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RESEARCH AND EDUCATION

Mechanical and biocompatibility testing of zirconia and lithium disilicate ceramics: An in vitro study

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ABSTRACT

Statement of problem. High-translucency zirconia and lithium disilicate are among the most used materials in contemporary fixed prosthodontics because of their excellent esthetic and mechanical properties. However, their different cementation techniques, physical properties, and biocompatibility profiles can influence the clinician's choice.

Purpose. The purpose of this in vitro study was to evaluate the mechanical strength and adhesive cementation techniques for zirconia and lithium disilicate materials through mechanical testing, including compressive and pull-out tests. The biocompatibility of these materials was also assessed.

Material and methods. A total of 72 human maxillary molars that were free from damage were embedded in acrylic resin and prepared 1 mm occlusal to the enamel-cementum junction. The specimens were divided into 3 groups: lithium disilicate (CAD), zirconia High-Translucency HT (CAD), and lithium disilicate (PRESS). Following the recommended cementation protocols, compression and pull-out tests were performed. Twelve disks of each group were fabricated to test the integration of gingival fibroblasts. Human gingival fibroblasts were isolated from gingival biopsies and cultured in Dulbecco modified Eagle medium (DMEM). Cell viability was determined using the 3-(4,5-di-methylthiazol-2-yl)–2,5-diphenyltetrazolium bromide (MTT) assay, serving as an indicator of cellular respiration.

Results. Zirconia was the most mechanically efficient material, with a high resistance value (2081.4 ±405.4 N). The pull-out test determined that CAD-CAM lithium disilicate had similar adhesive cementation strength (244.3 ±29.3 N) to the other groups. In terms of biocompatibility, all materials demonstrated good results, with lithium disilicate CAD emerging as the most biocompatible material. Statistically significant differences were observed between the zirconia HT material and lithium disilicate PRESS (P=.006) and between lithium disilicate CAD and lithium disilicate PRESS (P=.002).

Conclusions. All the monolithic restorations analyzed have shown excellent results in terms of mechanical properties, adhesion, and biocompatibility. Zirconia exhibited outstanding mechanical properties, making it an ideal choice for applications requiring high strength. To achieve adhesion results comparable with those of lithium disilicate, a protocol using airborne-particle abrasion and silane with 10-MDP is recommended. This approach ensures optimal adhesion, enhancing the longevity and effectiveness of the restoration. (J Prosthet Dent 2025;133:593.e1-e8)

Popular dental ceramics include esthetic glass-ceramics, resin-matrix ceramics, and high-strength zirconia ceramics.[1,2] Lithium disilicate is a popular option with excellent optical, mechanical, and biocompatibility attributes.[3–9] In addition to the heat-pressed method, the widespread adoption of computer-aided design and computer-aided manufacturing (CAD-CAM) technologies have led to the marketing of ceramic blocks to

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Clinical Implications

All the analyzed monolithic restorations exhibited excellent mechanical and biocompatibility properties. High-translucency zirconia combines good esthetic properties with excellent mechanical and adhesive characteristics. Specifically, the use of airborne-particle abrasion, silanization, and the use of a 10-methacryloyloxydecyl-dihydrogen-phosphate (10-MDP)-containing cement enhanced the adhesive cementation of zirconia, reaching levels comparable with those of the glass-ceramics.

produce milled restorations, including chairside fabrication.[9,10]

Yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) is also a popular material for indirect restorations. Zirconia is a heterogeneous, highly durable, polycrystalline ceramic, with favorable mechanical properties, good optical characteristics, and excellent biocompatibility.[3,11–14] However, unlike glass-ceramics, it is not responsive to conventional acid-etching techniques and, as a result, is unsuitable for traditional adhesive bonding procedures.[15] More translucent partially stabilized zirconia ceramics (4Y-TZP and 5Y-TZP) have been introduced to meet esthetic expectations by addressing the opacity of 3Y-TZP restorations.[3,11,16–18]

Adhesive bonding to lithium disilicate is achieved through micromechanical etching (20 seconds with 5% hydrofluoric acid) and chemical bonding mechanisms.[19] Airborne-particle abrasion with aluminum oxide or laser etching may result in excessive material loss.[20,21] Silane application improves lithium disilicate adhesion with siloxane linkages between the resin-based agent and the ceramic surface.[22,23]

The combination of silane with a phosphate functional monomer, specifically 10-methacryloyloxydecyl-dihydrogen-phosphate (10-MDP), which creates an acidic environment, has been reported to further enhance the bond strength of resin-based luting cement to lithium disilicate ceramics.[24] Also, the long-term bonding of zirconia prostheses requires the appropriate pretreatment of the adhesive surface, the use of 10-MDP, and the selection of suitable resin luting agents.[25,26]

Aging impacts the clinical efficacy of a restoration. Oral mechanical stresses, temperature, and humidity degrade the bond to zirconia restorations,[20] and thermocycling and varying durations of water storage have been used as artificial aging methods.[17,27–29] However, in vitro studies analyzing the mechanical aspects, adhesive properties, and biocompatibility of monolithic restorations are sparse.

The primary objective of this study was to assess the compression strength of monolithic restorations. In addition, adhesive properties were evaluated by using a pull-out test. A secondary objective was to assess the biocompatibility of these different dental materials by evaluating the reaction of human keratinized mucosa fibroblast cells and the production of collagen cell viability with a 3-(4,5-dimethylthiazol-2-yl)–2,5-diphenyltetrazolium bromide (MTT) assay cytotoxicity test. The null hypothesis was that no differences in terms of adhesion and mechanical resistance would be found between the monolithic restorations analyzed in the study.

MATERIAL AND METHODS

A total of 72 intact human maxillary molars with no carious lesions or other defects were collected. Power analysis using a software program (G*Power Ver. 3.0.10; Heinrich Heine University Düsseldorf) was used to determine the sample size. Specimens per group were set considering power=.95, α=.5, and effect size=.8. A total of 36 specimens for each test were fabricated, 12 specimens in each material.

The study was conducted in accordance with the Declaration of Helsinki, and the protocol was approved by the Inter Institutional Ethics Committee of the 'G. d'Annunzio' University of Chieti-Pescara. The teeth had been stored at 4 °C in a physiological saline solution and were divided into 2 groups: 36 teeth for pull-out testing and 36 teeth embedded in acrylic resin (Dentalon; Kulzer GmbH) for compression testing. The specimens were further divided into 3 groups for pull-out and compressive testing: lithium disilicate (CAD) (CEREC Tessera HT; Dentsply Sirona), zirconia (CAD) (Cercon High-Translucency HT, 5% Yttria; Dentsply Sirona), and lithium disilicate (PRESS) (Vision-LD. Press; Wohlwend AG Dental Manufakter) (Fig. 1). To ensure the tooth preparations were consistent, they were prepared with a 1-mm chamfer finish, 2-mm occlusal reduction, and 1.5-mm axial reduction by a single researcher (B.S,) under ×4 magnification (Eyemag Pro F; Zeiss).[30] The preparations were evaluated with silicone indices (Soft Putty; 3M ESPE) and measured with a periodontal probe. The procedures were evaluated by 2 researchers (G.T., E.X.) to ensure accuracy in the axis, taper, and other features.

For the zirconia crowns, each tooth was optically scanned (Medit 710; Meditcorp), and the crowns were designed using a software program (Plovdiv2; exocad GmbH) with a 1.5-mm occlusal thickness. Zirconia blocks (Cercon HT; Dentsply Sirona) were then milled (Ceramill Motion 2; Amann Girrbach AG) and sintered in a furnace (HT-S 2; Mihm-Vogt). To optimize bonding, the intaglio surfaces of the crowns were airborne-particle abraded using 50-µm aluminum oxide at 0.25 MPa for a maximum of 15 seconds at 10 mm.[25]

For the lithium disilicate CAD-CAM crowns, each tooth was scanned (Cerec Primescan; Dentsply Sirona), and the

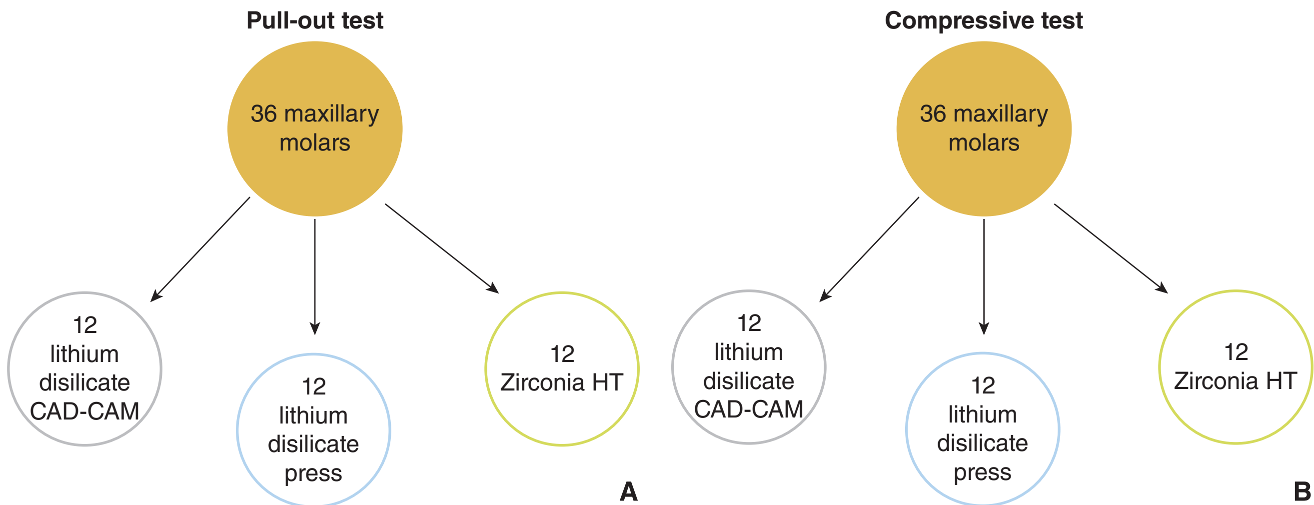

Figure 1. Specimen preparation. A, Pull-out test. B, Compressive test.

crowns were designed with a 1.5-mm occlusal thickness using a software program (CEREC AC; Dentsply Sirona), milled (CEREC MC X5; Dentsply Sirona) in a precrystallized state, and crystalized for 24 minutes in a furnace (Programat CS2; Ivoclar AG). For the lithium disilicate PRESS crowns, wax patterns (Geo classic opak; Renfert) were fabricated, invested (Pressvest speed; Ivoclar AG), wax eliminated, and pressed (Vision-LD. Press; Wohlwend AG Dental Manufakter) by a single experienced dental laboratory technician. Subsequently, lithium disilicate PRESS ingots were used in a pressing furnace (Programat EP 3000; Ivoclar AG).

For the crown adhesive procedure, the specimens were cleaned and prepared for total etching and bonding. A 2-step etch-and-rinse adhesive system (Clearfill, Universal Bond Quick; Kuraray Dental) was used as follows. The dental surface was etched with 37% phosphoric acid for 20 seconds, rinsed using a water spray for 20 seconds, and air dried for 5 seconds. A primer (Panavia V5 tooth primer; Kuraray Dental) was applied with a microbrush (Microbrush Applicator Panavia; Kuraray Dental) and gentle brushing motions for 20 seconds (Fig. 2). The primer-coated area was then air dried for 3 seconds. An adhesive resin (Adhese

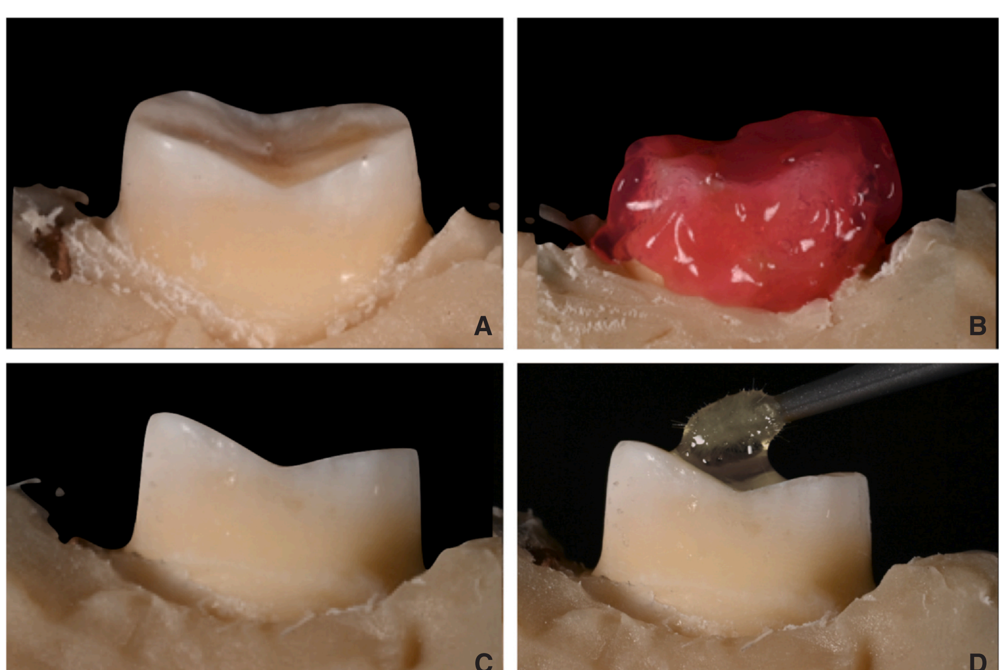

Figure 2. Dental abutment preparation protocol. A, Tooth preparation. B, Surface treatment with phosphoric acid 37% for 20 s (Etch Ena, Micerium). C, Washing and drying. D, Application of Tooth Primer for 20 s and drying (Panavia V5).

Universal; Ivoclar AG) was applied with gentle brushing motions for 10 seconds. The adhesive resin was air thinned for 3 seconds, and the bonding resin was polymerized using a multiwave-length light-emitting diode polymerizing lamp (BluePhase-G2; Ivoclar AG) for 40 seconds at 800 mW/cm², (wavelength of 395–480 nm) positioned 3 mm from the specimen (Fig. 2).

For zirconia cementation, the intaglio surface was airborne-particle abraded with 50-µm aluminum oxide at 0.25 MPa for a maximum of 15 seconds at 10 mm (Figs. 3 and 4). To remove airborne particle residue, the specimens were ultrasonically cleaned (Eurosonic 3D, Euronda) in 99% isopropyl alcohol (isopropyl alcohol; Feroca) for 5 minutes; subsequently, the intaglio surface was thoroughly air dried.[26] A ceramic primer (Clearfill plus Panavia V5; Kuraray Dental) was applied to the intaglio surface with a microbrush (Microbrush Applicator Panavia; Kuraray Dental). The cement (Panavia V5; Kuraray Dental) was dispensed using an automix syringe and photopolymerized for 40 seconds at 800 mW/cm² (BluePhase-G2; Ivoclar AG).

The CAD and PRESS lithium disilicate restorations were surface treated with 4.9% HF (Vita Ceramics Etch; Vita Zahnfabrik) for 20 seconds[19] Subsequently, Clearfill ceramic primer plus was applied with a microbrush (Microbrush Applicator Panavia; Kuraray Dental), followed by the application of resin cement (Panavia V5; Kuraray Dental) using automix syringes. This was followed by restoration seating, photopolymerization (BluePhase-G2; Ivoclar AG), and the removal of excess cement.

After cementation, all the specimens underwent pull-out retention tests according to D'Addazio et al[31] and compression tests (Figs. 5 and 6) using a universal testing machine (LR30K PLUS; AMETEK). In pull-out tests, the abutment and cemented crown was fixed to the universal testing machine parallel with the devices secured to the base of the testing machine. The abutments, designed with a perforated cylinder on the occlusal surface, were fixed with a stainless-steel device to the hook and placed under tension. A preload of 6 N was set to prevent system displacement. The test was conducted at a constant speed of 5 mm/minute until the abutment and crown separated.

The compression test was conducted using the universal testing machine equipped with a 30-kN load cell. The force was applied via a tungsten carbide pilot punch with a radius of 3.18 mm, targeting the central fissure of

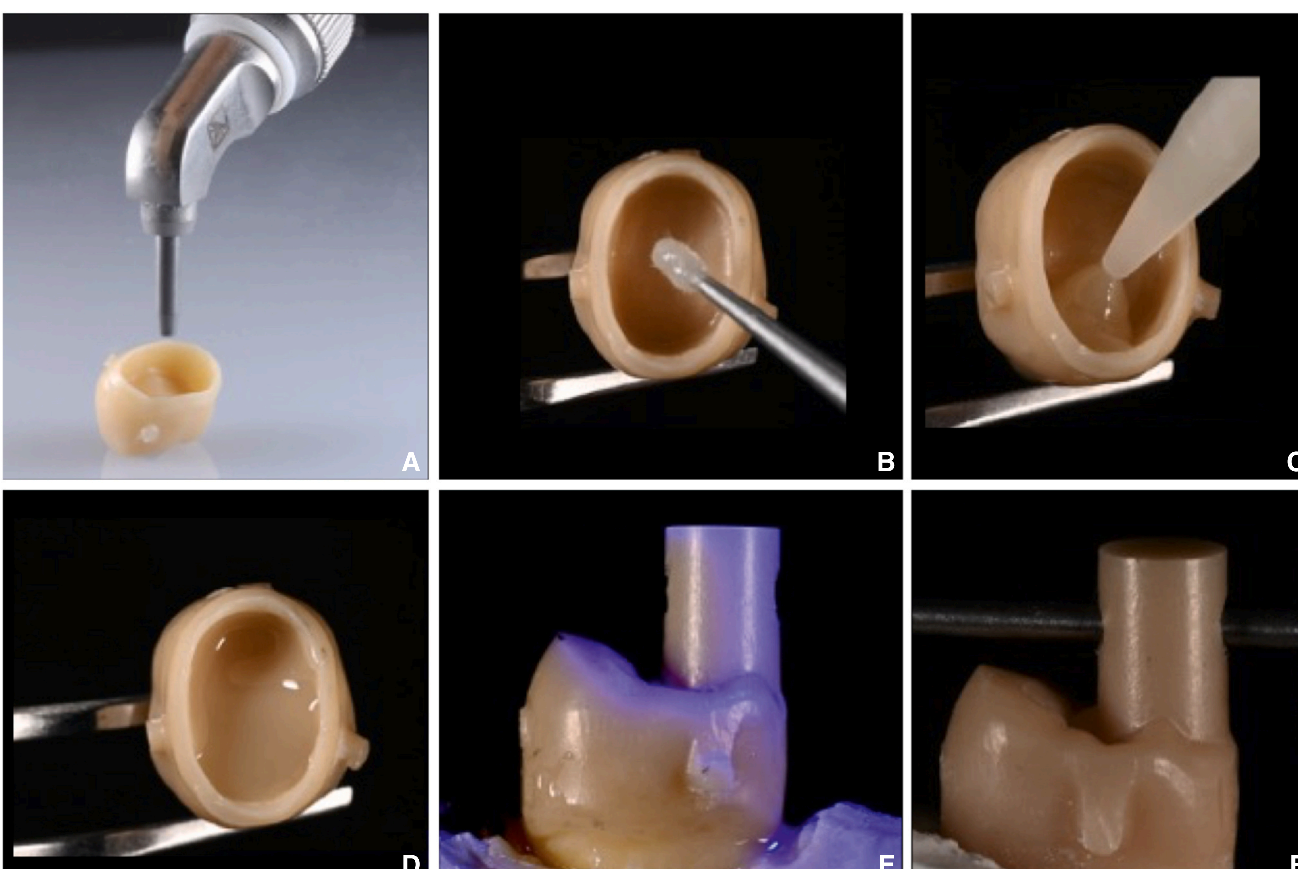

Figure 3. Cementation protocol zirconia prosthetic restoration. A, Airborne-particle abrasion with alumina oxide (Al_2O_3) at 0.25 MPa. B, Application of Clearfill ceramic primer plus Panavia V5. C, Application of Panavia V5 Paste cement. D, Panavia V5 Paste cement. E, Photopolymerization and excess removal. F, Pull-out test.

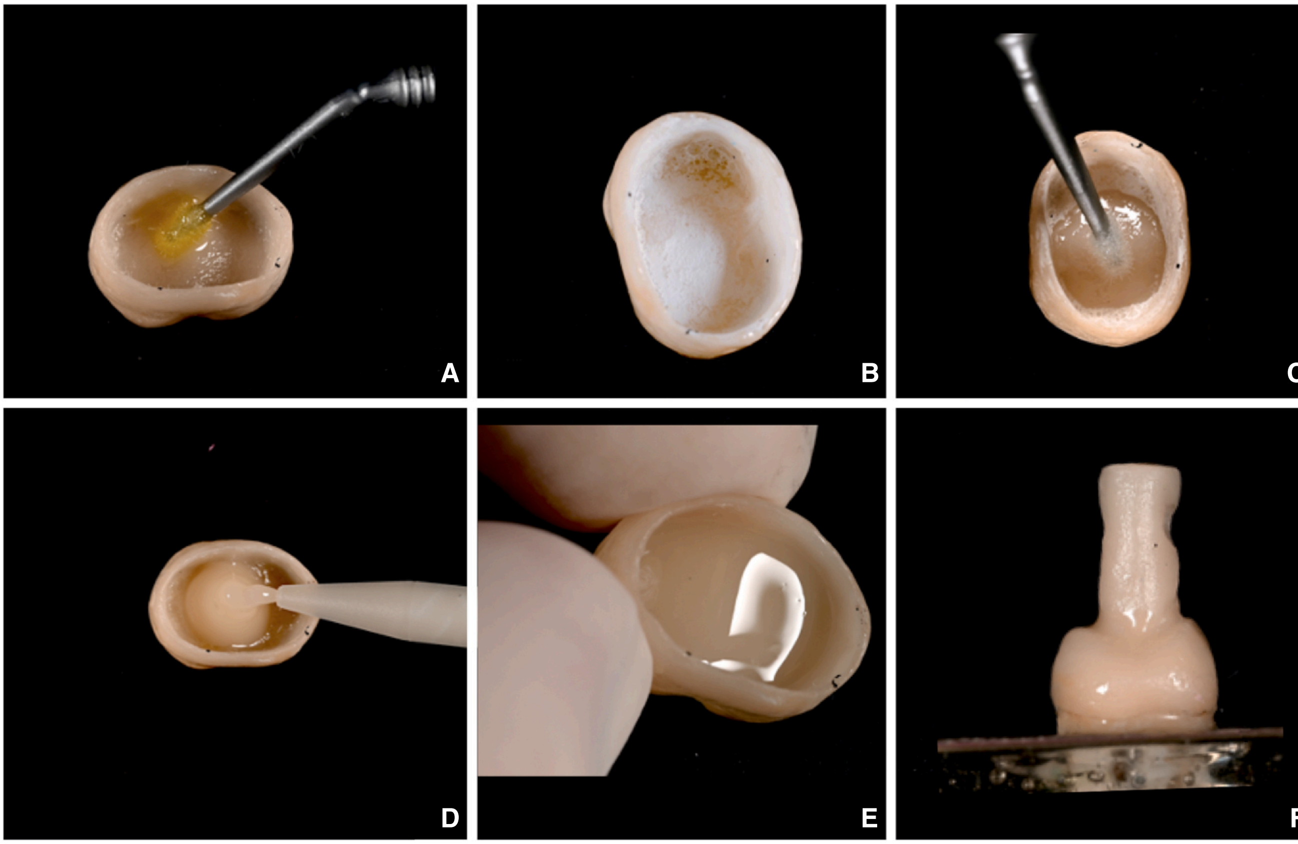

Figure 4. Cementation protocol lithium disilicate prosthetic restoration. A, Application of hydrofluoric acid, 4.9% for 20 s. B, Acid-etched surface. C, Application of Clearfill ceramic primer plus Panavia V5. D, Application of Panavia V5 Paste resin cement. E, Placement of restoration. F, Photopolymerization and excess removal.

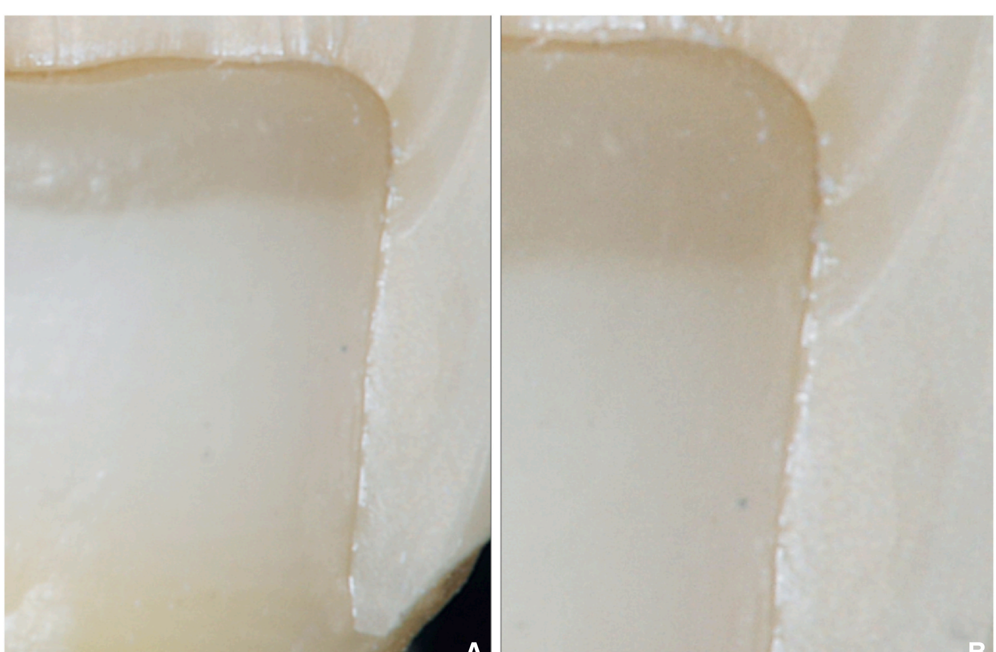

Figure 5. Representative fracture of zirconia crown after compressive test.

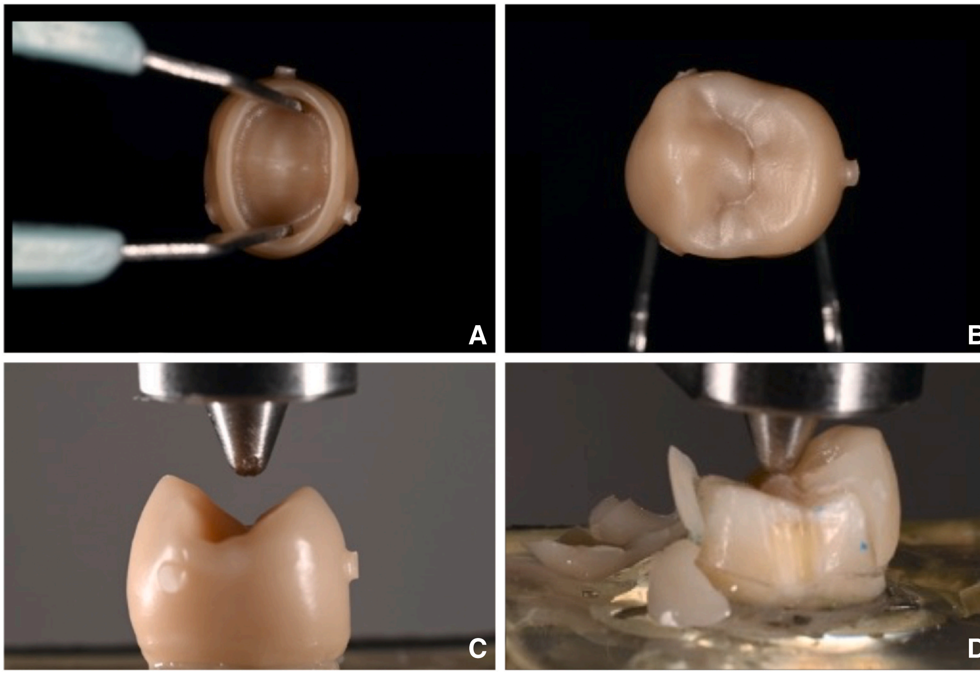

Figure 6. Zirconia crown cementation and lithium disilicate compressive test. A, Zirconia crown intaglio surface. B, Occlusal surface. C, Zirconia crown compressive test. D, Lithium disilicate compressive test.

the tooth to ensure 2-point contact, at a speed of 1 mm/minute. The maximum load before failure was recorded in N, and mean values were calculated for each group.

The biocompatibility tests were conducted to investigate the response of human gingival fibroblast cells to 12 disk specimens (Ø4×1.5 mm) of the 3 dental materials (Fig. 7). Ceramic materials were polished sequentially by using coarse (55 µm), medium (40 µm), fine (24 µm), and superfine (4 µm) grit diamond rotary instruments (Dialite ZR; Brasseler).

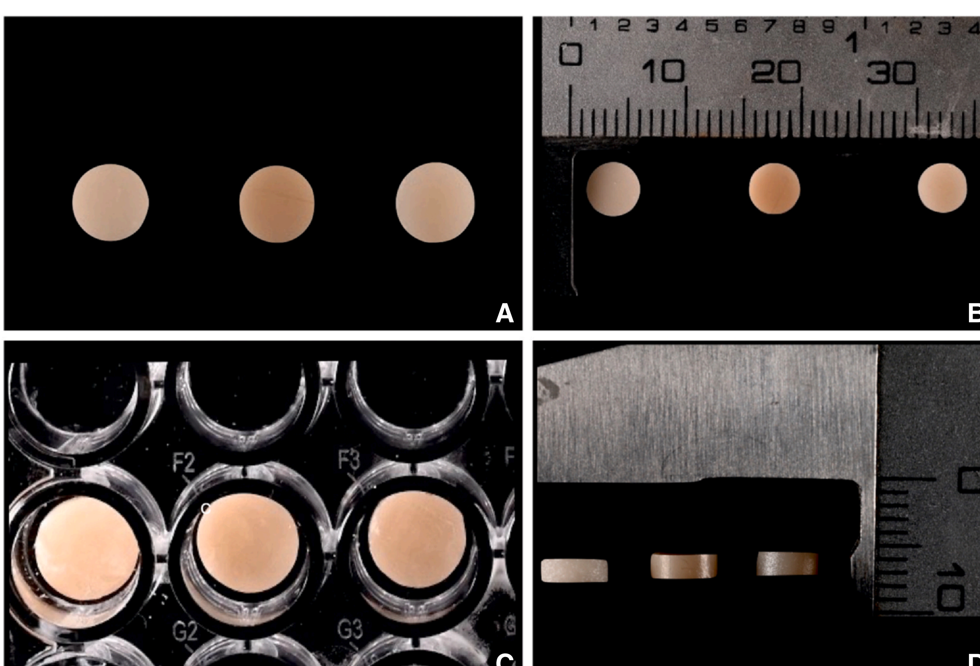

Figure 7. Biocompatibility test. A, Zirconia, lithium disilicate CAD, and lithium disilicate PRESS. B, Specimens in microtilter plate. C, Measurement of specimen width. D, Measurement of specimen thickness.

Human gingival fibroblasts were isolated from gingival biopsies and cultured in Dulbecco modified Eagle medium (DMEM) containing 10% of bovine fetal serum (FBS), 2 mM of L-glutamine, 1 mM of sodium pyruvate, ×1 of non-essential amino acids, and 50 U/mL of penicillin-streptomycin for 10 days. The cells were incubated in a humidified CO_2 incubator set (Thermo Scientific 8000 CO_2 Incubator; Fisher Scientific) at 37 °C to reach an 80% confluence, after HGFs had reached the 80% confluence, they were used for the experiment. Cells were plated at a density of 0.5×10^6 per well. The well contained sterilized experimental disks and was cultured for 24 hours. All the disks were analyzed in quadruplicate. An MTT assay (MTT Assay; Merck KGaA) was used to measure cellular respiration as an indicator of cell viability. Briefly, after 21 hours of incubation, 10 µL of MTT solution was added to the culture medium and incubated for 3 hours under the same conditions. After the incubation period, the MTT medium was removed and replaced with an equal volume of DMSO to break the cell membranes and solubilize the intracellular purple crystals.[7] The absorbance of the resulting purple solution was measured at 550 nm with a detection system (GloMax-Multi+; Promega). Cell viability was reported as the mean of the optical density obtained for each replica for the different biomaterials (Fig. 7). To analyze the differences between the biomaterials, a Student *t* test was conducted.

The data for the pull-out and compression test were statistically analyzed using 1-way ANOVA and post hoc Tukey tests with a software program (Mann Whitney U test calculator; Statistics Kingdom, 2017). The data are presented as mean ±standard deviation (SD), 95% confidence interval (C.I.) or percentage (Table 1). One-way ANOVA was used to assess if there were differences in adhesion for the 3 pull-out test groups and in fracture resistance for the 3 compression test groups (α=.05). The Kolmogorov–Smirnov normality test was applied for each of the postural variables considered to determine whether the data were normally distributed.

RESULTS

Zirconia showed the highest load resistance with a mean ±standard deviation resistance value of 2081.4 ±405.4 N, while lithium disilicate PRESS had the lowest load resistance at 1293.9 ±270.1N (Table 1). Lithium disilicate CAD had a mean ±standard deviation adhesive cementation load (244.3 ±29.3N), followed closely by zirconia HT (240.6 ±33.4 N) and lithium disilicate PRESS (188.7 ±17.4 N), with no statistical differences (P>.05) (Table 1). Regarding biocompatibility, all materials performed well, with lithium disilicate CAD demonstrating the highest biocompatibility. Significant differences were found between zirconia HT and lithium disilicate PRESS (P=.006), as well as between lithium disilicate CAD and PRESS (P=.002) (Table 1).

Regarding biocompatibility tests, after 24 hours of culture, the MTT assay revealed a higher number of viable cells on the lithium disilicate CAD compared with the other 2 disks. Statistically significant differences were observed between the zirconia HT material and lithium disilicate PRESS (P=.006), as well as between lithium disilicate CAD and PRESS (P=.002).

DISCUSSION

The null hypothesis, indicating that no statistically significant differences would be found in terms of adhesion and mechanical properties among the materials analyzed, was not rejected. All the analyzed monolithic restorations exhibited excellent mechanical and biocompatibility properties. HT zirconia can combine good esthetics with excellent mechanical and adhesive characteristics. The use of airborne-particle abrasion and silanization with 10-MDP enhances the adhesive cementation of zirconia, reaching levels comparable with those of glass-ceramics.[16,26] The data suggested that, with the application of a correct protocol, zirconia can achieve a level of adhesion comparable with that of lithium disilicate. Any observed variations between the materials could be associated with differences in protocol efficiencies; however, no statistically significant differences were detected in this study (P>.05).

HT zirconia can represent an excellent compromise in terms of biocompatibility, mechanical properties, and adhesive properties if airborne-particle abraded and treated with10-MDP.[32] The 4%mol yttria zirconia had adequate strength and toughness while maintaining good translucency.[17] A comparative in vitro study[6] reported that lithium disilicate improved cell viability and collagen secretion more than zirconia-reinforced lithium-silicate (ZLS) and zirconia, with a more pronounced difference compared with polymethylmethacrylate (PMMA). Consistent with this study, the present study found zirconia and CAD and PRESS lithium disilicate to have excellent biocompatibility.

Limitations of the present study included the in vitro conditions that did not fully simulate clinical situations and that the mechanical tests were static and not dynamic. In addition, the specimens were not aged or thermocycled.

Table 1. Force testing

Specimen	Zirconia HT	Lithium Disilicate CAD	Lithium Disilicate Press	*P*
Mean ±standard deviation compressive force (N)	2081.5 ±405.5	1737.4 ±428.5	1293.9 ±370.3	.617
Mean ±standard deviation pull-out force (N)	240.6 ±33.5	244.3 ±29.3	188.7 ±17.4	.304

Future research should evaluate these parameters over an extended period. Comparisons between survival rates and complications of these restorations require well-designed, long-term randomized controlled studies. In addition to compression and tensile strength, it is necessary to study wear properties, marginal microleakage, internal fit, and comparisons between materials.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Zirconia exhibited outstanding mechanical properties, making it an ideal choice for applications requiring high strength.
2. The zirconia cementation protocol with airborne-particle abrasion and MDP-10 performed as well as the protocol with lithium disilicate.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Ethics approval was not required for this study.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF DATA AND MATERIALS

All data are included into the manuscript.

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