha vida e me fazem querer sempre ser uma pessoa melhor, para mim e para eles.

Agradeço ao Alexandre, meu parceiro, meu aconchego, meu porto seguro diário, que tanto me incentiva a buscar meus objetivos e de correr atrás pra que eu possa alcançá-los.

Agradeço ao Fernando, que me estimulou a me inscrever e começar o doutorado, quando juntos decidimos que era hora de iniciar essa jornada.

Agradeço ao meu orientador e amigo Emmanuel, que sempre se colocou à disposição, não hesitando em ajudar, sempre que eu precisasse.

Agradeço a todos os meus colegas que cursaram comigo o doutorado, e que me ajudaram a chegar até aqui, foram parceiros em trabalhos e em aulas online. Sem a amizade de vocês e todo o incentivo, com certeza teria sido muito mais tedioso e sem graça. Vocês transformaram o curso em algo extremamente prazeroso.

Agradeço a todos os professores que de alguma maneira, transmitiram seus conhecimentos, para que hoje eu pudesse me tornar Doutora.

EPÍGRAFE

“A sabedoria em tese é conhecimento, o conhecimento aplicado é sabedoria.”

Brenon Salvador

ÍNDICE

RESUMO

Objetivos: A ampla variedade de instrumentos disponíveis no mercado endodôntico atual requer uma análise criteriosa e precisa para garantir a segurança e eficácia clínica. Portanto, o objetivo da presente tese foi realizar, a partir de quatro diferentes estudos, uma avaliação abrangente de diversos instrumentos endodônticos disponíveis no mercado odontológico por meio de uma abordagem multimétodo. **Materiais e Métodos:** No capítulo 1, 2 e 3, diferentes instrumentos foram comparados quanto às suas características de design, propriedades metalúrgicas, desempenho mecânico e a capacidade de modelagem. Os instrumentos analisados em cada capítulo foram: Genius Proflex, Vortex Blue e TruNatomy no capítulo 1; REX, Reciproc Blue e WaveOne Gold no capítulo 2; e HyFlex EDM, Neoniti, EDMax e ProTaper Gold no capítulo 3. Já no capítulo 4, as características de design, propriedades metalúrgicas e o desempenho mecânico do sistema ProTaper Ultimate foram comparados com instrumentos de dimensões similares dos sistemas ProGlider, ProTaper Gold e ProTaper Universal. Os ensaios foram analisados utilizando os testes estatísticos necessários, sempre com um nível de significância estabelecido em 5%. **Resultados:** De maneira geral, os instrumentos testados em um mesmo capítulo apresentaram diferenças significativas em seu design geral, fases de transição de temperatura e comportamento mecânico. No entanto, essas diferenças não afetaram a capacidade de modelagem dos mesmos. **Conclusões:** A adoção de uma abordagem multimétodo, combinando metodologias qualitativas e quantitativas, ampliou a capacidade de resposta aos objetivos propostos e proporcionou *insights* valiosos para a prática clínica. Em geral, todos os sistemas testados demonstraram semelhanças e diferenças em seus designs, propriedades metalúrgicas e mecânicas, porém exibiram capacidade de modelagem similar, sem erros clinicamente significativos. Compreender as características de cada instrumento auxilia na tomada de decisões adequadas para cada situação clínica.

Palavras-chave: Avaliação multimétodo; endodontia; instrumentos níquel-titânio.

ABSTRACT

Objectives: The wide variety of instruments available in the current endodontic market requires careful and accurate analysis to ensure clinical safety and efficacy. Therefore, the objective of this thesis was to carry out, from four different studies, a comprehensive evaluation of several endodontic instruments available in the dental market through a multimethod approach. **Materials and Methods:** In Chapter 1, 2 and 3, different instruments were compared regarding their design characteristics, metallurgical properties, mechanical performance, and formability. The instruments analyzed in each chapter were: Genius Proflex, Vortex Blue and TruNatomy in chapter 1; REX, Reciproc Blue and WaveOne Gold in Chapter 2; and HyFlex EDM, Neoniti, EDMax and ProTaper Gold in Chapter 3. In Chapter 4, the design characteristics, metallurgical properties and mechanical performance of the ProTaper Ultimate system were compared with instruments of similar dimensions from the ProGlider, ProTaper Gold and ProTaper Universal systems. The tests were analyzed using the necessary statistical tests, always with a significance level set at 5%. **Results**: In general, the instruments tested in the same chapter showed significant differences in their general design, temperature transition phases and mechanical behavior. However, these differences did not affect their modeling ability. **Conclusions:** The adoption of a multimethod approach, combining qualitative and quantitative methodologies, increased the responsiveness to the proposed objectives and provided valuable insights for clinical practice. In general, all tested systems demonstrated similarities and differences in their designs, metallurgical and mechanical properties, but exhibited similar modeling ability without clinically significant errors. Understanding the characteristics of each instrument helps in making appropriate decisions for each clinical situation.

Keywords: Multimethod evaluation; endodontics; nickel-titanium instruments.

1. INTRODUÇÃO E REVISÃO DE LITERATURA

A tecnologia por trás da metalurgia das ligas de níquel-titânio (NiTi) permitiu o desenvolvimento de novas limas endodônticas rotatórias com uma variedade de designs e maior eficiência e segurança (ARIAS & PETERS, 2022), visando reduzir contratempos iatrogênicos, como desvio ou perfuração (HÜLSMANN *et al*., 2005). Atualmente, os procedimentos de modelagem usando instrumentos rotatórios de NiTi são mais previsíveis, fáceis e com melhores resultados clínicos em comparação com a preparação convencional com limas manuais de aço inoxidável (ARIAS & PETERS, 2022; HÜLSMANN *et al*. 2005). Mesmo assim, os instrumentos de NiTi ainda são suscetíveis a deformações e/ou fraturas, eventos indesejados que podem representar um preditor de periodontite apical persistente e consequente falha no tratamento de dentes infectados (MCGUIGAN *et al*., 2013; NG *et al.,* 2011). Para superar esses problemas, os fabricantes desenvolveram várias estratégias para melhorar as propriedades da liga NiTi, incluindo mudanças na cinemática, design dos instrumentos e tratamento de superfície (MARTINS e*t al.,* 2022a).

As ligas de NiTi usadas para produzir instrumentos endodônticos têm uma proporção quase equiatômica de elementos de níquel e titânio (ZHOU *et al.,* 2013; GAVINI *et al.,* 2018), e podem ter três fases microestruturais, ou seja, austenita, fase R e martensita, responsáveis por seu comportamento mecânico (ZHOU *et al.,* 2013; ZUPANC *et al*., 2018). A liga NiTi superelástica convencional tem estrutura austenítica predominante, tanto na temperatura ambiente (20 ºC) quanto na temperatura corporal (37 ºC), e por isso é relativamente rígida, dura e tem flexibilidade limitada. Para superar essa limitação, novos processos de fabricação usando tratamento térmico foram desenvolvidos para produzir instrumentos endodônticos de NiTi com maiores quantidades da fase estável de martensita (ZUPANC *et al*., 2018). Em sua forma martensítica, a liga de NiTi é macia, dúctil e pode ser facilmente deformada (ZHOU *et al.,* 2013; ZUPANC *et al*., 2018), enquanto a transformação de fase R comumente aparece como uma fase intermediária na maioria dos fios de NiTi disponíveis comercialmente (MILLER & LAGOUDAS, 2001). Comparado aos instrumentos austeníticos, foi relatado que os instrumentos de NiTi tratados termicamente têm maior resistência à fadiga cíclica, força (DUKE *et al.,* 2015; SILVA *et al.,* 2016) e flexibilidade, apresentando menores cargas de flexão nos testes de flexibilidade (MARTINS *et al*., 2022a; MARTINS *et al.,* 2022b). Na última década, as propriedades otimizadas dos instrumentos de NiTi tratados termicamente levaram as empresas a lançarem vários novos sistemas rotatórios no mercado. Vortex Blue (Dentsply Sirona, Baillagues, Suíça) foi introduzido em 2011, e o tratamento térmico patenteado melhorou suas propriedades mecânicas em comparação com seu antecessor, fabricado com a liga MWire (DUKE *et al.,* 2015). Os instrumentos rotatórios TruNatomy tratados termicamente (Denstply Sirona, Ballaigues, Suíça) têm uma conicidade variável, com um desenho transversal de paralelogramo descentrado, e estudos relataram sua capacidade de preservar a dentina radicular durante a preparação mecânica do canal radicular (MORALES *et al.,* 2021; SILVA *et al.,* 2022). Genius Proflex (Medidenta, Las Vegas, NV, EUA) é um sistema rotatório multi-instrumentos lançado recentemente, composto por instrumentos com diferentes seções transversais e submetidos a tratamentos térmicos distintos, resultando em lâminas ativas com cores diferentes (roxo, azulado e amarelado), visando garantir um equilíbrio entre flexibilidade e resistência, dependendo da massa metálica de cada instrumento da série (https://bit.ly/3rgSqEH (acessado em 25 de maio de 2022)). O novo sistema rotatório PT Ultimate (Dentsply Sirona Endodontics) é a última geração da família PT e é um dos primeiros sistemas a tirar proveito de arranjos cristalográficos distintos induzidos por tecnologia de tratamento térmico específico para produzir um conjunto de instrumentos com diferentes comportamentos mecânicos, com o objetivo de garantir um equilíbrio entre flexibilidade e força. Segundo o fabricante, os 8 instrumentos que compõem esse sistema (Slider [16/.02v], SX [20/.03v], Shaper [20/.04v], F1[20/.07v], F2 [25/.07v], F2 [25/. 08v], F3 [30/.09v], FX [35/.12v] e FXL [50/.10v]) são fabricados com 3 ligas diferentes tratadas termicamente: M-wire (Slider), Gold-wire (SX, Shaper, F1, F2, F3), e fio tratado termicamente azul (FX e FXL) (Dentsply Sirona, 2022).

Nos últimos anos, os fabricantes também desenvolveram métodos de produção diferentes do método tradicional de retificação, tais como torção, conformação, corte a laser e usinagem por descarga elétrica (EDM) (ARIAS &

PETERS, 2022). Através do processo de EDM, os instrumentos são fabricados por uma erosão térmica sem contato através de faíscas controladas que ocorrem entre um eletrodo e uma peça metálica, na presença de um fluido dielétrico (ARIAS & PETERS, 2022; PIRANI *et al.,* 2016). Este processo “derrete” a superfície da liga de níquel-titânio, evaporando parcialmente pequenas porções do metal e abandonando uma superfície erodida. O instrumento é então tratado termicamente a temperaturas entre 300 e 600°C, por 10 minutos a 5 horas, antes ou depois da limpeza ultrassônica e banho de ácido (GAVINI *et al.,* 2018). Esse processo exclusivo não utiliza contato físico para remoção de material, mas a vaporização local do metal, evitando a formação de microtrincas, podendo otimizar a capacidade de corte, a flexibilidade e a resistência à fadiga cíclica de instrumentos rotatórios (ARIAS & PETERS, 2022; GAVINI *et al*., 2018; PEDULLÁ *et al.,* 2016; PIRANI *et al*., 2016). O primeiro instrumento rotatório de NiTi lançado no mercado e fabricado pelo processo EDM foi um alargador de orifício denominado *Initial* (Neolix SAS) (MALLET, 2012). No ano seguinte, a mesma empresa lançou o sistema Neoniti (Neolix SAS), um conjunto de instrumentos rotatórios também produzidos pelo método EDM (STANURSKI, 2013). O sistema HyFlex EDM (Coltene/Whaledent) foi lançado 2 anos depois (MÜLLER, 2015) e estudos iniciais demonstraram uma maior resistência à fadiga cíclica em comparação com outros instrumentos produzidos com ligas NiTi superelásticas ou martensíticas (GÜNDOĞAR & ÖZYÜREK, 2017; SILVA *et al.,* 2020; THU *et al.,* 2020). Recentemente, um estudo multimétodo não mostrou diferença entre o comportamento mecânico dos instrumentos HyFlex EDM e Neoniti (SILVA *et al*., 2020). No ano de 2022, foi introduzido no mercado o sistema EDMax (Neolix SAS), outro conjunto de instrumentos rotatórios produzidos pelo mesmo processo. No entanto, de acordo com o fabricante, este sistema apresenta diferenças marcantes em relação ao Neoniti, incluindo arestas de corte estriadas, seção transversal não retangular variável em paralelogramo com arestas de corte vivas e superfície endurecida e abrasiva (https://bit.ly/3SJPOef). Além disso, os instrumentos EDMax são submetidos a um tratamento térmico que resulta em lâminas ativas com coloração azulada, em contraste com a cor amarelada dos instrumentos Neoniti e HyFlex EDM. Estas modificações foram implementadas a este sistema com o objetivo de melhorar a sua eficiência mecânica e capacidade de modelagem.

Outro esforço para reduzir a ocorrência de fratura é a cinemática oscilatória assimétrica - comumente conhecida como movimento reciprocante. O movimento reciprocante alivia o estresse no instrumento por uma rotação especial no sentido anti-horário para cortar a dentina e uma pequena rotação no sentido horário para aliviar o instrumento (YARED, 2008). Em comparação com a rotação contínua, esta cinemática prolonga a vida útil do instrumento aumentando sua resistência à fadiga (DE DEUS *et al.,* 2010) e reduzindo a ocorrência de deformação plástica (CABALLERO-FLORES *et al*., 2019; DE DEUS *et al*., 2021; RUIVO *et al.,* 2021). Reciproc Blue (VDW, Munique, Alemanha) e WaveOne Gold (Dentsply Sirona Endodontics, Baillagues, Suíça) são exemplos de instrumentos reciprocantes compostos por quantidades substanciais de martensita obtidas por tratamentos térmicos proprietários da liga NiTi. Recentemente, foi lançado no mercado o sistema reciprocante REX (Medidenta, Las Vegas, NV, EUA) com a proposta de instrumentos de NiTi fabricados com diferentes tratamentos térmicos, similares ao anteriormente descrito Genius Proflex, fazendo com que flexibilidade e resistência sejam equilibradas de forma consistente dependendo da massa metálica de cada instrumento na série (https://bit.ly/ 3ZcKe EK). Este sistema inclui instrumentos para glidepath mecânico [REX Glide Path (17/.05v)], com a liga na cor púrpura, e instrumentos apresentando diferentes tonalidades amareladas para modelagem [REX 25 (25/.08v), REX 40 (40/.08v) e REX 40 (40/. 06v)].

Os procedimentos de teste de materiais geralmente seguem normativas que visam estandardizar as condições para garantir testes reprodutíveis e simular condições de trabalho importantes para aplicações específicas onde esse tipo de solicitação é relevante. Assim, a padronização dos procedimentos de teste visa torná-los comparáveis, mesmo quando executados por diferentes operadores e/ou utilizando diferentes instrumentos. Ao aproximar as condições de teste das configurações de trabalho reais, o procedimento de teste pode representar melhor o comportamento em serviço do material testado. A padronização dos procedimentos de teste geralmente é bem-sucedida na maioria das metodologias utilizadas atualmente. Infelizmente, abordar condições reais de trabalho de aplicações complexas dá origem a alguns problemas de engenharia. Estabelecer padrões procedimentais complexos para contornar esses problemas pode inviabilizar e/ou dificultar a reprodução dos testes. Esta situação ocorre com os diferentes tipos de ensaios mecânicos utilizados para caracterizar os instrumentos endodônticos. Notadamente, o ensaio de fadiga cíclica é talvez a situação mais crítica, mas não a única. A execução de tais testes levanta questões que são difíceis de responder tanto para o endodontista quanto para o ponto de vista do engenheiro de materiais.

Dadas essas limitações, relatar os resultados dos testes, ou seja, quando apenas uma variável é abordada, como é frequentemente o caso dos testes de fadiga, pode ser de relevância limitada para os clínicos e/ou pesquisadores (HÜLSMANN, 2019; DARVEN, 2020). Um editorial do *International Endodontic Journal* (HÜLSMANN, 2019) relatou que os manuscritos submetidos sobre a suscetibilidade à fratura de instrumentos de níquel-titânio e novas ligas, frequentemente baseados em testes de torção ou fadiga cíclica, preenchem várias edições da revista todos os anos. O mesmo editorial afirmava que, devido à diversidade de metodologias e às limitadas informações fornecidas pelos ensaios de fadiga cíclica, não seriam necessárias outras publicações com esta metodologia por aquela revista. Outro editorial do *Dental Materials* de DARVELL (2020) afirmou que é responsabilidade do pesquisador garantir que as metodologias sejam atualizadas, sólidas, relevantes e totalmente justificáveis e que é essencial investigar suposições e condições de validade. Uma possível tendência para superar essas dificuldades seria usar uma combinação cuidadosamente selecionada de técnicas de caracterização, que podem fornecer uma compreensão mais clara das características mecânicas, estruturais e geométricas de diferentes instrumentos e ajudar a identificar o melhor "perfil geral" de cada classe de instrumento. Considerando o espaço limitado que os periódicos de Odontologia de primeira linha disponibilizam para estudos sobre teste de instrumentos, vale a pena pensar sobre que tipo de metodologias são realmente necessárias.

A pesquisa multimétodo é baseada em um projeto de estudo usando várias metodologias qualitativas ou quantitativas (BREWER & ALBERT, 2006; HUNTER & BREWER, 2015). Caso ambas as metodologias (qualitativa e quantitativa) sejam usadas simultaneamente, ela também ganha uma característica de métodos mistos (BREWER & ALBERT, 2006; HUNTER & BREWER, 2015), que tem como principal vantagem contornar a fraqueza das medições quantitativas e qualitativas. Algumas avaliações quantitativas podem relatar diferenças que só podem ser compreendidas e explicadas quando integradas e contextualizadas com dados não quantificáveis (BREWER & ALBERT, 2006; HUNTER & BREWER, 2015). Esta simbiose entre abordagens qualitativas e quantitativas fornece à pesquisa ferramentas superiores para responder às questões e problemas do estudo, ao mesmo tempo em que fornece uma validação superior em ambos os sentidos (BREWER & ALBERT, 2006; HUNTER & BREWER, 2015).

2. JUSTIFICATIVA

A ampla variedade de instrumentos disponíveis no mercado endodôntico atual requer uma análise criteriosa e precisa para garantir a segurança e a eficácia clínica. Portanto, a justificativa para esta tese baseia-se na necessidade de uma avaliação abrangente de diversos instrumentos endodônticos por meio de uma abordagem multimétodo. Essa abordagem é baseada em um desenho de estudo que utiliza várias metodologias qualitativas e/ou quantitativas, aproveitando a sinergia entre as diferentes abordagens para fornecer ferramentas avançadas de pesquisa, capazes de responder às questões e problemas do estudo e oferecer validação sólida em ambos os aspectos. Por meio dessa pesquisa abrangente, espera-se promover a segurança e a qualidade dos tratamentos endodônticos, contribuindo para aprimorar os resultados clínicos e o bem-estar dos pacientes.

3. OBJETIVO(S)

A presente tese é composta por 4 capítulos que utilizaram uma abordagem multimétodo de avaliação para contemplar os seguintes objetivos:

(i) Avaliar as características de design, propriedades metalúrgicas, o desempenho mecânico e a capacidade de modelagem dos instrumentos rotatórios Vortex Blue, TruNatomy e Genius Proflex;

(ii) Avaliar as características de design, propriedades metalúrgicas, o desempenho mecânico e a capacidade de modelagem dos instrumentos reciprocantes REX, comparando com os sistemas Reciproc Blue e WaveOne Gold;

(iii) Avaliar as características de design, propriedades metalúrgicas, o desempenho mecânico e a capacidade de modelagem de três sistemas fabricados utilizando o processo EDM, nominalmente HyFlex EDM, Neoniti e EDMax, usando o sistema ProTaper Gold como referência para comparação;

(iv) Avaliar as características de design, propriedades metalúrgicas e o desempenho mecânico do sistema ProTaper Ultimate, que foram comparados com instrumentos de dimensões similares dos sistemas ProGlider, ProTaper Gold e ProTaper Universal.

4. CAPÍTULOS

4.1 CAPÍTULO 1

A MULTIMETHOD ASSESSMENT OF A NEW CUSTOMIZED HEAT-TREATED NICKEL-TITANIUM ROTARY FILE SYSTEM

Artigo publicado na revista *Materials (Basel)*

doi: 10.3390/ma15155288.

Classificação de periódicos quadriênio 2017-2020 – Odontologia – A3

Fator de impacto 3.748

materials

Article

A Multimethod Assessment of a New Customized Heat-Treated Nickel–Titanium Rotary File System

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Citation: Silva, E.J.N.L.; Martins, J.N.R.; Ajuz, N.C.; Antunes, H.S.; Vieira, V.T.L.; Braz Fernandes, F.M.; Belladonna, F.G.; Versiani, M.A. A Multimethod Assessment of a New Customized Heat-Treated Nickel–Titanium Rotary File System. *Materials* **2022**, *15*, 5288. https://doi.org/10.3390/ma15155288

Academic Editor: Luca Testarelli

Received: 19 June 2022
Accepted: 28 July 2022
Published: 31 July 2022

Abstract: This study aimed to compare three endodontic rotary systems. The new Genius Proflex (25/0.04), Vortex Blue (25/0.04), and TruNatomy (26/0.04v) instruments (n = 41 per group) were analyzed regarding design, metallurgy, and mechanical performance, while shaping ability (untouched canal walls, volume of removed dentin and hard tissue debris) was tested in 36 anatomically matched root canals of mandibular molars. The results were compared using one-way ANOVA, post hoc Tukey, and Kruskal–Wallis tests, with a significance level set at 5%. All instruments showed symmetrical cross-sections, with asymmetrical blades, no radial lands, no major defects, and almost equiatomic nickel–titanium ratios. Differences were noted in the number of blades, helical angles, cross-sectional design, and tip geometry. The Genius Proflex and the TruNatomy instruments had the highest and lowest R-phase start and finish temperatures, as well as the highest and lowest time and cycles to fracture ($p < 0.05$), respectively. The TruNatomy had the highest flexibility ($p < 0.05$), while no differences were observed between the Genius Proflex and the Vortex Blue ($p > 0.05$). No differences among tested systems were observed regarding the maximum torque, angle of rotation prior to fracture, and shaping ability ($p > 0.05$). The instruments showed similarities and differences in their design, metallurgy, and mechanical properties. However, their shaping ability was similar, without any clinically significant errors. Understanding these characteristics may help clinicians to make decisions regarding which instrument to choose for a particular clinical situation.

Keywords: differential scanning calorimetry; endodontics; energy-dispersive X-ray spectroscopy; micro-computed tomography; root canal therapy; scanning electron microscopy

1. Introduction

The technology behind the metallurgy of nickel–titanium (NiTi) alloys allowed for the development of new rotary endodontic files with a variety of designs and improved efficiency and safety [1], aiming to reduce iatrogenic mishaps, such as deviation or perforation [2]. Currently, shaping procedures using NiTi rotary instruments are more predictable and easier when compared to manual preparation with stainless-steel files [1,2]. The NiTi alloys used to produce endodontic instruments have an almost equiatomic ratio of nickel and titanium elements [3,4] and may have three microstructural phases, namely austenite, R-phase, and martensite, responsible for their mechanical behavior [3,5]. The conventional superelastic NiTi alloy has a predominant austenite structure at both room (20 °C) and body (37 °C) temperatures, and for this reason, it is relatively stiff, hard, and has limited flexibility. To overcome this limitation, new manufacturing processes using heat treatment have been developed to produce endodontic NiTi instruments with larger amounts of the stable martensite phase [5]. In its martensite form, the NiTi alloy is soft, ductile, and can be easily deformed [3,5], while the R-phase transformation commonly appears as an intermediate phase in most of the commercially available NiTi wires [6]. Compared to austenitic instruments, it has been reported that heat-treated NiTi instruments have increased cyclic fatigue resistance, strength [7–9], and flexibility, presenting lower bending loads in the bending tests [8–10].

In the last decade, the optimized properties of heat-treated NiTi instruments led companies to launch several new rotary systems on the market. Vortex Blue (Dentsply Sirona, Baillagues, Switzerland) was introduced in 2011, and the proprietary heat treatment improved its mechanical properties compared to its predecessor, manufactured with M-Wire alloy [7]. The heat-treated TruNatomy rotary instruments (Denstply Sirona, Ballaigues, Switzerland) have a variable taper with an off-centered parallelogram cross-sectional design, and studies have reported its ability to preserve the radicular dentin during root canal mechanical preparation [11,12]. Genius Proflex (Medidenta, Las Vegas, NV, USA) is a recently launched multi-file rotary system composed of instruments with different cross-sections and submitted to distinct heat treatments, resulting in active blades with different colors (purplish, blueish, and yellowish), aiming to ensure a balance between flexibility and resistance, depending on the metal mass of each instrument in the series (https://bit.ly/3rgSqEH (accessed on 25 May 2022)). Thus far, there is no available scientific evidence to support its efficiency or safety. Therefore, the aim of this study was, by using a multimethod approach, to evaluate the design, metallurgy, mechanical performance, and shaping ability of the Vortex Blue, TruNatomy, and Genius Proflex rotary instruments. The null hypothesis to be tested in the present research was that there would be no differences among these instruments regarding the evaluated properties.

1. Materials and Methods

New 25-mm NiTi instruments (n = 123) from 3 rotary systems (41 per group; Genius Proflex (25/0.04), TruNatomy (26/0.04v), and Vortex Blue (25/0.04)) (Figure 1) were compared in relation to design, metallurgical characteristics, and mechanical behavior. In addition, 48 instruments (16 per group) were employed for testing the shaping ability of each system in root canals of extracted mandibular molars. Instruments were previously examined under a stereomicroscope (×13.6 magnification; Opmi Pico, Carl Zeiss Surgical, Oberkochen, Germany) looking for defects that would exclude them from being tested, but none were excluded.

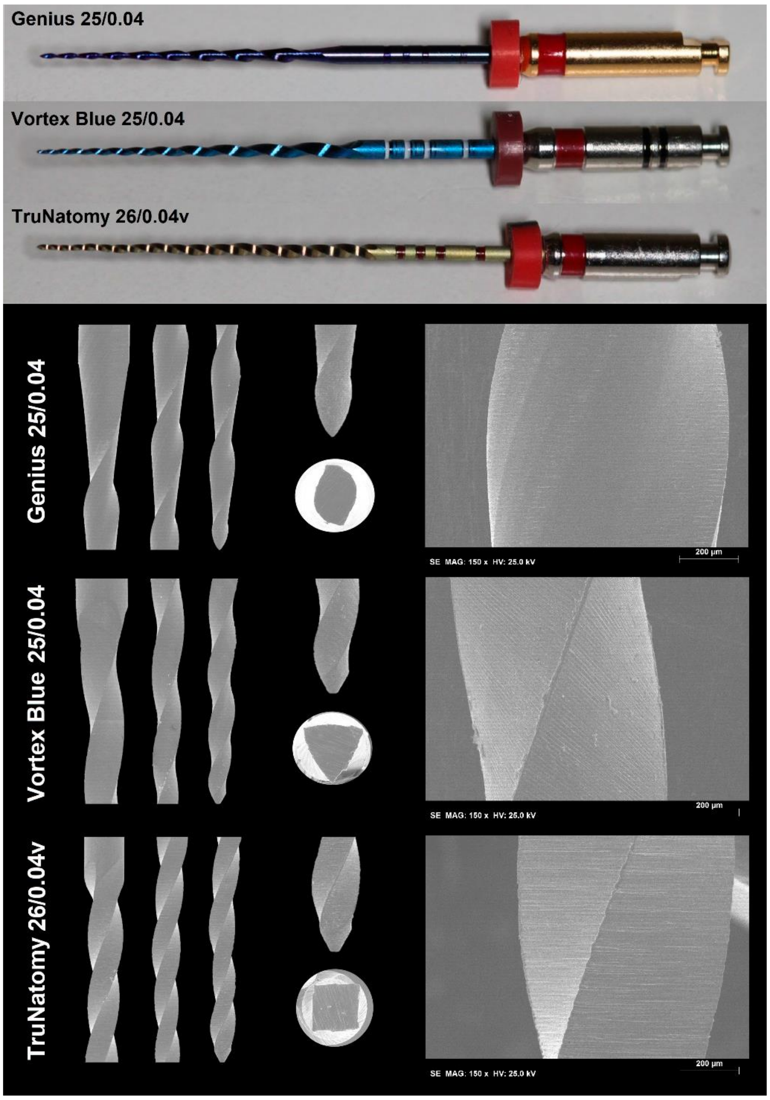

Figure 1. Tested instruments and their design and surface finishing. Macroscopic analyses of the tested instruments (top) showed a higher number of blades in the TruNatomy and distinct colors of the alloy among them. SEM evaluation (bottom) revealed that all instruments have asymmetrical blades, no radial lands and different symmetrical cross sections (square: TruNatomy; triangular: Vortex Blue; S-shaped: Genius Proflex). The tips were non-active, with distinct geometry and transition angles. All surfaces had parallel manufacturing marks, with few irregularities.

1.1. Instrument Design

The number of active blades (in units) and the helical angles (in degrees) at the 6 most coronal flutes of 6 randomly selected endodontic files from each system were assessed under stereomicroscopy (×13.6 magnification; Opmi Pico) using the ImageJ v1.50e software (Laboratory for Optical and Computational Instrumentation, Madison, WI, USA). These same instruments were further imaged in a conventional scanning electron microscope (Hitachi S-2400, Hitachi, Tokyo, Japan) at different magnifications (×100 and ×500) to evaluate their blade design (radial lands and symmetry), cross-sectional shape, tip geometry (active or non-active), and surface finishing.

1.1. Metallurgical Characterization

The semi-quantitative elemental analysis of 3 instruments from each tested system was carried out to evaluate the nickel and titanium ratio, or the presence of any other element, using a scanning electron microscope (S-2400; Hitachi) mounted with an energy-dispersive X-ray spectroscopy (EDS) device (Bruker Quantax; Bruker Corporation, Billerica, MA, USA) set at 20 kV and 3.1 A. The analysis was performed for each instrument at a 25-mm distance from a surface area of 400 μm^2 using a proper software with ZAF correction (Systat Software Inc., San Jose, CA, USA).

The differential scanning calorimetry (DSC) method (DSC 204 F1 Phoenix; Netzsch-Gerätebau GmbH, Selb, Germany) was used to determine the phase transformation temperatures of the NiTi alloy following the guidelines of the American Society for Testing and Materials [13]. Fragments of 2 to 3 mm in length (5–10 mg), removed from the coronal active blade of 2 instruments from each system, were exposed for 2 min to a chemical etching consisting of a mixture of 45% nitric acid, 25% hydrofluoric acid, and 30% distilled water. Then, they were mounted in an aluminum pan inside the DSC device, with an empty pan serving as control. The thermal cycle was performed under gaseous nitrogen atmosphere at a pace of 10 °C/min with temperatures ranging from −150 °C to 150 °C. Phase transformation temperatures were analyzed by the Netzsch Proteus Thermal Analysis software (Netzsch-Gerätebau GmbH). For each group, the DSC test was performed twice to confirm the results. Tested instruments included TruNatomy size 26/0.04v, Vortex Blue size 25/0.04, and the whole set of Genius Proflex instruments (sizes 25/0.06, 13/0.03, 17/0.05, 25/0.04, and 35/0.04) due to differences in their heat treatment, as claimed by the manufacturer (https://bit.ly/38DxX6J (accessed on 25 May 2022)).

1.2. Mechanical Tests

The mechanical performance of the selected systems was evaluated through cyclic fatigue, torsional resistance, and bending tests. For each test, the sample size was calculated with an alpha-type error of 0.05 and a power of 80%, based on the highest difference between 2 systems after 6 initial measurements. For the time to fracture (TruNatomy vs. Genius Proflex; effect size of 217.8 ± 118.8), maximum torque (TruNatomy vs. Vortex Blue; effect size of 0.15 ± 0.22), angle of rotation (TruNatomy vs. Genius Proflex; effect size of 6.2 ± 48.2), and maximum bending load (TruNatomy vs. Vortex Blue; effect size of 67.7 ± 37.2), the final sample sizes of 6, 36, 949, and 6 instruments were determined, respectively. Even though 36 and 949 instruments were calculated for the maximum torque and angle of rotation, a final sample size of 10 instruments per group was defined for each parameter, since a difference only identifiable in that large a sample size can be considered of little clinic relevance.

The cyclic fatigue test was conducted on a non-tapered stainless steel curved tube apparatus (radius of 6 mm and 86° degree angle) using glycerin as a lubricant, according to previous studies [8,9,14]. The tested instruments were adapted to a 6:1 reduction handpiece (Sirona Dental Systems GmbH, Bensheim, Germany) and activated at static mode by a torque-controlled motor (VDW Silver; VDW GmbH) set at 400 rpm and 2.0 N (Genius Proflex), 500 rpm and 1.5 N (TruNatomy), and 500 rpm and 1.0 N (Vortex Blue), according to the manufacturers' directions. The test was conducted at room temperature (20 °C) following the guidelines of the American Society for Testing and Materials regarding tension testing of superelastic NiTi materials [15]. Fracture was detected by both auditory and visual inspection. The time to fracture was recorded in seconds using a digital chronometer, and the fragment size was measured in millimeters with a digital caliper for experimental control. Torsional and bending resistance tests were performed according to international standards [16,17]. In the torsional test, instruments were clamped 3 mm from their tip and rotated clockwise at a constant pace of 2 rotations per minute to assess the maximum torque (measured in N.cm) and the angle of rotation (recorded in degrees) prior to fracture. In the bending test, each instrument was mounted in the file holder of the motor and positioned at 45° in relation to the floor, while it was attached to a wire (3 mm from its tip) connected to a universal testing machine (Instron 3400; Instron Corporation, Canton, MA, USA). The maximum load needed for a 45° displacement of the instrument, using a load of 20 N and 15 mm/min of constant speed, was recorded in gram-force (gf).

1.1. Shaping Ability

After approval of this research project by the local ethics committee (Protocol CE-FMDUL 13/10/20), 120 two-rooted mandibular molars with fully formed apices were randomly selected from a pool of extracted teeth and initially scanned at a pixel size of 11.93 μm in a micro-computed tomographic device (micro-CT) (SkyScan 1173; Bruker-microCT, Kontich, Belgium) set at 70 kV, 114 μA, rotation of 360° with steps of 0.7°, using a 1 mm thick aluminum filter. The first step in the image acquisition involved fixing the specimen on a sample holder with dental wax to avoid movement during scanning. The acquired projections were reconstructed into axial cross-sections using standardized parameters of smoothing (1), attenuation coefficient (0.05–0.007), beam hardening (20%), and ring artifact (5) corrections (NRecon v.1.6.9; Bruker-microCT). A three-dimensional (3D) model of the internal anatomy of each tooth was created (CTAn v.1.14.4; Bruker-microCT) and qualitatively evaluated (CTVol v.2.2.1; Bruker-microCT) regarding root canal configuration. Then, and considering teeth with the same working length from cementoenamel junction to the apex, and the same volume and surface area from the mesial and distal canals, were calculated, within these two anatomic landmarks. Based on these parameters, specimens were anatomically matched to create 3 groups of 4 teeth (12 canals per group) that were randomly assigned to an experimental group according to the preparation system: Genius Proflex, TruNatomy, and Vortex Blue.

After access cavity preparation, apical patency was confirmed with a size 10 K-file (Dentsply Sirona Endodontics) and the glide path was performed using a size 15 K-file (Dentsply Sirona Endodontics) up to the working length (WL), established 1 mm from the apical foramen. In the Genius Proflex group, coronal flaring was performed with a size 25/0.06 instrument (350 rpm, 2.5 N.cm), followed by instruments in sizes 13/0.03 (250 rpm, 1.5 N.cm) and 25/0.04 (400 rpm, 2 N.cm) up to the WL. In the TruNatomy group, all instruments were used at 500 rpm and 1.5 N.cm. After coronal flaring with a size 20/0.08 instrument, instruments of 17/0.02v (Glider) and 26/0.04v (Prime) were used up to the WL. In the Vortex Blue group, instruments of sizes 15/0.04 (500 rpm, 0.7 N.cm), 20/0.04 (500 rpm, 0.7 N.cm), and 25/0.04 (500 rpm, 1 N.cm), were sequentially used up to the WL. Then, in all groups, the distal canals were further enlarged with instruments in sizes 35/0.05 (Genius Proflex group; 400 rpm, 2.5 N.cm), 36/0.03v (TruNatomy group), 30/0.04 and 35/0.04 (Vortex Blue group; 500 rpm, 1.0 N.cm, and 1.3 N.cm, respectively). Instruments were activated by an electric motor (VDW Silver; VDW, Munich, Germany) and used in a slow in-and-out pecking motion of about 3 mm amplitude with light pressure in the apical direction. After 3 pecking motions, the instrument was removed from the canal and cleaned. The WL was reached after 3 waves of instrumentation. Each instrument was used in one tooth and then discarded. Irrigation was performed with a total of 15 mL of 2.5% NaOCl per canal, followed by a final rinse with 5 mL of 17% EDTA (3 min) and 5 mL of distilled water using a syringe fitted with a 30-G NaviTip needle (Ultradent, South Jordan, UT, USA) positioned 2 mm from the WL. All procedures were performed by an experienced operator under magnification (×12.5; ZEISS OPMI Pico, Jena, Germany).

The canals were slightly dried with paper points and a final scan and reconstruction were performed using the previously mentioned parameters. Datasets before and after preparation were co-registered (3D Slicer 4.3.1 software; http://www.slicer.org (accessed on 25 May 2022)) and the shaping ability was assessed by measuring 3 parameters: the volume of dentin removed after preparation (in mm^3), the volume of hard tissue debris created by the preparation protocols (in mm^3), and the percentage of unprepared canal walls [18,19]. An examiner blinded to the shaping protocols performed all analyses by excluding canal interconnections and accessory anatomies.

1.1. Statistical Analysis

The Shapiro–Wilk and Lilliefors tests were used to verify the normality of the data. Depending on data distribution, results were summarized as mean (standard deviation) or median (interquartile range) values. One-way ANOVA and post hoc Tukey tests were carried out to compare the angle of rotation, untouched canal walls, volume (root canal, removed dentine, hard tissue debris), and surface area (root canal) of the mesial canals, while the Kruskal–Wallis test, combined with the Dunn test, was used to compare the helical angle, time to fracture, maximum torque to fracture, maximum bending load, and volume of removed dentine and hard tissue debris in the distal canal. The significance level was set at 5% (SPSS v25.0 for Windows; SPSS Inc., Chicago, IL, USA).

2. Results

2.1. Instrument Design

The instrument stereomicroscopic analysis of both number of blades and helical angles showed that the Vortex Blue (11 blades; 17.8° (17.3–18.9°)) had a significantly lower helical angle degree when compared to the TruNatomy (17 blades; 21.3° (19.5–22.1°)) and the Genius (9 blades; 21.7° (19.8–23.1°)) ($p < 0.05$). SEM analysis (Figure 1) revealed that all instruments had asymmetrical blades, with no radial lands, and symmetrical cross sections, with squared (TruNatomy), convex (Vortex Blue), and S-shaped (Genius Proflex) profiles. None of the tips could be identified as active, and the overall geometry and transition angles of the blade varied among the instruments. While the tips of TruNatomy and Vortex Blue instruments were flat at their ends, the Genius Proflex had a bullet-like shape. Under higher magnification, all instruments showed similar surface finishing, with a pattern of parallel marks created by the grinding manufacturing process. It was also possible to observe some metal rollovers on the blades, but Vortex Blue showed more irregularities than the others (Figure 1).

2.2. Metallurgical Characteristics

EDS/SEM analysis revealed a nearly equiatomic ratio of nickel and titanium elements in the Genius Proflex (1.061), TruNatomy (1.014) and Vortex Blue (1.016) instruments, without any other traceable metal element. DCS analyses (Figure 2A) showed distinct transformation–temperature curves. Although no instrument had full austenitic characteristics at the test temperature (20 °C), Vortex Blue and TruNatomy showed this feature at body temperature (36 °C). The highest (45.4 °C) and lowest (25.9 °C) R-phase start and finish (34.6 °C and 13.5 °C) temperatures were observed in the Genius Proflex and the TruNatomy, respectively (Figure 2A). The Vortex Blue had the lowest austenitic start temperature (3.3 °C) and the Genius Proflex showed the highest austenitic finish temperature (50.3 °C). DSC tests of the Genius Proflex instruments (Figure 2B) demonstrated similar heat treatment among them, with minor differences in R-phase transformation temperatures, in the cooling transformation of martensitic B19^{t}, and in the austenitic transformation during heating curves. Major differences were observed in the heating of the Genius Proflex 13/0.03, with a lower austenitic start (3.6 °C) compared to that of the other instruments (Figure 2B).

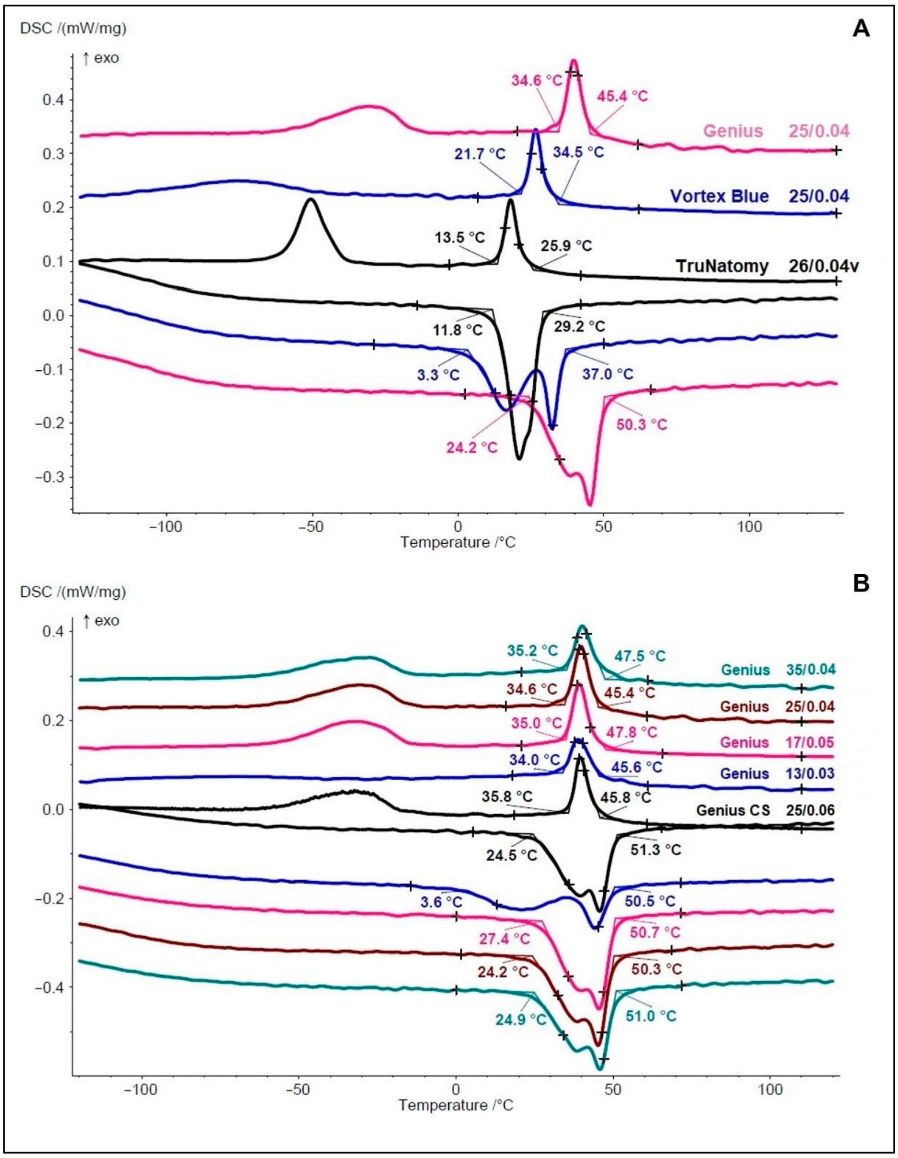

Figure 2. DSC charts showing the phase transformation temperatures at cooling on the top (reads from right to left) and at heating on the bottom (reads from left to right). (**A**) Genius Proflex showed the highest R-phase start (45.4 °C) and finish (34.6 °C) temperatures, while TruNatomy had the lowest (25.9 °C and 13.5 °C, respectively). Genius Proflex also had the highest austenitic start (24.2 °C) and finish (50.3 °C) temperatures. (**B**) Phase transformation temperatures of the Genius Proflex system. Except for the 13/0.03 instrument, which showed a distinct R-phase to martensite B19^{t} transformation at cooling, all other instruments had similar curves.

1.1. Mechanical Performance

The Genius Proflex had the highest time (252 s) and cycles (1680) to fracture ($p < 0.05$), while the lowest time (41 s) and cycles (341.7) to fracture were observed with the TruNatomy ($p < 0.05$). The maximum torque and angle of rotation prior to fracture revealed no significant differences among groups ($p > 0.05$). The TruNatomy showed the highest flexibility (108.5 gf) compared to the other tested instruments ($p < 0.05$) (Table 1).

Table 1. Mechanical behavior of tested instruments shown as mean (standard deviation) and median (interquartile range) values.

System	Cyclic Fatigue		Torsional Test		Bending Test
	Time to Fracture (s)	Cycles to Fracture (NCF)	Maximum Torque (N.cm)	Angle of Rotation (°)	Maximum Load (gf)
TruNatomy	41.0 (± 8.6) [a]	341.7 (± 71.7) [a]	0.76 (± 0.12) [a]	633.6 (± 40.9) [a]	108.5 (± 9.5) [a]
26/0.04v	43.5 [31.8–48.0]	362.5 [264.6–400.0]	0.70 [0.70–0.83]	620.5 [602.5–662.3]	108.0 [99.5–119.0]
Vortex Blue	80.0 (± 9.1) [b]	666.7 (± 76.2) [b]	0.93 (± 0.13) [a]	589.8 (± 29.0) [a]	178.8 (± 13.7) [b]
25/0.04	77.5 [72.3–88.8]	645.9 [602.1–739.6]	0.90 [0.90–1.00]	593.5 [556.0–610.5]	180.0 [167.5–186.0]
Genius	252.0 (± 53.7) [c]	1680.0 (± 357.7) [c]	0.79 (± 0.27) [a]	587.3 (± 78.6) [a]	167.4 (± 16.4) [b]
25/0.04	257.0 [199.8–290.8]	1713.4 [1331.7–1938.3]	0.70 [0.58–0.93]	609.5 [509.8–659.0]	162.0 [158.5–180.5]

Different superscript letters in the same column represent statistically significant differences ($p < 0.05$) among instruments.

1.1. Shaping Ability

The homogeneity of the groups regarding the volume and surface area of the mesial and distal canals was confirmed ($p > 0.05$) (Table 2). No statistically significant differences were observed among the groups in all the tested parameters ($p > 0.05$). Mean percentages of unprepared canal areas ranged from 50.5% to 60.4% in the mesial canal, and from 57.8% to 68.7% in the distal canal (Table 2, Figure 3).

Table 2. Pre- and post-operative parameters (mean, standard deviation, and range interval) evaluated in mesial (n = 24) and distal (n = 12) root canals of mandibular molars after preparation protocols using 3 rotary systems.

Canal	Parameters		Genius	TruNatomy	Vortex Blue
Mesial	Volum	Before	4.7 ± 1.7 (2.3–6.3)	4.6 ± 1.8 (2.3–6.3)	3.4 ± 1.5 (1.6–5.3)
		After	5.8 ± 1.1 (4.1–6.7)	5.9 ± 1.4 (4.4–7.4)	4.5 ± 1.3 (2.9–6.3)
	Surface area	Before	68.9 ± 11.8 (53.3–81.4)	58.4 ± 16.5 (36.5–71.3)	55.1 ± 17.1 (31.1–70.8)
		After	69.9 ± 12.6 (53.4–83.5)	61.6 ± 12.3 (47.1–73.8)	56.9 ± 16.3 (37.3–74.1)
	Removed dentin	After	1.5 ± 0.6 (0.6–2.3)	1.6 ± 0.4 (1.1–2.2)	1.3 ± 0.07 (1.2–1.4)
	Debris	After	0.037 ± 0.035 (0.003–0.073)	0.013 ± 0.009 (0.004–0.025)	0.014 ± 0.012 (0.002–0.030)
	Unprepared area	After	60.4 ± 17.3 (44.9–77.9)	50.5 ± 24.4 (25.5–75.9)	54.2 ± 24.5 (17.4–69.1)
Distal	Volume	Before	6.1 ± 1.8 (3.9–8.5)	8.2 ± 3.6 (4.7–13.3)	4.6 ± 0.5 (4.1–5.2)
		After	7.4 ± 1.2 (6.3–9.2)	8.8 ± 3.7 (5.9–14.4)	5.7 ± 0.6 (5.1–6.5)
	Surface area	Before	60.5 ± 3.8 (56.8–65.4)	57.3 ± 20.4 (41.4–86.6)	45.3 ± 6.2 (40.9–54.5)
		After	61.9 ± 6.6 (54.9–70.6)	62.4 ± 19.4 (50.9–90.8)	48.2 ± 9.1 (42.1–61.8)
	Removed dentin	After	1.5 ± 1.4 (0.7–3.6)	0.9 ± 0.6 (0.3–1.8)	1.2 ± 0.8 (0.5–2.4)
	Debris	After	0.007 ± 0.010 (0.000–0.021)	0.001 ± 0.003 (0.000–0.005)	0.002 ± 0.003 (0.000–0.007)
	Unprepared area	After	63.6 ± 9.1 (53.4–73.81)	68.7 ± 14.8 (46.8–79.1)	57.8 ± 12.0 (46.6–72.2)

Volume (mm^3); surface area (mm^2); removed dentin (mm^3); debris (mm^3); unprepared area (%).

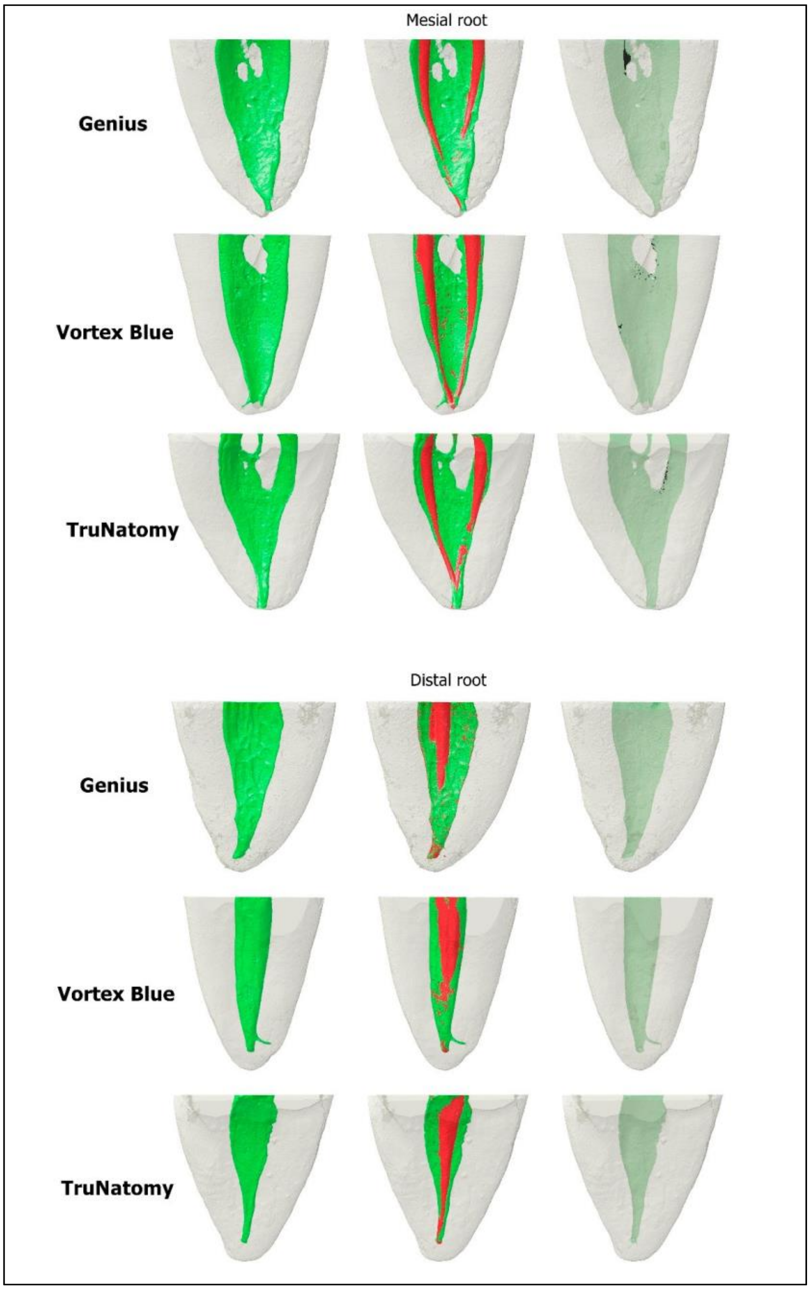

Figure 3. Representative micro-CT 3D models of mesial and distal canals of mandibular molars showing the root canals before (green color) (left column) and after (red color) preparation (central column) using the Genius Proflex, TruNatomy, and Vortex Blue systems. None of the shaping protocols were able to prepare the entire surface area of the root canal walls. Accumulated hard-tissue debris is depicted in black (right column).

1. Discussion

The present investigation, using a multimethod research approach, assessed the overall geometric design, elemental composition, phase transformation temperatures, mechanical behavior, and shaping ability of 3 heat-treated NiTi rotary systems (Genius Proflex, TruNatomy, and Vortex Blue). This methodological approach allows for a more comprehensive assessment regarding the properties of the tested instruments, as it avoids 'knowledge compartmentalization,' a phenomenon in which knowledge structures about a specific domain are composed of several separate parts [20].

All tests followed strict international guidelines [13,15–17] or methodologies with high internal validity [14,18,21,22], enabling a more robust and trustworthy understanding of the systems' performance. While similarities were observed among the instruments regarding nickel and titanium composition, torsional response (Table 1), and shaping ability (Table 2, Figure 3), differences were observed in the helical angles, number of blades, cross- sections, tip geometry (Figure 1), temperature transition phases (Figure 2), cyclic fatigue, and bending resistance tests (Table 1). Therefore, the null hypothesis was rejected.

Differences in the mechanical behavior of tested instruments should be analyzed considering multiple factors, which may be relevant depending on the test. Since all of the instruments were made from almost equiatomic NiTi alloys, their mechanical behavior may be explained by differences in the design and crystallographic arrangements [3,5], depicted by their distinct phase transformation temperatures (Figure 2A). Considering that all mechanical tests were performed at room temperature (20.0 ± 1 °C), which is inside the instrument's service temperature range, and in accordance with ASTM recommendations [15], the Rs temperatures of the Genius Proflex (45.4 °C), Vortex Blue (34.5 °C), and TruNatomy (25.9 °C), indicates that none of them had full austenitic characteristics at the test temperature. On the other hand, this baseline temperature tends to increase and approach body temperature (around 36 °C) under clinical conditions. In such cases, the Vortex Blue and TruNatomy instruments may suffer a crystallographic rearrangement lead- ing to a higher increase in the amount of austenitic phase compared to the Genius Proflex. Therefore, the higher martensitic composition and smaller metal core (represented by the S-shaped cross-section and fewer number of blades) of the Genius Proflex instruments, compared to the TruNatomy and the Vortex Blue, could explain its higher cyclic fatigue resistance (Table 1). Unfortunately, the results of the Genius Proflex cannot be compared to the literature, as there is still no scientific publication on its mechanical properties. On the other hand, comparisons between the TruNatomy and the Vortex Blue have shown con- trasting results. While in one study [23], no statistical difference was observed in the mean cycles to fracture in the Vortex Blue (523.9) and TruNatomy (436.8), in another study [24], the TruNatomy showed a higher mean number of cycles to fracture (1238.8) compared to the Vortex Blue (529.5). These studies were conducted at body temperature (35–37 °C), and these dissimilarities could be explained by differences in the angles of curvature of the simulated canals (90° vs. 60°).

Although differences were observed in the cyclic fatigue test, the instruments showed similar results in the torsional resistance assay. This test followed ISO 3630-3631 guidelines [17] that recommend measuring the torsional resistance of an instrument only at 3 mm from its tip. This methodological aspect may explain the observed similarities since, at this specific level, minor differences among the instruments regarding taper (0.04v for TruNatomy, and 0.04 for Vortex Blue and Genius Proflex) are compensated by their dissimilar cross-sectional design and metal core. While little debate exists regarding this methodological aspect, it is possible that analyses of torsional resistance performed at other levels of the instruments may result in different outcomes from those obtained herein.

In this study, an interesting finding was observed in the bending test. While it would be expected that highly flexible instruments would perform better in the cyclic fatigue resistance test, the TruNatomy was the most flexible instrument, but had the lowest cycles to fracture (Table 1). This apparent contradictory result may be explained because of differences in the small diameter of the NiTi wire used to produce the TruNatomy (0.8 mm) compared to the Genius Proflex and Vortex Blue (1.0 mm and 1.2 mm, respectively). Considering that in the bending test, all instruments are fixed in the file holder, the smaller wire can have a direct influence on this result.

The idea behind the Genius Proflex instruments is to take advantage of different crystallographic phases of the NiTi alloy, depending on the clinical needs. For instance, it would be expected that, during glide path, the instrument suffers a torsional overload, requiring a high torque resistance to avoid unexpected fracture while, for the apical enlargement, especially in curved canals, flexural fatigue resistance would be more relevant than torsional overload. In this way, if all instruments in a set were submitted to the same heat treatment, the accomplished metallurgical changes would be more beneficial to some instruments than to others. Thus, the present study also aimed to analyze all sets of instruments of the Genius Proflex system (25/0.06; 13/0.03; 17/0.05; 25/0.04; 35/0.04) regarding their phase transformation temperatures (Figure 2B). The different transformation temperature profiles in the Genius Proflex customized heat-treated instruments were shown by the glide path instrument (13/0.03), which presented a very distinct R-phase to martensite $B19^{t}$ transformation at cooling (Figure 2B), compared to the 25/0.06 (yellowish blade color) and 35/0.04 (bluish blade color).

In addition to the mechanical tests, this study also assessed the shaping ability of the selected rotary systems using the non-destructive micro-CT gold-standard technology. This analytical tool allows for the standardization of specimen selection, avoiding bias related to root canal morphology, and the assessment of several morphometric parameters after root canal preparation [18,19,21]. Although differences were observed in the design and mechanical behavior among the tested instruments (Table 1), all preparation protocols were similar in terms of dentin removed after preparation, hard tissue debris created by the preparation protocols, and unprepared canal walls. Moreover, no instrument fracture or significant deviation from the original canal path could be observed. The similar tip and taper sizes of the tested instruments might explain these results, which are in line with previous studies using instruments with equivalent sizes and tapers [22,25]. In the literature, both the TruNatomy [11,12] and the Vortex Blue [26,27] systems have been evaluated regarding their shaping ability using micro-CT technology. While different methodological strategies were used in these studies, taken together, their outcomes were similar to the present research regarding the large percentage areas of untouched canals walls (TruNatomy: 50%; Vortex Blue: 58.8%) [11,26], the low amount of dentin removal after canal reparateion [12,26], and the small accumulation of hard tissue debris (Vortex Blue system: 0.16 mm^3) [26].

The multimethod research may be seen as one of the main strengths of the present research, which allowed for a more comprehensive assessment of the instruments' profiles and behaviors. Additionally, the use of DSC allowed a broader understanding of the temperature issue, when compared to tests based on a single temperature, whatever it may be. Among the limitations of the present study are the fact that other relevant tests, such as cutting efficiency, microhardness, and buckling resistance, were not conducted. Future studies using the multimethod approach should include these additional tests to compare and justify this new trend of manufacturers to produce sets of instruments with customized heat-treated NiTi alloys. Knowing the characteristics of these instruments may help the clinicians to take a better decision regarding which instruments to select in a particular clinical situation.

1. Conclusions

The Genius Proflex, Vortex Blue, and TruNatomy instruments showed differences regarding the number of blades, helical angles, cross-sectional design, tip geometry, phase transformation temperatures, cyclic fatigue resistance, and flexibility, but were similar in terms of nickel–titanium ratios, maximum torque, angle of rotation prior to fracture, and shaping ability.

Author Contributions: Conceptualization, E.J.N.L.S., J.N.R.M. and M.A.V.; methodology, E.J.N.L.S., J.N.R.M., N.C.A., H.S.A., V.T.L.V. and F.G.B.; software, F.G.B. and M.A.V.; validation, E.J.N.L.S., F.M.B.F. and M.A.V.; formal analysis, F.G.B.; investigation, J.N.R.M., N.C.A., H.S.A. and V.T.L.V.; resources, E.J.N.L.S. and F.M.B.F.; data curation, E.J.N.L.S. and F.G.B.; writing—original draft preparation, E.J.N.L.S. and J.N.R.M.; writing—review and editing, F.M.B.F. and M.A.V.; visualization, N.C.A., H.S.A. and V.T.L.V.; supervision, F.M.B.F. and M.A.V.; project administration, E.J.N.L.S.; funding acquisition, F.M.B.F. All authors have read and agreed to the published version of the manuscript.

Funding: Funding was provided by CENIMAT/i3N through national funds obtained through the FCT-Fundação para a Ciência e a Tecnologia, I.P., within the scope of Multiannual Financing of R&D Units, reference UIDB/50025/2020-2023, and is acknowledged by F.M.B.F. Fernanda Carvalho is acknowledged for running the DSC tests of the files. This study was also partly funded by the Fundação de Amparo à Pesquisa do Estado do Rio de Janeiro (FAPERJ), reference E-26/201.249/2021 and by the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), reference 403655/2021-8.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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4.2. CAPÍTULO 2

DESIGN, METALLURGY, MECHANICAL PROPERTIES AND SHAPING ABILITY OF 3 HEAT-TREATED RECIPROCATING SYSTEMS: A MULTIMETHOD INVESTIGATION

Artigo publicado na revista *Clinical Oral investigations*

doi: 10.1007/s00784-023-04899-2

Classificação de periódicos quadriênio 2017-2020 – Odontologia – A1

Fator de impacto 3.606

Clinical Oral
https://doi.org/10.1007/s00784-023-04899-2

RESEARCH

Design, metallurgy, mechanical properties, and shaping ability of 3 heat-treated reciprocating systems: a multimethod investigation

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Received: 9 April 2022 / Accepted: 1 February 2023

Abstract

Objective This study aimed to compare 3 reciprocating systems regarding design, metallurgy, mechanical properties, and shaping ability.

Materials and methods New Reciproc Blue R25, WaveOne Gold Primary, and REX 25 instruments (n=41 per group) were analyzed regarding design, metallurgy, and mechanical performance, while shaping ability (untouched canal walls, volume of removed dentin, and hard tissue debris) was tested in 36 anatomically matched root canals of mandibular molars. Results were compared using one-way ANOVA post hoc Tukey and Kruskal–Wallis tests with a significant level set at 5%.

Results All instruments showed symmetrical cross sections with asymmetrical blades, no radial lands, no major defects, and an almost equiatomic nickel and titanium ratio. The highest R-phase start temperatures were observed with WaveOne Gold (46.1°C) and REX (44.8°C), while Reciproc Blue had the lowest R-phase start (34.5°C) and finish (20°C) temperatures. WaveOne Gold had the lowest time to fracture (169 s) and the highest maximum load (301.6 gf) (P <0.05). The maximum torque of Reciproc Blue (2.2 N.cm) and WaveOne Gold (2.1 N.cm) were similar (P >0.05), but lower than REX (2.6 N.cm) (P <0.05). No statistical differences were observed among instruments in the angle of rotation (P >0.05) and in the shaping ability in both mesial and distal canals (P >0.05).

Conclusion Although the overall design, temperature transition phases and mechanical behavior parameters were different among tested instruments, they were similar in terms of shaping ability.

Clinical relevance All tested heat-treated NiTi reciprocating systems showed similar shaping ability, without clinically significant errors.

Keywords Canal preparation · Cyclic fatigue · Endodontics · Preparation outcome · Micro-CT · Mechanical features · Nickel-titanium alloy

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Published online: 07 February 2023

Introduction

In the last years, efforts to reduce the fracture occurrence [1, 2] of NiTi instruments resulted in two major technological advancements: the asymmetric oscillatory kinematic — commonly known as reciprocating motion — and the heat treatment of the NiTi alloy. The reciprocating motion relieves stress on the instrument by a special counterclockwise motion rotation to cut dentine and a short clockwise rotation to relieve the instrument [3]. In comparison to continuous rotation, this kinematic extends the instrument lifespan by increasing its resistance to fatigue [4] and reducing the occurrence of plastic deformation [5–7]. The heat treatment, in its turn, allowed the development of NiTi instruments with the crystalline structure in intermediate stages between austenitic and martensitic phases, but with substantial stable martensite phase under body temperature [8, 9]. The alteration in the crystalline microstructure of the NiTi alloy has a significant influence in its mechanical properties once the martensitic phase has greater elasticity and can reach higher deformation with relatively low stress compared to the austenitic one [10].

Reciproc Blue (VDW, Munich, Germany) and WaveOne Gold (Dentsply Sirona Endodontics, Baillagues, Switzerland) are examples of reciprocating instruments composed of substantial amounts of martensite obtained by proprietary heat treatments of the NiTi alloy. Several research studies have confirmed the increased fatigue resistance and flexibility of these heat-treated systems compared to conventional NiTi instruments [11–15]. Recently, the REX reciprocating system (Medidenta, Las Vegas, NV, USA) was launched into the market with the proposal of having NiTi instruments made with different heat treatments, making flexibility and resistance to be consistently balanced depending on the metal mass of each instrument in the series (https://bit.ly/3ZcKeEK). This system includes instruments for mechanical glidepath [REX Glide Path (17/.05v)], with the alloy in purplish color, and instruments presenting different yellowish tonalities for shaping [REX 25 (25/.08v) and REX 40 (40/.06v)]. To date, there is no scientific evidence to support the efficacy or safety of these new instruments. Therefore, this study aimed to use a multimethod approach to compare the design characteristics, metallurgical features, mechanical performance, and shaping ability of REX instruments with the well-known Reciproc Blue and WaveOne Gold systems. The null hypothesis tested was that there would be no differences amongst the tested instruments regarding the evaluated properties.

Material and methods

A total of 123 new 25-mm NiTi instruments (41 per group) from 3 reciprocating systems [Reciproc Blue R25 (25/.08v), WaveOne Gold Primary (25/.07v), and REX 25 (25/.08v)] were analyzed regarding design, metallurgical characteristics, and mechanical performance. In addition, twenty-four instruments (8 per group) were employed for testing the shaping ability of Reciproc Blue [4 R25 and 4 R40 (40/.06v)], WaveOne Gold [4 Primary and 4 Large (45/.06v)], and REX [4 REX 25 and 4 REX 40 (40/.06v)] systems in root canals of extracted mandibular molars. Before their use, the selected instruments were examined under a stereomicroscope (×13.6 magnification; Opmi Pico, Carl Zeiss Surgical, Germany) looking for defects that would exclude them from being tested, but none was excluded.

Instrument Design

The number of active blades (in units) and the helical angles (in degrees) at the 6 most coronal flutes of 6 ran- domly selected instruments from each system were assessed under a stereomicroscope (×13.6 magnification; Opmi Pico) using the ImageJ v1.50e software (Laboratory for Optical and Computational Instrumentation, Madison, WI, USA). These same instruments were further evaluated under scan- ning electron microscopy (SEM) at ×100 and ×500 magnifications (Hitachi S-2400, Hitachi, Tokyo, Japan) regarding their active blade design (radial lands and symmetry), cross-sectional shape, tip geometry (active or non-active), surface finishing, deformations, and defects.

Metallurgical Characterization

The semi-quantitative elemental analysis of 3 instruments from each tested system was carried out to evaluate the proportions of nickel, titanium, or any other relevant element, using a scanning electron microscope (S-2400; Hitachi) equipped with an energy-dispersive X-ray spectroscopy (EDS) (Bruker Quantax; Bruker Corporation, Billerica, MA, USA) set at 20 kV and 3.1 A. The analysis was performed at a 25-mm distance from the surface (400 μm^2) of each instrument using a dedicated software with ZAF correc- tion (Systat Software Inc., San Jose, CA, USA). Differential scanning calorimetry (DSC) method (DSC 204 F1 Phoenix; Netzsch-Gerätebau GmbH, Selb, Germany) was used to determine the phase transition temperatures of instruments' alloy following the guidelines of the American Society for Testing and Materials [16] and a previously documented protocol [13]. Phase transformation temperatures were analyzed by the Netzsch Proteus Thermal Analysis software (Netzsch-Gerätebau GmbH). In each group, DSC test was performed twice to confirm the results. Tested instruments include Reciproc Blue R25, WaveOne Gold Primary, REX Glide Path, REX 25, and REX 40. Unlike Reciproc Blue and WaveOne Gold systems, all set of REX instruments were tested due to differences in their heat treatment, as claimed by the manufacturer (https://bit.ly/3ZcKeEK).

Mechanical Tests

The mechanical performance of the selected systems was evaluated through cyclic fatigue, torsional resistance, and bending tests. The sample size calculation was based on the highest difference of 2 of the tested systems after 6 initial measurements considering an alpha-type error of 0.05 and a power of 80%. For the time to fracture, maximum torque and angle of rotation (WaveOne Gold vs. REX), final sample sizes of 6, 10 and 70 instruments were determined based on effect sizes of 111.8 (± 62.2), 0.6 (± 0.5) and 31.3 (± 47.2), respectively, while, for the maximum load in the bending test (WaveOne Gold vs. Reciproc Blue), an effect size of 59.6 (± 36.7) resulted in a final sample size of 8 instruments. Although sample size calculation determined that 70 instruments would be needed to evaluate the angle of rotation, this high value can be considered of low clinical meaning and therefore the sample size was set at 10 for all parameters.

The cyclic fatigue test was conducted on a non-tapered stainless steel curved tube apparatus (radius of 6 mm and 86° angle) according to a previous reported methodology [13, 17], using glycerin as lubricant. The tested instruments were adapted to a 6:1 reduction handpiece (Sirona Dental Systems GmbH, Bensheim, Germany) powered by a torque-controlled motor (VDW Silver; VDW GmbH) set at RECIPROC ALL (Reciproc Blue and REX) or WAVEONE ALL (WaveOne Gold) modes and activated at a static position. The test was performed at room temperature (20 °C) following the guidelines of the American Society for Testing and Materials applied to superelastic NiTi materials [18]. Fracture was detected by visual and auditory inspection. The time to fracture (in seconds) was recorded using a digital chronometer, and the fragment size (in mm) was measured with a digital caliper for experimental control.

Torsional and bending resistance tests were performed according to international standards [19, 20]. In the torsional test, instruments were clamped at their apical 3 mm and rotated counterclockwise at a constant pace of 2 rotations per minute to assess the maximum torque (in N.cm) and the angle of rotation (in degrees) prior to fracture. In the bending test, each instrument was mounted in the file holder of the motor and positioned at 45° in relation to the floor, while its apical 3 mm was attached to a wire connected to a universal testing machine (Instron 3400; Instron Corporation, Canton, MA, USA). The maximum load needed for a 45° displacement of the instrument, using a load of 20 N and 15 mm/min of constant speed, was recorded in gram/force (gf).

Shaping Ability

After approval of the local Ethics Committee (Protocol CE-FMDUL 13/10/20), ninety-four two-rooted mandibular molars with fully formed apices were randomly selected from a pool of extracted teeth and scanned at pixel size of 11.93 μm in a micro-computed tomographic device (micro-CT) (SkyScan 1173; Bruker-microCT, Kontich, Belgium) set at 70 kV, 114 mA, rotation of 360° with steps of 0.7°, using a 1-mm-thick aluminum filter. The acquired projections were reconstructed into axial cross-sections using standardized parameters of smoothing (1), attenuation coefficient (0.05–0.007), beam hardening (20%), and ring artifact (5) corrections (NRecon v.1.6.9; Bruker-microCT). A three-dimensional (3D) model of the internal anatomy of each tooth was created (CTAn v.1.14.4; Bruker-microCT) and qualitatively evaluated (CTVol v.2.2.1; Bruker-microCT) regarding root canal configuration. Then, volume and surface area the mesial and distal canals were calculated from the cementoenamel junction to the apex. Based on these parameters, specimens were anatomically matched to create 3 groups of 4 teeth (n = 12 canals). Then, each set of teeth was randomly assigned to an experimental group according to the preparation system: Reciproc Blue, WaveOne Gold, and REX.

After conventional access cavity preparation, apical patency was confirmed using a size 10 K-file (Dentsply Sirona Endodontics). Glide path was then performed with a size 15 K-file (Dentsply Sirona Endodontics) up to the working length (WL), established 1 mm from the apical foramen. All canals were initially prepared with instruments size 25, according to each group (Reciproc Blue R25, WaveOne Gold Primary, and REX 25) and then distal canals were further enlarged with instruments size 40 (Reciproc Blue R40 and REX 40) or size 45 (WaveOne Gold Large). Instruments were activated in reciprocating motion powered by an electric motor (VDW Silver; VDW) set at "RECIPROC ALL" (Reciproc Blue and REX) or "WAVEONE ALL" (WaveOne Gold) modes. Each instrument was moved to the apical direction using a slow in-and-out pecking motion of about 3-mm amplitude with light pressure. After 3 pecking motions, the instrument was removed from the canal and cleaned. The WL was reached after 3 waves of instrumentation. Each instrument was used in one tooth and discarded. Irrigation was performed with a total of 15 mL of 2.5% NaOCl per canal, followed by a final rinse with 5 mL of 17% EDTA (3 min) and 5 mL of distilled water using a syringe fitted with a 30-G NaviTip needle (Ultradent, South Jordan, UT, USA) positioned 2 mm from the WL. All procedures were performed by an operator with a large experience using reciprocating systems.

After slightly drying the root canals with paper points (VDW), a final scan and reconstruction were performed using the previously mentioned parameters followed by the co-registration of datasets acquired before and after preparation (3D Slicer 4.3.1 software; http://www.slicer.org). Shaping ability was assessed by measuring 3 parameters: (i) the volume (in mm^3) of dentin removed after preparation, (ii) the volume (in mm^3) of hard tissue debris created by the preparation protocols, and (iii) the percentage of unprepared canal walls, according to methodologies published in previous studies [21, 22]. All analyses were done by an examiner blinded to the shaping protocols. Canal interconnections and accessory anatomies were excluded for the analyses.

Statistical Analysis

The Shapiro-Wilk and Lilliefors tests were used to verify the normality of the data. One-way ANOVA and post hoc Tukey tests were carried out to compare the helical angle, time to fracture, angle of rotation, maximum bending load, root canal volume and surface area, volume of removed dentine, hard tissue debris in the mesial canals, and untouched canal walls, while Kruskal–Wallis test was used to evaluate the maximum torque to fracture and the volume of hard tissue debris in the distal canals, with a significance level set at 5% (SPSS v25.0 for Windows; SPSS Inc., Chicago, IL, USA). Depending on data distribution, results were summarized as mean (standard deviation) or median (interquartile range) values.

Results

Instrument Design

The stereomicroscopic assessment revealed similar number of blades and helical angles in the REX and WaveOne Gold instruments (Table 1). SEM analysis (Fig. 1) showed that all instruments had symmetrical cross sections with asymmetrical blades and no radial lands. WaveOne Gold instrument had an offset parallelogram-shaped cross section, whilst REX and Reciproc Blue had an inverted S-shaped profile. None of the tips could be identified as active, and the overall geometry and transition angles to the blade varied amongst instruments. While the tip of Reciproc Blue and WaveOne Gold was flat at its end, it showed a bullet-like shape in the REX instrument. In higher magnification, all instruments showed similar surface finishing with a pattern of parallel horizontal marks created by the grinding manufacturing process. In the REX instruments, it was also possible to observe some metal rollovers on the blades.

Metallurgical Characteristics

EDS/SEM analysis revealed a nearly equiatomic composition of nickel and titanium elements in all instruments (Ni/Ti Ratio 1.016, 1.032, and 1.028 for Reciproc Blue, WaveOne Gold, and REX instruments, respectively), without any other traceable metal element. Cooling and heating curves of tested instruments obtained by the DCS analyses are depicted in Fig. 2. Comparison among systems (Fig. 2a) showed distinct transformation temperature curves suggesting the presence of R-phase in all of them at testing temperature (20 °C). The highest R-phase start temperatures were observed in WaveOne Gold Primary (46.1 °C) and REX 25 (44.8 °C) instruments. Reciproc Blue R25 had the lowest R-phase start (34.5 °C) and R-phase finish (20 °C) temperatures (Figure 2a). The lowest (8.5 °C) and high- est (51.3 °C) austenitic start and finish temperatures were observed in the WaveOne Gold Primary instrument. DSC test of REX instruments (Fig. 2b) demonstrated similar heat treatment between REX Glide Path and REX 25 with minor differences in R-phase transformation temperatures, in the cooling transformation of martensitic B19', and in the austenitic transformation during heating curves. On the other hand, REX 40 showed major differences mostly on cooling (R-phase to martensite B19' transformation) and on heating, with an almost perfectly overlapped martensitic B19' and R-phase transformations to austenite-B2 (Fig. 2b).

Mechanical Performance

WaveOne Gold had the lowest time to fracture and the highest maximum load ($P < 0.05$), while no statistical differences were observed in these parameters between Reciproc Blue and REX instruments ($P > 0.05$). The maximum torque values of Reciproc Blue and WaveOne Gold were similar ($P > 0.05$), but lower than REX instrument (*P*

Table 1 Stereomicroscopic assessment of instruments expressed as mean (standard deviation) or median [interquartile range]

System	Design		Cyclic fatigue	Torsional test		Bending test
	Blades (*n*)	Helical angle (°)	Time to fracture (s)	Maximum torque (N.cm)	Angle of rotation (°)	Maximum load (gf)
Reciproc Blue R25	8	152.9 (±1.5)[a]	255.3 (±36.7)[a]	2.2 [2.0–2.2][a]	488.5 (±45.4)[a]	247.4 (±18.0)[a]
WaveOne Gold Primary	7	155.3 (±0.8)[b]	169.1 (±41.7)[b]	2.1 [1.9–2.1][a]	493.4 (±35.5)[a]	301.6 (±17.9)[b]
REX 25	7	155.2 (±0.5)[b]	244.7 (±51.5)[a]	2.6 [2.4–2.8][b]	460.4 (±43.3)[a]	259.8 (±12.7)[a]

Different superscript letters in the same column represent statistically significant difference ($p < 0.05$)

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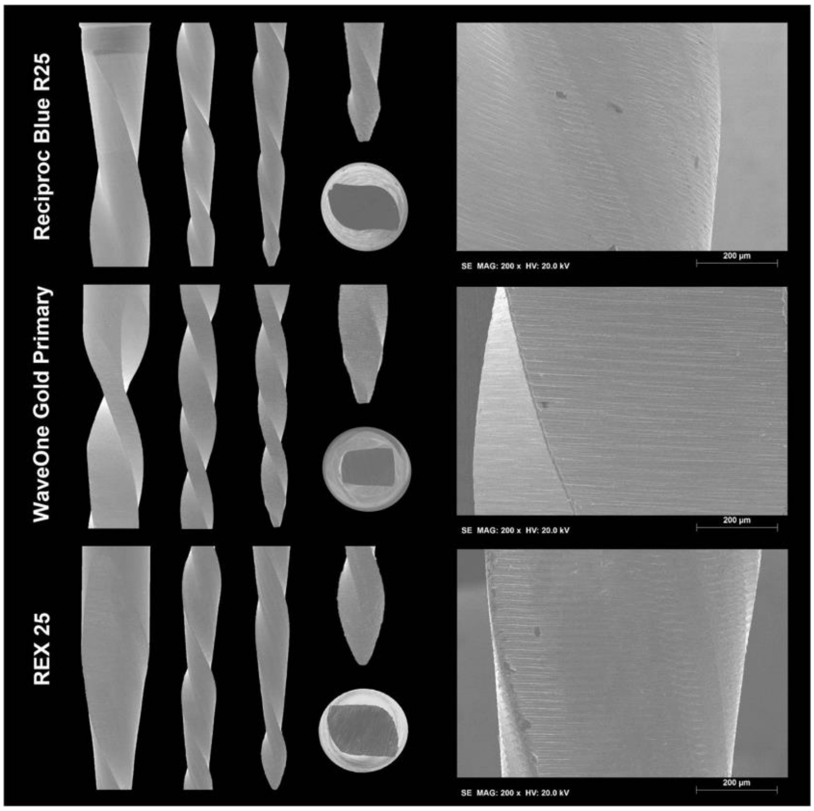

Fig. 1 SEM representative images of the tested instruments depicting that all instruments have symmetrical cross sections with asymmetrical blades and no radial lands. REX and Reciproc Blue showed an inverted S-shaped profile, while WaveOne Gold had an offset parallelogram-shaped cross section. Tips were non-active with differences in the overall geometry and transition angles to the blade. All surfaces had parallel manufacturing marks with few irregularities. In the REX instruments, it is possible to observed metal rollovers on the blades

< 0.05). No difference amongst instruments was observed in the angle of rotation ($P > 0.05$) (Table 1).

Shaping Ability

The homogeneity of groups regarding morphometric parameters of volume and surface area in mesial and distal root canals was confirmed ($P > 0.05$) (Table 2). No statistical differences were observed among the tested systems regarding the volume of hard tissue debris ($P > 0.05$), the dentin removed after preparation ($P > 0.05$), and the percentage of untouched canal walls in both mesial and distal canals ($P > 0.05$). None of the preparation protocols was able to prepare all root canal surfaces (Fig. 3) or render root canals free from hard tissue debris (Table 2). The mean percentages of unprepared canal walls were 21.8% (Reciproc Blue), 17.4% (REX), and 21.5% (WaveOne Gold) in the mesial canals (Table 2), and 16.8% (Reciproc Blue), 13.6% (REX) and 17.0% (WaveOne Gold) in the distal canals (Table 2).

Discussion

The present investigation provides answers to a series of questions about the mechanical behavior of 3 reciprocating systems through the use of a multimethod research analysis. The main advantage of this approach is the possibil- ity to offset the weaknesses of each test providing more information, better understanding, and superior internal and external validation [23]. In addition, this approach avoids the phenomenon of “knowledge compartmentali- zation”, i.e., the knowledge about a specific domain com- posed of several separate, not intertwined parts, usually obtained in single or double assessment methods [24]. In this study, overall design, manufacturing quality, elemen- tal composition, and phase transformation temperatures of Reciproc Blue, WaveOne Gold, and REX reciprocat- ing NiTi systems were assessed in order to achieve a bet- ter comprehension of the results obtained in the cyclic fatigue, torsional resistance, bending load, and shaping ability tests. Notwithstanding similarities regarding the

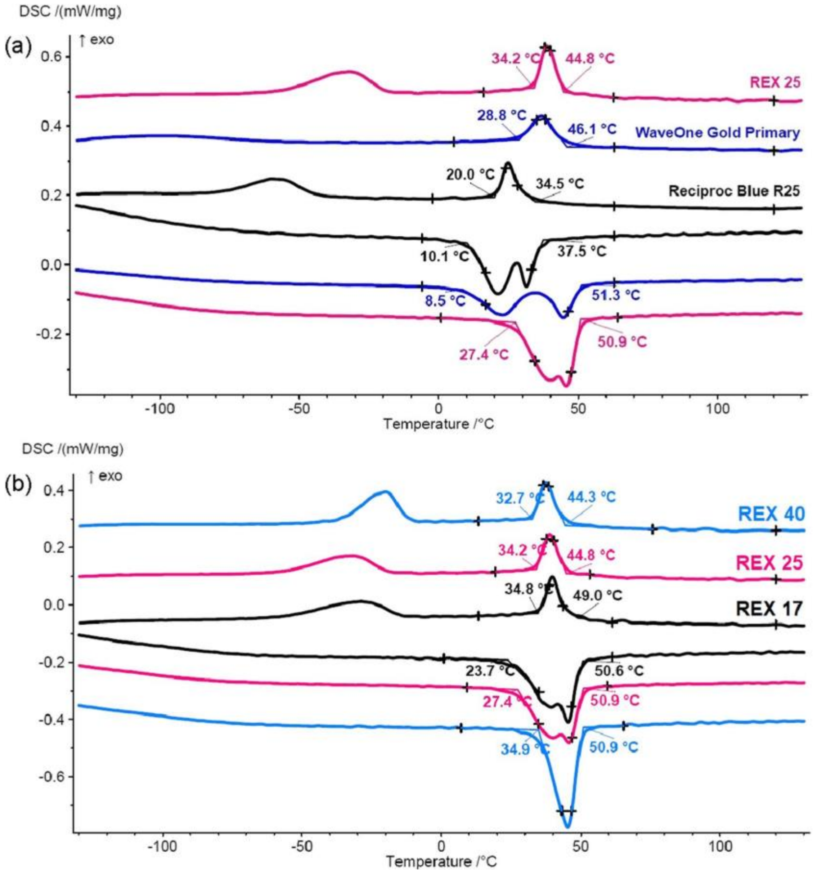

Fig. 2 DSC charts showing the phase transformation temperatures of the assessed files, with the cooling curves on top (reads from right to left) showing the R-phase start (Rs) and finish (Rf) temperatures and heating curves on bottom (reads from left to right) detailing the austenitic start (As) and finish (Af) temperatures. **a** The highest R-phase start temperatures were observed with WaveOne Gold Primary (46.1 °C) and REX 25 (44.8 °C), while Reciproc Blue R25 had the lowest R-phase start (34.5 °C) and R-phase finish (20 °C) temperatures. The lowest (8.5 °C) and highest (51.3 °C) austenitic start and finish temperatures were observed in the WaveOne Gold Primary instrument. REX 25 showed DSC curves completely different from both others, while all of them appear to be at R-phase at test temperature (20° C). **b** Transformation temperatures of the REX system instruments. REX 17 (Glide Path) and REX 25 had similar transformation temperatures with minor differences in R-phase transformation temperatures and in the cooling transformation of martensitic B19'. The REX 40 had the most visible differences with an almost perfectly overlapped martensitic B19' and R-phase transformations to austenite-B2

helical angles (Table 1), elemental constitution, and shaping ability (Table 2, Fig. 3), significant differences were observed in the overall design (Fig. 1), mechanical properties (Table 1), and temperature transition phases (Fig. 2), and the null hypothesis was partially rejected.

The analysis of the mechanical performance of NiTi preparation systems must be done taken into account several factors. Since the alloys of tested instruments in this study were similar in terms of elemental constitution, information regarding their phase transformation temperatures (austenitic and martensitic crystallographic arrangements) and design are of utmost importance to explain their mechanical behavior [9, 10]. Considering differences on the dimensions of instruments available in each tested system, the first DSC analysis was performed only in instruments with a tip size 25 and revealed the presence of R-phase alloy in all of them at test temperature (20 °C) (Fig. 2a). The R-phase alloy is characterized as an intermediate crystalline phase that occurs along a very narrow temperature range on the heating or cooling curve between martensitic and austenitic forms. This phase change in the crystal structure of the alloy results in lower resistance to elastic deformation (high flexibility and low rigidity), increasing its resistance to cyclic fatigue, while reducing its torsional resistance when compared

Table 2 Pre- and post-operative parameters (mean, standard deviation, and range interval) evaluated in mesial (*n*=24) and distal (*n*=12) root canals of mandibular molars after preparation protocols with 3 reciprocating instruments

Canal	Parameters		Reciproc Blue	WaveOne Gold	REX
Mesial	Volume (mm^3)	Before	4.0 ± 2.1 (2.5–7.2)	4.4 ± 1.5 (2.3–5.7)	5.1 ± 2.1 (2.8–7.6)
		After	8.8 ± 1.0 (7.5–10.1)	9.0 ± 1.4 (7.5–10.9)	10.1 ± 1.7 (8.1–12.3)
	Surface area (mm^2)	Before	61.5 ± 12.5 (53.4–80.1)	68.4 ± 23.6 (43.5–98.6)	74.2 ± 19.1 (50.1–94.3)
		After	77.0 ± 8.5 (69.9–89.3)	82.6 ± 18.7 (68.0–108.1)	88.8 ± 15.3 (72.3–107.3)
	Removed dentin (mm^3)	After	4.5 ± 1.5 (2.4–5.9)	4.2 ± 1.4 (3.0–5.8)	4.6 ± 1.9 (1.8–6.1)
	Debris (mm^3)	After	0.030 ± 0.025 (0.001–0.051)	0.035 ± 0.026 (0.013–0.071)	0.036 ± 0.040 (0.002–0.093)
	Unprepared area (%)	After	21.8 ± 11.7 (8.0–33.2)	21.5 ± 7.9 (15.2–32.9)	17.4 ± 9.8 (5.1–28.5)
Distal	Volume (mm^3)	Before	6.9 ± 2.7 (5.3–10.0)	6.8 ± 2.0 (5.1–9.7)	8.0 ± 2.3 (5.1–10.4)
		After	10.3 ± 3.1 (7.7–13.8)	10.0 ± 1.6 (7.8–11.8)	10.4 ± 2.1 (7.5–12.3)
	Surface area (mm^2)	Before	72.6 ± 17.0 (60.6–92.2)	73.6 ± 7.5 (63.9–81.9)	73.4 ± 16.0 (50.7–88.2)
		After	78.4 ± 21.3 (63.0–102.8)	76.6 ± 14.5 (55.9–88.0)	76.0 ± 17.0 (52.4–90.0)
	Removed dentin (mm^3)	After	3.0 ± 0.8 (2.0–3.6)	2.9 ± 1.2 (1.7–4.3)	2.0 ± 0.4 (1.6–2.6)
	Debris (mm^3)	After	0.010 ± 0.009 (0.001–0.017)	0.057 ± 0.047 (0.000–0.096)	0.063 ± 0.083 (0.012–0.186)
	Unprepared area (%)	After	16.8 ± 4.3 (13.0–21.5)	17.0 ± 11.3 (4.3–32.0)	13.6 ± 7.1 (6.6–23.5)

to conventional austenitic alloys [25]. The intermediate R-phase has specific temperatures for its formation represented by R_s, for the beginning of phase formation, and R_f for the end [10, 26]. In the present study, REX had the highest R_f temperature (34.2 °C), followed by WaveOne Gold (28.8 °C) and Reciproc Blue (20 °C) (Fig. 2a). Considering that the mechanical tests were conducted in accordance to an international standard for testing transformation temperature of nickel-titanium alloys at room temperature (20 °C) [18], it would be expected that all instruments had martensitic characteristics during the test. In contrast, at body temperature (36 °C), the instrument that more quickly approached the austenitic crystallographic arrangement would be the Reciproc Blue. Therefore, depending on test temperature, instruments may present changes in their behavior. Since this is the first study evaluating the REX system, the second DSC analysis was done in its set of instruments (Fig. 2b) and confirmed the manufacturer claim that these instruments are made with different customized heat treatments. However, DSC results suggest only minor differences in the R-phase and martensitic B19’ transformation temperatures on both cooling and heating.

Notwithstanding DSC analysis revealed that REX instrument had higher martensitic composition than Reciproc Blue at room temperature (20 °C) (Fig. 2a), no differences were observed between them in the cyclic fatigue, angle of rotation (torsional test), and bending resistance tests (Table 1), findings that can be explained by the larger metal core (Fig. 1) and greater number of blades (Table 1) of REX instruments. Differences in the design also help to explain the highest maximum torque to fracture observed during torsional resistance test of REX instruments (Table 1). On the other hand, even though WaveOne Gold also had a high martensitic composition (Fig. 2a), it showed lower time to fracture (cyclic fatigue) and flexibility (bending resistance) than REX and Reciproc Blue (Table 1). Again, the design of the WaveOne Gold with its large cross-sectional design and taper (Fig. 1) may explain the results. Although only minor differences were observed in the heat treatments of REX instruments (Fig. 2b), they might influence their clini- cal behavior. For instance, at the test temperature (20° C), the lower A_s of REX Glide Path indicates a more austenitic composition compared to other instruments, which may be translated as a better resistance to torsion. In its turn, REX 40 had the highest A_s amongst REX instruments. It means that this large-tapered heat-treated instrument may present a high torque strength and flexibility during shaping procedures, an important aspect that may prevent fracture by torsional stress.

In the last years, there seems to be a tendency of industry to develop proprietary heat treatments of the NiTi alloy in order to create ultraflexible instruments with superior amount of martensitic crystallographic arrangement at temperatures above 30 °C [15, 17] and/or by changing the design with an increased number of spirals and reduced metal core [14]. In the laboratory, these changes usually improve some mechanical properties of the instrument including cyclic fatigue resistance, angle of rotation, and flexibility (low bending load resistance), but, on the other hand, they may compromise its torsional strength [14]. Besides, in the clinical setting, ultra flexible instruments usually needs to apply more apical pressure to reach the working length [17], which may lead to early plastic deformation or fracture [14]. Therefore, considering the unfeasibility of creating a single instrument that combines all of the best metallurgic and mechanical features with the available technology, the latest generations of rotary systems are including, in the same set, instruments with

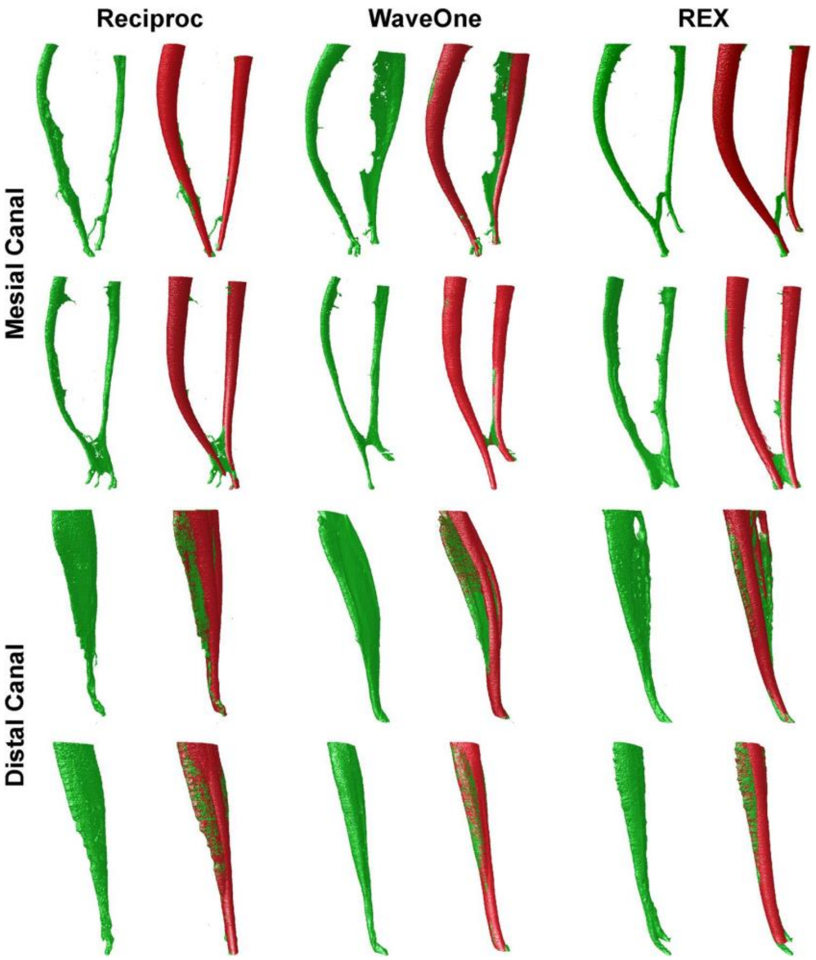

Fig. 3 Representative micro-CT 3D models of mesial and distal canals mandibular molars showing the root canals before (green color) and after (red color) preparation with Reciproc, WaveOne Gold and REX systems. Mesial canals were prepared with instruments size 25, while the enlargement of distal canals were done with instruments size 40 (Reciproc Blue R40 and REX 40) or size 45 (WaveOne Gold Large). None of the shaping protocols were able to prepare the entire surface area of the root canal walls

different designs and crystallographic arrangements. In theory, it allows to customize an instrument in order to improve its fracture resistance and/or flexibility depending on the canal morphology or treatment phase. This is, for example, the proposal of some recently launched systems including the EdgeSequel Sapphire (EdgeEndo, Albuquerque, NM), the ProTaper Ultimate (Dentsply Sirona Endodontics, Baillagues, Switzerland), the Genius Proflex (Medidenta, Las Vegas, NV), the One Endo File (NanoEndo LCC, Chattanooga, TN), and the REX system evaluated in this study. Although the DSC analysis demonstrated differences in the heat treatment among REX instruments, it did not translate into a better shaping performance in extracted teeth when compared to the other tested systems (Table 2). The multimethod research applied to this study included not only the evaluation of metallurgical and mechanical properties of instruments, but also the assessment of several shaping ability parameters obtained from the root canal preparation of extracted molars using micro-CT imaging, an analytical tool that allows the longitudinal track of a sample at different time points. Preliminary efforts were made to ensure the anatomical matching of the specimens in each group according to some morphometric parameters in order to create a reliable baseline and enhance the internal validity of the study [27]. Although differences could be observed in the overall design of the instruments (Fig. 1) and in their mechanical behavior (Table 1), no significant differences were observed among them regarding the volume of hard tissue debris, the dentin removed after preparation, and the percentage of untouched canal walls in both mesial and distal canals (Table 2). Besides, no instrument fracture or significant deviation of the original canal was observed. These findings can be explained by the use of instruments with similar dimensions, preparation protocols, and kinematics in anatomically balanced specimens, corroborating recent micro-CT studies [13, 14, 17, 28–31]. None of the preparation protocols was able to prepare all root canal surfaces or render root canals free from hard tissue debris, which is also in accordance with previous publications [32, 33]. Besides, this outcome agrees with other studies that also showed no difference in the percentage of untouched canal areas in extracted teeth after using Reciproc Blue and WaveOne Gold [17, 34]. No comparison could be made with REX system as this is the first study that assessed its shaping ability.

The strengths of the present study relies on the multimethod assessment of different reciprocating instruments using methodologies validated by international standards [16, 18–20] or previously methods with high internal validity [13, 27, 31] which allowed a robust and trustworthy understanding of their mechanical performance. The limitations include the lack of other tests such as cutting efficiency, microhardness, buckling resistance, and measurements of instruments' dimensions. Therefore, future studies should include additional methodologies to evaluate other rotary or reciprocating NiTi systems with different designs and crystallographic arrangements.

Conclusions

Under the conditions of this multimethod study, Reciproc Blue, WaveOne Gold, and REX reciprocating systems were similar regarding elemental composition and shaping ability, but showed significant differences in their overall design, temperature transition phases, and mechanical behavior.

Acknowledgements FMBF acknowledges the funding of CENIMAT/i3N by national funds through the FCT-Fundação para a Ciência e a Tecnologia, I.P., within the scope of Multiannual Financing of R&D Units, reference UIDB/50025/2020-2023. Fernanda Carvalho is acknowledged for running the DSC tests of the files.

Authors' contributions All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Emmanuel J. N. L. Silva, Jorge N.R. Martins, Natasha C. Ajuz, Henrique dos Santos Antunes, Victor T. L. Vieira, Francisco Manuel Braz-Fernandes, Felipe G. Belladona, and Marco A. Versiani. The first draft of the manuscript was written by Emmanuel J.N.L. Silva, Jorge N.R. Martins, and Marco A. Versiani, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Funding This work was partially supported by the FAPERJ and CNPq.

Declarations

Ethical approval All procedures performed in studies involving human participants were in accordance with the ethical standards of the institutional and/or national research committee and with the 1964 Helsinki declaration and its later amendments or comparable ethical standards.

Informed consent For this type of study, formal consent is not required.

Conflict of interest The authors declare no competing interests.

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4.3. CAPÍTULO 3

MULTIMETHOD ANALYSIS OF THREE ROTARY INSTRUMENTS PRODUCED BY ELECTRIC DISCHARGE MACHINING TECHNOLOGY

Artigo publicado na revista *International Endodontic Journal*

doi: 10.1111/iej.13915

Classificação de periódicos quadriênio 2017-2020 – Odontologia – A1

Fator de impacto 5.165

Received: 17 October 2022 | Accepted: 16 March 2023
DOI: 10.1111/iej.13915

ORIGINAL ARTICLE

INTERNATIONAL ENDODONTIC JOURNAL | WILEY

Multimethod analysis of three rotary instruments produced by electric discharge machining technology

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Funding information
Conselho Nacional de Desenvolvimento Científico e Tecnológico; Fundação Carlos Chagas Filho de Amparo à Pesquisa do Estado do Rio de Janeiro; Fundação para a Ciência e a Tecnologia

Abstract

Aim: This study aimed to compare three rotary instruments produced by the EDM process with the heat-treated ProTaper Gold system regarding design, metallurgy, mechanical properties and shaping ability.

Methodology: HyFlex EDM (25/~), Neoniti (25/.06), EDMax (25/.06) and ProTaper Gold (25/.08v) instruments (n = 58 per group) were compared regarding design, metallurgy and mechanical performance. Unprepared canal areas were calculated for each system after preparation of mesiobuccal, mesiolingual and distal canals of mandibular molars (15 canals per group) using micro-CT technology. Statistical analyses were performed using One-way anova post-hoc Tukey and Kruskal–Wallis post-hoc Dunn's tests ($\alpha = 5\%$).

Results: All instruments had asymmetrical blades, no radial lands, no major defects and almost equiatomic nickel/titanium ratios, but different cross-section designs, tip geometries and surface appearances. Although instruments had distinct transformation temperature curves, they showed crystallographic martensitic arrangement at 21°C and mixed austenite plus R-phase at body temperature. Neoniti and HyFlex EDM showed similar results in all mechanical tests ($p > .05$), while EDMax and ProTaper Gold had similar time to fracture ($p = .841$), maximum bending load ($p = .729$), and cutting ability ($p = .985$). ProTaper Gold showed the highest torque to failure ($p < .001$) and HyFlex EDM had the lowest buckling resistance ($p < .001$). Mean percentages of unprepared canal areas ranged from 20.4% to 25.7% in the mesial canals, and from 20.8% to 26.2% in the distal canal, with no statistical differences among systems ($p > .05$).

Conclusions: Instruments' geometry and phase transformation temperatures influenced the results of the mechanical tests, but not their shaping ability.

KEYWORDS

differential scanning calorimetry, endodontics, mechanical properties, micro-CT, nickel–titanium alloy, scanning electron microscopy

INTRODUCTION

Improvements in the nickel–titanium (NiTi) metallurgy allowed the development of a variety of new endodontic instruments with different designs, which ended up in an enhanced efficiency not only in the control of iatrogenic mishaps, such as deviation and perforation but also in the root canal shaping, making it faster, easier and with better clinical outcomes compared with conventional preparation with stainless-steel hand files (Bürklein & Arias, 2022). Even so, NiTi instruments are still susceptible to deformations and/or fractures, an unwanted event that may represent a predictor of persistent apical periodontitis and consequent failure when treating infected teeth (McGuigan et al., 2013; Ng et al., 2011). To overcome these problems, manufacturers have developed several strategies to improve the properties of the NiTi alloy including changes on kinematics, instruments' design and surface treatment (Martins, Martins, et al., 2022). In the last years, manufacturers have also developed different production methods to the traditional grinding method such as twisting, shape-setting, laser-cutting and electric discharge machining (EDM) (Arias & Peters, 2022). Through the process of EDM, instruments are manufactured by a non-contact thermal erosion through controlled sparks that occur between an electrode and a metal workpiece in the presence of a dielectric fluid (Arias & Peters, 2022; Pirani et al., 2016). This process “melts” the surface of the nickel–titanium alloy, partially evaporating small portions of the metal and leaving behind an eroded surface. The instrument is then heat-treated at temperatures between 300 and 600°C for 10 min to 5 h, before or after ultrasonic cleaning and acid bath (Gavini et al., 2018). This unique process does not use physical contact for material removal, but the local vaporization of the metal preventing the formation of microcracks, and may optimize the cutting ability, flexibility and cyclic fatigue resistance of rotary instruments (Arias & Peters, 2022; Gavini et al., 2018; Pedullá et al., 2016; Pirani et al., 2016).

The first NiTi rotary instrument launched in the market and manufactured using the EDM process was an orifice opener named Initial (Neolix SAS) (Mallet, 2012). In the following year, the same company launched the Neoniti system (Neolix SAS), a set of rotary instruments also produced by the EDM method (Stanurski, 2013). HyFlex EDM (Coltene/Whaledent) was launched 2 years later (Müller, 2015) and initial studies demonstrated a higher cyclic fatigue resistance compared with other instruments produced with superelastic or martensitic NiTi alloys (Gündoğar & Özyürek, 2017; Silva et al., 2020; Thu et al., 2020). More recently, a multimethod study showed no difference between the mechanical behaviour of HyFlex EDM and Neoniti instruments (Silva et al., 2020). In the current year, the EDMax system (Neolix SAS), another set of rotary instruments produced by the same process, was introduced into the market. However, according to the manufacturer, this system has striking differences compared with Neoniti, including streaked cutting edges, non-rectangular variable parallelogram cross-section with sharp cutting edges, and hardened and abrasive surface (https://bit.ly/3SJPOef). In addition, EDMax instruments are submitted to a heat treatment that results in active blades with blueish colour, in contrast to the yellowish colour of Neoniti and HyFlex EDM instruments. These modifications were implemented to this system aiming to improve their mechanical efficiency and shaping ability; but, thus far, there is no scientific evidence to support this statement. Therefore, this study aimed to use a multimethod approach to evaluate design, metallurgy, mechanical performance and shaping ability of 3 preparation systems manufactured using the EDM process (HyFlex EDM, Neoniti, and EDMax), using the ProTaper Gold system (Dentsply Sirona) as reference for comparison. The null hypothesis tested was that there would be no differences among tested instruments regarding their mechanical properties.

MATERIALS AND METHODS

The manuscript of this laboratory study has been written according to Preferred Reporting Items for Laboratory studies in Endodontology (PRILE) 2021 guidelines (Nagendrababu et al., 2021) (Figure 1). A total of 232 new

FIGURE 1 PRILE flowchart.

25-mm NiTi instruments (58 per group) from HyFlex EDM (25/~), Neoniti (25/0.06), EDMax (25/0.06) and ProTaper Gold (25/0.08v) rotary systems were tested regarding design, metallurgical characteristics and mechanical performance, while 100 instruments (HyFlex EDM [n = 20], Neoniti [n = 25], EDMax [n = 25], and ProTaper Gold [n = 30]) were additionally employed to compare the shaping ability of each system in root canals of extracted mandibular molars, using the instrumentation sequence recommended by the manufacturers. Instruments were previously examined under a stereomicroscope (×13.6 magnification; Opmi Pico, Carl Zeiss Surgical) looking for defects that would exclude them from being tested, but none was excluded.

Design

Three new 25-mm instruments per system (n = 12) were evaluated under conventional scanning electron microscopy (SEM) (S-2400, Hitachi) regarding the symmetry of the blade (symmetrical or asymmetrical) (×20 magnification), the tip geometry (active or non-active) (×40), the cross-sectional shape (×80) and the presence of surface marks, deformations or defects produced by the manufacturing process (×200).

Metallurgy

The semi-quantitative elemental analysis was carried out in 3 instruments from each tested system to evaluate the ratio of nickel and titanium in the alloy, or the presence of other elements, using a scanning electron microscope (S-2400; Hitachi) equipped with energy-dispersive X-ray spectroscopy (EDS) (Bruker Quantax; Bruker Corporation) set at 20 kV and 3.1 A. The analysis was performed in each instrument at a 25-mm distance from a surface area of 400 µm^2 using dedicated software with ZAF correction (Systat Software Inc.). Differential scanning calorimetry (DSC) method (DSC 204 F1 Phoenix; Netzsch-Gerätebau GmbH) was applied to determine the phase transformation temperatures of the NiTi alloy (ASTM International, 2004). Fragments of 2 to 3 mm in length (5–10 mg) acquired from the coronal active blade of two instruments from each system were exposed to a chemical etching (25% hydrofluoric acid, 45% nitric acid and 30% distilled water) for 2 min and mounted on an aluminium pan inside the DSC device, with an empty pan serving as control. Each thermal cycle was performed under gaseous nitrogen atmosphere at a pace of 10°C/min with temperatures ranging from −150°C to 150°C, and the phase transformation temperatures were analysed by the Netzsch Proteus Thermal Analysis software (Netzsch-Gerätebau GmbH). In each group, the DSC test was performed twice.

Mechanical tests

The mechanical performance of the selected systems was assessed by cyclic fatigue, cutting ability, torsional, bending and buckling resistance tests. Sample sizes were calculated based on the highest difference obtained by two instruments after six initial measurements with an alpha-type error of 0.05 and power of 80%. For the time to fracture (Neoniti vs. EDMax; effect size of 0.80), maximum torque (Neoniti vs. EDMax; effect size of 1.0), angle of rotation (Neoniti vs. EDMax; effect size of 0.85), maximum bending load (Neoniti vs. EDMax; effect size of 0.87), buckling resistance (Neoniti vs. EDMax; effect size of 0.54), and cutting ability (Neoniti vs. EDMax; effect size of 0.88) parameters, sample sizes were 5, 4, 5, 5, 10, and 5, respectively. Therefore, a total of 10 instruments per group was defined for each dependent variable.

The cyclic fatigue test followed the methodology reported in a previous study (Martins, Silva, et al., 2022) and was conducted at room temperature in accordance with the recommendations of the American Society for Testing and Materials (ASTM International, 2004) and a current ISO norm proposal (Peters et al., 2020). All instruments were activated at static mode by a torque-controlled motor (VDW Silver; VDW) set at 300 rpm and 1.5 N. Fracture was detected by visual and auditory inspection, time to fracture (in seconds) was recorded using a digital chronometer, and the fragment size (in mm) was measured with a digital calliper (resolution of 0.01 mm; Mitutoyo) for experimental control. Torsional and bending resistance tests were performed according to international standards (ISO 3630-3631, 2008) to assess the maximum torque (in N.cm), the angle of rotation prior to fracture (in degrees), and the maximum load needed for a 45° displacement of the instrument (in gram/force; gf), respectively. The buckling test was performed according to a previous publication (Lopes et al., 2012). A diagram of load (N) × deformation (mm) was obtained for each instrument and the maximum load needed to induce the elastic displacement of the instrument up to 1 mm was calculated. The cutting efficiency test was done following the methodology proposed by Plotino et al. (2014). Each instrument was powered by an electric motor (Reciproc Silver; VDW GmbH) mounted to a free-falling holder and activated (300 rpm; 3.0 N) in direct contact with a Plexiglass block for 1 min. The analysis of the cutting depth in the blocks was performed using a digital calliper (Mitutoyo).

Shaping ability

After approval of this research project by the local Ethics Committee (Protocol CAAE 57369521.9.0000.5283), 20 two-rooted mandibular molars presenting mesial and distal root canals with moderate curvature (Schneider, 1971) and Vertucci's Type IV and I configurations, respectively, were selected. Inclusion criteria also comprised teeth with fully formed apices, no internal resorption, calcification, previous endodontic treatment or root fracture. All teeth were imaged in a micro-CT device (SkyScan 1174v.2; Bruker-MicroCT) and reconstructed (NRecon v.1.6.9; Bruker-microCT) using standardized parameters, according to a previous study (Silva et al., 2020). Then, information about volume (in mm^3), surface area (in mm^2) and configurations of mesial and distal root canals (CTAn v.1.14.4; Bruker-microCT), were obtained to create 4 anatomically-matched groups (n = 15 canals per group). After conventional access cavity preparation, apical patency was confirmed with a size 10 K-file (Dentsply Sirona Endodontics) and glide path performed using a size 15 K-file (Dentsply Sirona Endodontics) up to the working length (WL), established 1 mm from the apical foramen. One set of each tested system was used to prepare 1 tooth (3 canals) according to the following protocols:

- *HyFlex EDM system*: After coronal flaring (instrument 25/.12; 500 rpm, 2.5 N.cm), instruments 10/.05 (300 rpm 1.8 N.cm) and 25/~ (500 rpm, 2.5 N.cm) were used up to the WL.
- *Neoniti system*: After coronal flaring (instrument 25/.12; 300 rpm, 1.5 N.cm), instruments 15/.03 (300 rpm, 1.5 N.cm), 20/.06 (300 rpm, 1.5 N.cm) and 25/.06 (300 rpm, 1.5 N.cm) were used up to the WL.
- *EDMax system*: after coronal flaring (instrument 20/.10; 500 rpm, 1.5 N.cm), instruments 15/.03 (500 rpm, 1.5 N.cm), 20/.06 (500 rpm, 1.5 N.cm), and 25/.06 (500 rpm, 1.5 N.cm) were used up to the WL.
- *ProTaper Gold system*: After coronal flaring (instrument SX 19/.04v; 300 rpm, 5.0 N.cm), instruments S1 (18/.02v; 300 rpm, 1.5 N.cm), S2 (20/.04v; 300 rpm, 1.5 N.cm), F1 (20/.07v; 300 rpm, 1.5 N.cm), and F2 (25/.08v; 300 rpm, 3.0 N.cm) were used up to the WL.

Considering that the physiological diameter of distal canals of mandibular molars at the apical third has been reported to range from 0.39 and 0.46 mm (Wolf et al., 2017), these canals were further enlarged using the size 40 instrument of each system (HyFlex EDM 40/.04, Neoniti 40/.04, EDMax 40/.04 and ProTaper Gold 40/.06v).

Instruments were activated by an electric motor (VDW Silver; VDW) and used in a slow in-and-out pecking motion of about 3 mm amplitude with light pressure to the apical direction. After three pecking motions, the instrument was removed from the canal and cleaned. The WL was reached after 3 waves of instrumentation. Each instrument was used in one tooth and discarded. Irrigation was performed with a total of 15 mL of 2.5% NaOCl per canal, followed by a final rinse with 5 mL of 17% EDTA (3 min) and 5 mL of distilled water using a syringe fitted with a 30-G NaviTip needle (Ultradent) positioned 2 mm from the WL. All procedures were performed by an experienced operator under magnification (×12.5; Zeiss OPMI Pico). After preparation, the canals were slightly dried with paper points and a final scan and reconstruction were performed using the previously mentioned parameters. Datasets acquired before and after preparation were co-registered and the root canals were evaluated regarding volume, surface area and unprepared surface areas. The latter was determined by the formula (Au/Ab)*100, where Au and Ab represent the unprepared area and the canal area before preparation, respectively.

Statistical analysis

The Shapiro–Wilk and Lilliefors tests were used to verify the normality of the data. Depending on data distribution, results were summarized as mean (standard deviation) or median (interquartile range) values. One-way anova and post-hoc Tukey tests were carried out to compare time to failure, angle of rotation, bending resistance, buckling resistance, cutting ability, volume, surface area and untouched canal areas, while Kruskal–Wallis and post-hoc Dunn's tests with Bonferroni correction to adjust for multiple comparisons were used to compare the maximum torque to failure. Significance level was set at 5% (SPSS v25.0 for Windows; SPSS Inc.).

RESULTS

Design

SEM analyses revealed that all instruments had asymmetrical blades with no radial lands. The cross-section designs of EDMax (non-rectangular parallelogram with a slight positive rake angle), Neoniti (parallelogram with a rectangle-like shape) and ProTaper Gold (convex triangular) were symmetrical, while HyFlex EDM was asymmetrical (trapezoidal/irregular convex hexagon). EDMax, Neoniti and ProTaper Gold instruments showed a slightly rounding tip-transition angle, while HyFlex EDM showed a different tip feature, more active. HyFlex EDM, Neoniti and EDMax instruments had regularly distributed craters, a typical isotropic surface observed in materials submitted to the electrical discharge machining process. In contrast, ProTaper Gold displayed a very distinct surface finish with marks resulting from the manufacturing process (grinding). Only small defects, such as barbs in the cutting edge, were observed in all instruments (Figure 2).

Metallurgy

EDS/SEM analyses revealed a nearly equiatomic nickel/titanium ratio in HyFlex EDM (1.062), Neoniti (1.065), EDMax (1.028) and ProTaper Gold (1.001) instruments, without any other traceable metal element. DCS analyses showed distinct transformation temperature curves (Figure 3). HyFlex EDM and Neoniti had comparable results on cooling R-phase to martensite B19′ transformation, which was different from EDMax and ProTaper Gold. HyFlex EDM and Neoniti also showed an almost perfectly overlapped martensitic B19′ and R-phase transformations to austenite-B2 on heating, while the

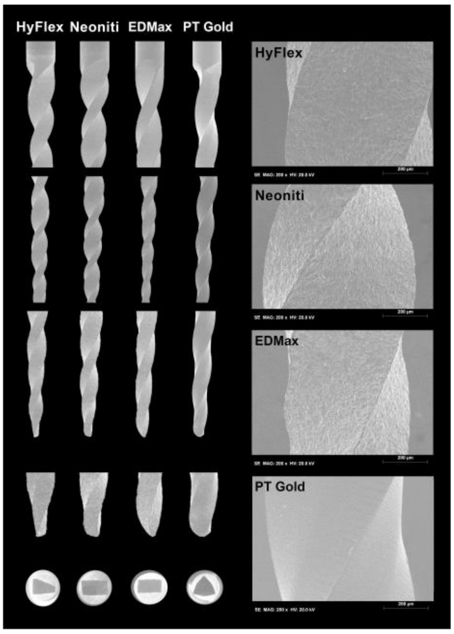

FIGURE 2 SEM analyses of the active blade, cross-section design, tip geometry, and surface finishing of tested instruments. All instruments had asymmetrical blades, no radial lands, and different cross-section designs and tip geometries. HyFlex EDM, Neoniti and EDMax instruments had isotropic surface with regularly distributed crater, while ProTaper Gold had parallel griding marks.

other instruments had a double peak transformation. The cooling R-phase start (Rs) and R-phase finishing (Rf) transformation temperatures were distinct among instruments, ranging from 44.8°C (ProTaper Gold) to 46.7°C (HyFlex EDM), and from 28.7°C (ProTaper Gold) to 35.2°C (HyFlex EDM), respectively. All tested instruments had a crystallographic R-phase arrangement at test temperature (21°C) and a mixed austenite plus R-phase at body temperature. At heating, the lowest and highest Austenite start (As) temperatures were noted with ProTaper Gold (10.1°C) and HyFlex EDM (42.7°C), respectively, while the highest Austenite finish (Af) temperatures were observed in HyFlex EDM (56.8°C) and Neoniti (57.2°C) instruments.

Mechanical performance

Neoniti and HyFlex EDM showed similar results in all mechanical tests ($p > .05$), while EDMax and ProTaper Gold had similar time to fracture ($p = .841$), maximum bending load ($p = 0.729$), and cutting depth ($p = 0.985$). The highest time to fracture and angle of rotation was observed in Neoniti and HyFlex EDM instruments ($p < .001$), while EDMax and ProTaper Gold presented the highest bending load (lower flexibility) and cutting depth ($p < .001$). ProTaper Gold showed the highest torque to failure ($p < .001$) and HyFlex EDM had the lowest buckling resistance ($p < .001$) (Table 1).

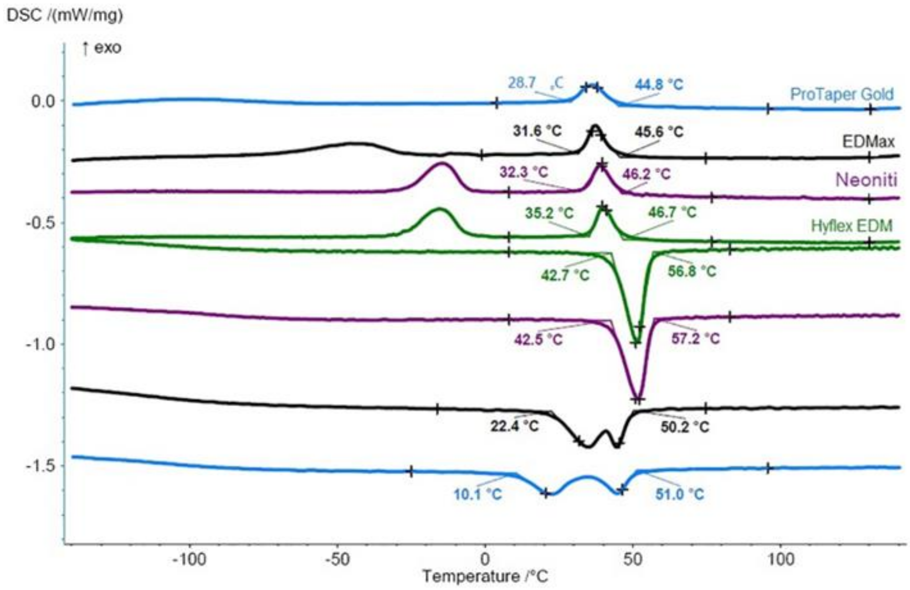

FIGURE 3 DSC charts showing the phase transformation temperatures at cooling on top (reads from right to left) and at heating on bottom (from left to right). The cooling R-phase start (Rs) and R-phase finishing (Rf) transformation temperatures were distinct among instruments. At heating, the lowest and highest Austenite start (As) temperatures were noted with ProTaper Gold and HyFlex EDM, respectively, while the highest Austenite finish (Af) temperatures were observed in HyFlex EDM and Neoniti instruments.

TABLE 1 Mean (standard deviation) or median [interquartile range] results of time to fracture (s), maximum torque (N.cm), angle of rotation (°), maximum load (gf), and cutting depth (mm) parameters obtained after testing four different rotary instruments

	Cyclic fatigue	**Torsional resistance**		**Bending resistance**	**Buckling resistance**	**Cutting ability**
Rotary instruments	**Time to fracture**	**Maximum torque**	**Angle of rotation**	**Maximum load**	**Maximum load**	**Cutting depth**
HyFlex EDM 25/.~	174.2 (±71.3)[a]	1.5 (1.5–1.6)[a, b]	614.5 (±99.3)[a]	217.0 (±20.8)[a]	177.4 (±25.5)[a]	8.83 (±1.19)[a]
Neoniti 25/.06	202.9 (±58.9)[a]	1.5 (1.5–1.6)[a, b]	567.1 (±48.7)[a]	230.4 (±23.0)[a]	202.2 (±19.7)[ab]	9.06 (±0.97)[a]
EDMax 25/.06	84.9 (±11.2)[b]	1.3 (1.1–1.4)[a]	363.4 (±77.3)[c]	307.5 (±24.4)[b]	223.1 (±21.1)[b]	12.14 (±1.29)[b]
ProTaper Gold 25/.08v	102.4 (±16.6)[b]	1.6 (1.6–1.7)[b]	453.5 (±40.7)[b]	319.1 (±30.9)[b]	375.5 (±27.2)[c]	12.33 (±1.44)[b]

Note: Different superscript letters represent statistically significant differences among instruments ($p < 0.05$). Abbreviation: EDM, electric discharge machining.

TABLE 2 Pre- and post-operative parameters evaluated in both mesial and distal root canals of mandibular molars after preparation with four different NiTi systems.

	HyFlex EDM	Neoniti	EDMax	ProTaper g
Parameters	**Mesial canal**			
Volume				
Before (mm^3)	4.3 ± 2.2	5.5 ± 2.8	4.1 ± 1.0	5.4 ± 2.3
After (mm^3)	8.8 ± 3.6	11.8 ± 3.0	9.3 ± 2.7	9.0 ± 3.7
Surface area				
Before (mm^2)	42.5 ± 16.6	53.8 ± 21.2	43.7 ± 9.0	42.3 ± 9.2
After (mm^2)	57.6 ± 18.7	71.0 ± 16.5	59.4 ± 11.8	55.7 ± 16.6
Unprepared area				
After (%)	25.7 ± 10.9	24.1 ± 17.1	24.3 ± 9.2	20.4 ± 13.9
	Distal canal			
Volume				
Before (mm^3)	6.6 ± 4.1	5.7 ± 2.4	8.9 ± 1.9	8.3 ± 1.9
After (mm^3)	8.4 ± 3.8	8.6 ± 2.9	10.3 ± 2.8	9.8 ± 1.9
Surface area				
Before (mm^2)	43.2 ± 14.9	40.6 ± 13.0	51.3 ± 18.0	48.8 ± 7.5
After (mm^2)	47.7 ± 16.7	48.8 ± 15.2	53.1 ± 17.1	49.2 ± 7.2
Unprepared area				
After (%)	26.2 ± 6.7	20.8 ± 6.2	25.2 ± 6.8	25.6 ± 6.9

Abbreviation: EDM, electric discharge machining.

Shaping ability

The homogeneity of groups regarding volume and surface area of mesial and distal canals was confirmed ($p > .05$). Mean percentages of unprepared canal areas ranged from 20.4% to 25.7% in the mesial canals, and from 20.8% to 26.2% in the distal canal, with no statistical differences among tested instruments ($p > .05$) (Table 2, Figure 4).

DISCUSSION

This study presents original results comparing the mechanical behaviour of three rotary instruments produced by the EDM process with the well-known ProTaper Gold system regarding cyclic fatigue, cutting ability, torsional, bending and buckling resistance. A comprehensive understanding of the results, however, was only possible because of the further evaluation of their

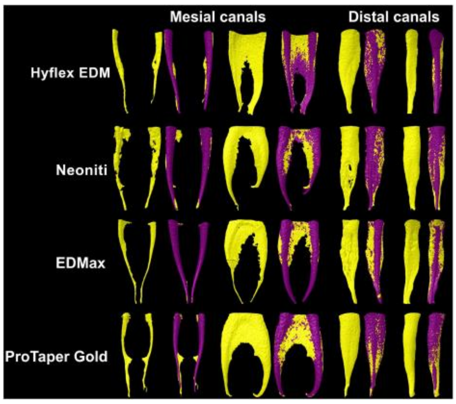

FIGURE 4 Micro-CT analyses. Representative 3D models of 8 mesial and 8 distal root canal systems of mandibular molars before (yellow colour) and after (purple colour) preparation with HyFlex EDM, Neoniti, EDMax, and ProTaper Gold rotary canal systems. In mesial and distal canals, the mean percentage of unprepared canal walls ranged from 20.4% to 25.7% and from 20.8% to 26.2%, respectively.

overall designs, surface finishings and metal alloy crystallographic arrangements, performed in accordance with international guidelines (ASTM International, 2004; ISO 3630-3631, 2008) or well-established and validated methodologies (Lopes et al., 2012; Plotino et al., 2014; Versiani et al., 2018). This multimethod approach avoids the phenomenon of 'knowledge compartmentalization' while provides comprehensive knowledge of each method by taking advantage of their strength and minimizing their weaknesses in order to improve the internal validation of the research (Hunter & Brewer, 2015). In this study, Neoniti and HyFlex EDM showed similar results in all mechanical tests (Table 1), corroborating a previous study (Silva et al., 2020) in which these two instruments were also compared regarding cyclic fatigue and torsional resistance. These findings might be explained considering that, although they might present small differences in their cross-sectional designs, these instruments have comparable dimensions (tip and taper), quality of manufacturing (Figure 2), and metallurgical properties, as demonstrated by EDS and DSC assays (Figure 3). On the other hand, results revealed differences in their mechanical properties compared to EDMax and ProTaper Gold instruments (Table 1), and the null hypothesis was rejected. It may be assumed that alloy composition had no impact on the mechanical performance of instruments, considering that all systems were made with similar amounts of nickel and titanium elements, without traces of other metals. On the other hand, the combination of the overall geometry, evaluated by stereomicroscopy and SEM, and the alloy crystallographic arrangement, determined by the DSC phase transformation temperature analysis, can partially explain almost all mechanical findings.

During the preparation of curved canals, NiTi instruments are submitted to consecutive cycles of tension and compression that can reduce their life cycle by creating surface microcracks that may propagate, a phenomenon that can be simulated by the cyclic fatigue test, a method that uses a well-defined set of experimental conditions. In these same clinical conditions, the flexibility, assessed by the bending resistance test, is also important as it allows instruments to keep the original canal pathway while performing its enlargement. In addition, during root canal preparation, sometimes it is necessary to apply a light pressure along the axis of the instrument to allow its progression toward the apex. This property is assessed by the buckling test, a method developed to evaluate the ability of an instrument to sustain a compressive load in the direction of its own axis (Martins, Martins, et al., 2022). In this study, cyclic fatigue and flexibility of HyFlex EDM and Neoniti were similar, but higher than ProTaper Gold and EDMax (Table 1). The main variables that affect the results of these tests are the metallurgical properties and the size of instruments (Martins, Martins, et al., 2022) and, therefore, the results of cyclic fatigue and bending resistance tests can be explained by the small dimensions of Neoniti and HyFlex EDM (25/.06), the lowest Austenite start temperature of the ProTaper Gold (10.1°C) (Figure 3), and by the EDM process, which usually produces high flexible instruments (Pedullá et al., 2016; Pirani et al., 2016), corroborating the results of previous studies (Kaval et al., 2016; Silva et al., 2020). On the other hand, although EDMax was also produced by the EDM method and had similar dimensions (Figure 1), it showed less time to fracture and flexibility than Neoniti and HyFlex EDM (Table 1), a finding that can be explained by differences in their thermal treatments (Figure 3). The new thermal treatment applied to the EDMax changed its transformation temperature curve by reducing its Austenite start temperature to 22.4°C when compared to Neoniti (42.5°C) and HyFlex EDM (42.7°C) (Figure 3), indicating that its alloy converts to austenite closed to the test temperature (21°C), makes it less flexible and relatively stiffer than Neoniti and HyFlex EDM. These differences in the heating curve (Figure 3) also help to explain the higher buckling resistance of EDMax compared to Neoniti and HyFlex EDM, while the largest dimensions of ProTaper Gold justifies its highest buckling resistance values (Table 1).

One of the main objectives of NiTi endodontic instruments is the removal of infected dentine during root canal shaping procedures (Martins, Martins, et al., 2022). Thus, during the progression of the instru- ment to apical direction, its cutting efficiency, a property related to the capacity of an instrument to advance into the root canal and to provide lateral cut, is of utmost importance. This property depends on some features including metallurgy, surface treatment, cross-sectional design, sharpness of flute and tip design. In this study, however, the lowest cutting depth of Neoniti and HyFlex EDM (Table 1) can be explained by their phase transformation temperatures. Because of their high Austenitic start temperatures (Figure 3), these instruments present more martensitic behaviour than EDMax and ProTaper Gold. When the instrument is in its martensite form, it is soft and ductile and can easily be deformed, which may affect its efficiency for cutting (Arias & Peters, 2022), as observed herein.

The torsional strength is determined by the maximum torque before fracture, a characteristic that is relevant to prepare narrow or constricted root canals, and the angle of rotation, related to the capacity to sustain deformation before fracture under a torsional stress (Martins, Martins, et al., 2022). This property is highly relevant during the mechanical action of cutting dentine, as it is the main mechanism that may lead instruments to fracture (Sattapan et al., 2000). This mechanical property may be affected by several factors including the thermomechanical process applied during manufacturing, the cross-sectional design, the alloy composition and the dimension of the instrument (Martins et al., 2021; Martins, Martins, et al., 2022). In this test, however, the apical 3 mm of the instrument is locked in a chuck and rotated at a constant pace until fracture (ISO 3630-3631, 2008), a methodological aspect that may partially explain the present results. In general, large-sized instruments at this specific level tend to sustain higher torque (Martins, Martins, et al., 2022), which justifies the highest maximum torque observed in the ProTaper Gold instrument (Table 1). Likewise, similarities of HyFlex, Neoniti and EDMax instruments in terms not only on their manufacturing process (EDM) but also in their size and surface finishing at this level, were the main reasons to explain their comparable results. The angle of rotation represents the maximum rotation that an instrument would be able to support before its fracture by torsion. Higher values on this parameter are usually observed in large instruments, since they tend to sustain high torque, as well as, in heat-treated instruments because of the increased deformation ability resulting from their high ductility and flexibility (Ninan & Berzins, 2013). In this study, highest angles of rotation were observed in HyFlex and Neoniti instruments possibly because of their higher flexibility as a result of their distinct transformation-temperature curves (Figure 3).

Although many studies rely on mechanical parameters to assess the performance of rotary NiTi systems, a more comprehensive understanding should also include the assessment of their effectiveness in preparing the root canal system. Therefore, a multimethod approach combining the results of different mechanical tests and the shaping ability of different NiTi systems would be of benefit for a better interpretation of their performance and, consequently, a more precise translation of preclinical findings to guide the clinical use (Silva et al., 2020). In this study, tested systems were compared regarding the percentage of untouched canal walls left after the preparation of mesial and distal root canals of mandibular molars and evaluated using the gold standard micro-CT technology. This parameter has a high clinical relevance since untouched canal areas may harbour residual bacteria and serve as a potential cause of persistent infection, which ultimately may lead to post-treatment disease (Arias & Peters, 2022; Bürklein & Arias, 2022). In fact, the present findings are an ever-present condition in clinical practice since no preparation system was able to touch all root canal walls (Gagliardi et al., 2015; Martins et al., 2021; Versiani et al., 2013, 2018). As a consequence of this suboptimal performance, it is important to emphasize that current canal shaping protocols are still largely dependent on the action of irrigation procedures for intracanal disinfection.

Preliminary efforts were made to ensure comparability of the groups by anatomically matching the specimens based on morphometric parameters of the root canal system including configuration, volume and surface area. This process reduces the anatomical bias that usually confound the outcomes in this type of study and creates a reliable baseline, enhancing the internal validity of the study (Versiani et al., 2013). While the metallurgical and design dissimilarities of tested instruments were clearly reflected in the results of the mechanical tests, micro-CT evaluation revealed no difference among systems in the percentage of unprepared areas (Figure 4), with values ranging from 20.4% to 25.7% in the mesial canals and from 20.8% to 26.2% in the distal canal (Table 2). These findings corroborate previous micro-CT studies (Gagliardi et al., 2015; Martins et al., 2021; Silva et al., 2020; Stringheta et al., 2019; Versiani et al., 2018) and might be attributed to the previous balancing of groups regarding their internal morphology and the inherent anatomical complexity of the root canal system of mandibular molars (Martins et al., 2021). The analysis of centring ability (canal transportation/centroid shift) was not performed in the present study because the selection criteria included only root canals with moderate curvature and tested instruments had a very high flexibility, demonstrated by their transformation temperature curves (Figure 3) and bending load results (Table 1). In these conditions, it is unlikely that clinical relevant changes in the original canal curvature could be noticed, as previously reported (Gagliardi et al., 2015; Silva et al., 2023; Silva, Lima, et al., 2022;

Silva, Martins, et al., 2022). Therefore, future studies are suggested to compare the ability of instruments produced by EDM technology to prepare severely curved canals.

The main strength of this study was the use of a multimethod research approach that allowed the interconnection of the results and a better understanding of the influence of each factor on the overall performance of tested systems, which highlight the importance of assessing several variables and not relying on one single aspect of the instrument behaviour. As limitations, the real dimensions of instruments were not assessed and other tests, such as microhardness and electron probe x-ray microanalysis (EPMA), could have been also used in this multimethod protocol, which are recommended to be included in further studies.

CONCLUSIONS

This multimethod research allowed to obtain noteworthy information of the main set instruments of three rotary systems produced by electric discharge machine technology through different perspectives in order to compare their mechanical performance and shaping efficiency with the well-known heat-treated ProTaper Gold system. Overall, it was observed that instruments' geometry and phase transformation temperatures influenced the results of the mechanical tests, but not their shaping ability.

AUTHOR CONTRIBUTIONS

EJNLS: conceptualization, analysis, writing, review and editing (lead); NCA: sample selection, experimental procedures, micro-CT scans, writing, review and editing (lead); JNRM: conceptualization, writing, analysis, review and editing (lead); BRA: experimental procedures; COL: experimental procedures, micro-CT scans, review and editing (supporting); VTLV: review and editing (supporting); FMB-F: experimental procedures; MAV: conceptualization, writing, analysis, review and editing (lead).

ACKNOWLEDGEMENTS

The authors deny any conflicts of interest related to this study. This study was partially funded by CNPq and FAPERJ. FMBF acknowledges the funding of CENIMAT/i3N by national funds through the FCT-Fundação para a Ciência e a Tecnologia, I.P., within the scope of Multiannual Financing of R&D Units, reference UIDB/50025/2020-2023. Fernanda Carvalho is acknowledged for running the DSC tests on the instruments.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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How to cite this article: Silva, E.J.N.L., Ajuz, N.C., Martins, J.N.R., Antunes, B.R., Lima, C.O., Vieira, V.T.L. et al. (2023) Multimethod analysis of three rotary instruments produced by electric discharge machining technology. *International Endodontic Journal*, 56, 775–785. Available from: https://doi.org/10.1111/iej.13915

4.4. CAPÍTULO 4:

CHARACTERIZATION OF THE FILE-SPECIFIC HEAT-TREATED PROTAPER ULTIMATE ROTARY SYSTEM

Artigo publicado na revista *International Endodontic Journal*

doi: 10.1111/iej.13880

Classificação de periódicos quadriênio 2017-2020 – Odontologia – A1

Fator de impacto 5.165

Received: 2 August 2022 | Accepted: 9 December 2022
DOI: 10.1111/iej.13880

ORIGINAL ARTICLE

INTERNATIONAL ENDODONTIC JOURNAL | WILEY

Characterization of the file-specific heat-treated ProTaper Ultimate rotary system

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Funding information
Fundação para a Ciência e a

Abstract

Aim: To compare design, metallurgy and mechanical performance of the ProTaper (PT) Ultimate system with instruments of similar dimensions from the ProGlider, PT Gold and PT Universal systems.

Methodology: New PT Ultimate instruments (n = 248) were compared with instruments of similar dimensions from ProGlider (n = 31), PT Gold (n = 155) and PT Universal (n = 155) systems regarding their number of spirals, helical angle, blade symmetry, tip geometry, surface finishing, nickel/titanium ratio, phase transformation temperatures and mechanical performance. One-way anova and nonparametric Mood's median tests were used for statistical comparison (α = 5%).

Results: All instruments had symmetrical blades without radial lands or flat sides, similar surface finishing and an almost equiatomic nickel/titanium ratio, whilst the number of spirals, helical angles and the tip geometry were different. PT Ultimate instruments showed 3 distinct heat treatments that matched with the colour of their metal wire. Slider and ProGlider instruments had similar R-phase start (Rs) and R-phase finish (Rf) temperatures. SX, F1, F2, F3 and Shaper instruments showed equivalent heat treatments (Rs ~45.6°C and Rf ~28.3°C) that were similar to their PT Gold counterparts (Rs ~47.9°C and Rf ~28.2°C), but completely distinct to the PT Universal ones (Rs ~16.2°C and Rf ~−18.2°C). Amongst the PT Ultimate instruments, the lowest maximum torques were observed in the SX (0.44 N cm), Slider (0.45 N cm) and Shaper (0.60 N cm) instruments, whilst the highest was noted in the FXL (4.90 N cm). PT Ultimate Slider and ProGlider had similar torsional (~0.40 N cm) and bending loads (~145.0 gf) (p = 1.000), whilst the other PT Ultimate instruments showed statistically significantly lower maximum torque, higher angle of rotation and lower bending load (higher flexibility) than their counterparts of the PT Universal and PT Gold systems.

Conclusions: The PT Ultimate system comprises instruments with 3 distinct heat treatments that showed similar phase transformation temperatures to their heat-treated analogues. PT Ultimate instruments presented lower torsional strength and

Tecnologia, Grant/Award Number: UIDB/50025/2020-2023

superior flexibility than their counterparts, whilst maximum torque, angle of rotation and bending loads progressively increased with their sizes.

KEYWORDS

bending load, differential scanning calorimetry, endodontics, nickel-titanium alloy, scanning electron microscope, torsional strength

INTRODUCTION

Nickel-titanium (NiTi) instruments have been widely used to perform the mechanical enlargement of the root canal system. Over several years, successive improvements have been introduced in these instruments including different heat treatments employed during the manufacturing process (Rubio et al., 2022; Zupanc et al., 2018). These changes may lead to distinct crystallographic arrangements of the NiTi alloy at specific temperatures, ultimately influencing the mechanical behaviour of these instruments (Martins et al., 2022).

A few examples of heat-treated alloys are the M-wire (Dentsply Tulsa Dental), which incorporates a heat treatment before the alloy production, and the Gold and Blue heat-treated wires (Dentsply Tulsa Dental) which receive a post-grinding heat treatment (Zupanc et al., 2018). According to Gao et al. (2012), different mechanical behaviours are expected when addressing instruments of similar dimensions manufactured from austenitic NiTi, M-wire or Blue heat-treated alloys. In such cases, M-wire instruments tend to have higher maximum torques, whilst Blue heat-treated wires present lower bending resistance (high flexibility) and higher cyclic fatigue strength and degree of rotation under twist stress (De-Deus et al., 2017; Duke et al., 2015). Likewise, Gold heat-treated instruments usually present superior cyclic fatigue strength and flexibility, but lower torsional strength when compared with conventional (austenitic) NiTi alloy instruments of similar dimensions (Elnaghy & Elsaka, 2016; Plotino et al., 2017). These improvements can be considered relevant in a clinical setup as they may extend the lifespan of instruments, whilst simultaneously preserve the original pathway of the main root canal (Zupanc et al., 2018). Besides, the development of instruments with different features gives to the clinicians the opportunity to choose the most appropriate to a specific root or canal morphology.

Rotary NiTi instruments from the ProTaper (PT) family are probably the most well-known and long-lasting available systems currently on the market. In 2001, when the first generation of this system was launched, instruments were made of conventional NiTi alloy with an innovative design utilizing multiple increasing or decreasing percentage tapers on a single file (Ruddle, 2005). This system originally comprised 3 shaping (SX [19/.04v], S1 [18/.02v] and S2 [20/.04v]) and 3 finishing (F1 [20/.07v], F2 [25/.08v] and F3 [30/.09v]) instruments with sharp cutting edges and no radial lands. Later, 2 larger finishing instruments (F4 [40/.06v] and F5 [50/.05v]) were added to this set and the system changed its name to PT Universal (Dentsply Maillefer). The next generation was launched in 2013, the PT Next (Dentsply Sirona Endodontics), and comprised 5 instruments (sizes 17/.04v, 25/.06v, 30/.07v, 40/.06v and 50/.06v) manufactured in M-wire and designed to have an offset design to improve flexibility and minimize the engagement between the instrument and dentine (Ruddle et al., 2013). Taking advantage of technological advancements in metallurgy, the PT Universal system evolved to PT Gold (Dentsply Sirona Endodontics) in 2014, a system in which instruments have the same geometries, but the alloy is thermomechanically treated (Gold Wire), resulting in an improved flexibility and resistance to cyclic fatigue (Elnaghy & Elsaka, 2016). In this same year, the ProGlider (16/.02v) (Dentsply Sirona Endodontics), an auxiliary rotary instrument that utilizes M-Wire technology, was also introduced for mechanical glidepath preparation (Ruddle et al., 2014).

The novel PT Ultimate rotary system (Dentsply Sirona Endodontics) is the latest generation of the PT family and is one of the first systems to take advantage of distinct crystallographic arrangements induced by specific heat treatment technology to produce a set of instruments with different mechanical behaviours, aiming to ensure a balance between flexibility and strength. According to the manufacturer, the 8 instruments that comprise this system (Slider [16/.02v], SX [20/.03v], Shaper [20/.04v], F1 [20/.07v], F2 [25/.08v], F3 [30/.09v], FX [35/.12v] and FXL [50/.10v]) are manufactured using 3 different heat-treated alloys: M-wire (Slider), Gold-wire (SX, Shaper, F1, F2, F3) and Blue heat-treated wire (FX and FXL) (Dentsply Sirona, 2022). Considering the lack of knowledge regard- ing this system, a multimethod research approach was conducted to compare the design, metallurgical charac- teristics and mechanical performance of the PT Ultimate system with instruments of similar sizes from ProGlider, PT Gold and PT Universal systems. The null hypothesis to be tested was that there would be no difference in the me- chanical behaviour amongst these different instruments.

MATERIAL AND METHODS

Sample selection

A total of 248 new randomly selected NiTi rotary instruments from the novel PT Ultimate (31 instruments of each size – Slider, SX, Shaper, F1, F2, F3, FX, FXL – distributed among design, metallurgical and mechanical assessments) were compared regarding their design, metallurgical characteristics and mechanical behaviour to similar instruments of the ProGlider (n = 31), PT Gold (n = 155; 31 instruments of each size – SX, S2, F1, F2, F3) and PT Universal (n = 155; 31 instruments of each size – SX, S2, F1, F2, F3) systems after being previously checked for major deformations (such as unwinding or major blade discontinuity) that would exclude them from the study. All instruments were 25 mm long, except the SX (19 mm). No major deformation was observed under operative microscope (×13.6) (OPMI Pico; Carl Zeiss Surgical) in any instrument and therefore none of them was excluded.

Design

The microscopic assessment of the design was conducted at ×13.6 magnification (OPMI Pico) in 6 instruments of each size from all tested systems in which the number of blades and the mean helical angles from the 6 most coronal spirals were determined (Image J v1.50e; Laboratory for Optical and Computational Instrumentation). These same instruments were additionally evaluated by scanning electron microscopy (SEM) (Hitachi S-2400; Hitachi) to investigate the symmetry of the blades, the presence of radial lands or flat sides (×20), and the design and type (active or nonactive) of the tips (×40). The surface finishing was also evaluated (×150) regarding the existence of microdefects, such as metal rollovers or spiral discontinuities.

Metallurgy

Energy-dispersive X-ray spectroscopy (EDS) was conducted in 3 instruments of each tested system on a conventional SEM unit (DSM-962 Carl Zeiss Microscopy GmbH) equipped with an Inca X-act EDS detector (Oxford Instruments NanoAnalysis) and set at 20 kV and 3.1 amperes. The initial vacuum was conducted for 10 min, and data acquisition was accomplished in an area of 500 × 400 µm for 1 min at a working distance of 25 mm. Analyses used the ZAF correction and the proportions of metal elements were obtained in a dedicated software (Microanalysis Suite v.4.14 software; Oxford Instruments NanoAnalysis). Differential scanning calorimetry (DSC) tests (DSC 204 F1 Phoenix; NETZSCH-Gerätebau GmbH) were also conducted to determine the phase transformation temperatures (ASTM F2004-17, 2004) using 2 instruments of each size from all tested systems. A fragment of 4–5 mm in length (weighting 5–10 mg) was obtained from the active blade of each instrument and submitted to an etching bath (45% of nitric acid, 25% hydrofluoric acid and 30% of distilled water) for 2 min. After that, the acid solution was neutralized with distilled water and each specimen was mounted on an aluminium pan inside the DSC device, having an empty pan as a control. Each individual thermal cycle had 1 h 40 min duration and ran under gaseous nitrogen (N_2) protection. The cycle temperatures ranged from −150°C to 150°C with a pace of 10°C per minute. The DSC results and charts were obtained using the NETZSCH Proteus Thermal Analysis software (NETZSCH -Gerätebau GmbH). A second test was conducted to confirm the results of the first test.

Mechanical tests

The mechanical behaviour of instruments was evaluated by testing their torsional and bending resistances according to international specifications (ANSI/ADA Specification No. 28, 2002; ISO 3630-3631, 2008). Sample size calculations for the mechanical tests were determined taking into account the highest differences in the results obtained by 2 of the assessed instruments from the PT Ultimate system after 6 initial measurements. Considering an alpha-type error of 0.05 and a power of 80%, the determined sample sizes for maximum torque (effect size: 4.45 ± 2.38; Slider vs. FXL), angle of rotation (effect size: 279.88 ± 162.04; Shaper vs. FXL) and maximum bending load (effect size: 245.42 ± 129.27; Shaper vs. FX) were 6, 7 and 6 instruments, respectively. The final sample size for each test was set as 10 instruments for all groups.

In the torsional test, instruments were mounted in a straight position on a torsiometer (TT100; Odeme Dental Research) and clamped at their apical 3 mm. Then, they were rotated on a constant pace of 2 rpm in a clockwise direction until fracture. The maximum torque sustained prior to rupture (in N cm) and the angle of rotation (in degrees) were assessed with a dedicated software (Odeme Analysis TT100, Odeme Dental Research). In the bending test, instruments were mounted in the file holder and positioned at 45° in relation to the floor, whilst their apical 3 mm were attached to a wire connected to a universal testing machine (DL-200 MF; EMIC). The test was conducted using a 20 N load applied at a 15 mm/min constant pace until the instrument accomplished a 45° displacement. The maximum load required to induce this displacement was recorded in gram/force (gf) using the Tesc v3.04 software (Mattest Automação e Informática).

Statistical analysis and reporting

Data normality was assessed using the Shapiro-Wilk test and presented as mean (standard deviation) or median (interquartile range) depending on their distribution. One-way anova *post hoc* Tukey tests were used to assess differences in the mean helical angles, whilst the nonparametric Mood's median test was employed to compare maximum torque, angle of rotation and maximum bending load amongst instruments (SPSS v22.0 for Windows; SPSS Inc.). The level of significance was set at 5%. The present manuscript was written according to Preferred Reporting Items for Laboratory studies in Endodontology (PRILE) 2021 guidelines (Figure 1) (Nagendrababu et al., 2021).

RESULTS

Design

Table 1 summarizes the analyses of design, whilst Figure 2 shows the SEM images of the assessed instruments. All tested files had symmetrical blades without radial lands or flat sides.

PT Ultimate Slider was similar to ProGlider in terms of tip size, surface finishing and helical angle, but had a shorter active area with a smaller number of blades and a parallelogram cross-section, whilst the ProGlider had a square horizontal cross-section. The number of blades of the PT Ultimate Shaper and Finishers (F1, F2 and F3) decreased (from 18 to 12), as the diameter in- creased, and was higher than their counterparts, whose spirals also decreased from 11 (S2) to 9 (F3). Overall, he- lical angles were similar amongst instruments, however PT Ultimate F1 and F2 showed significant lower angles than their equivalent PT Universal and PT Gold instruments (Table 1). PT Ultimate Shaper and Finishers had an off-centred parallelogram cross-section, whilst all analogue instruments had a convex cross-sectional triangular shape, except for the F3 instruments that had a concave triangular cross-section. PT Ultimate FX and FXL had the smallest number of blades and helical angles amongst tested systems, but similar cross-sections to the other PT Ultimate instruments. The tips of the PT Ultimate Shaper and Finishers were similar, but different from the Slider, FX and FXL, whilst in the other systems, the geometry of the tips was distinct from each other. None of the tips could be clearly identified as active.

Visual and microscopic analyses of all instruments revealed no major deformations or defects. In general, surface finishing was similar with manufacturing parallel marks in all instruments and only very few micro defects.

Metallurgy

Energy-dispersive X-ray spectroscopy tests showed an almost equiatomic nickel/titanium elements ratio in all instruments with no other metal element. DSC analyses of the 8 instruments of the PT Ultimate system revealed 3 distinct heat treatments that matched with the colour of their metal alloy (Figure 3). The Slider and the ProGlider instruments had similar R-phase start (Rs) and R-phase finish (Rf) temperatures. SX, F1, F2, F3 and Shaper instruments showed equivalent heat treatments (Rs ~45.6°C and Rf ~28.3°C) that were similar to their PT Gold counterparts (Rs ~47.9°C and Rf ~28.2°C), but completely distinct to the PT Universal ones (Rs ~16.2°C and Rf ~−18.2°C). PT Ultimate FX and FXL instruments showed similar DSC curves with phase transformation temperatures ranging from 29.4°C (Rs) and 19.8°C (Rf) at cooling, and 7.7°C (austenitic start [As]) and 36.4°C (austenitic finish [Af]) at heating (Table 2, Figure 3).

Mechanical tests

Amongst the PT Ultimate instruments, the lowest maximum torques were observed in the SX (0.44 N cm), Slider (0.45 N cm) and Shaper (0.60 N cm) instruments, whilst the highest was noted in the FXL (4.90 N cm) (Table 1). The lowest and highest angles of rotation were observed in the Shaper (418°) and FXL (712°) instruments, respectively. Although the bending test revealed an overall tendency of instruments to become less flexible as they increased in size, the largest instrument of this system (FXL) showed a maximum load significantly lower (294.4 gf) than the FX instrument (410.9 gf), which was the least flexible amongst the 25-mm instruments (Table 1, Figure 4). PT Ultimate Slider and ProGlider had similar torsional (p = 1.000) and bending load (p = 1.000) results, whilst, in general, the other PT Ultimate instruments showed statistically significantly lower maximum torque, higher angle of rotation and lower bending load (higher flexibility) than their counterparts of the PT Universal and PT Gold systems (Table 1, Figure 4).

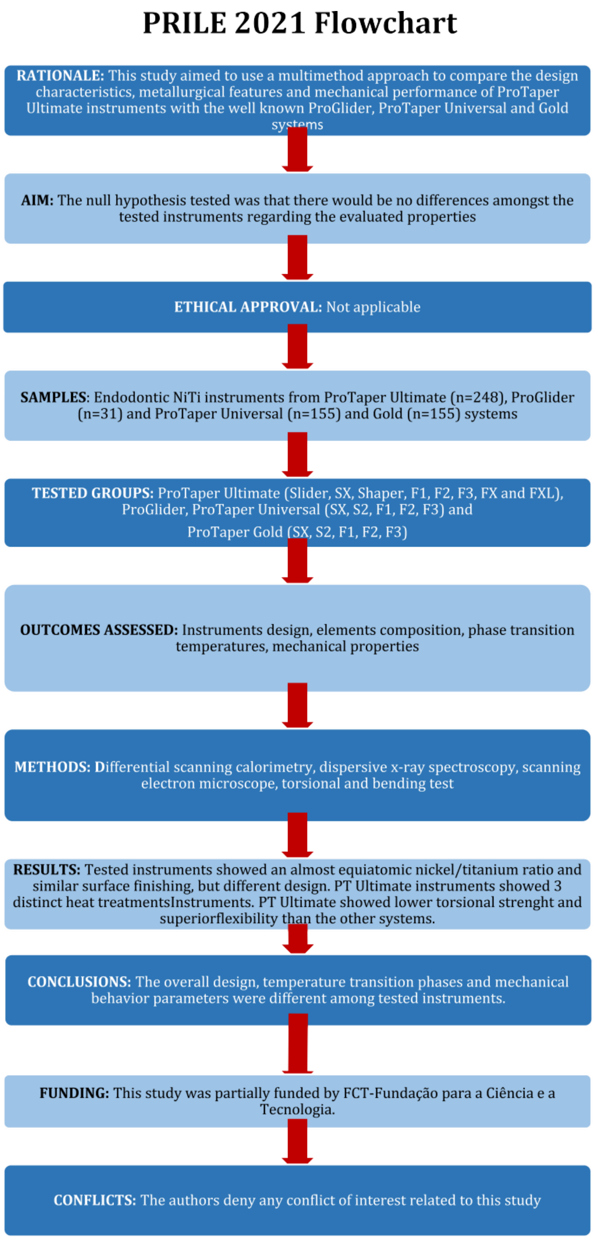


FIGURE 1 PRILE (2021) flowchart (Nagendrababu et al., 2021).

DISCUSSION

This study presents original data regarding the recently launched PT Ultimate file-specific heat-treated rotary system using the multimethod research concept, an approach that provides more information, better understanding and superior internal and external validation than a single or double method assessment (Martins et al., 2021c). Overall, the concept of the PT Ultimate system seems to combine several features from previous instruments developed by the same company including the variable taper (ProTaper), the so-called 'Deep Shape' concept or

TABLE 1 Design characteristics and mechanical test results of ProTaper (PT) Ultimate, Gold, Universal and ProGlider instruments expressed as mean (standard deviation) or median [interquartile range]

	Design		**Torsional test**		**Bending test**
Instruments (tip size/taper, wire)	**Number of blades**	**Helical angle (°)**	**Maximum torque (N cm)**	**Angle of rotation (°)**	**Maximum load (gf)**
PT Ultimate Slider (16/.02v, M-Wire)	19	21.4 (±1.9)	0.45 [0.38–0.50]	435.4 [404.9–478.7]	149.7 [139.9–176.9]
ProGlider (16/.02v, M-Wire)	21	21.2 (±0.8)	0.40 [0.30–0.50]	429.0 [419.3–463.0]	144.8 [136.9–149.6]
PT Ultimate SX (20/.03v, Gold)	12	16.9 (±1.7)	0.44 [0.38–0.50][a]	422.1 [343.3–507.0]	538.3 [511.9–570.4][a]
PT Gold SX (19/.04v, Gold)	9	19.9 (±3.3)	0.55 [0.48–0.63][b]	387.6 [330.0–455.5]	490.1 [471.5–504.9][b]
PT Universal SX (19/.04v, Austenitic)	9	20.6 (±3.0)	0.31 [0.20–0.40][a]	405.3 [384.8–435.8]	876.0 [833.3–926.9][c]
PT Ultimate Shaper (20/.04v, Gold)	18	21.9 (±2.9)	0.60 [0.48–0.73][a]	408.9 [364.2–485.3][a]	229.1 [208.9–236.4][a]
PT Gold S2 (20/.04v, Gold)	11	22.1 (±1.2)	1.00 [0.88–1.20][b]	506.5 [471.0–542.8][b]	255.1 [241.6–260.7][b]
PT Universal S2 (20/.04v, Austenitic)	11	22.4 (±2.5)	0.70 [0.58–0.80][a]	408.0 [362.8–455.3][a]	546.9 [524.5–574.6][c]
PT Ultimate F1 (20/.07v, Gold)	16	18.4 (±2.9)[a]	1.05 [1.00–1.20][a]	478.9 [417.2–584.9][a]	200.9 [188.7–210.7][a]
PT Gold F1 (20/.07v, Gold)	12	25.3 (±1.1)[b]	1.30 [1.20–1.40][b]	486.0 [447.5–534.8][a]	261.0 [237.7–274.5][b]
PT Universal F1 (20/.07v, Austenitic)	12	25.6 (±0.8)[b]	1.30 [1.08–1.40][a,b]	359.5 [304.3–390.8][b]	393.3 [389.7–406.8][c]
PT Ultimate F2 (25/.08v, Gold)	16	19.1 (±1.5)[a]	1.40 [1.30–1.40][a]	489.1 [449.9–618.2][a]	204.1 [192.8–219.5][a]
PT Gold F2 (25/.08v, Gold)	10	21.9 (±1.3)[b]	1.50 [1.40–1.53][b]	414.0 [390.0–452.3][b]	249.4 [241.2–251.1][b]
PT Universal F2 (25/.08v, Austenitic)	10	22.4 (±1.4)[b]	1.80 [1.58–1.83][c]	310.0 [289.0–329.0][c]	494.1 [485.6–509.3][c]
PT Ultimate F3 (30/.09v, Gold)	12	18.7 (±2.2)	1.45 [1.28–2.13][a]	632.1 [457.6–791.6][a]	254.9 [240.2–272.3][a]
PT Gold F3 (30/.09v, Gold)	9	20.7 (±3.8)	1.70 [1.60–1.85][a,b]	639.0 [618.0–711.8][a]	279.7 [269.5–304.4][a]
PT Universal F3 (30/.09v, Austenitic)	9	20.9 (±4.6)	2.10 [1.80–2.50][b]	469.5 [438.0–481.0][b]	681.7 [661.5–698.1][b]
PT Ultimate FX (35/.12v, Blue)	8	18.1 (±3.4)	3.35 [1.38–3.83]	659.9 [486.3–746.1]	416.1 [397.4–428.8]
PT Ultimate FXL (50/.10v, Blue)	5*	19.0 (±2.0)	4.90 [4.53–5.23]	712.3 [645.1–807.4]	294.4 [290.8–301.4]

Note: Different letters in the same column related to the same group of analogue instruments mean statistically significant differences ($p < .05$).

*Only 5 blades were measured instead of 6.

increased apical taper (ProTaper), the off-centred parallelogram cross-section (PT Next, TruNatomy), the large-tapered FXL auxiliary instrument (ProFile GT) and the use of M-Wire (ProGlider, PT Next), Gold wire (PT Gold, WaveOne Gold) and Blue wire (Vortex Blue, Reciproc Blue) heat-treated metal alloys. Amongst the PT Ultimate instruments, it was observed that the maximum torque sustainable prior to fracture and maximum bending loads increased with instruments' size (Table 1, Figure 4), an expected result considering previous studies on multi-file systems reporting higher torques and less flexibility in larger instruments (Kramkowski & Bahcall, 2009; Ninan & Berzins, 2013; Pedulla et al., 2018; Viana et al., 2010; Wycoff & Berzins, 2012). In contrast, no pattern could be demonstrated in the angle of rotation according to instruments' size, but mixed results in this mechanical parameter have been also reported by several authors (Kramkowski & Bahcall, 2009; Ninan & Berzins, 2013; Pedulla et al., 2018; Wycoff & Berzins, 2012). The different crystallographic arrangements of PT Ultimate instruments, however, did not seem to influence their mechanical behaviour since these results could be mostly explained by differences in the dimensions of instruments. An exception was observed in the largest instrument of the PT Ultimate system, the FXL (50/.10v), which was more flexible than the FX (35/.12v), an instrument made with the same heat treatment alloy, but with small dimensions (Table 1, Figure 3). This apparent contradictory result may be explained considering that the active part of the FXL has only 7 mm in length, and, therefore, the result of the bending test reflected the cross-sectional diameter of its nonactive portion, which is smaller (1 mm) than the FX instrument (1.2 mm at D16).

The mechanical performance of the tested instruments can be partially explained by the dissimilarities observed

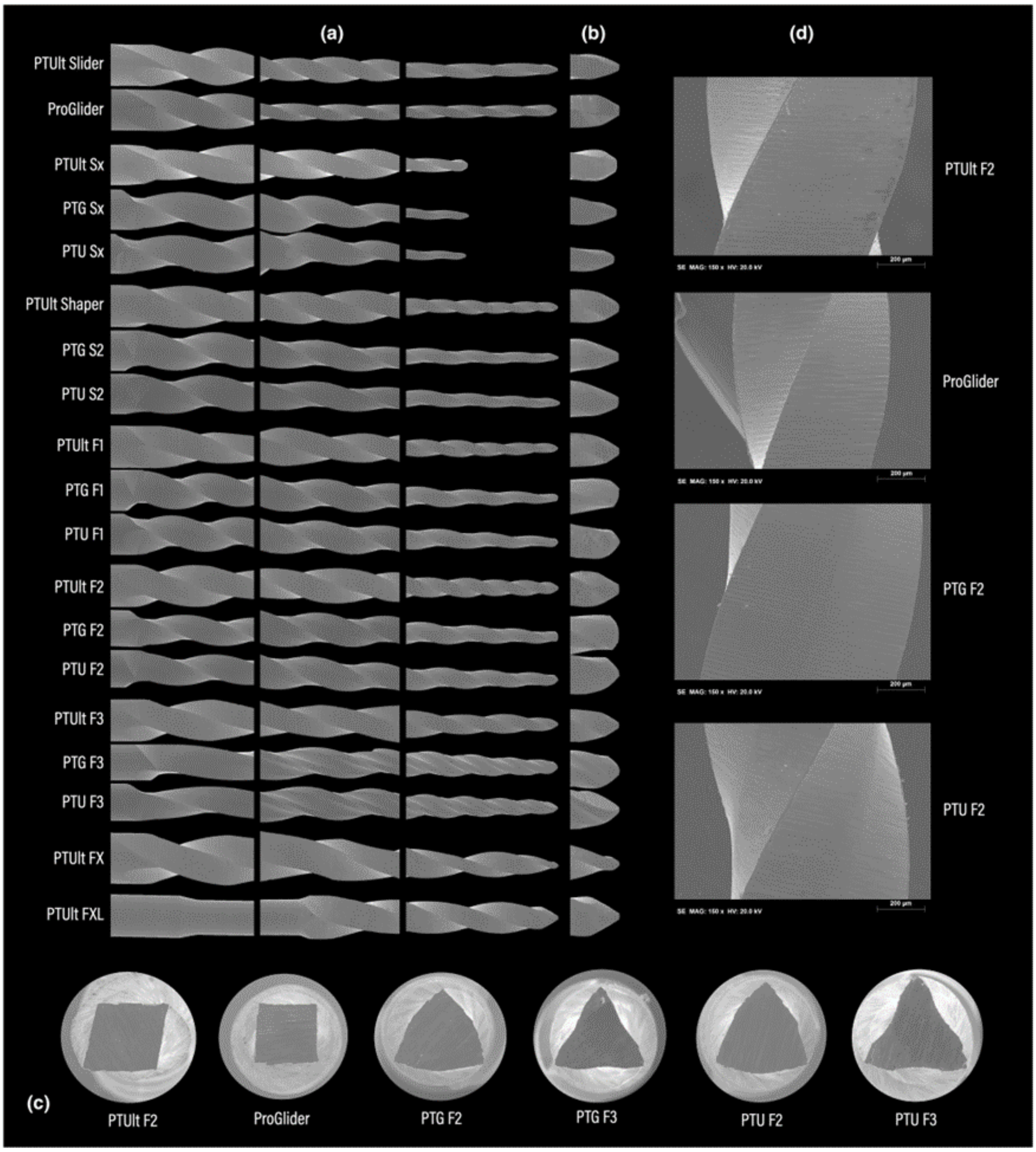

(a)
(b)
(d)
PTUlt Slider
ProGlider
PTUlt Sx
PTG Sx
PTU Sx
PTUlt Shaper
PTG S2
PTU S2
PTUlt F1
PTG F1
PTU F1
PTUlt F2
PTG F2
PTU F2
PTUlt F3
PTG F3
PTU F3
PTUlt FX
PTUlt FXL
PTUlt F2
ProGlider
PTG F2
PTU F2
(c)
PTUlt F2
ProGlider
PTG F2
PTG F3
PTU F2
PTU F3

FIGURE 2 Scanning electron microscopic analyses of ProGlider, ProTaper Ultimate (PTUlt), ProTaper Gold (PTG) and ProTaper Universal (PTU) instruments. Representative images of the (a) active blades (×20), (b) tips (×40), (c) cross-sections (at D10) (×80) and (d) surface finishing (×150). Except for the FXL, the design of the other PTUlt instruments was similar, but different from their equivalent counterparts. Surface finishing (d) was similar amongst instruments with the presence of parallel manufacturing mark and few micro defects.

in their geometry, mostly because changes in the design of the novel PT Ultimate system do not allow to compare one-to-one with the old versions of ProTaper instruments, highlighting the importance of a multimethod analysis to properly understand their mechanical behaviour. The present results demonstrated that PT Ultimate Shaper and Finishers (F1, F2 and F3) had lower torsional strength and superior flexibility (higher angle of rotation and lower bending load) compared with their counterparts (Table 1, Figure 4) and the null hypothesis was rejected. Considering the similarities of tested instruments in terms of nickel/titanium ratio and surface finishing, results of these PT Ultimate instruments may be mostly explained not only by their different designs, such as the high number of spirals (McSpadden, 2007) (Table 1) and the off-centred parallelogram cross-section (Martins et al., 2020) (Figure 2), but also by their crystallographic arrangement compared to the full austenitic PT Universal, once the alloy of the PT

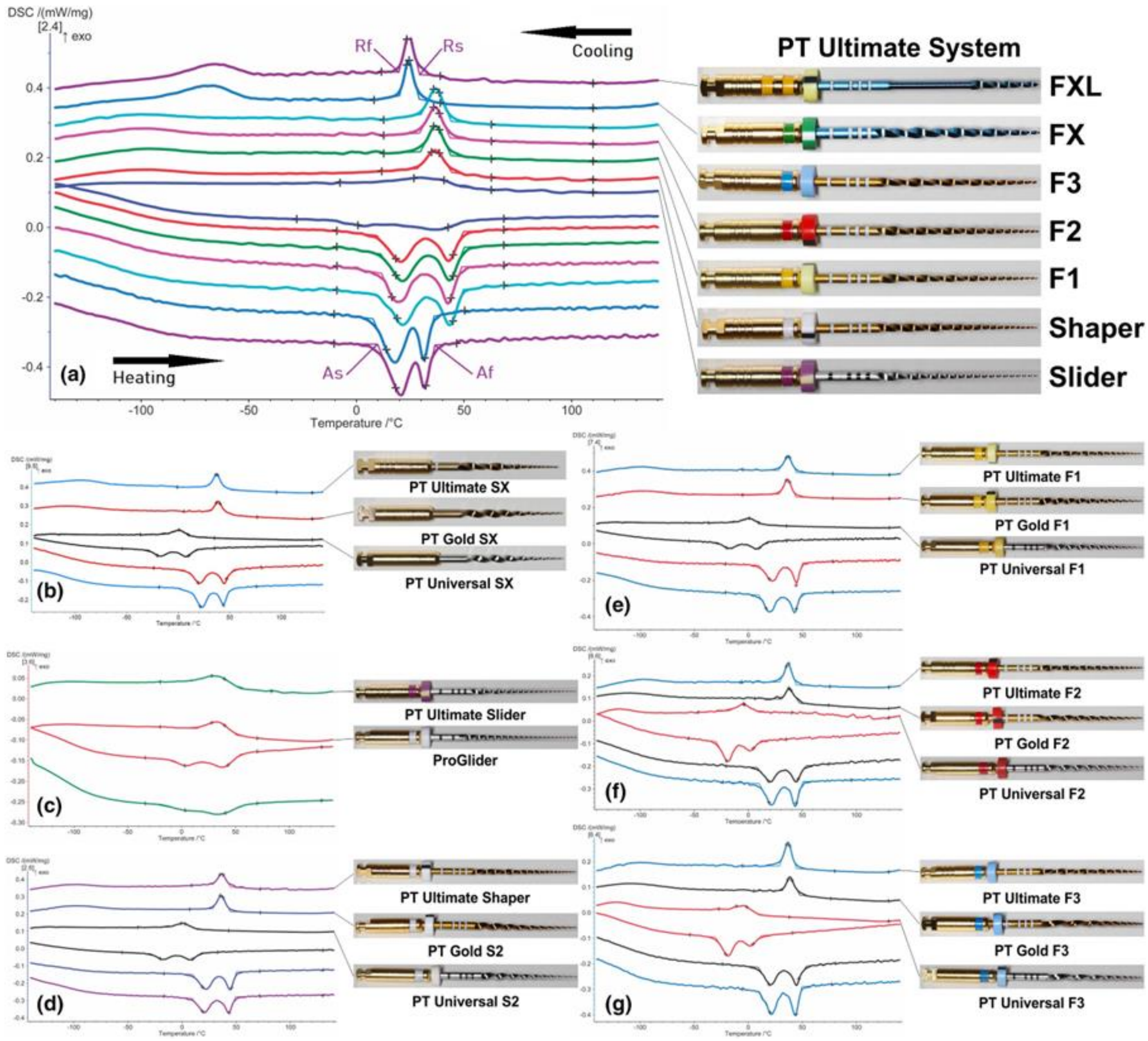

DSC /(mW/mg)
[2.4] ↑ exo
Rf
Rs
Cooling
PT Ultimate System
FXL
FX
F3
F2
F1
Shaper
Slider
0.4
0.2
0.0
-0.2
-0.4
(a)
Heating
As
Af
-100
-50
0
50
100
Temperature /°C
PT Ultimate SX
PT Gold SX
PT Universal SX
(b)
PT Ultimate Slider
ProGlider
(c)
PT Ultimate Shaper
PT Gold S2
PT Universal S2
(d)
PT Ultimate F1
PT Gold F1
PT Universal F1
(e)
PT Ultimate F2
PT Gold F2
PT Universal F2
(f)
PT Ultimate F3
PT Gold F3
PT Universal F3
(g)

FIGURE 3 Comparison of DSC curves and phase transformation temperatures amongst the ProTaper (PT) Ultimate (a) instruments and their equivalent counterparts (b–g), depicting their macroscopic views in which differences in their alloy colours suggest distinct heat treatments. (a) The analyses of the PT Ultimate system revealed 3 different curve patterns: (1) Slider had a unique curve with the highest and lowest phase transformation temperatures at both cooling (top curve, reading from right to left) and heating (bottom curve, reading from left to right); (2) SX, Shaper, F1, F2 and F3 instruments share similar DSC curves (Rs ~44.0°C; Rf ~29.0°C), as well as, (3) FX and FXL instruments on cooling (Rs ~29.0°C; Rf ~20°C) and heating (As 7.7–9.8°C; Af ~36.0°C). (b–g) PT Ultimate instruments showed equivalent DSC curves and phase transformation temperatures to their counterparts, except for the ProTaper Universal instruments. (As Austenitic start; Af Austenitic finish; Rs R-phase start; Rf R-phase finish).

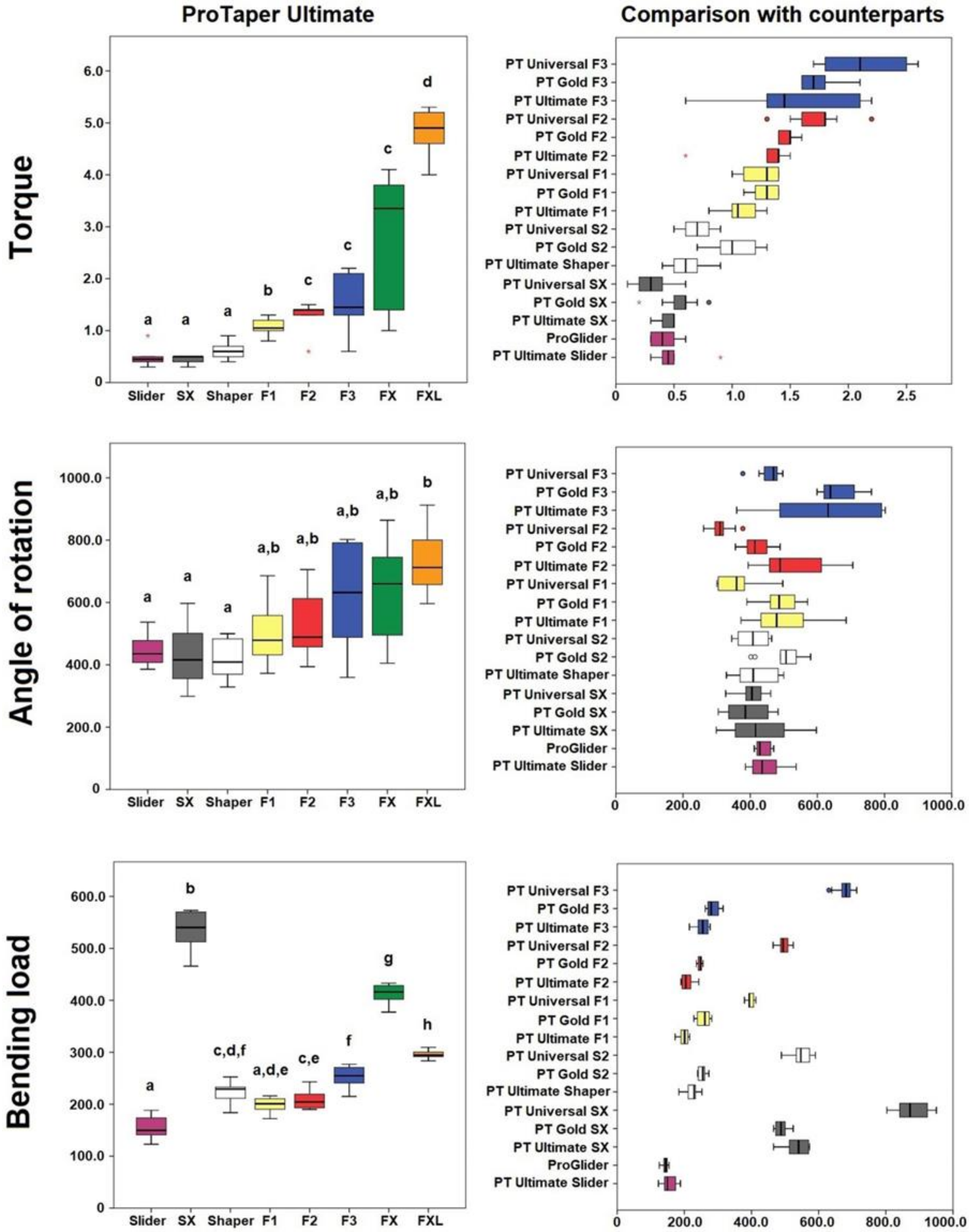
Gold system has similar heat treatment (Figure 3, Table 2). Compared with the other tested instruments, the reduced flexibility of the SX instruments (Table 1, Figure 4) may be related to their shorter lengths (19 mm) which led to an exponential increase in the stress necessary to apply the force during the standardized bending test.

NiTi alloys may have three distinct microstructural phases named austenite, R-phase and martensite, which can directly influence the mechanical behaviour of endodontic instruments (Elnaghy & Elsaka, 2016; Plotino et al., 2017; Zupanc et al., 2018). The austenitic phase of the NiTi alloy is relatively stiff, hard and has limited flexibility. When stress is applied to this type of instrument, a transformation from the austenitic to the martensitic crys- tallographic arrangement may occur in a process named stress-induced martensitic transformation This atomic re- organization leads to a feature known as superelasticity, characterized by a form rearrangement that may spring back the instrument to its original form without any de- finitive deformation when the induced stress is stopped or reduced (Shen et al., 2011), meaning that its lower elastic modulus, compared with stainless-steel instruments, pro- vides superior flexibility (Zupanc et al., 2018). The auste- nitic form, and its superelasticity features, characterizes the NiTi conventional alloy that has been used in systems such as the ProTaper Universal tested in this study.

Instrument (tip size/ taper, wire)	R-Phase start (Rs)	R-Phase finish (Rf)	Austenitic start (As)	Austenitic finish (Af)
PT Ultimate Slider (16/.02v, M-Wire)	48.4	11.5	−13.3	52.1
ProGlider (16/.02v, M-Wire)	51.2	13.9	−14.4	54.6
PT Ultimate SX (20/.03v, Gold)	45.6	29.1	11.5	50.6
PT Gold SX (19/.04v, Gold)	47.7	31.6	9.6	54.1
PT Universal SX (19/.04v, Austenitic)	16.2	−13.5	−28.9	17.9
PT Ultimate Shaper (20/.04v, Gold)	44.3	28.3	9.4	49.7
PT Gold S2 (20/.04v, Gold)	44.4	28.2	9.6	50.4
PT Universal S2 (20/.04v, Austenitic)	14.1	−12.5	−28.9	18.0
PT Ultimate F1 (20/.07v, Gold)	43.6	29.3	11.7	48.9
PT Gold F1 (20/.07v, Gold)	43.9	28.4	9.4	50.0
PT Universal F1 (20/.07v, Austenitic)	16.2	−13.3	−28.7	17.6
PT Ultimate F2 (25/.08v, Gold)	43.9	29.8	11.2	50.1
PT Gold F2 (25/.08v, Gold)	47.5	31.0	8.1	53.1
PT Universal F2 (25/.08v, Austenitic)	9.8	−17.7	−29.8	11.7
PT Ultimate F3 (30/.09v, Gold)	44.0	30.1	9.6	49.7
PT Gold F3 (30/.09v, Gold)	47.9	31.7	9.3	53.8
PT Universal F3 (30/.09v, Austenitic)	10.7	−18.2	−30.1	12.4
PT Ultimate FX (35/.12v, Blue)	29.2	20.0	7.7	36.1
PT Ultimate FXL (50/.10v, Blue)	29.4	19.8	9.8	36.4

TABLE 2 Phase transformation temperatures (in °C) of ProTaper (PT) Ultimate, Gold, Universal and ProGlider instruments

The crystallographic arrangement of the NiTi alloy observed in a higher temperature range is defined as the austenitic phase and is characterized by a B2 type lattice (cubic symmetry). When the alloy temperature decreases below the transformation temperature range, the martensitic transformation occurs from the austenitic phase to the martensitic one. This martensitic phase displays a monoclinic lattice (B19' type) that can be reverted to the B2 type lattice by heating the alloy above the transformation temperature range (Thompson, 2000). This phenomenon of changing the physical properties to allow a deformed NiTi alloy recovers its original shape when heated is known as shape memory (Zupanc et al., 2018). Companies take advantage of this property to produce martensitic instruments that are heat treated during their manufacture to raise their phase transformation temperatures. As a result, these instruments are softer, more ductile and have superior flexibility, cyclic fatigue resistance and lower strength to torsional stress than instruments with austenitic crystallographic arrangements. Several designations have been given to these heat-treated NiTi alloys, such as M-wire, CM wire, Gold wire, Blue wire, or MaxWire (Zupanc et al., 2018). Notwithstanding



ProTaper Ultimate
Comparison with counterparts
Torque
Angle of rotation
Bending load
Slider
SX
Shaper
F1
F2
F3
FX
FXL
PT Universal F3
PT Gold F3
PT Ultimate F3
PT Universal F2
PT Gold F2
PT Ultimate F2
PT Universal F1
PT Gold F1
PT Ultimate F1
PT Universal S2
PT Gold S2
PT Ultimate Shaper
PT Universal SX
PT Gold SX
PT Ultimate SX
ProGlider
PT Ultimate Slider

FIGURE 4 Comparison of the mechanical behaviour amongst the ProTaper (PT) Ultimate instruments and their equivalent counterparts. (On the left column) In the PT Ultimate system, the maximum torque, angle of rotation and bending load progressively increased with instruments' sizes, except for the FXL that was more flexible than the FX instrument. (On the right column) PT Ultimate showed lower torsional strength, superior flexibility and higher angle of rotation than PT Gold and PT Universal counterparts. Slider and ProGlider had similar mechanical behaviour. Different letters in the charts represent statistically significant differences ($p < 0.05$).

the fact that all of them share similar martensitic characteristics, they have distinct crystallographic arrangements at service temperature and, consequently, different mechanical behaviours (Zupanc et al., 2018), as depicted by the present results (Table 1, Figures 3 and 4). Another type of martensitic transformation, which occurs between full austenitic and full martensitic forms, is the R-phase transformation, which may also be considered a martensitic form (Kuhn & Jordan, 2002). It consists of a rhombohedral atomic disposition with thermoelastic martensitic characteristics and, similarly to the martensitic phase, can be stress- or temperature-induced. Many manufacturers have used this R-phase transformation to produce instruments with some ductility, but enhanced flexibility and cyclic fatigue strength, compared to conventional NiTi instruments (Zhou et al., 2013; Zupanc et al., 2018).

One of the innovations of the PT Ultimate system was the file-specific heat treatment based on instruments' dimensions featuring M-wire (Slider), Gold (SX, Shaper and Finishers F1, F2 and F3) and Blue (Auxiliary Finishers FX and FXL) heat-treated wires, that is, instruments presenting 3 distinct crystallographic arrangements of their metal alloys (mixed austenitic, R-phase and martensitic forms depending on the temperature of the instrument) in the same system, a feature confirmed in this study (Figure 3). The idea behind this approach is to take advantage of different crystallographic phases of the NiTi alloy to create instruments with enhanced properties according to their use requirements. The Slider and ProGlider instruments showed equivalent DSC curves that were consistent to M-wire instruments (Martins et al., 2021a; Martins et al., 2021b), but distinct to the other instruments of the PT Ultimate system (Table 2, Figure 3). The Slider has an austenitic plus R-phase crystallographic arrangement at both room and body temperatures and, therefore, minor changes in its mechanical behaviour may be expected in that service temperature range. The Shaper and Finishers (F1, F2 and F3) of the PT Ultimate system appear to present a martensitic crystallographic arrangement at room temperature after manufacturing and tend to acquire a mixed austenitic plus R-phase characteristics when enclosing the body temperature meaning that, at higher temperatures, instruments may develop some characteristics of the austenitic alloy. These instruments present a R-phase transformation at cooling (between 44.3°C [Rs] and 28.3°C [Rf]) with a transition to B19' at a very low temperature (under −50°C) but with a DSC double curve from B19' to R-phase to B2 on heating in a more proximal temperature range (between 9.4°C and 50.1°C) (Figure 3). These transformation temperatures were similar to their analogues PT Gold instruments, but distinct from the PT Universal ones (Table 2, Figure 3), and followed previous reports testing gold wire instruments (Martins et al., 2021b).

The auxiliary FX and FXL instruments of the PT Ultimate system showed DSC curves and phase transformation temperatures between 29.4°C (Rs) and 19.8°C (Rf) on cooling and 7.7°C (As) and 36.4°C (Af) on heating (Table 2), corroborating with previous studies testing Blue heat-treated wire instruments (Martins et al., 2021b). These 2 instruments present a martensitic crystallographic arrangement at room temperature, which tends to change to an austenitic form at body temperature. Therefore, the incorporation of more austenitic characteristics in these instruments is expected if their temperature rises during root canal preparation procedures, decreasing their flexibility (Oh et al., 2020) and their ability to sustain high maximum torque (Silva et al., 2018). These results, however, raise doubts regarding the decision of the manufacturer to use the Blue heat-treated wire in the FX and FXL auxiliary instruments. One possible argument would be the intention of increasing their austenite phase, consequently improving their torsional strength resistance. But this makes no sense since both instruments are recommended to be used only in anatomically straight and large canals that were previously enlarged by the other instruments (Ruddle, 2022), a condition in which they are submitted only to a low torsional stress. Therefore, a proper explanation from the manufacturer regarding the advantage of using the Blue heat-treated wire in these auxiliary instruments is still missing. Considering that PT Gold and PT Universal systems do not have instruments with similar dimensions of FX and FXL, no comparisons with other instruments could be made.

The main limitations of this study include not evaluating parameters such as cyclic fatigue strength, cutting efficiency and shaping ability, which should be included in future studies. Besides, it was also not possible to determine the real influence of the different cross-sections in the mechanical properties of tested instruments. On the other hand, the major strengths were to provide essential information about the design, metallurgy and mechanical behaviour of the recently launched PT Ultimate, a system that comprises instruments with specific heat treatments and distinct crystallographic arrangements of their metal alloys, through a multimethod research using well-established international guidelines (ANSI/ADA Specification No. 28, 2002; ASTM F2004-17, 2004; ISO 3630-3631, 2008). This methodological approach allows for a more comprehensive understanding of the results since it overcomes the inherent limitations of each test. Considering that the novel PT Ultimate system showed lower torsional strength and higher flexibility than their counterparts, clinicians may benefit from this system in clinical cases that requires these characteristics, such as curved and nonconstricted root canals, instead of PT Universal or PT Gold; however, considering the lack of information about this recently launched system, further studies are still needed to guide clinical recommendations.

CONCLUSIONS

The novel PT Ultimate system comprises instruments with three distinct heat treatments that showed different design, but similar surface finishing, nickel/titanium ratios and phase transformation temperatures to their heat-treated analogues. Whilst Slider and ProGlider had similar mechanical behaviour, the other PT Ultimate instruments showed lower torsional strength and superior flexibility than their counterparts, whilst maximum torque, angle of rotation and bending loads progressively increased with their sizes.

AUTHOR CONTRIBUTIONS

J.N.R. Martins: Conceptualization; Data curation; Formal analysis; Investigation; Methodology; Writing – original draft presentation. E.J.N.L. Silva: Conceptualization; Data curation; Formal analysis; Investigation; Methodology; Writing – original draft presentation. D. Marques: Data curation; Methodology; Supervision; Writing – review & edit. N. Ajuz: Investigation; Methodology; Validation. M. Rito Pereira: Investigation, Resources; Writing – review & edit. R. Pereira da Costa: Data curation, Resources; Writing – review & edit. F.M. Braz Fernandes: Data curation; Formal analysis; Funding acquisition; Writing – review & edit. M.A. Versiani: Conceptualization; Methodology; Supervision; Writing – review & edit.

ACKNOWLEDGEMENTS

FMBF acknowledges the funding of CENIMAT/i3N by national funds through the FCT-Fundação para a Ciência e a Tecnologia, I.P., within the scope of Multiannual Financing of R&D Units, reference UIDB/50025/2020-2023.

CONFLICT OF INTEREST

The authors deny any conflicts of interest.

DATA AVAILABILITY STATEMENT

Data sharing is not applicable to this article as no new data were created or analyzed in this study.

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How to cite this article: Martins, J.N.R., Silva, E.J.N.L., Marques, D., Ajuz, N., Rito Pereira, M. & Pereira da Costa, R. et al. (2023) Characterization of the file-specific heat-treated ProTaper Ultimate rotary system. *International Endodontic Journal*, 56, 530–542. Available from: https://doi.org/10.1111/iej.13880

5. DISCUSSÃO

A presente tese é composta de quatro diferentes estudos, que utilizaram uma abordagem de pesquisa multimétodo para avaliar desenho geométrico geral, composição elementar, temperaturas de transformação de fase, comportamento mecânico e capacidade de modelagem de diferentes instrumentos endodônticos. Essa abordagem metodológica permite uma avaliação mais abrangente sobre as propriedades dos instrumentos testados, pois evita a "compartimentalização do conhecimento", fenômeno no qual as estruturas de conhecimento sobre um domínio específico são compostas por várias partes separadas (SCHOENFELD, 1986). Esse modelo de avaliação multimétodo pode ser visto como um dos principais pontos fortes da presente tese e de seus artigos, por permitir uma ampla avaliação dos perfis e comportamentos dos instrumentos. Todos os testes seguiram diretrizes internacionais rígidas (ASTM, 2004; ASTM, 2007; ANSI/ADA, 2002; ISO 3630-3631, 2008) ou metodologias com alta validade interna (SILVA *et al.,* 2020; PAQUÉ *et al*., 2009; VERSIANI *et al*., 2013; MARTINS *et al.,* 2021a), permitindo uma compreensão mais robusta e confiável do desempenho dos sistemas.

Uma das principais vantagens da abordagem multimétodo é a sua capacidade de triangulação de dados. Ao utilizar vários métodos, os pesquisadores podem validar e corroborar os achados, aumentando a robustez das conclusões. A triangulação ajuda a mitigar as limitações dos métodos individuais e proporciona uma visão mais holística do tópico de pesquisa. Outra vantagem da abordagem multimétodo é a sua flexibilidade e adaptabilidade. Os pesquisadores podem adaptar a combinação de métodos de acordo com a questão da pesquisa específica, o contexto e os recursos disponíveis. Isso permite uma investigação mais personalizada e apropriada ao contexto, aumentando a validade e a generalizabilidade dos resultados (SCHOENFELD, 1986).

A abordagem multimétodo é particularmente útil ao estudar fenômenos complexos que não podem ser totalmente compreendidos por meio de um único método. Ela permite que os pesquisadores capturem diferentes aspectos, perspectivas e dimensões do fenômeno, levando a uma compreensão mais abrangente. Além disso, o uso de múltiplos métodos pode ajudar a superar as limitações e os vieses inerentes aos métodos individuais, resultado em uma análise mais objetiva e abrangente (HUNTER & BREWER, 2015).

No entanto, a abordagem multimétodo também apresenta algumas limitações, o que pode demandar tempo e recursos, uma vez que requer expertise em múltiplos métodos de pesquisa e técnicas de coleta de dados. Os pesquisadores precisam planejar e coordenar cuidadosamente os diferentes métodos para garantir sua integração e complementariedade. Além disso, a análise e a síntese de dados provenientes de várias fontes podem ser desafiadoras pois demandam habilidade especializada em integração e interpretação de dados (HUNTER & BREWER, 2015).

O desenvolvimento de sistemas de níquel-titânio (NiTi) para o preparo do canal radicular tem permitido aos clínicos o acesso a instrumentos dotados de notável desempenho (KUHN *et al.,* 1997). No entanto, uma preocupação primária em relação ao seu uso em ambiente clínico tem sido a possibilidade de fratura inesperada. Consequentemente, os fabricantes têm se esforçado para melhorar o desempenho mecânico dos instrumentos, alterando suas características. Notavelmente, um avanço significativo foi a produção de instrumentos tratados termicamente, resultando em resistência à fadiga e flexibilidade acentuadamente melhoradas (ELNAGHY & ELSAKA, 2016). Além disso, o desempenho mecânico dos instrumentos de NiTi pode ser significativamente afetado por vários aspectos de seu projeto, incluindo geometrias de seção transversal, ângulo helicoidal e o número de espirais (MCSPADDEN, 2007). Essa influência é ainda evidenciada pelos efeitos do acabamento superficial (ANDERSON *et al.,* 2007) e do arranjo cristalográfico da liga metálica (MARTINS *et al.,* 2021b). No entanto, apesar desses avanços significativos, a questão da fratura de instrumentos ainda representa um desafio que requer atenção.

A ocorrência de fratura de instrumentos pode ser atribuída a dois mecanismos: fadiga cíclica e falha torsional (SILVA *et al.,* 2018). Outra preocupação em relação ao uso de instrumentos de NiTi é a preservação da integridade anatômica do canal radicular e a minimização de desvios do trajeto original durante os procedimentos de preparo, o que pode ser resolvido por sua maior flexibilidade. O princípio que se aplica ao teste de fadiga cíclica também se estende ao teste de torção, que avalia a capacidade de um instrumento de resistir a altos níveis de tensão de torção, particularmente em cenários como o travamento da ponta em canais radiculares estreitos. O teste de flexão opera em um princípio semelhante aos testes mencionados, mas visa avaliar a flexibilidade de um instrumento (MARTINS e*t al.,* 2022a). A flexibilidade desempenha um papel fundamental, particularmente na obtenção de uma abordagem mais conservadora ao moldar canais curvos. Este princípio pode ser extrapolado para vários outros testes mecânicos, como flambagem, microdureza ou eficiência de corte. Em resumo, embora uma extensa pesquisa tenha influenciado as práticas atuais em procedimentos técnicos, há uma necessidade de melhorias metodológicas nos testes de sistemas de NiTi para obter uma compreensão abrangente dos fatores subjacentes que influenciam na resposta de instrumentos endodônticos específicos (MARTINS e*t al.,* 2022a).

Vários estudos têm debatido a influência da temperatura de teste no comportamento dos instrumentos. Embora especificar uma temperatura para conduzir um teste possa ser considerado muito limitado e simplista devido à faixa de temperatura de trabalho do instrumento, vale mencionar as possíveis alterações devido ao aumento da exposição à temperatura dos instrumentos contemporâneos. Todos os quatro estudos da presente tese foram conduzidos de acordo com diretrizes internacionais (ANSI/ADA 2002; ISO 2008) e os ensaios mecânicos foram realizados em temperatura ambiente, em conformidade com a maioria dos estudos disponíveis na literatura (DOSANJH *et al., 2*017; BURKLEIN *et al.*, 2021; ALBERTON *et al.,* 2020). Além disso, uma avaliação utilizando testes de calorimetria diferencial de varredura (DSC) foi realizada para compreender as possíveis mudanças de comportamento sob múltiplas temperaturas e faixas de temperaturas.

Embora muitos estudos dependam de parâmetros mecânicos para avaliar o desempenho dos sistemas de instrumentação de NiTi, um entendimento mais abrangente também deve incluir a avaliação de sua eficácia no preparo do sistema de canais radiculares. Portanto, é importante que na abordagem multimétodo os resultados de diferentes testes mecânicos sejam combinados com a capacidade de modelagem dos sistemas de NiTi para uma melhor interpretação de seu desempenho e, consequentemente, uma tradução mais precisa dos achados pré-clínicos para orientar o uso clínico (SILVA *et al.,* 2020). Em 3 dos 4 artigos apresentados, os sistemas testados foram comparados em relação à porcentagem de paredes intactas, após o preparo dos canais radiculares, e avaliadas usando uma tecnologia padrão ouro, a microtomografia computadorizada (ARIAS & PETERS, 2022; BÜRKLEIN & ARIAS, 2022). Este parâmetro tem uma alta relevância clínica, uma vez que as áreas não tocadas do canal podem abrigar bactérias residuais e servir como uma causa potencial de infecção persistente, que pode levar à doença pós-tratamento (ARIAS & PETERS, 2022; BÜRKLEIN & ARIAS, 2022).

6. CONCLUSÃO

As conclusões da presente tese foram:

(i) Os sistemas Vortex Blue, TruNatomy e Genius Proflex foram semelhantes em relação à composição elementar e capacidade de modelagem, mas mostraram diferenças significativas em seu design geral, fases de transição de temperatura e comportamento mecânico;

(ii) Os sistemas Reciproc Blue, WaveOne Gold e REX foram semelhantes em relação à composição elementar e capacidade de modelagem, mas mostraram diferenças significativas em seu design geral, fases de transição de temperatura e comportamento mecânico;

(iii) Os sistemas HyFlex EDM, Neoniti, EDMax e ProTaper Gold apresentaram diferenças em seu design e temperaturas de transformação de fase, que influenciaram diretamente nos resultados dos testes mecânicos, mas não na sua capacidade de modelagem;

(iv) O novo sistema PT Ultimate compreende instrumentos com três tratamentos térmicos distintos que mostraram designs diferentes, mas acabamentos de superfície, proporções de níquel/titânio e temperaturas de transformação de fase semelhantes aos seus análogos tratados termicamente.

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8. ANEXOS

8.1 ANEXO – Comitê de Ética

PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Avaliação das características, design e preparo dos canais radiculares utilizando diferentes instrumentos.
Pesquisador: NATASHA CAMARA AJUZ DEMIER

Área Temática: Equipamentos e dispositivos terapêuticos, novos ou não registrados no País;

Versão: 1
CAAE: 57369521.9.0000.5283
Instituição Proponente:UNIVERSIDADE

UNIGRANRIO **Patrocinador Principal:**

Financiamento Próprio

DADOS DO PARECER

Número do Parecer: 5.355.157

Apresentação do Projeto:
O presente estudo possui modelo de caráter experimental, in vitro, e será desenvolvido a partir de uma amostra de 4 grupos de 5 dentes cada, avaliados através da microtomografia computadorizada (micro-CT). Cada grupo irá ser utilizado um sistema de lima n=5, sendo 15 canais para cada sistema: ED Max NEO NiTi A1, Hyflex CM e Protaper Gold. Os dentes serão instrumentados em manequim odontológico e em seguida, serão reescaneados por micro-CT para avaliar a qualidade do preparo dos sistemas em relação a porcentagem de área não preparada, acúmulo de debris e volume de dentina removida. Além disso, todas as limas de cada sistema serão avaliadas seguindo outros 3 parâmetros: qualidade e design de fabricação, testes mecânicos e caracterização metalúrgica de cada instrumento.

Objetivo da Pesquisa:

Avaliar a qualidade do preparo dos canais radiculares após a instrumentação com os sistemas EDMax, Neo NiTi A1, Hyflex CM e Protaper Gold , através da micro-CT, bem como avaliar as propriedades metalúrgicas e mecânicas dos instrumentos.

Avaliação dos Riscos e Benefícios:

Riscos: Existe risco mínimo previsível. Os voluntários doadores de dentes não serão afetados por nenhum procedimento da metodologia desta pesquisa. A única situação que pode vir a afetar o doador do elemento dentário é o procedimento de exodontia que pode resultar em sangramento excessivo, dor e/ou desconforto.

Benefícios: O trabalho beneficiará tantos os cirurgiões-dentistas como os pacientes. Os profissionais terão o conhecimento técnico sobre diferentes instrumentos endodônticos e suas respectivas capacidades de preparo e de remoção de estrutura dentária dos canais radiculares durante o tratamento endodôntico. Os pacientes terão o beneficio da utilização de uma técnica que permita um melhor preparo do canal e possivelmente, maior chance de sucesso do tratamento endodôntico.

Comentários e Considerações sobre a Pesquisa: O estudo utiliza um modelo consagrado na literatura e o orientador proponente possui dezenas de publicações sobre o assunto utilizando esta metodologia. Porém o trabalho é valido pois investiga novos sistemas recém lançados comercialmente.

Considerações sobre os Termos de apresentação obrigatória:
TCLE - OK
INFORMAÇÕES BÁSICAS DO PROJETO - OK
CRONOGRAMA - OK
ORÇAMENTO - OK
PROJETO - OK
ANUÊNCIA - OK
FOLHA DE ROSTO - OK

Recomendações:
Recomenda-se que o projeto seja aprovado e que os resultados sejam publicados em periódicos científicos para que os clínicos tomem conhecimento das evidências geradas.

Conclusões ou Pendências e Lista de Inadequações:
Não existem pendências e inadequações.

O presente projeto, seguiu nesta data para análise da CONEP e só tem o seu início autorizado após a aprovação pela mesma.

Este parecer foi elaborado baseado nos documentos abaixo relacionados:

Tipo Documento	Arquivo	Postagem	Autor	Situação
Informações Básicas do Projeto	PB_INFORMAÇÕES_BÁSICAS_DO_PROJETO_1867263.pdf	30/03/2022 17:09:24		Aceito
TCLE / Termos de Assentimento /	tcleprojetodoutorado.pdf	30/03/2022 17:08:41	NATASHA CAMARA AJUZ DEMIER	Aceito

Justificativa de Ausência	tcleprojetodoutorado.pdf	30/03/2022 17:08:41	NATASHA CAMARA AJUZ DEMIER	Aceito
Cronograma	cronograma.docx	30/03/2022 17:08:22	NATASHA CAMARA AJUZ DEMIER	Aceito
Orçamento	orcamentoprojetodoutorado.pdf	30/03/2022 17:07:59	NATASHA CAMARA AJUZ DEMIER	Aceito
Projeto Detalhado / Brochura Investigador	projetodoutoradocomite.docx	10/12/2021 10:46:30	NATASHA CAMARA AJUZ DEMIER	Aceito
Outros	cartadeanuenciaprojeto.pdf	10/12/2021 10:21:41	NATASHA CAMARA AJUZ DEMIER	Aceito

Folha de Rosto	folhaderostoprojeto.pdf	08/12/2021 16:07:57	NATASHA CAMARA AJUZ DEMIER	Aceito

Situação do Parecer:

Aprovado

Necessita Apreciação da CONEP:

Sim

DUQUE DE CAXIAS, 18 de Abril de 2022

Assinado por:
SERGIAN VIANNA CARDOZO
(Coordenador(a))