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Influence of sintering protocol and translucency-enhancing liquid on the mechanical performance of anterior zirconia crowns

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ABSTRACT

Aim: This study aimed to evaluate the effects of a translucency-enhancing liquid (TEL) and high-speed sintering (HS) on the mechanical behavior, with a focus on the survival rates and fracture forces of anterior zirconia crowns with different yttria contents.

Materials and methods: Anterior crowns (16 groups, n = 8 per group) were fabricated using four different zirconia materials (3Y-TZP, 4Y-TZP, 5Y-TZP, and multilayer (ML)). The 3Y and 4Y groups were treated with a TEL in three variations: no treatment (NL), two-layer penetration (2L), and four-layer penetration (4L). The 5Y-TZP and ML zirconia crowns served as reference groups without TEL treatment. All specimens underwent either conventional (CS) or high-speed (HS) sintering before being subjected to thermocyclic and mechanical loading (TCML), followed by a load-to-fracture test. Statistics: ANOVA, Bonferroni test, Shapiro-Wilk test, Pearson correlation; $\alpha = 0.05$.

Results: Three specimens in group 5Y-NL-CS and one in group 4Y-NL-HS failed during TCML. Fracture forces varied significantly between different types of zirconia and decreased with increasing yttria content. While mean fracture forces were not affected by HS or TEL treatment, HS of ML and particularly 5Y-TZP crowns was associated with a higher frequency of crown fractures, indicating material- and condition-specific tendencies of potential clinical relevance.

Conclusion: Within the limitations of this in-vitro study - including the use of PMMA abutments, failures during TCML, and material- and failure-specific differences - all specimens withstood forces exceeding the physiological loads expected in the anterior region. Under these conditions, TEL treatment and HS of 3Y-TZP and 4Y-TZP zirconia appear to allow the rapid fabrication of anterior crowns while maintaining their mechanical performance. As optical parameters were not assessed, any potential aesthetic implications of TEL or HS cannot be inferred.

1. Introduction

Computer-aided design and manufacturing (CAD/CAM) enable the efficient fabrication of fixed dental prostheses and have become an integral part of modern dentistry. Chairside CAD/CAM-fabricated restorations allow for immediate treatment, but achieving high aesthetic quality, particularly in the anterior region, remains a challenge. To address these aesthetic shortcomings, a variety of all-ceramic materials have been developed, each with inherent trade-offs between aesthetics and mechanical strength. Monolithic lithiumsilicate ceramics provide an appealing visual outcome but have moderate flexural strength (Rosentritt et al., 2020), which limits their clinical range of indication. Veneered zirconia frameworks improve aesthetics but are prone to chipping (Edelhoff et al., 2008; Sailer et al., 2018). Monolithic zirconia restorations offer the highest mechanical strength but are often more opaque, which diminishes their natural appearance (Camposilvan et al., 2018; Carrabba et al., 2017; Fathy et al., 2021; Rosentritt et al., 2020; Zhang et al., 2016). One option to improve the aesthetic appearance of zirconia is to increase its yttria content. This procedure improves translucency but reduces mechanical strength (Camposilvan et al., 2018; Carrabba et al., 2017; Fathy et al., 2021; Rosentritt et al., 2020; Zhang et al., 2016). Multilayer (ML) zirconia addresses these challenges by incorporating a built-in translucency gradient. However, its translucency distribution is pre-defined by the blank's structure and cannot be individually adjusted. Ideally, translucency should be selectively enhanced in specific areas while maintaining strength and opacity

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elsewhere. Translucency-enhancing liquids (TEL) contain dissolved yttrium ions such as yttrium(III)-nitrate hexahydrate (Greitens and Heilemann, 2015). The liquid is designed to penetrate the porous pre-sintered zirconia blank and to locally increase the yttria content to up to 10 wt% (Greitens and Heilemann, 2015). During sintering, this procedure promotes the formation of cubic zirconia crystals and results in increased grain size (Ban et al., 2023; Greitens and Heilemann, 2015; Hauptmann et al., 2013). Translucent zirconia with a high proportion of cubic crystals and large grains has reduced mechanical properties (Camposilvan et al., 2018; Carrabba et al., 2017; Fathy et al., 2021; Rosentritt et al., 2020; Zhang et al., 2016). Therefore, TEL can be selectively applied to enhance aesthetics of the incisal areas of a crown, while the opacity and strength of the core is preserved.

In addition to the challenge of achieving high aesthetics, the prolonged sintering process is another limitation of zirconia restorations (Stawarczyk et al., 2014). Recent advances in high speed sintering (HS) reduced processing times from several hours to less than half an hour. Although this innovation accelerates the fabrication process, it requires higher temperatures. Higher sintering temperatures and extended dwell times promote grain growth in zirconia, reducing the number of grain boundaries and typically enhancing translucency (Jansen et al., 2019; Kim et al., 2013; Stawarczyk et al., 2013; Zhang, 2014). However, in HS, the significantly shortened dwell time coincide with decreased translucency, despite the observed increase in grain growth (Alshahrani et al., 2024; Jansen et al., 2019; Lawson and Maharishi, 2020). This reduction in translucency may be attributed to microstructural irregularities, such as residual pores and the simultaneous presence of different grain sizes (Alshahrani et al., 2024; Lawson and Maharishi, 2020; Mayinger et al., 2024; Zhang, 2014). Notably, biaxial flexural strength and fracture load values are not negatively affected by HS (Alshahrani et al., 2024; Elisa Kauling et al., 2020; Jansen et al., 2019; Mayinger et al., 2024), but low mechanical reliability has been reported (Cokic et al., 2020; Jerman et al., 2020).

It remains to be determined whether HS, when combined with TEL, alters the mechanical properties of zirconia. The manufacturer indicates that the liquid produces lighter colour outcomes at sintering temperatures higher than 1450 °C, confirming its temperature sensitivity. Consequently, a potential influence on mechanical properties and therefore clinical performance cannot be excluded. To investigate this, all specimens were subjected to TCML, as aging tests under thermo-mechanical load provide insight into clinical performance (Rosentritt et al., 2009). A subsequent load-to-fracture test evaluated differences in fracture force among the various materials and assessed the impact of different processing techniques.

Improving the aesthetics of zirconia crowns using TEL and reducing processing time through HS are clinically relevant objectives. Despite extensive research on the optical and mechanical performance of zirconia, the combined effect of TEL and HS on its mechanical behavior has not been systematically investigated. This study addresses this gap by evaluating how TEL and HS influence the mechanical resilience of anterior crowns made from zirconia with different yttria content. Accordingly, the hypothesis of this study was that TEL and HS affect the mechanical properties of anterior zirconia crowns with varying yttria content, leading to lower fracture forces and shorter survival rates after TCML.

2. Material and methods

An artificial maxillary first incisor (FDI #11, Frasaco GmbH, D) was prepared with a chamfer preparation ensuring a circular reduction of 1.5 mm with 6° angulation, incisal reduction of 1.5 mm and a minimum abutment height of 4 mm (Rosentritt et al., 2020). The prepared tooth was digitized with an intraoral scanner (STL-file; Cerec Primescan, Dentsply Sirona, USA). PMMA tooth duplicates (Shaded PMMA Disc, Dentsply Sirona, USA) were milled (Cerec MC X5, Dentsply Sirona, USA) and the sprue locations were refined. Based on the STL-file, anatomical crowns were designed following CAD software recommendations (radial minimal thickness of 500 μm, occlusal minimum thickness of 700 μm, margin thickness of 50 μm, margin ramp angle of 60°, margin ramp width of 150 μm; InLabCAD SW 20.0.4, Dentsply Sirona, USA). The designed restorations were milled (Cerec MC X5, Dentsply Sirona, USA) from four different zirconia materials (see Table 1) with a radial and occlusal spacer of 80 μm. ML restorations were positioned at a depth of 2.5 mm within the blank in accordance with the manufacturers' nesting recommendations. For 3Y-TZP (DD BioZX2, Dental Direkt GmbH, D) and 4Y-TZP (DD Cube One ML, Dental Direkt GmbH, D) zirconia, three subgroups were tested: no liquid (NL), two-layer liquid penetration (2L; application to the crowns' upper half (~5 mm from incisal edge) and upper third (~3 mm from incisal edge)), and four-layer liquid penetration (4L; additional double application to the crowns' upper half and upper third) with a translucency-enhancing liquid (DD Incisal X, Dental Direkt GmbH, D) containing 45–55 wt% ytterbiumtrinitrat pentahydrate and <0.5 wt% hydrogen chloride. The TEL was applied manually according to the manufacturer's instructions in thin, consecutive layers over the full width of the incisal area, vestibularly and orally. Each layer was applied with a fully saturated microbrush (DD Bio Z Applikator, Dental Direkt GmbH, D) using controlled, uniform strokes, with re-saturation before each application to ensure consistent dosage. The application strokes were directed parallel to the incisal edge. All TEL applications were performed by the same operator. TEL induces a yellowish coloration of the zirconia blank, which enables visual assessment of the coverage area and the intensity of the application. Treated crowns were dried at 90 °C for 20 min (TSU-S, Mihm-Vogt, D). 5Y-TZP (DD Cube X2 ML, Dental Direkt GmbH, D) and ML 3Y-TZP + 5Y-TZP (Nacera Pearl, Dental Direkt GmbH, D) zirconia crowns served as reference and were not treated with TEL (see Fig. 1). In this study, the term multilayer (ML) zirconia refers to a material combining 3Y-TZP and 5Y-TZP layers within one blank, resulting in both compositional and translucency gradients. All groups underwent either a conventional sintering (CS; inFire HTC speed, Dentsply Sirona, USA) or HS (Cerec Speedfire, Dentsply Sirona, USA) process (see Fig. 1). The CS program followed the manufacturer's recommendations. HS was performed using a standard program suitable for zirconia (see Table 2). This reflects typical clinical conditions, as the HS furnace cannot be freely programmed. The crowns were polished in a two-stage protocol using a

Table 1
Materials: Zirconia specimens.

Material	Manufacturer	Type	Composition [wt. %]	Flexural strength [MPa]
DD Bio ZX^2 High Translucent Zirconia	Dental Direkt GmbH, G	3Y-TZP	ZrO_2, HfO_2, Y_2O_3 < 6, Al_2O_3 ≤ 0,15, other <1	1150 ± 150
DD cube ONE ML® Multilayer – High Strength Cubic Zirconia System	Dental Direkt GmbH, G	4Y-TZP	ZrO_2, HfO_2, Y_2O_3 < 8, Al_2O_3 < 0,15, other <1	1200 ± 150
DD cubeX^2® ML Multilayer - Cubic Zirconia System	Dental Direkt GmbH, G	5Y-TZP	ZrO_2, HfO_2, Y_2O_3 < 10, Al_2O_3 ≤ 0,01, other <1	700 ± 100
Nacera® Pearl Natural Multilayer	Dental Direkt GmbH, G	5Y-TZP + 3Y-TZP (ML)	ZrO_2, HfO_2, Y_2O_3 < 10, Al_2O_3 = 0,10, other <1	incisal: 1000, cervical: 1200

Zirconia materials investigated, including manufacturer, composition and flexural strength. All data provided by the manufacturer (Dental Direkt GmbH, G; Rev. 21_2024/03).

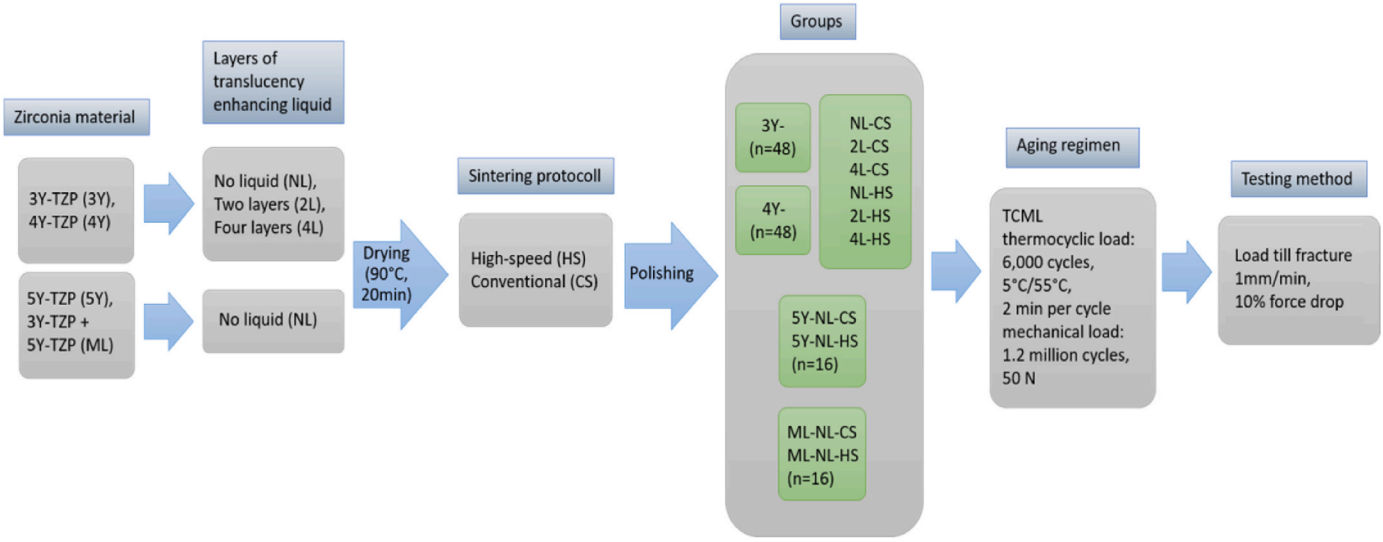

Fig. 1. Study design and groups: Schematic overview of the experimental workflow. Zirconia crowns of four material types were processed with or without TEL and subjected to either CS or high-speed sintering HS. All specimens underwent TCML followed by a load-to-fracture test.

Table 2
Sintering protocol.

Zirconia type	3Y-TZP, 4Y-TZP, 5Y-TZP	ML	3Y-TZP, 4Y-TZP, 5Y-TZP, ML
Sintering method	Conventional	Conventional	High-speed
Sintering furnace	inFire HTC speed, Dentsply Sirona, USA	inFire HTC speed, Dentsply Sirona, USA	Cerec Speedfire, Dentsply Sirona, USA
Starting temperature in °C	20	20	25
Heating rate 1 in °C/min	8	8	129,6
Heating time 1 in min	110:00	185:00	12:00
Holding time 1 in min	30:00	120:00	4:00
Heating rate 2 in °C/min	3	–	–
Heating time 2 in min	183:00	–	–
Holding time 2 in min	120:00	–	–
Final temperature in °C	1450	1500	1580
Cooling rate in °C/min	10	8	163
Total holding time in min	150:00	120:00	4:00

Overview of the conventional sintering (CS) and high-speed sintering (HS) programs used for each zirconia type. The table specifies furnace models, peak temperatures, dwell times, and total sintering durations.

handpiece (Typ 4941, KaVo Dental GmbH, D) according to the manufacturer's instructions (8000 rpm; Polishing Kit 4622, Komet Dental/-Gebr. Brasseler GmbH & Co. KG, D). Each polishing step was applied for 60 s per surface under a constant applied pressure of ~2 N. The rotation was maintained parallel to the incisal edge and performed in a uniform direction throughout. All polishing procedures were carried out by the same operator. The quality of the polishing was verified visually to ensure a smooth surface. The crowns inner surfaces were airborne-particle abraded (50 μm Al_2O_3, 1 bar), cleaned, and conditioned with an MDP-containing primer (Ceramic Bond, Voco Dental, D) (Rosentritt et al., 2020). The restorations were bonded to the PMMA abutments using a dual-cure resin-based composite cement (Bifix QM, Voco Dental, D). Excess cement was removed, followed by additional light curing of 20 s (1200 mW/cm^2; 385–515 nm; Bluephase G4, Ivoclar Vivadent AG, FL).

To enable TCML and subsequent fracture testing, custom bases were fabricated, simulating a natural jawbone (see Figs. 2 and 3). The artificial alveolus was set at a 135° angle to the loading axis (Albashaireh

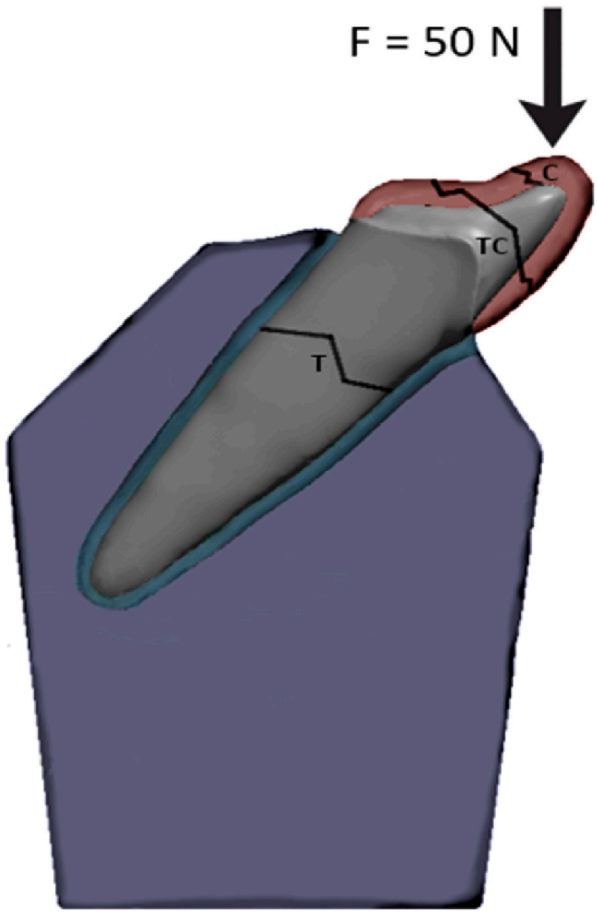

Fig. 2. Experimental setup and fracture patterns (C: Isolated crown fracture, T: Isolated tooth fracture, TC: Combined fracture).

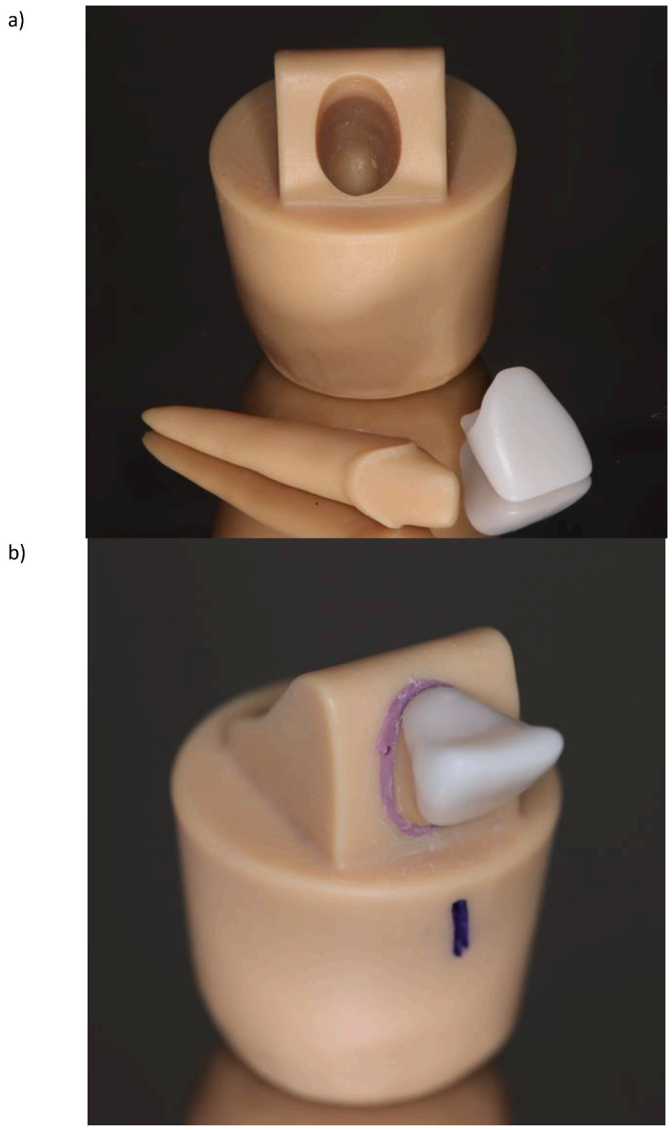

Fig. 3. Specimen setup (above: individual parts of the specimens (a), below: sampled specimen (b)).

and Sbeih, 2024). A 0.75 mm periodontal ligament space was simulated using polyether impression material (Impregum, 3M Oral Care, D) (Rosentritt et al., 2011). The designed bases were 3D-printed (Form 3B+, Formlabs GmbH, USA; Model Resin V2, Formlabs GmbH, USA). Specimens underwent TCML (EGO, Regensburg, D) with a combination of thermal cycling (TC: 2x3,000 cycles, 5 °C/55 °C, 2 min per cycle, temperature change during cycling within 30 s; H_20 dist.) and mechanical loading (ML: 1.2 million cycles at 50 N, f = 1.6 Hz) (see Figs. 1 and 2) to simulate clinical service (Rosentritt et al., 2009). The incisal edges of the crowns were centrally loaded with a steatite antagonist (d = 6 mm, CeramTec, D). Contrary to a physiological load situation palatally below the incisal edge, the force was applied directly to the incisal edge in the present test. This made it possible to avoid limitations in the test setup due to the mobile abutments and the angled alignment and the resulting shifts in the load point and angle. The load on the central incisal edge thus enabled a reproducible and clinically approximate load. However, the extended lever arm resulting from this force causes an increased load compared to the clinical situation and thus possibly an underestimation of the stability of the crowns. After mastication simulation, surviving specimens were subjected to a load-to-fracture test (Z010, ZwickRoell AG, D). Similarly to TCML, the incisal edges of the crowns were centrally loaded (see Fig. 2), placing a 0.3 mm thick tin foil between the crown and loading stamp (ø = 4 mm) to ensure an even distribution of forces. A compressive load (v = 1 mm/min) was applied until a 10 % force drop or acoustic monitoring indicated fracture. Fracture patterns (FP) during simulation or fracture test were classified into tooth fracture (T), crown fracture (C), or combined fractures (TC) (see Figs. 2, Figs. 4 and Fig. 6).

Calculations and statistical analysis were performed using SPSS 29.0 for Windows (SPSS Inc., Chicago, IL, USA). Data distribution was verified using the Shapiro-Wilk test. Mean values and standard deviations were analyzed with one-way ANOVA ($\eta^2 = 0.543$) and the Bonferroni post-hoc test. The significance level was set at $\alpha = 0.05$. A Power calculation (G*Power 3.1.3, Kiel, G) indicated an estimated power of >85 % using eight specimens per group.

3. Results

The results of the fracture test are shown in Fig. 5 and Table 3.

Fig. 4. Fracture patterns (above: FP C: isolated crown fracture (a); middle: FP T: isolated tooth fracture (b); below: FP TC: combined fracture (c)).

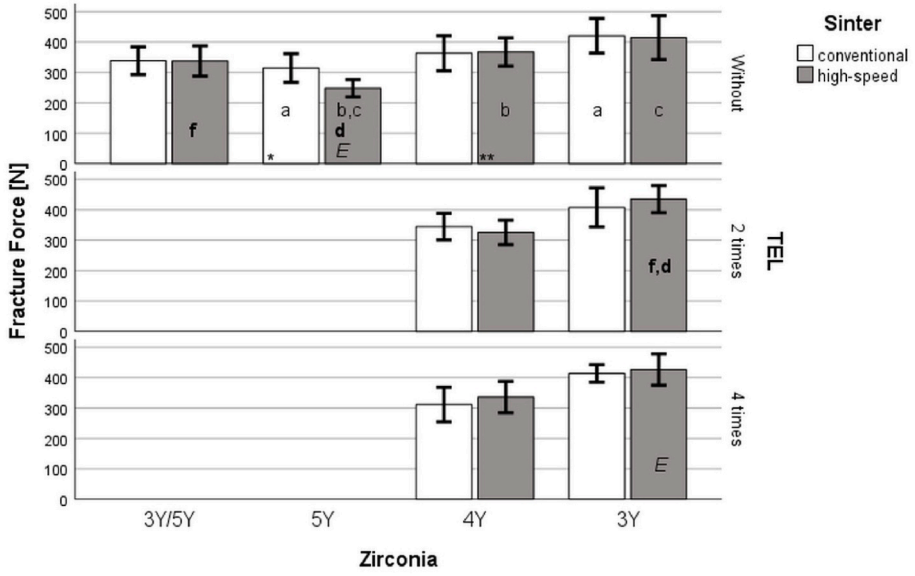

Fig. 5. Fracture force [N] (mean, error bars represent standard deviations; identical letters indicate significant differences ($p < 0.05$); n = 8 except for: *n = 5; **n = 7).

Table 3
– Fracture patterns.

	Group	3Y-NL-CS	3Y-NL-HS	3Y-2L-CS	3Y-2L-HS	3Y-4L-CS	3Y-4L-HS	4Y-NL-CS	4Y-NL-HS	4Y-2L-CS	4Y-2L-HS	4Y-4L-CS	4Y-4L-HS	ML-NL-CS	ML-NL-HS	5Y-NL-CS	5Y-NL-HS
Failures during TCML	FP T	–	–	–	–	–	–	–	1	–	–	–	–	–	–	1	–
	FP TC	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
	FP C	–	–	–	–	–	–	–	–	–	–	–	–	–	–	2	–
Load to Fracture	FP T	6	7	7	8	8	8	8	7	8	7	8	7	6	4	–	–
	FP TC	2	–	1	–	–	–	–	–	–	1	–	1	2	–	1	–
	FP C	–	1	–	–	–	–	–	–	–	–	–	–	–	4	4	8

Summary of observed fracture patterns among all groups after TCML and load-to-fracture testing. Fracture types are categorized as crown fracture (C), tooth abutment fracture (T), or combined fracture (CT). Values are expressed as frequencies.

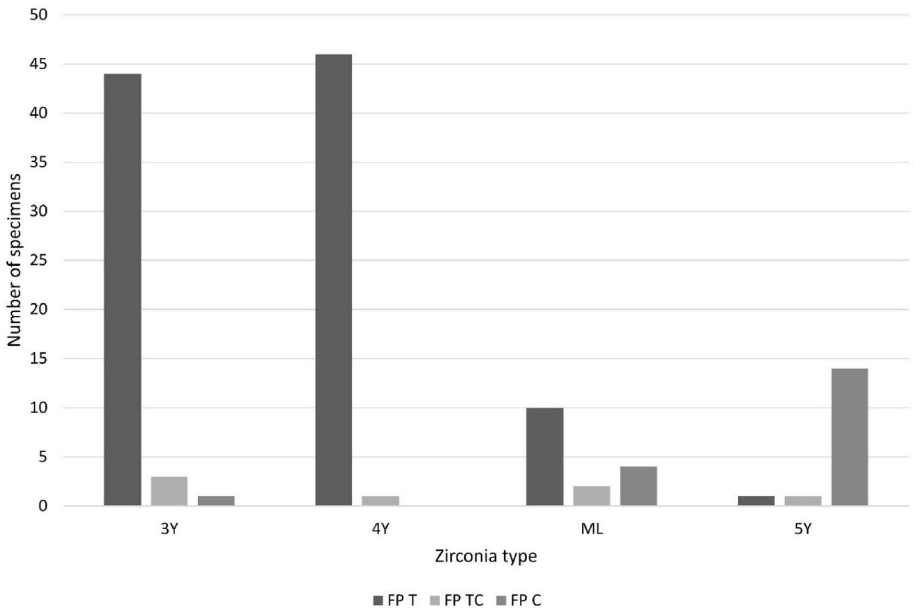

Fig. 6. Frequency of fracture patterns depending on zirconia type (fracture pattern (FP) are categorized as crown fracture (C), tooth abutment fracture (T), or combined fracture (TC)).

3.1. Conventional sintering (CS) vs. highspeed sintering (HS): groups without TEL

CS groups without TEL-treatment: There were no failures during TCML in group 3Y-NL-CS, 4Y-NL-CS, and ML-NL-CS. Three out of eight specimens failed in the 5Y-NL-CS group, two of which were isolated crown fractures (FP C) and one isolated fracture of the tooth abutment (FP T).

The mean fracture forces decreased with increasing yttria content, with values ranging between 314.4 N (5Y), 338.6 N (ML), 363.0 N (4Y), and 420.5 N (3Y). The 5Y-NL-CS group showed significantly ($p = 0.043$) lower fracture forces than the 3Y-NL-CS group (see Fig. 5, letter a). There were no significant ($p \geq 0.188$) differences between the other groups.

Tooth abutment fractures (Table 3, Fig. 6) were more frequent in 3Y (6x FP T, 2x FP TC, 0x FP C), 4Y (8x FP T, 0x FP TC, 0x FP C), and ML groups (6x FP T, 2x FP TC, 0x FP C). For 5Y-TZP zirconia, all failures were associated with crown fractures (0x FP T, 1x FP TC, 4x FP C).

HS groups without TEL-treatment: There were no failures during TCML in group 3Y-NL-HS, 5Y-NL-HS, and ML-NL-HS. One out of eight specimens failed in the 4Y-NL-HS group. This failure was an isolated tooth abutment fracture (FP T).

Mean fracture forces ranged between 247.9 N (5Y), 337.6 N (ML), 367.1 N (4Y), and 414.4 N (3Y). The 5Y-NL-HS group had significantly lower fracture forces compared to the groups 4Y-NL-HS ($p = 0.002$) (see Fig. 5, letter b) and 3Y-NL-HS ($p < 0.001$) (see Fig. 5, letter c). No significant ($p \geq 0.067$) differences were identified in the other group comparisons. No statistically significant ($p = 1.000$) fracture forces were detected between any CS and HS groups within the same material and treatment conditions.

Tooth abutment fractures (Table 3, Fig. 6) were more frequent in 3Y (7x FP T, 0x FP TC, 1x FP C) and 4Y groups (7x FP T, 0x FP TC, 0x FP C). Specimens fabricated from ML zirconia showed an increased occurrence of fractures involving the crown (4x FP T, 0x FP TC, 4x FP C). For 5Y-TZP zirconia, all failures were isolated crown fractures (0x FP T, 0x FP TC, 8x FP C).

3.2. Conventional sintering (CS) vs. highspeed sintering (HS): zirconia 4Y-TZP

CS 4Y: There were no failures during TCML in the CS 4Y-groups (NL, 2L, 4L).

Specimens of groups 4Y-NL-CS (363.0 N), 4Y-2L-CS (344.8 N), and 4Y-4L-CS (311.4 N) showed no significant ($p = 1.000$) differences in fracture forces. Fracture force decreased with an increasing number of TEL layers.

All fractures were tooth abutment fractures (24 x FP T, Table 3, Fig. 6).

HS 4Y: One failure during TCML was observed in the 4Y-NL-HS group.

Fracture forces did not differ significantly ($p = 1.000$) between specimens of the 4Y-NL-HS (367.1 N), 4Y-2L-HS (325.8 N), and 4Y-4L-HS (336.0 N) groups. Two fractures in the 2L and 4L group were combined crown and tooth abutment fractures (2 x FP TC).

All other fractures were isolated tooth abutment fractures (21 x FP T, Table 3, Fig. 6).).

3.3. Conventional sintering (CS) vs. highspeed sintering (HS): 3Y-TZP

There were no failures during TCML in the CS 3Y- or HS 3Y-groups.

CS 3Y: Fracture forces in groups 3Y-NL-CS (420.5 N), 3Y-2L-CS (407.9 N), and 3Y-4L-CS (413.6 N) were not significantly ($p = 1.000$) different.

Two fractures in group 3Y-NL-CS and one fracture in group 3Y-2L-CS involved crown and tooth abutment (3 x FP TC). All other fractures were isolated tooth abutment fractures (21 x FP T; Table 3, Fig. 6).

HS 3Y: Specimens of group 3Y-NL-HS showed lower mean fracture forces (414.4 N) compared to those of group 3Y-2L-HS (435.3 N) and 3Y-4L-HS (426.4 N), which were not statistically significant ($p = 1.000$).

There was one isolated crown fracture (1 x FP C) in group 3Y-NL-HS. All other fractures were isolated tooth abutment fractures (23 x FP T Table 3, Fig. 6).

3.4. Untreated ML and 5Y groups vs. TEL-treated 3Y and 4Y groups

Fracture forces of TEL-treated 4Y-TZP zirconia (311.4 N–344.8 N) were similar to that of ML zirconia (337.6 N; 338.6 N). TEL-treated 3Y-TZP zirconia showed higher fracture forces (407.9 N–435.3 N), while 5Y-TZP zirconia displayed the lowest fracture forces (247.9 N; 314.4 N). Significant differences were identified between group 3Y-2L-HS and ML-NL-HS ($p = 0.022$) (see Fig. 5, letter f), 3Y-2L-HS and 5Y-NL-HS ($p < 0.001$) (see Fig. 5, letter d), 3Y-4L-HS and 5Y-NL-HS ($p < 0.001$) (see Fig. 5, letter e). There were no significant ($p \geq 0.083$) differences in the other group comparisons.

Pearson correlation revealed significant ($r = 0.539$, $p < 0.001$) differences across different zirconia types. No significant differences were identified between TEL subgroups (NL, 2L, 4L) ($r = 0.135$, $p = 0.134$) and between CS and HS specimens ($r = -0.040$, $p = 0.663$).

4. Discussion

The hypothesis of this study suggesting that TEL and HS affect the mechanical properties of anterior zirconia crowns with varying yttria content, leading to differences in fracture forces and survival rates after TCML, could only be partially confirmed.

4.1. TCML

TCML simulated a clinical service of five years (Rosentritt et al., 2009). In a clinical scenario, fractures of natural teeth would not be common within five years, but during TCML, two 5Y-TZP zirconia crowns (group 5Y-NL-CS) and two PMMA tooth abutments (group 4Y-NL-HS and 5Y-NL-CS) fractured. These failures of the standardized PMMA abutments might be attributed to their lower mechanical strength compared to natural dentin (Arnetzl and Arnetzl, 2015), which might be regarded as a limitation of the current investigation. However, the two crown fractures observed in the CS-5Y group during TCML indicate that 5Y-TZP zirconia may yield reduced clinical reliability compared to the other zirconia types. In 3Y- and 4Y-TZP zirconia, mechanical and thermal loading may induce martensic phase transformation – a transition from tetragonal to the monoclinic phase – which is supposed to hinder crack propagation (Rosentritt et al., 2020). This toughening mechanism may be less pronounced in 5Y-TZP zirconia due to its high content of cubic phase, which could make it more susceptible to crack growth under fatigue conditions (Rosentritt et al., 2020). HS and the application of TEL did not seem to influence the in vitro performance. But, since TEL in 3Y- and 4Y-TZP, appears to reduce the amount of transformable tetragonal phase, it can be speculated that the effectiveness of the transformation toughening mechanism during TCML is diminished in these cases (Ban et al., 2023; Cokic et al., 2020; Greitens and Heilemann, 2015; Nonaka et al., 2022; Rosentritt et al., 2020).

4.2. Fracture force

Influence of HS on zirconia without TEL-treatment: An about 20 % reduction in fracture force was observed for HS 5Y-TZP zirconia compared to CS 5Y-TZP zirconia. This decrease may be associated with microstructural changes induced by the HS process. HS has been reported to promote grain growth and reduce the amount of transformable tetragonal phase in 5Y-TZP zirconia, which could lead to reduced fracture forces (Cokic et al., 2020; Nonaka et al., 2022). In addition, previous studies have shown increased porosity within the grain structure

of HS 5Y-TZP zirconia (Alshahrani et al., 2024; Lawson and Maharishi, 2020; Nonaka et al., 2022). These porosities may directly lead to lower fracture forces, but also impair mechanical reliability. A lower mechanical reliability for HS zirconia has already been reported (Cokic et al., 2020; Jerman et al., 2020). Since other studies have reported no negative impact of HS on 5Y-TZP zirconia (Alshahrani et al., 2024; Cokic et al., 2020; Jansen et al., 2019; Lawson and Maharishi, 2020; Liu et al., 2022), low mechanical reliability could explain the variable outcomes for HS of 5Y-TZP zirconia, because failures are less predictable. Differences in study results may also be related to whether the zirconia material is specifically designed and approved by the manufacturer for HS protocols (Cokic et al., 2020; Lawson and Maharishi, 2020; Liu et al., 2022). The 5Y-TZP zirconia used in the present study was not specifically indicated for HS, which suggests that differences in the process parameters may contribute to changes in the materials's structure. SEM or XPS might be performed to prove this assumption. For 3Y-TZP and 4Y-TZP zirconia, HS tended to affect fracture force. These materials appear to be less sensitive to HS than 5Y-TZP zirconia (Alshahrani et al., 2024; Cokic et al., 2020; Jansen et al., 2019; Mayinger et al., 2021, 2024). Recent studies suggest that HS 3Y-TZP and 4Y-TZP zirconia exhibit fewer porosities, less grain growth, and a more homogeneous grain structure than HS 5Y-TZP zirconia (Alshahrani et al., 2024; Cokic et al., 2020). Furthermore, a high content of transformable tetragonal phase could be preserved, which would be essential for resisting crack propagation (Alshahrani et al., 2024; Cokic et al., 2020). No influence of HS on the fracture forces was observed for ML zirconia as well. The incisal layer of the ML zirconia crowns is composed of 5Y-TZP zirconia, which should react more sensitively to the HS process. However, this procedure did not seem to impact fracture forces as the main load is borne by the basal portion of the crown (Mayinger et al., 2024; Zacher et al., 2020). The mechanical properties of this basal layer do not appear to be negatively affected by HS, since it is composed of 3Y-TZP zirconia (Alshahrani et al., 2024; Cokic et al., 2020; Jansen et al., 2019; Mayinger et al., 2021, 2024).

Influence of HS on zirconia with TEL-treatment: Group 4Y-2L-HS showed about 6 % reduction in fracture force after HS, whereas the other three groups showed increases in fracture forces ranging between 3 and 8 %. TEL is meant to induce grain growth and increases the amount of cubic phase within zirconia, mimicking the properties of zirconia with high yttria contents (Ban et al., 2023; Greitens and Heilemann, 2015; Hauptmann et al., 2013). As mentioned above, zirconia with a high yttria content can be more sensitive to HS processes (Cokic et al., 2020; Nonaka et al., 2022). However, TEL is applied only to the incisal portion of the crown, while the load-bearing cervical area remains untreated, similar to the structural design of ML zirconia (Mayinger et al., 2024; Zacher et al., 2020). Therefore, it is plausible that HS had little effect on the fracture force of TEL-treated zirconia. The variation in results, both positive and negative, may be attributed to the manual application of TEL.

Influence of yttria-content: In the CS groups without TEL application, 3Y-TZP zirconia exhibited the highest fracture forces, followed by 4Y-TZP, ML, and, finally, 5Y-TZP zirconia. An increase in yttria content is associated with larger grains, a higher amount of cubic phase, and therefore lower mechanical strength (Jansen et al., 2019; Rosentritt et al., 2020; Stawarczyk et al., 2017). Similar results were observed for groups without TEL application that underwent a HS process: fracture forces appeared to decrease with increasing yttria content. Also, the aluminum oxide content of the investigated zirconia materials decreases with increasing yttria content in the following order: 3Y-TZP, 4Y-TZP, ML, and 5Y-TZP zirconia hindering grain growth and reducing ageing effects, finally resulting in a more resilient restoration (Jansen et al., 2019; Rosentritt et al., 2020; Stawarczyk et al., 2017). Notably, a significant drop in fracture forces was observed for 5Y-TZP zirconia, highlighting its sensitivity to the HS process (Cokic et al., 2020; Jansen et al., 2019; Mayinger et al., 2021, 2024; Nonaka et al., 2022).

Influence of treatment with TEL: CS and HS 4Y-TZP zirconia exhibited a reduction in fracture force of up to 17 % following the application of TEL. For CS 3Y-TZP zirconia, the application of TEL resulted in a loss of fracture force of up to 3 %. A decrease in fracture force with increasing TEL layers seems plausible, as the TEL contains yttrium ions that promote the formation of cubic phase and grain growth in zirconia (Ban et al., 2023; Greitens and Heilemann, 2015; Hauptmann et al., 2013), which could negatively affect the material's mechanical performance (Jansen et al., 2019; Rosentritt et al., 2020; Stawarczyk et al., 2017). The effect of TEL treatment on zirconia's fracture force seemed to be more pronounced in 4Y-TZP than in 3Y-TZP zirconia, as 4Y-TZP zirconia typically contains a lower amount of tetragonal phase (Alshahrani et al., 2024; Rosentritt et al., 2020). TEL treatment might further reduces this tetragonal phase content, which would impair the material's ability to hinder crack propagation (Ban et al., 2023; Chevalier et al., 2009; Greitens and Heilemann, 2015; Hauptmann et al., 2013; Rosentritt et al., 2020).

Comparison between ML and 5Y-TZP zirconia with TEL-treated 3Y-TZP and 4Y-TZP zirconia: Among these groups, TEL-treated 3Y-TZP zirconia exhibited the highest fracture force. This outcome is consistent with the previously published correlation between decreased yttria content and increased mechanical strength (Jansen et al., 2019; Rosentritt et al., 2020; Stawarczyk et al., 2017). While both TEL-treated 3Y-TZP and ML zirconia have a load-bearing basal layer composed of 3Y-TZP, TEL-treated 3Y-TZP still outperformed ML zirconia. TEL was individually applied to each crown, potentially resulting in TEL layers of varying extent. Since TEL has been shown to reduce fracture forces, its distribution is of particular relevance. The further the TEL layer extends into the cervical region, the more it is likely to affect the crown's fracture force. For ML zirconia specimens, the nesting position is essential for their mechanical properties. Restorations located closer to the top surface of the blank typically exhibit inferior mechanical performance (Winter et al., 2022). It is conceivable that the TEL application did not extend as far cervically as the high-yttria incisal layers of the ML zirconia. Consequently, the load-bearing basal regions of TEL-treated crowns are less intrigued compared to those of ML zirconia crowns. A reduced performance of HS 5Y-TZP may be linked to previously reported microstructural irregularities such as grain growth and increased porosity (Alshahrani et al., 2024; Cokic et al., 2020; Lawson and Maharishi, 2020; Nonaka et al., 2022). Since the basal portion of the 3Y-TZP and 4Y-TZP zirconia crowns was not TEL-treated, it could have retained its original microstructure during HS (Alshahrani et al., 2024; Cokic et al., 2020; Jansen et al., 2019; Mayinger et al., 2021, 2024). Consequently, HS may have had a reduced effect on fracture forces in these groups.

4.3. Fracture pattern

The observed shift of fracture patterns from type T to C or TC with lower yttria content was accompanied with decreased fracture forces of the crown. The influence of TEL application and sintering technique on the fracture pattern could be considered secondary. However, in ML zirconia a trend was observed, indicating that HS increased the risk of crown fractures. Specifically, fracture pattern type C occurred four out of eight times in the HS ML group. This observation mirrors the typical fracture behaviour seen in HS 5Y-TZP zirconia. In contrast, fractures in the CS ML group consistently involved the tooth abutments, indicating that the incisal 5Y-TZP portion of ML zirconia may be particularly sensitive to the HS process. As the testing design directs the applied force to this area of the crown, failure is more likely. However, no reduction in fracture forces was observed, which may be due to the 3Y-TZP load-bearing basal layer remaining unaffected by HS (Alshahrani et al., 2024; Cokic et al., 2020; Jansen et al., 2019; Mayinger et al., 2021, 2024). Despite the predominantly abutment-related fractures observed in both 3Y- and 4Y-TZP, 4Y-TZP zirconia exhibited lower fracture force values than 3Y-TZP zirconia, which suggests that 3Y-TZP reinforces the overall system. A similar trend was observed in TEL-treated specimens.

Crowns without TEL exhibited the highest fracture forces, even though fracture pattern type T was predominant. One potential explanation might be the elastic mounting of the specimens in artificial periodontal sockets (see Figs. 2 and 3). Load application to the incisal edge occasionally resulted in crown margin displacement beneath the crestal edge of the alveolar socket. Although similar fracture patterns were found, 3Y zirconia appears to be superior in the subcrestal area due to its higher strength. PMMA tooth abutments have a lower elastic modulus than natural dentin, which might contribute to the high prevalence of fracture pattern, especially for the fracture type T (see Table 3 and Fig. 6) (Arnetzl and Arnetzl, 2015). The experimental design might lead to an overestimation of the prevalence of tooth abutment failures compared to the expected clinical performance, especially in the higher strength 3Y- and 4Y-TZP groups. In contrast, ML and particularly 5Y-TZP zirconia crowns showed more crown-related fractures (types C and TC) (see Table 3 and Fig. 6). Based on the in-vitro results, crowns made from 5Y-TZP and ML zirconia, especially in HS configuration, may be more prone to fracture before the tooth structure is damaged.

5. Study limitations

All interpretations should be considered exploratory, as actual clinical conditions were not addressed. No optical or microstructural analyses were performed to quantify the effects of TEL treatment or HS on translucency or grain structure. This represents a limitation of the present study, as conclusions regarding aesthetic appearance and microstructural changes must therefore be interpreted with caution. The effects of TEL treatment and HS on the microstructure of the different zirconia types were determined by measuring changes in fracture forces and patterns. Additional SEM and XPS analyses would be necessary to confirm and expand upon these findings. The design of the specimens and TCML can only approximate intraoral conditions especially in terms of long-term clinical performance. Due to the varying structure of zirconia and the manual application of TEL, deviations in infiltration depths and distribution cannot be ruled. Similarly, the polishing protocol was performed using a standardized two-step system with defined application time and constant pressure. However, the manual nature of those procedures may introduce variability in surface smoothness and TEL infiltration, which could potentially affect zirconia's mechanical properties. The load application on the central incisal edge enabled a reproducible and clinically approximate load. However, the extended lever arm resulting from this force causes an increased load compared to the clinical situation and thus possibly an underestimation of the stability of the crowns. The load-to-fracture tests involved forces that exceed maximum bite forces typically encountered in the anterior region (Ferrario et al., 2004). While such testing enables a standardized comparison of material strength, it does not necessarily represent clinically relevant loading scenarios or failure behavior. All tested materials featured sufficient strength to withstand the physiological forces expected in the anterior zone (Ferrario et al., 2004). However, given the variability in the results and the simplified in vitro testing setup, these findings should be interpreted cautiously, and clinical safety margins may be lower than suggested by the mean values.

6. Conclusions

Different types of zirconia materials.

- exhibited progressively lower fracture forces with increasing yttria content (3Y-TZP > 4Y-TZP > ML > 5Y-TZP).

The application of TEL.

- had only minor effects on fracture forces of CS and HS 3Y-TZP zirconia.
- led to a reduction in fracture forces of up to 17 % for CS and HS 4Y-TZP zirconia.

The HS process.

- did not significantly affect fracture forces of 3Y-TZP and 4Y-TZP zirconia – regardless of TEL application.
- had no significant effect on fracture forces of ML zirconia.
- led to a 20 % reduction in fracture force for 5Y-TZP zirconia.
- increased the number of crown fractures in 5Y-TZP and ML zirconia groups.

CRediT authorship contribution statement

Nikolaus Kronwitter: Writing – review & editing, Writing – original draft, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Angelika Rauch:** Writing – review & editing, Validation, Methodology. **Sebastian Hahnel:** Writing – review & editing, Resources, Investigation. **Martin Rosentritt:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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